INNOVATION IN METALLURGICAL INDUSTRIAL AND LABORATORY EQUIPMENT, TECHNOLOGIES AND MATERIALS

ИННОВАЦИИ В МЕТАЛЛУРГИЧЕСКОМ ПРОМЫШЛЕННОМ И ЛАБОРАТОРНОМ ОБОРУДОВАНИИ, ТЕХНОЛОГИЯХ И МАТЕРИАЛАХ

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Development of a methodology for determining the content of non-metallic inclusions in steel

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Abstract. As part of the study, a method was proposed for assessing the metal purity for non-metallic inclusions using optical emission spectrometry. To assess the content of non-metallic inclusions in the slabs, two columns of metal were selected from two slabs of low-alloy metal deoxidized with aluminum. Each column was divided into seven samples in the direction from the small radius of the continuously cast ingot to the large one. We studied these samples to assess metal contamination with non-metallic inclusions using quantitative optical metallography according to ASTM E1245-03, fractional gas analysis (FGA) and optical emission spectral analysis PDA. Analysis of the samples according to ASTM E1245-03 standard showed that in all samples the percentage of oxides and sulfides is on average 10 and 90 %, respectively. According to the results of FGA, it was concluded that such non-metallic oxide inclusions as aluminates predominate in the metal samples of both ingots. A comparison was made between the results of the determination of oxygen in non-metallic inclusions obtained by FGA method and the number of sparks in inclusions at the analysis by PDA method; analysis of the dependencies showed that there are two clearly defined point distributions. To carry out PDA analysis, a program was developed that allows determining the number of inclusions of various types and calculate their volume fraction.

Keywords: non-metallic inclusions, ASTM E1245-03, fractional gas analysis, PDA

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Разработка методики определения содержания неметаллических включений в стали

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Аннотация. В рамках исследования предложена методика оценки чистоты металла по неметаллическим включениям (НВ) с использованием оптико-эмиссионной спектрометрии. Для оценки содержания НВ в слябах отобраны по два столбика металла от двух слябов низколегированного металла, раскисленного алюминием. Каждый столбик поделен на семь образцов в направлении от малого радиуса непрерывнолитой заготовки к большому. На данных образцах проведены исследования по оценке загрязненности металла НВ методами количественной оптической металлографии по стандарту ASTM E1245-03, фракционного газового анализа (ФГА) и оптико-эмиссионного спектрального PDA анализа. Исследования по стандарту ASTM E1245-03 показали, что во всех образцах процентное соотношение содержания оксидов и сульфидов в среднем составляет 10 и 90 % соответственно. По результатам ФГА

сделан вывод о том, что в пробах металла обоих слитков преобладают такие оксидные НВ, как алюминаты. Проведено сравнение результатов определения содержания кислорода в НВ, полученного методом ФГА, и количества попаданий искр (спарков) во включения при анализе методом PDA. Анализ зависимостей показал, что есть два четко выраженных распределения точек. Для проведения анализа методом PDA разработана программа, позволяющая определить количество НВ различных типов в образцах металла и рассчитать их объемную долю.

Ключевые слова: неметаллические включения, ASTM E1245-03, фракционный газовый анализ, PDA

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Introduction

The quality of steel is significantly influenced by nonmetallic inclusions (NMI). The presence of NMI in the finished metal disrupts its homogeneity, degrades surface properties, fatigue strength, and plastic characteristics of the metal [1 – 3]. NMIs act as stress concentrators during deformation, rolling, and stamping of the steel sheet, which subsequently leads to the formation of surface defects [4 – 7]. A negative effect of increased NMI content in molten steel is also the clogging of steel casting nozzles, which drastically reduces casting speed and impairs production efficiency [8].

Various production factors affect the quantity, shape, size, and type of oxide NMIs in steel:

– chemical composition, oxidation state, temperature of the steel and slag;

– chemical and fractional composition of deoxidizers, slag-forming and alloying materials, and their introduction regime;

– inert gas blowing regime of the melt;

– vacuum treatment technology;

– chemical composition of the lining.

Inclusions larger than 50 μm are unevenly distributed and are significantly fewer in number. However, they substantially impact the quality of the finished product, as fatigue failure always occurs in the vicinity of large NMIs regardless of their composition [4; 9 – 11].

Most researchers consider NMIs as initiation sites for hydrogen cracking [12 – 14]. There is a correlation between $CaO-Al_2O_3$, $MgO-Al_2O_3$ inclusions and internal and external irregularity defects in the metal [15; 16].

