THE ELECTRON-MICROSCOPIC AND X-RAY SPECTRAL ANALYSIS OF PHASE COMPOSITION OF CGI INOCULANT STRUCTURE

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ABSTRACT

The paper presents an analysis of both the phase and chemical compositions as well as the structure of 3 types of inoculants having a similar chemical composition but produced by two completely different technological processes. All investigated inoculants serve to produce vermicular graphite iron of a similar chemical composition. A qualitative identification of inoculant particles phase composition and surface microanalysis is carried out by electron-microscopic examination and X-ray spectral microanalysis. The investigations used can serve as an effective way of inoculants ranking according to their qualitative and performance characteristics.

Keywords: an electron-microscopic examination, an X-ray spectral microanalysis, an inoculant, a phase composition.

INTRODUCTION

Nowadays compacted graphite iron (CGI) is cast in foundries with the use of alloys composed of magnesium, rare-earth metals and a significant number of other functionally different elements. In addition to the modifying impact on the melt these chemical elements (alkaline earth metals (Mg, Ca, Ba, Sr) and rare-earth metals (Y and lanthanides – La, Ce, etc.) have also micro-alloying and refining functions. Actually, multi-purpose complex additives act not so much as inoculants but in greater part as complex alloys [1] of their own specific structure. Their influence on the process of alloy crystallization and the technological processes of crushing, dusting and moisture accumulation are scarcely taken into account.

The chemical compositions of the inoculants and the alloys as well as the grain fineness are up to date the main criteria of their efficiency in practice of inoculating liquid melts. However, a great number of effects in the process of cast formation are indicative of the interference of the inoculant structure in the crystallizing process.

The changes of cast Fe-Ni-Mg alloys structure caused by modified and accelerated cooling processes are investigated [2]. The impact of the microstructure of cast Fe-Ni-Mg-REE-alloys on the graphite morphology formation in high-test cast iron is also studied.

In general there is a lack of scientific and technical information on the correlation between the inoculant smelting and its structure as well as on the quality of the final casting and the technical specifications of the inoculant usage.

The present work focuses on the study of the structure and the chemical composition of inoculants produced by two completely different technological processes, such as ore smelting and remelting.

RESULTS AND DISCUSSION

It is common for the world practice to cast inoculants and alloys according to the following standards of the metallurgical technologies: a full cycle ore-thermal process with final dedusting treatment after crushing and sizing (I) and a simplified process of remelting (II).

The detailed comparative analysis of both technological processes of inoculant materials casting is shown in Table 1. In general they should affect the quality of the finished product. The inoculant cast by the ore-thermal process is expected to have a higher quality in comparison with the one made by remelting.

To confirm the hypothesis the particles of crushed inoculants for CGI are examined under the following symbols: 1 - a domestic producer; a remelting technology; 2 - a foreign producer; an ore-thermal process; 3 - a foreign producer; a remelting technology. As seen, the chemical composition of the examined inoculants of the three different producers is similar – 44 % - 48 % of Si; 5 % - 6 % of Mg; 1,8 % - 2,3 % of Ca; 5,5 % - 6,5 % of P3M; Al - 1 %; Fe (the rest).

The electron-microscopic images and the results of X-ray spectral analysis of the particles of a graphitizing inoculant № 1 are shown in Figs. 1-7; those of the particles of inoculant № 2 are shown in Figs. 8-11, while those of the particles of the inoculant № 3 are presented in Figs. 12-16. The electron-microscopic images of the back-scattered electrons are also received.

The grey color ratio in the given images characterizes the chemical composition of the examined substance (the lighter the ratio is the greater is the atomic number of the elements comprising the substance according to Mendeleev's Periodic Table). The numerical results of

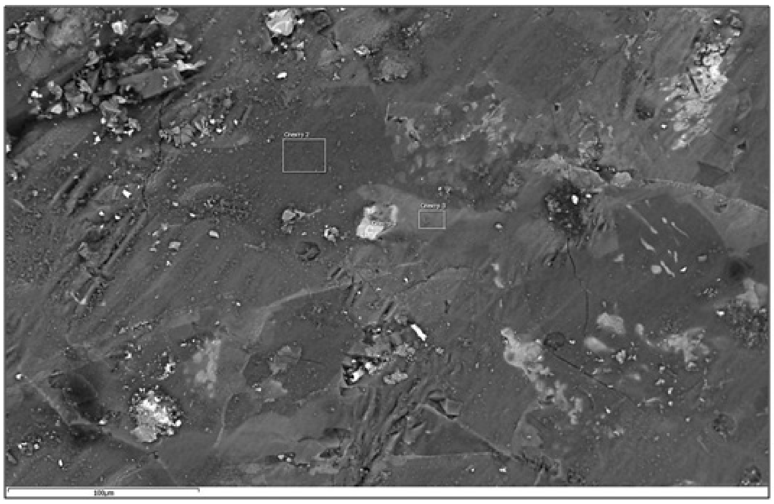

Fig. 1(a). SEM micrograph of the surface of inoculant ingot № 1.

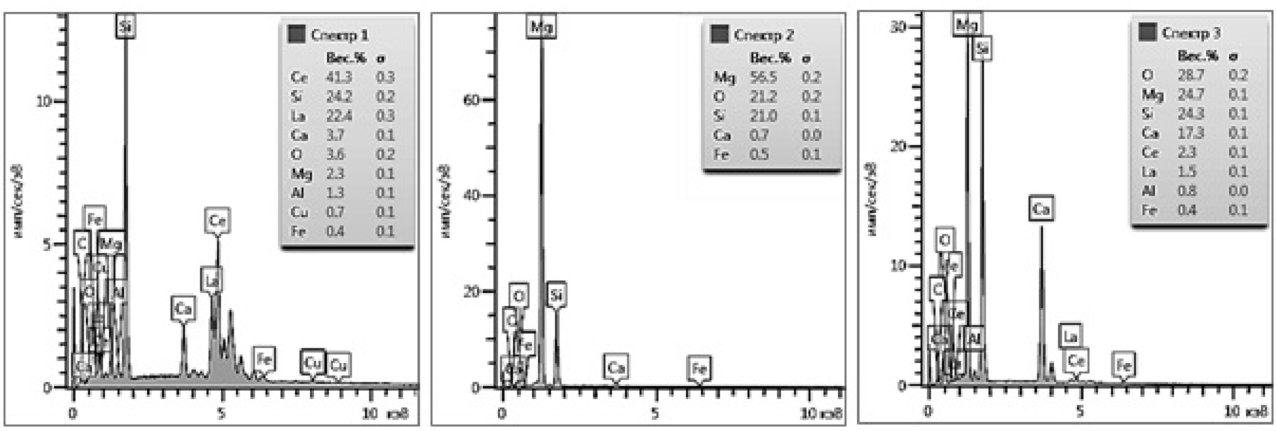

Fig. 1(b). Spectra of specific surface areas.

Table 1. The technological process of getting inoculants / a comparative parameter.

I	*II*
Dedusting of inoculants after crushing into commercial fraction	
Crushing and inoculant sizing is a multi-stage process – from a piece to the required fracture with a gradual decrease of particle size. At every stage, whichever the fraction is, the material goes through dedusting.	Dedusting takes place neither at intermediate crushing stages nor with the finished fraction.
A type and metal consumption of melting facilities for melting inoculants	
An induction crucible furnace.	An induction crucible furnace of ISC type, an arc furnace. The complete technological cycle of getting inoculants from quartz rock by melting is missing. The used melting facilities provide only remelting of the finished Si-iron with the addition of active elements.
Treatment of the inoculant melt including treatment by slag and flux	
The treatment of Si-iron melt (alloyed with elements required to get the target type of an inoculant) is carried out in the same ladle on a special stand. A protective coating is not necessary to prevent the metal from oxidation. The alloying and molding are accompanied by continuous bottom and side jet degassing of liquid metal (by Ar) to provide alloy elements blending.	The treatment of the inoculant melt by special slag, flux and jet degassing by Ar in the course of its preparation is missing. The inoculation into Si-iron melt occurs directly in the melting furnace.
A type of the inoculant melting process	
A carbo-thermal ore-smelting quartz rock process is carried out in industrial ferroalloy furnaces of an open (Fe-Si45 for Fe-Si mg) and enclosed type (Fe-Si 75 for graphitize inoculants). The process provides inoculants from a new-melt refined Si-iron without remelting. There are a number of advantages in comparison with remelting of ordinary finished Si-iron. The finished product is less oxidized and more effective when merging in liquid iron.	The process of an inoculant production is based on remelting ordinary final Si-iron either by inoculation, in a pure form or as an alloy. The cost of the cast inoculant increases as a result of the energy consumption growth in the process of its production. Inoculants increased oxidation and contamination by oxidizing species takes place.
A material of a mold used for casting a new inoculant melt	
Prior to the crushing and sizing the plant metal is cast in a casting machine. The mold material is CGI.	The mold material is cast iron.
An width of the produced inoculant ingot	
The tailored width of the ingot is ≤32 mm. It is determined on the basis of long-term manufacturing practice. It entirely excludes the possibility of element elimination in the liquid metal crystallizing process.	There are no standard requirements in respect to the ingot width. A manufacturer, for instance, provides a 44 mm width of an inoculant layer in a mold. The cooling procedure that causes its excessive oxidation refers to: cooling of the layer by a water jet; pouring of a liquid inoculant over the first solidified layer; formation of a second layer which is then cooled by a water jet.

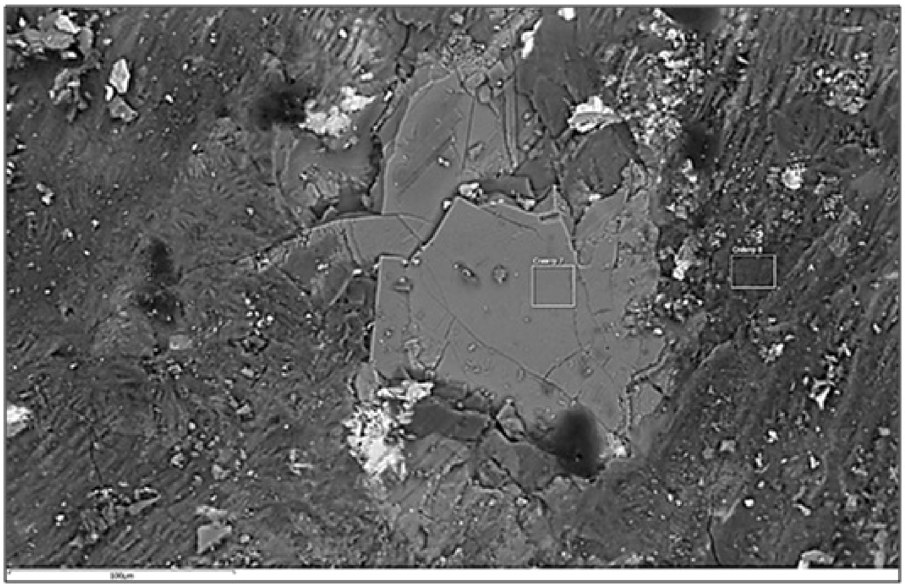

Fig. 2 (a). SEM micrograph of the surface of inoculant ingot № 1.

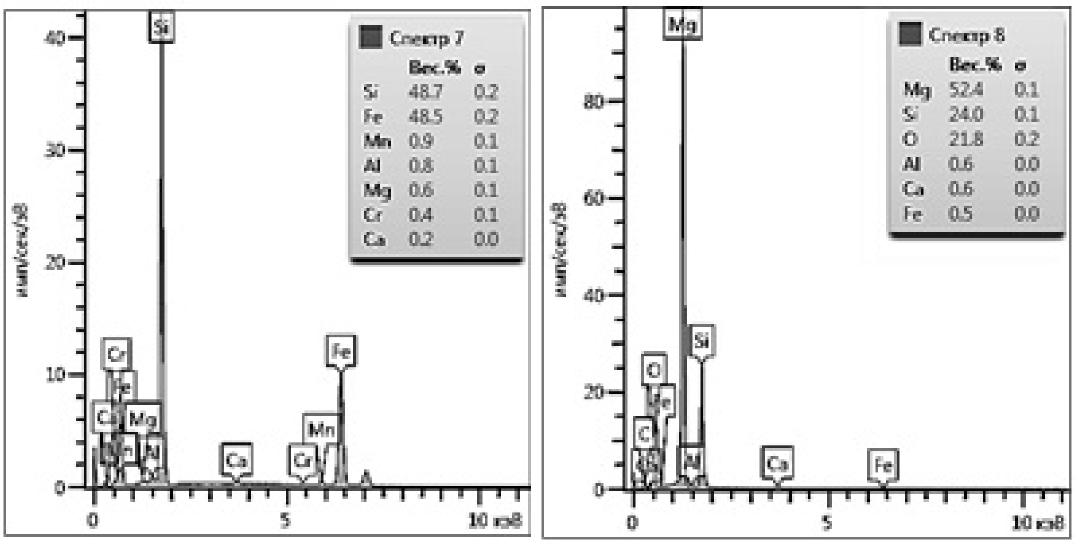

Fig. 2 (b). Spectra of specific surface areas.

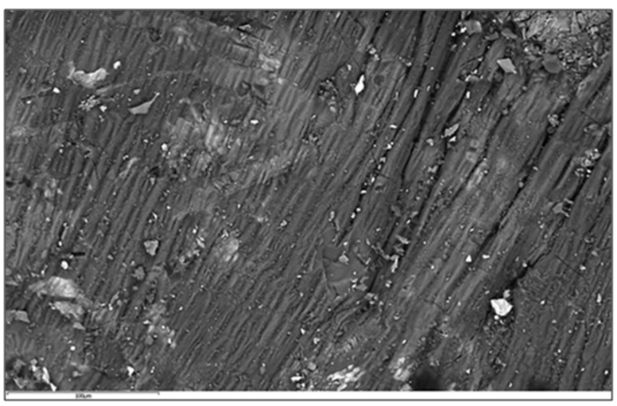

Fig. 3. SEM micrograph of an undulating wrinkle oxide film on the surface of inoculant ingot № 1.

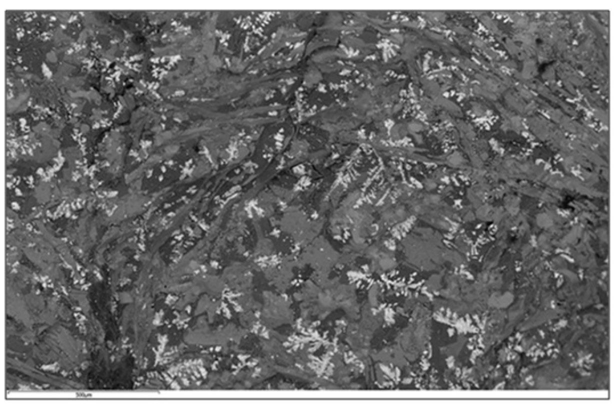

Fig. 4. SEM micrograph of the fracture of inoculant ingot №1.