The aim of this study is to compare methods for determining the contamination of metal by NMIs. One of the most common methods for quantitatively assessing NMI content in steel is metallographic analysis – comparing the sizes and shapes of NMIs found in the metal with standard scales using point-counting scales and quantitative optical microscopy as per ASTM E1245-03 standard, determining the volume fraction and size distribution of inclusions [17 – 20].

The fractional gas analysis (FGA) method allows determining the total oxygen content in various types of oxide NMIs and their volume fraction, providing a more comprehensive picture of the various types of NMIs present in the steel [21 – 23].

Assessing metal contamination by NMIs using optical microscopy as per ASTM E1245-03

The assessment of metal contamination by NMIs using optical microscopy according to ASTM E1245-03 standard was conducted with an optical microscope on a properly prepared polished specimen. Images were captured with a camera. The recognition and identification of inclusions were based on differences in grey level intensity when compared with each other and with the unetched matrix. The measurements and classification of NMIs depended on the nature (oxides, sulfides) of the identified elements in the image. These measurements were performed in each selected field of view. The polished specimen surface needed to be sufficiently large (at least 160 mm^2) to measure at least 100 fields of view at the required magnification.

For the study of NMI content, two columns from two continuously cast billets were selected. One column was taken from the middle of the continuously cast billet, and the other from the 1/4 width zone of the ingot. Each column was divided into seven samples. The sampling scheme is shown in Fig. 1. Table 1 presents the sample labeling.

Samples *1*, *2*, *4*, *6*, and *7* from each column were analyzed. Samples *3* and *5* were not examined.

To prepare samples for evaluating metal contamination by NMIs using optical microscopy as per ASTM E1245-03 standard, metal samples were ground and polished to achieve the required surface quality. The analysis per ASTM E1245-03 standard was conducted on a 200 mm^2 surface area for each sample. As a result of the analysis, two main types of NMIs were identified: oxides and sulfides. Oxide-sulfide compounds were also observed as oxysulfides. These oxysulfide inclusions were divided into two parts based on grey shades: oxide and sulfide. Table 2 presents the results

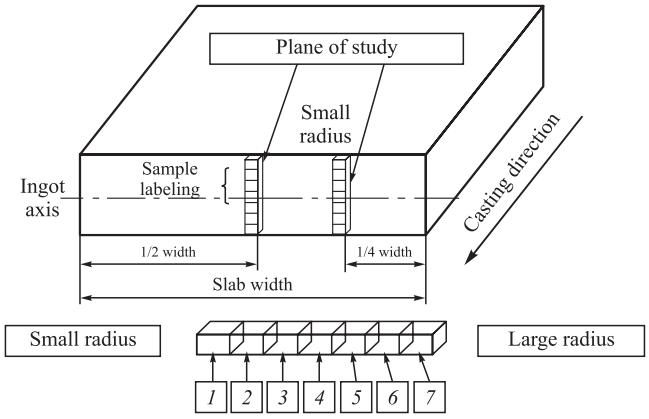

Fig. 1. Sampling scheme

Рис. 1. Схема отбора образцов

Table 1. **Sample labeling**

Таблица 1. **Маркировка образцов**

Sample label	Sample description
11X	Column from 1/2 width of slab *1*, *X* – sample number in the column
12X	Column from 1/4 width of slab *1*, *X* – sample number in the column
21X	Column from 1/2 width of slab *2*, *X* – sample number in the column
22X	Column from 1/4 width of slab *2*, *X* – sample number in the column

of the calculation of the total volume fraction of NMIs and separately the volume fractions of oxide and sulfide inclusions.

The study results indicated that the average percentage content of oxides and sulfides in the metal across all samples was 10 and 90 %, respectively. However, in samples 111 and 121, the relative oxide content was higher (31 and 17 % sulfides, respectively), indicating uneven distribution of NMIs. Uneven distribution of NMIs was also observed in samples located closer to the small radius (111, 121). The upper zone of the samples showed minimal NMI content, with their size not exceeding 10 μm, and the majority of inclusions located below 1/3 of the sample height. Thus, the upper 1/3 of the samples were cleaner in terms of inclusions than the lower 2/3. Samples (111, 112, 121, 122, 211, 212, 221, 222) closest to the small radius had a higher total volume fraction of NMIs compared to other samples. Figs. 2 – 5 compare the results of total volume fraction calculations of NMIs obtained from the same samples in the laboratories of PJSC "Novolipetsk Metallurgical Plant" (PCL and R&D) and Laboratory No. 17 of IMET RAS.