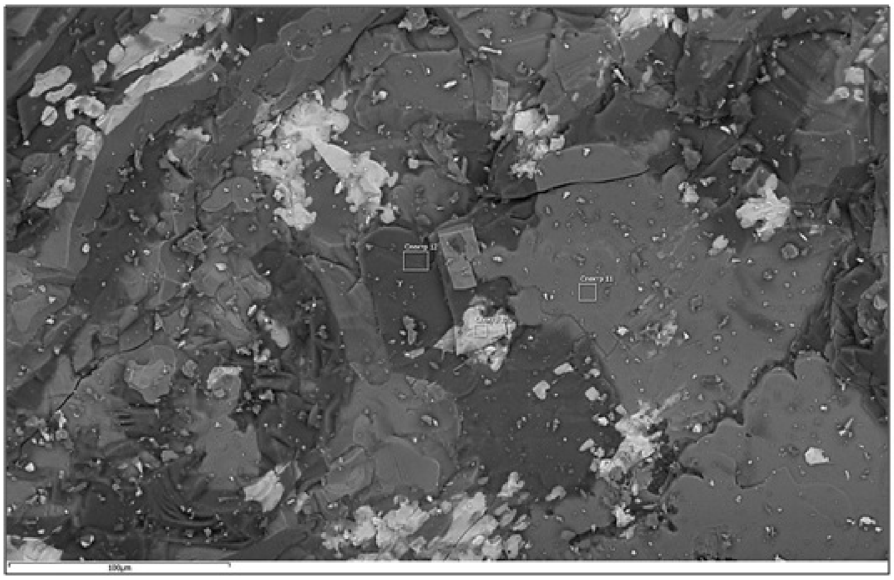

Fig. 5 (a). SEM micrograph of the fracture of inoculant ingot №1.

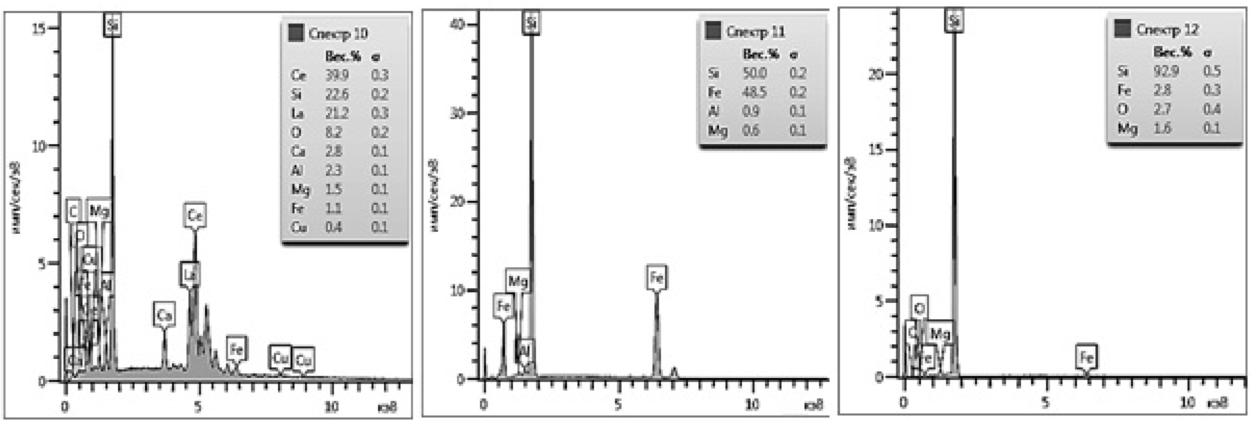

Fig. 5 (b). Spectra of specific surface areas.

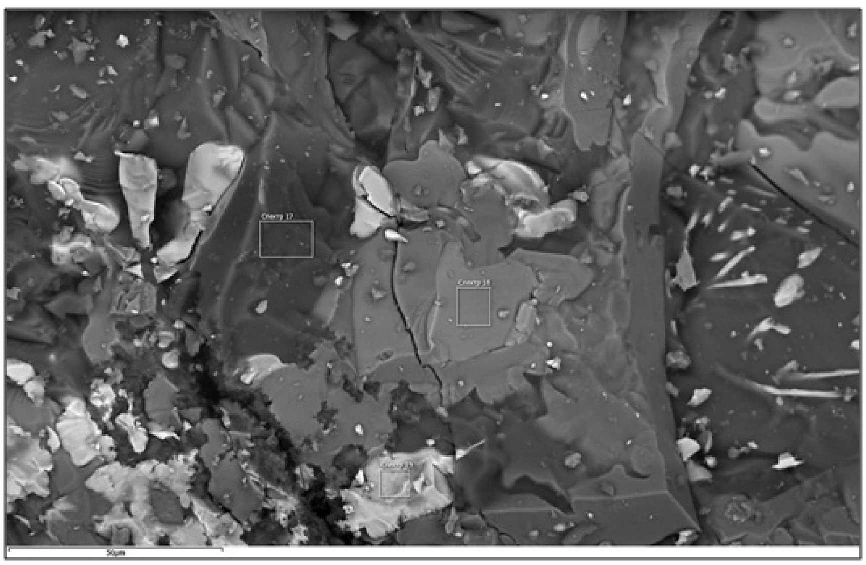

Fig. 6 (a). SEM micrograph of the fracture of inoculant ingot №1.

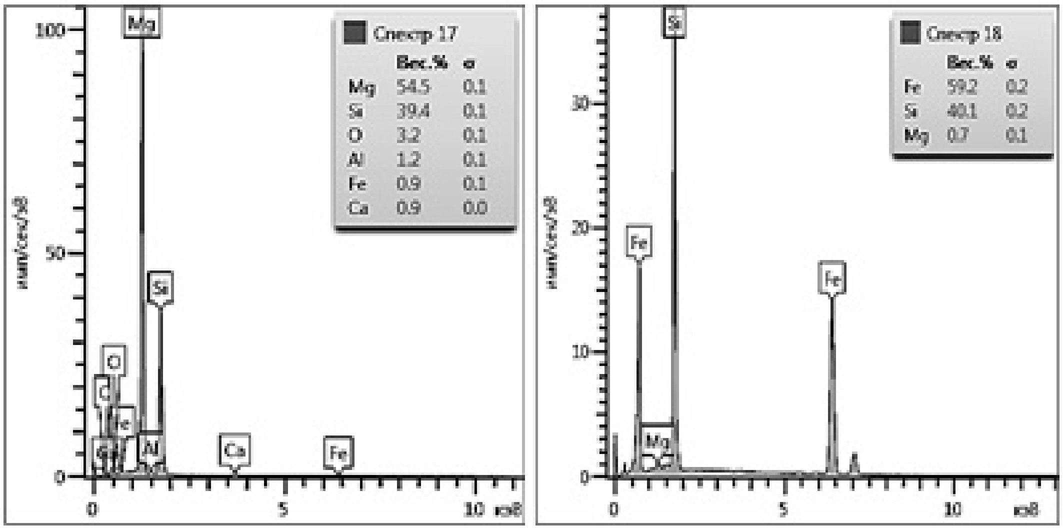

Fig. 6 (b). Spectra of specific surface areas.

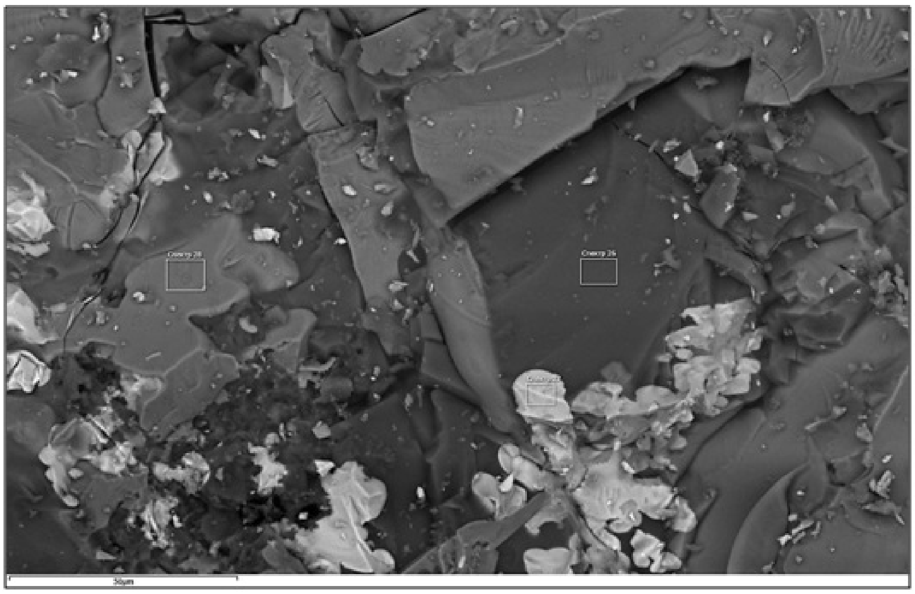

Fig. 7 (a). SEM micrograph of the fracture of inoculant ingot №1.