Figs. 2 – 5 show that the metal purity by NMIs in the second slab was slightly higher than in the first slab. The total volume fraction of NMIs in the metal of the second slab did not exceed 0.035 %, whereas in the first slab it varied from 0.020 to 0.055 %. It can be concluded that the second slab was more homogeneous in NMI content throughout its height and contained fewer inclusions than the first slab.

Determining metal contamination by NMIs using fractional gas analysis (FGA)

To determine the content of oxide NMIs formed in the steel, fractional gas analysis (FGA) was conducted on the selected metal samples. The FGA method allows the determination of total oxygen and nitrogen content, the amount of oxygen in various types of oxide NMIs, and the calculation of the volume fraction of different types of oxide NMIs. The main advantage of the FGA method is that it provides rapid information on the total oxygen and nitrogen content in the metal, as well as the oxygen distributed in different types of oxide NMIs.

Table 2. **Total volume fraction of non-metallic inclusions in the samples and volume fraction of oxide and sulfide inclusions, %**

Таблица 2. **Объемная доля всех НВ в образцах и объемная доля оксидных и сульфидных включений, %**

Sample	NMIs total volume fraction	Oxide NMIs	Sulfide NMIs
111	0.0444	0.0139	0.0305
112	0.0447	0.0047	0.0400
114	0.0230	0.0020	0.0210
116	0.0220	0.0020	0.0200
117	0.0104	0.0007	0.0097
121	0.0553	0.0093	0.0460
122	0.0391	0.0031	0.0360
124	0.0379	0.0029	0.0350
126	0.0286	0.0026	0.0260
127	0.0219	0.0019	0.0200
211	0.0274	0.0024	0.0250
212	0.0298	0.0018	0.0280
214	0.0215	0.0025	0.0190
216	0.0265	0.0025	0.0240
217	0.0200	0.0020	0.0180
221	0.0250	0.0020	0.0230
222	0.0264	0.0014	0.0250
224	0.0292	0.0022	0.0270
226	0.0204	0.0024	0.0180
227	0.0186	0.0016	0.0170

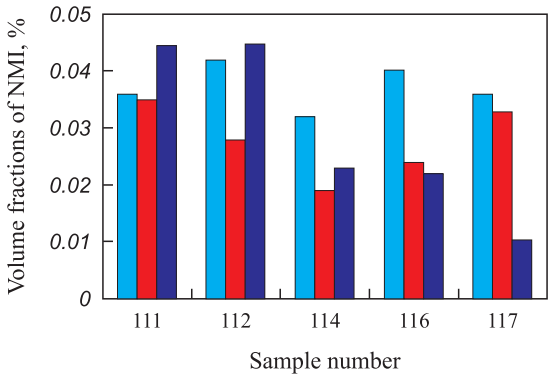

Fig. 2. Comparison of the results of calculating the volume fractions of non-metallic inclusions for the samples from the central column of *1* ingot:
– PCL; – R&D; – Laboratory No. 17

Рис. 2. Сравнения результатов расчета объемных долей НВ для образцов, отобранных от центрального столбика слитка *1*:
– ЦЛК; – R&D; – Лаборатория № 17

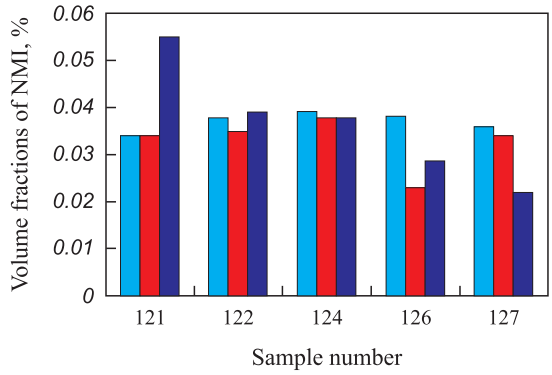

Fig. 3. Comparison of the results of calculating the volume fractions of non-metallic inclusions for the samples from the outer column of *1* ingot:
– PCL; – R&D; – Laboratory No. 17

Рис. 3. Сравнения результатов расчета объемных долей НВ для образцов, отобранных от крайнего столбика слитка *1*:
– ЦЛК; – R&D; – Лаборатория № 17

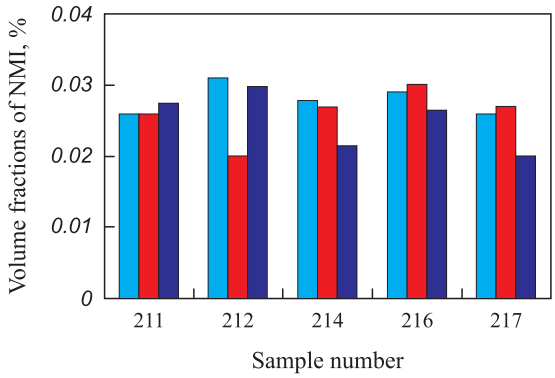

Fig. 4. Comparison of the results of calculating the volume fractions of non-metallic inclusions for the samples from the central column of *2* ingot:
– PCL; – R&D; – Laboratory No. 17

Рис. 4. Сравнения результатов расчета объемных долей НВ для образцов, отобранных от центрального столбика слитка *2*:
– ЦЛК; – R&D; – Лаборатория № 17

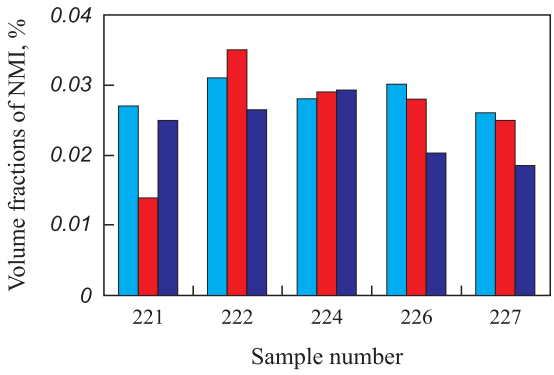

Fig. 5. Comparison of the results of calculating the volume fractions of non-metallic inclusions for the samples from the outer column of *2* ingot:
– PCL; – R&D; – Laboratory No. 17

Рис. 5. Сравнения результатов расчета объемных долей НВ для образцов, отобранных от крайнего столбика слитка *2*:
– ЦЛК; – R&D; – Лаборатория № 17

FGA is a modification of the reduction melting method in a graphite crucible under a stream of carrier gas at a specified linear heating rate of the sample. The analysis method is based on the difference in the temperature dependence of the thermodynamic stability of oxides, which contain the majority of the bound oxygen in the metal. As the melt temperature increases, oxides are reduced by carbon, and oxygen is extracted from the melt as carbon monoxide according to the reaction

$$Z_xO_y(k) + y\text{C(graph.)} = x[Z](\text{Fe} - \text{C(sat.)}) + y\text{CO(g)}.$$

The reduction of oxide NMIs contained in the metal is a complex process involving several stages, such as:

– melting of the sample and spreading of the melt over the graphite crucible;

– diffusion of carbon from the graphite crucible into the sample material;

– dissociation and reduction of oxide inclusions by carbon in the melt with the formation of CO molecules and bubbles;

– internal mass transfer of reaction products to the sample surface;

– removal of reaction products from the reaction surface and mass transfer in the gas phase.

A typical curve of carbon dioxide emission intensity from a metal sample depending on the melt temperature is shown in Fig. 6. The FGA results, processed using the proprietary software “Oxide Separation Pro” are shown in Fig. 7.

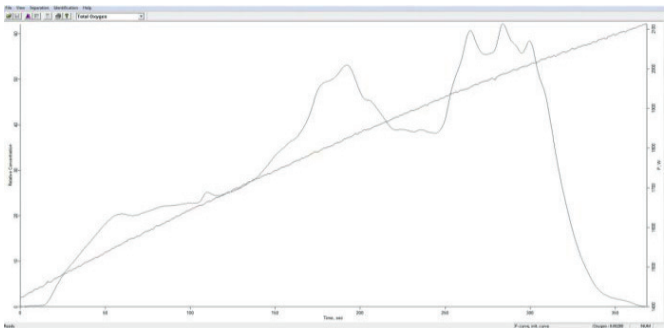

Fig. 6. Gas emission curve from the sample (evologram)

Рис. 6. Кривая газовыделения из образца (эвологрaмма)