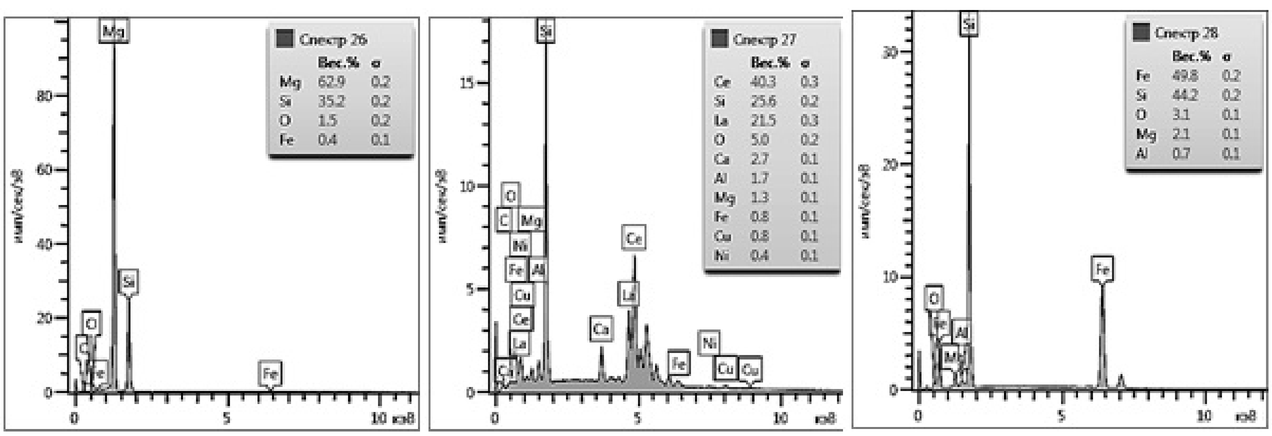

Fig. 7 (b). Spectra of specific surface areas.

Fig. 8 (a). SEM micrograph of the surface of inoculant ingot № 2.

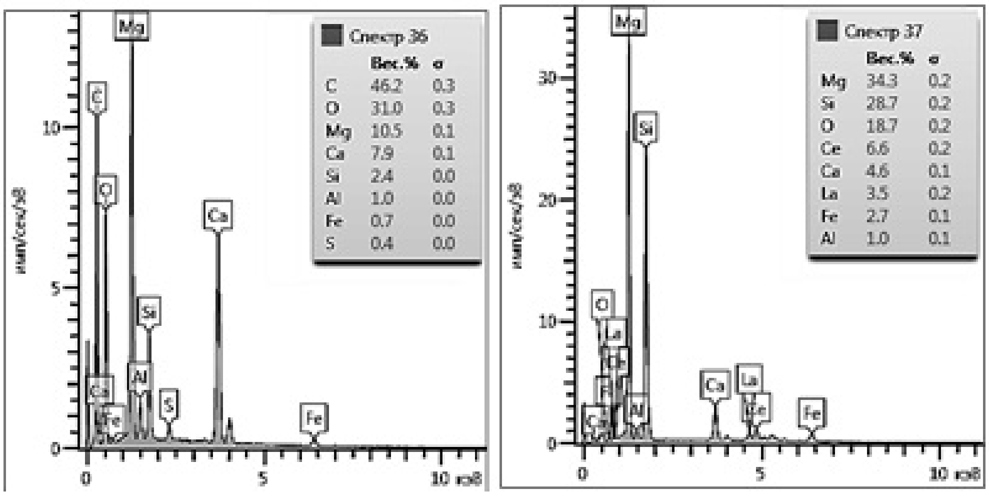

Fig. 8 (b). Spectra of specific surface areas.

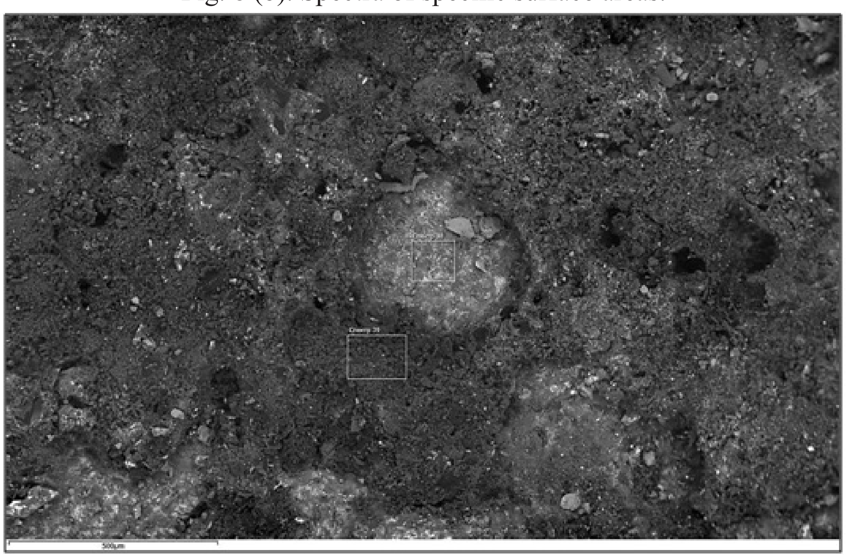

Fig. 9 (a). SEM micrograph of the surface of inoculant ingot № 2.

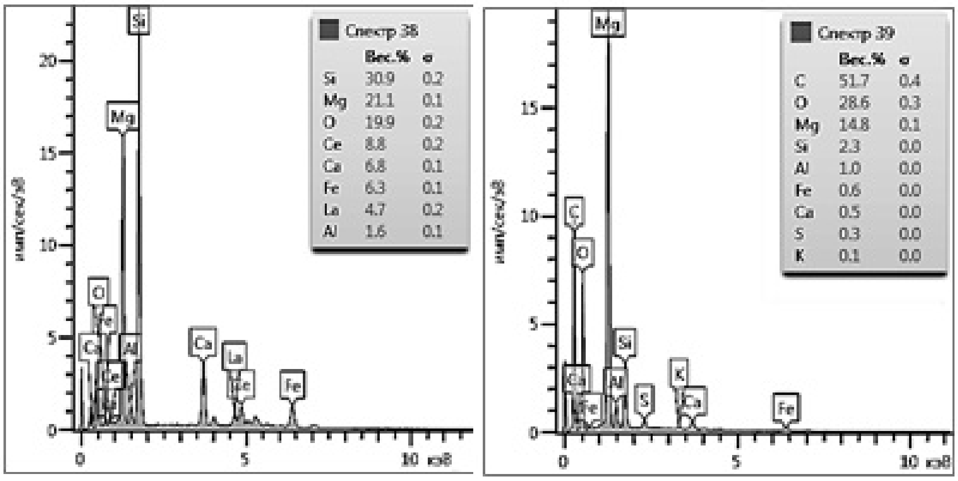

Fig. 9 (b). Spectra of specific surface areas.

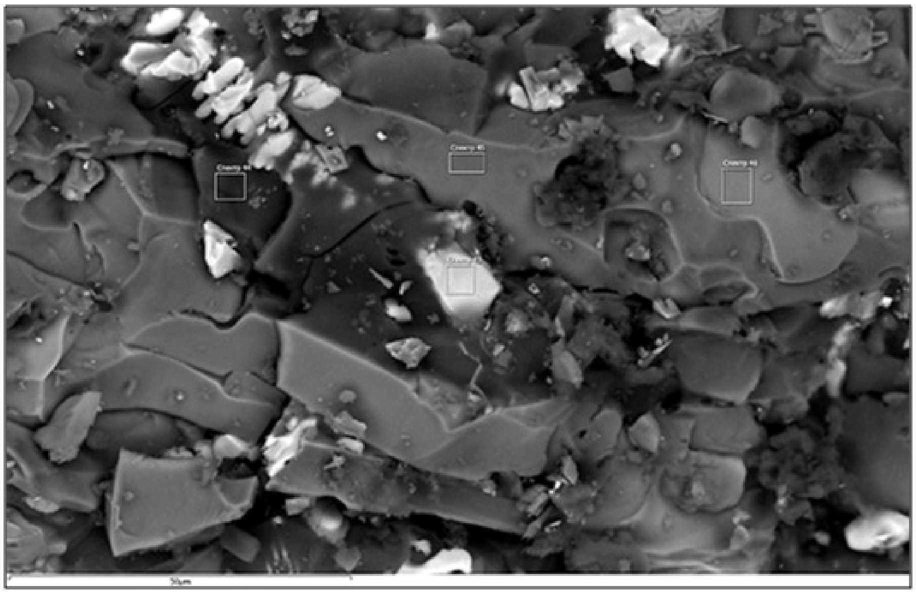

Fig. 10 (a). SEM micrograph of the fracture of inoculant ingot №2.

the elements' weight content are qualitative according to the X-ray spectral analysis carried out.

The electron-microscopic analysis of the inoculant particles surface indicates their dense, mostly solid texture with homogeneous phase distribution. There are three following specific phases in the inoculant particles:

- a binary Si-Mg phase with a different random content of Fe, Ca, Al, REE and O. Of an identical morphology the phase fields of an increased Mg content and a reduced Si content look darker, while those of a reduced Mg content and an increased Si content look lighter in the electron-microscopic images.

- a binary Fe-Si phase of a different correlation of Fe-Si and a random content of Al, Mg and O. The phase can look like both a sponge type mass of an increased Mg content and a separate dispersion crystals and lamellar fragments.

- a binary Si-REE phase with REE $\geq$ Si and a different random content of Fe, Ca, Al, Mg and O. Morphologically it has a form of a skeleton and blur like dendrites localized in both Fe-Si and Si-Mg phases.

- a mono phase of crystalline Si containing different impurities and a random content of Fe, Mg and O. Visually it looks like a Fe-Si phase with an increased

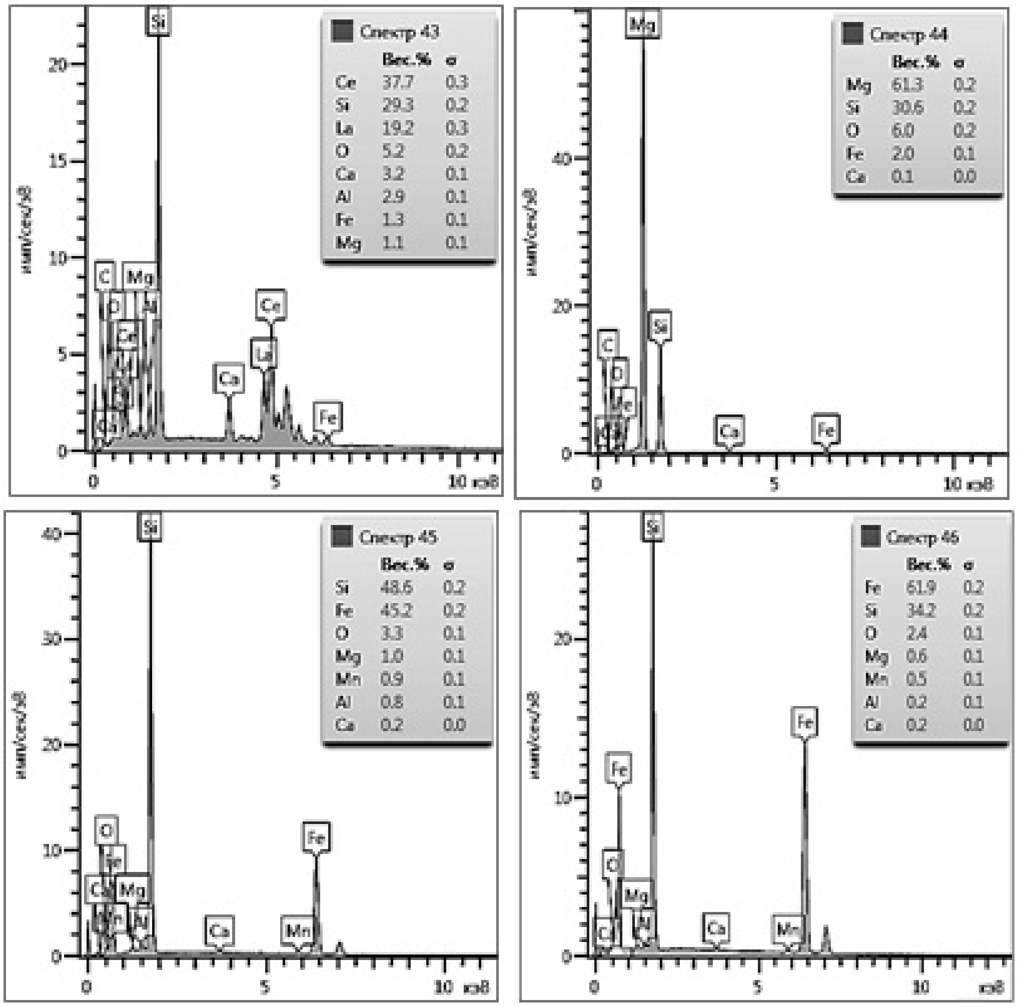

Fig. 10 (b). Spectra of specific surface areas.

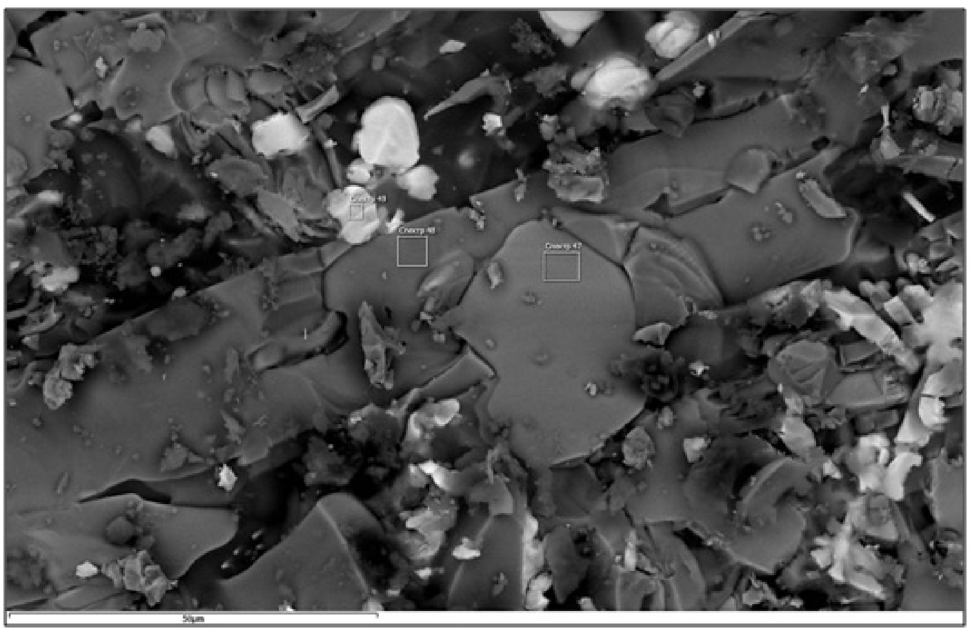

Fig. 11 (a). SEM micrograph of the fracture of inoculant ingot №2.