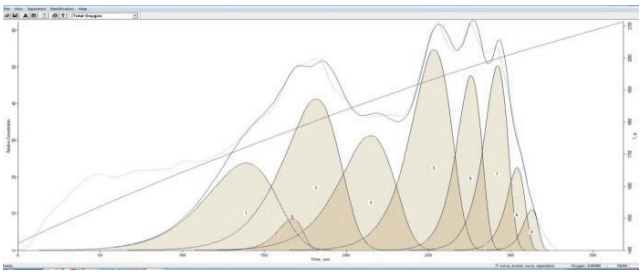

Fig. 7. Processing PGA results in Oxide Separation Pro program

Рис. 7. Обработка результатов ФГА в программе Oxide Separation Pro

For the FGA studies, three samples weighing 1.0 – 1.5 g were cut from each metal sample. Their surface was cleaned with a file to remove the oxide film and contaminants. After mechanical cleaning, the samples were washed with alcohol and dried. The FGA results are presented in Figs. 8 – 11.

The average values and standard deviations (SD) of the results for total oxygen and nitrogen, as well as

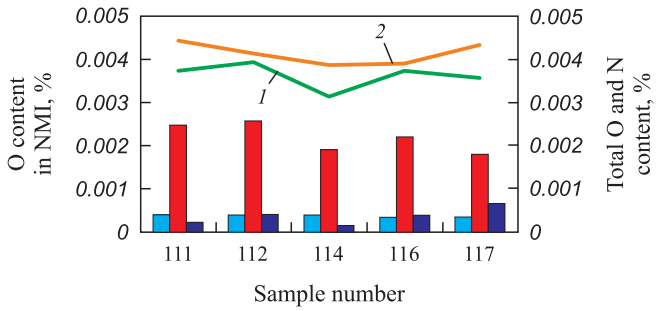

Fig. 8. FGA results of the samples from the central column of ingot *1*:
■ – silicates; ■ – aluminates; ■ – spinel;
1 – oxygen; *2* – nitrogen

Рис. 8. Результаты ФГА образцов от центрального столбика слитка *1*:
■ – силикаты; ■ – алюминаты; ■ – шпинель;
1 – кислород; *2* – азот

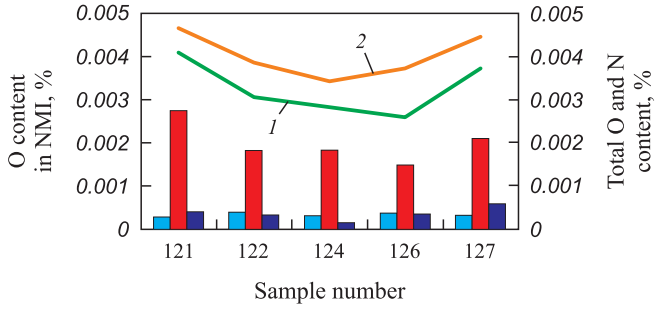

Fig. 9. FGA results of the samples from the outer column (1/4 width) of ingot *1*:
■ – silicates; ■ – aluminates; ■ – spinel;
1 – oxygen; *2* – nitrogen

Рис. 9. Результаты ФГА образцов крайнего столбика (1/4 ширины) слитка *1*:
■ – силикаты; ■ – алюминаты; ■ – шпинель;
1 – кислород; *2* – азот

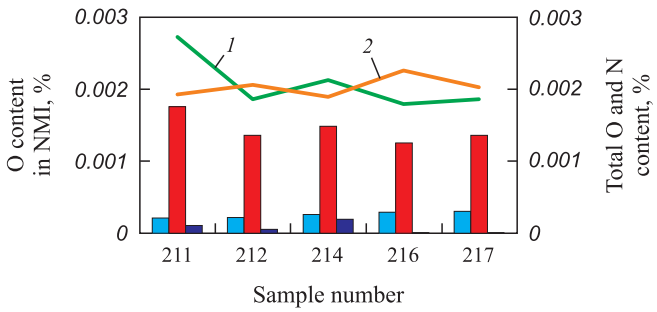

Fig. 10. FGA results of the samples from the central column of ingot *2*:
■ – silicates; ■ – aluminates; ■ – spinel;
1 – oxygen; *2* – nitrogen

Рис. 10. Результаты ФГА образцов центрального столбика слитка *2*:
■ – силикаты; ■ – алюминаты; ■ – шпинель;
1 – кислород; *2* – азот