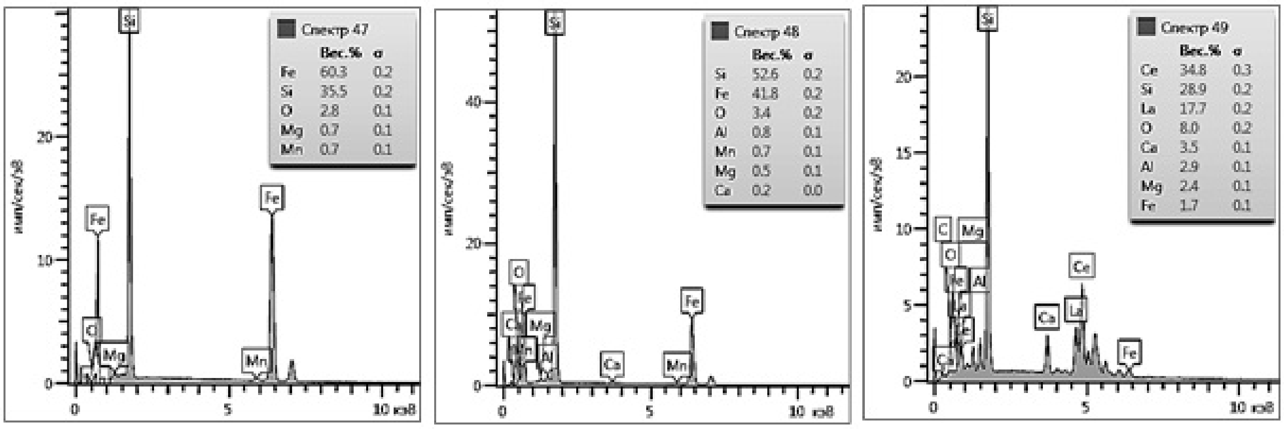

Fig. 11 (b). Spectra of specific surface areas.

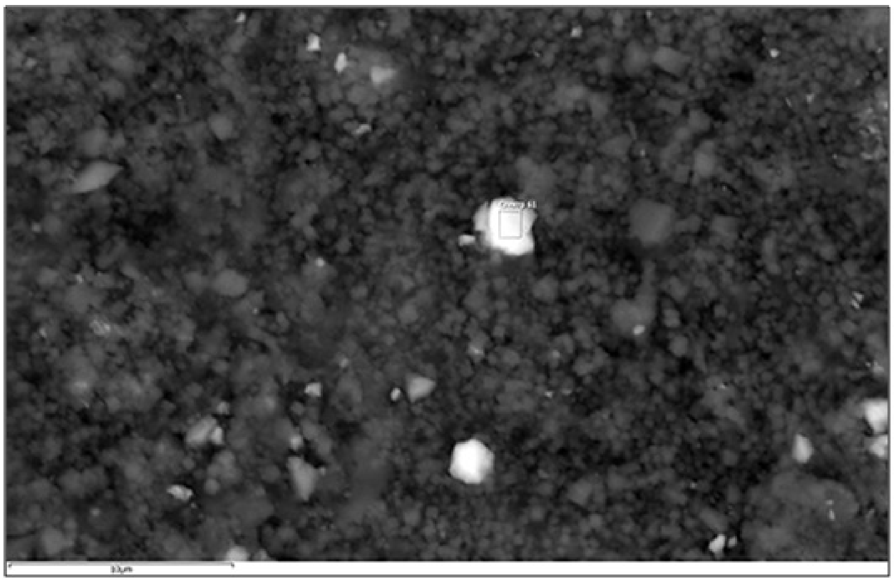

Fig. 12 (a). SEM micrograph of the surface of inoculant ingot № 3.

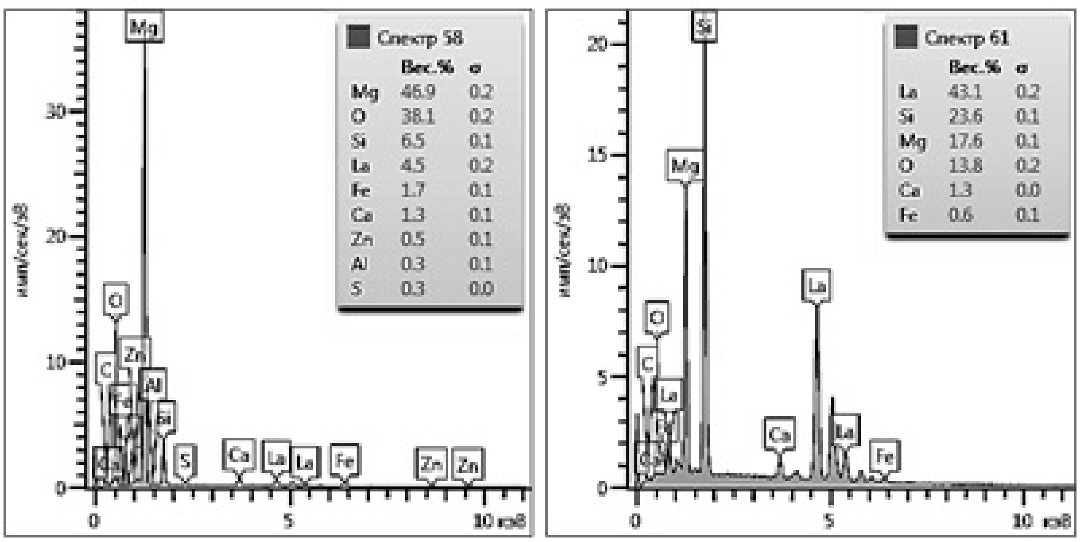

Fig. 12 (b). Spectra of surface areas.

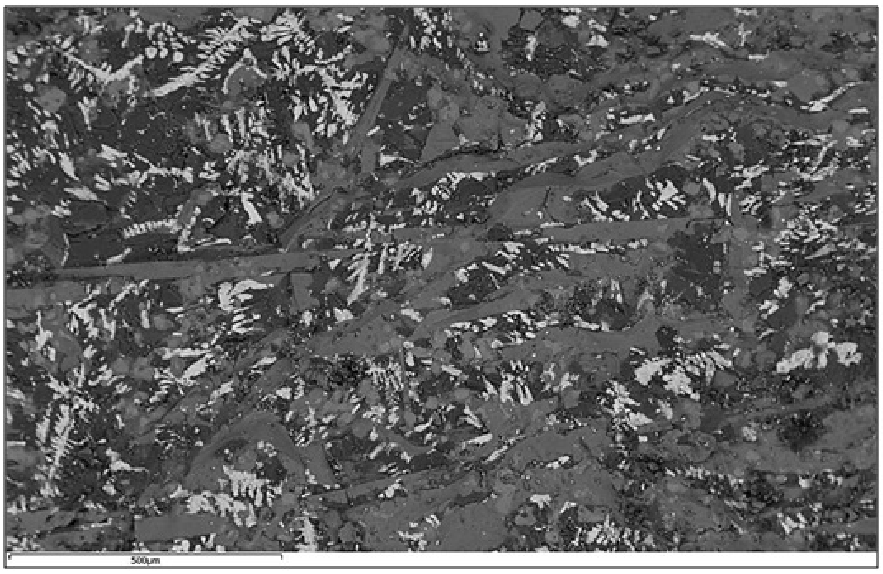

Fig. 13. SEM micrograph of the fracture of inoculant ingot № 3.

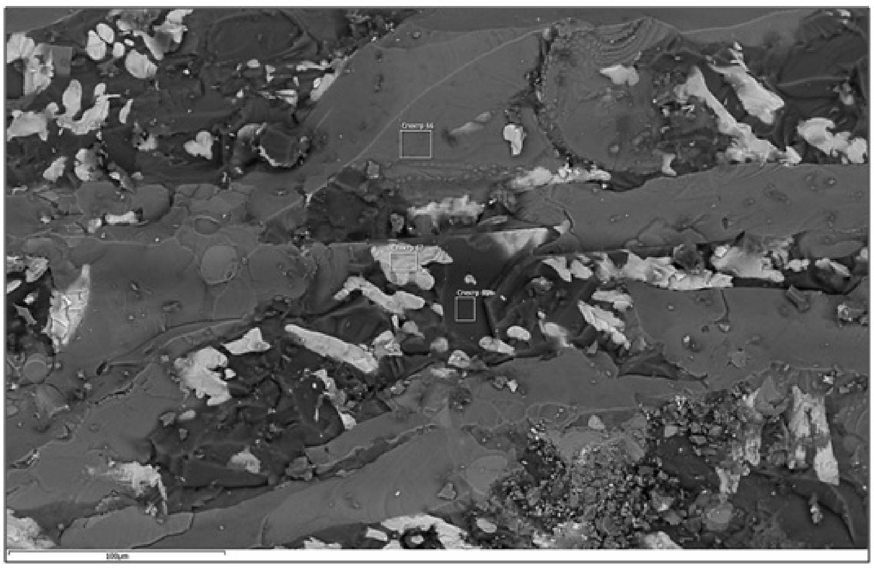

Fig. 14 (a). SEM micrograph of the fracture of inoculant ingot № 3.

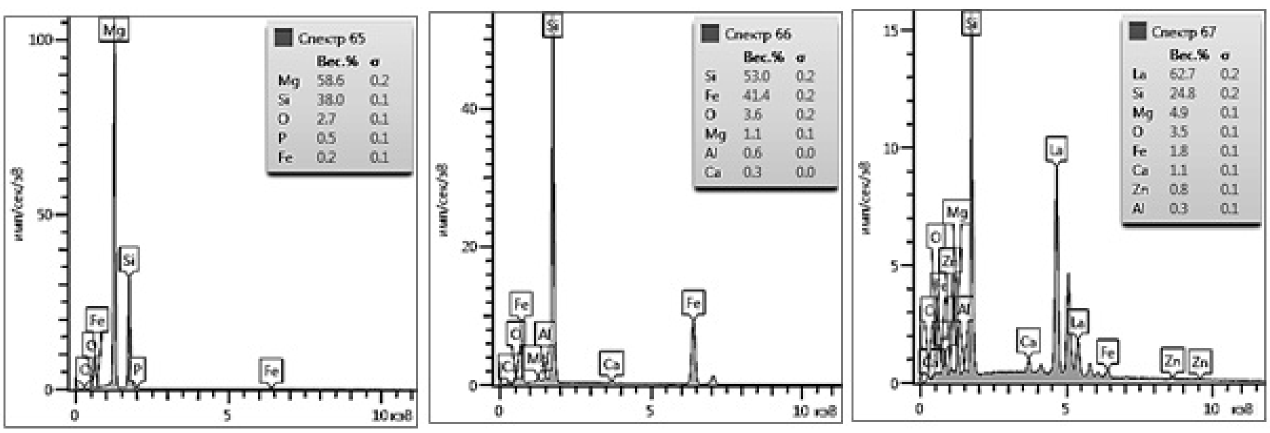

Fig. 14 (b). Spectra of specific surface areas.

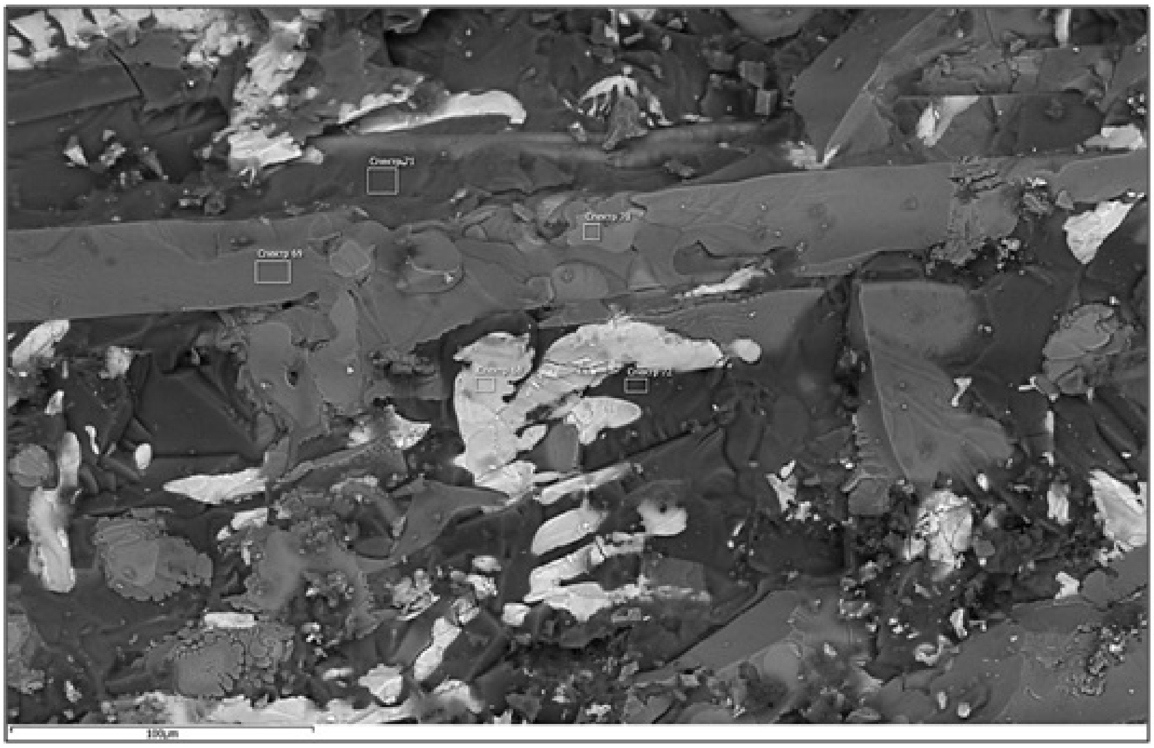

Fig. 15 (a). SEM micrograph of the fracture of inoculant ingot № 3.

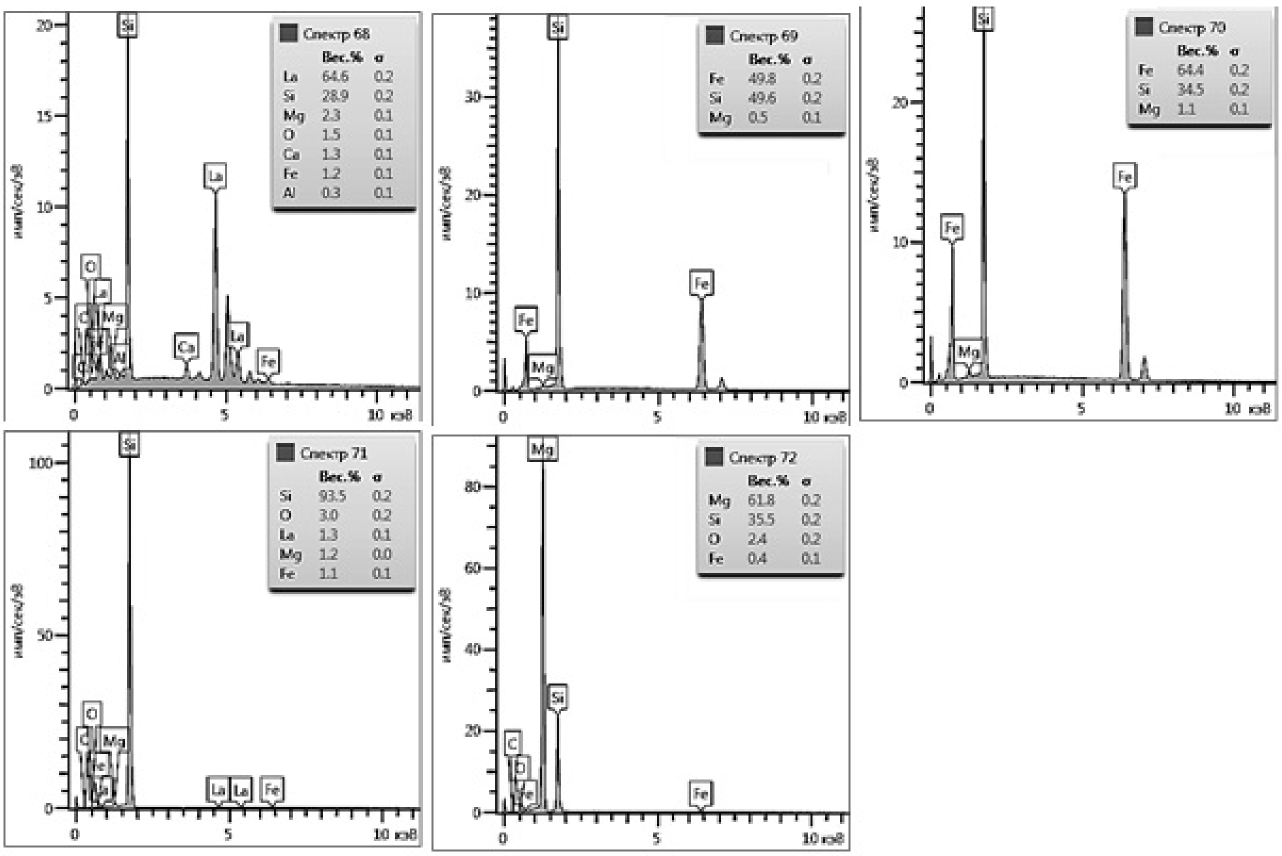

Fig. 15 (b). Spectra of specific surface areas.

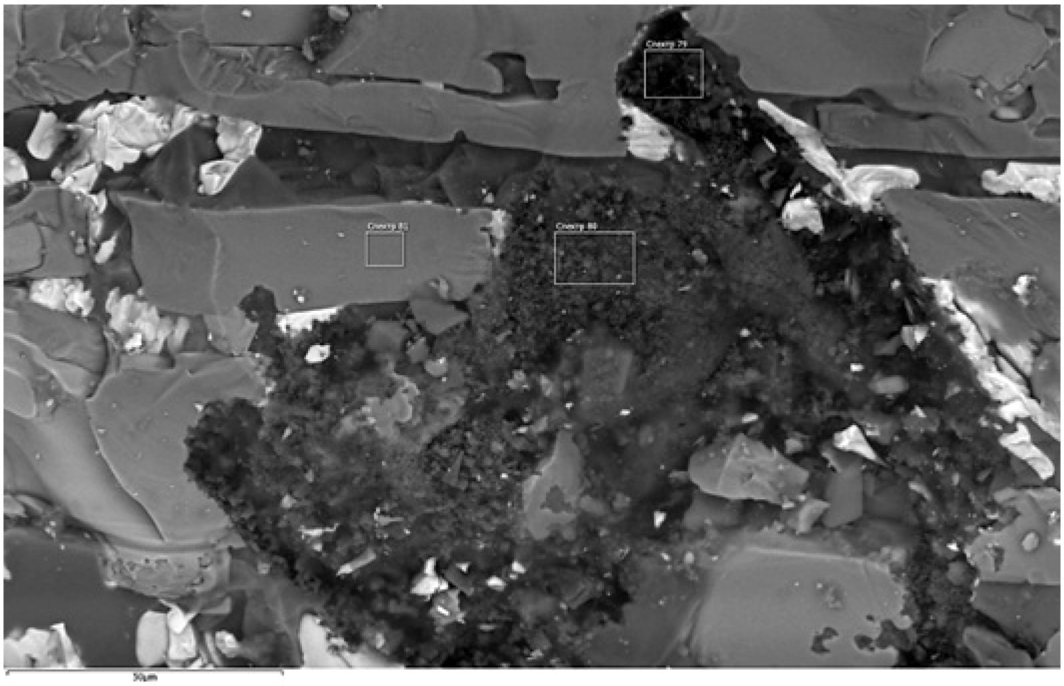

Fig. 16 (a). SEM micrograph of the fracture of inoculant ingot № 3.

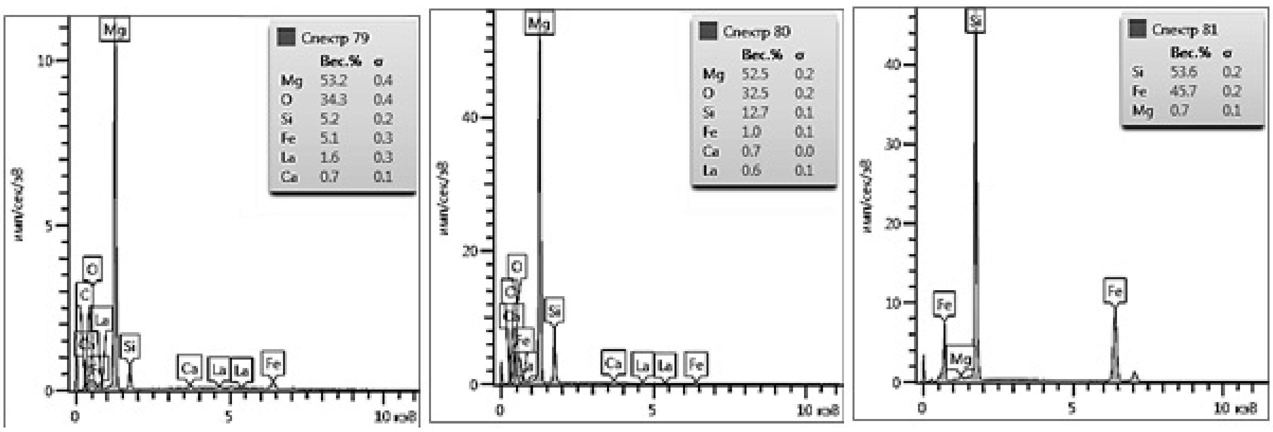

Fig. 16 (b). Spectra of specific surface areas.

Mg content but it's darker than Fe-Si phase.

All chemical elements in Fe-Si graphitizing inoculants can be divided into three groups:

1) An element-transporter → silicon (pure silicon can provide only a weak homogeneous mechanism of forming graphite inclusions);

2) Main inoculant elements (alkali-earth elements) → calcium, barium, strontium;

3) Useful elements → cerium, zirconium, titanium, bismuth, aluminium, sulfur, oxygen.

The inoculant elements can be separately ranked in groups in ascending order according to their efficiency:

(Ca + Al)
↓
(Ca + Ti)
↓
(Ca + Ba)
↓
(Ca + Zr)
↓
(Sr)
↓
(Ca + Ce + S + O)
↓
(Bi + P3M)

CONCLUSIONS

Regarding the three binary phases presented the base of all three alloys contains the quantitatively prevailing Si-Mg and Fe-Si phases with Si-REE phase inclusion.

The fragments of an undulating wrinkle oxide film are present only on the surface of the inoculant particles and are missing in their fracture.

There are cracks and cleavages on both the surface and in the fracture of the inoculant particles. They serve as evidence of a high degree of brittleness. There are no such casting defects as flux contaminations, voids and blowholes.

The following specific features of the inoculants are revealed:

Inoculant №2:

- The surface is characterized by a great graphite-based micro-porosity with a random content of Si, Mg and O. All the phases are characterized by higher dispersion ability than those of inoculants №1 and № 3.

- There are Fe-Si phases in inoculant № 2. They are of a different Fe/Si correlation with Fe>Si and Si>Fe separated by a clear interphase boundary.

- REE are presented as Σ (Ce + La) in inoculants №1 and № 2. La is present only in inoculant №3.

Thus, inoculant № 2 possesses the best quality of a micro-structure. It successfully combines a high chemical purity with a surface micro-porosity of inoculant particles that definitely contributes to better smelting of an inoculant in cast iron due to its more effective grinding and atomization in a liquid phase.

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