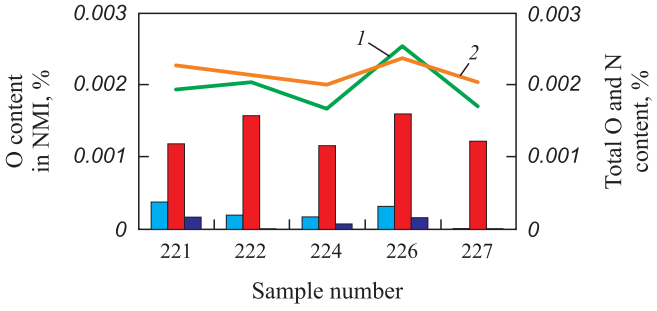

Fig. 11. FGA results of the samples from the outer column (1/4 width) of ingot *2*:
■ – silicates; ■ – aluminates; ■ – spinel;
1 – oxygen; *2* – nitrogen

Рис. 11. Результаты ФГА образцов крайнего столбика (1/4 ширины) слитка *2*:
■ – силикаты; ■ – алюминаты; ■ – шпинель;
1 – кислород; *2* – азот

Table 3. **Average values and SD of total oxygen and nitrogen, oxygen in non-metallic inclusions for ingots *1* and *2*, %**

Таблица 3. **Средние значения и СКО общего кислорода и азота, кислорода в НВ для слитков *1* и *2*, %**

Ingot	O	O (SD)	N	N (SD)	O in NMIs	O in NMIs (SD)
1	0.0034	0.0005	0.0041	0.0004	0.0028	0.0004
2	0.0020	0.0004	0.0021	0.0002	0.0016	0.0002

oxygen in NMIs, for samples from ingots *1* and *2* are presented in Table 3.

The FGA results indicate that the metal samples from both ingots predominantly contain oxide NMIs such as aluminates (Figs. 8 – 11). Fig. 8 and 9 for ingot *1* show an increasing trend in spinel content from the central part of the columns (samples 114, 124) to the large radius of the ingot (samples 116, 117, 126, 127). In the samples of ingot *2* (Figs. 10, 11), unlike ingot *1*, spinel-type inclusions are virtually absent. The average total oxygen content in ingot 1 is 0.0036 % for samples 11X and 0.0033 % for samples 12X. The average nitrogen content is 0.0041 and 0.0040 %, respectively. In ingot *2*, the average total oxygen content is 0.0021 % for samples 21X and 0.0020 % for samples 22X. The average nitrogen content is 0.0020 and 0.0022 %, respectively.

Based on the average oxygen, nitrogen, and oxygen in NMIs, it can be concluded that ingot *2* is cleaner than ingot *1*. This corresponds to the results obtained when calculating the volume fractions of inclusions using the metallographic method. Fig. 12 and 13 present correlations between the oxygen content in NMIs obtained by the FGA method in the studied samples and the number of sparks in inclusions during optical-emission spectrometric PDA analysis for identical samples on two different spectrometers (R&D and PCL). Fig. 14 shows the correlation between the total oxygen content in NMIs and the oxygen content in aluminates obtained by the FGA method. Fig. 15 shows the correlation between the oxygen content in NMIs obtained by the FGA method and the oxide content obtained from polished specimens analyzed by optical microscopy according to ASTM E1245-03 standard in Laboratory No. 17.

Fig. 12 and 13 highlight two regions of data points. The first region corresponds to the results from ingot *2*, and the second to ingot *1*.

A clear correlation is observed between the oxygen content in NMIs and the oxygen content in aluminates obtained by FGA (Fig. 14).

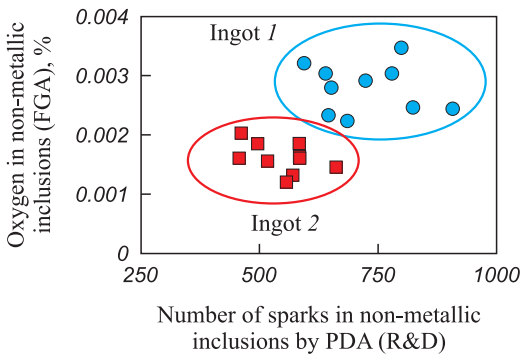

Fig. 12. Correlation between the oxygen content in non-metallic inclusions obtained by FGA method and number of sparks in inclusions by PDA (R&D) method for the samples of different ingots

Рис. 12. Корреляция между содержанием кислорода в НВ, полученного методом ФГА, и количеством попаданий спарков во включения методом PDA (R&D) для образцов разных слитков

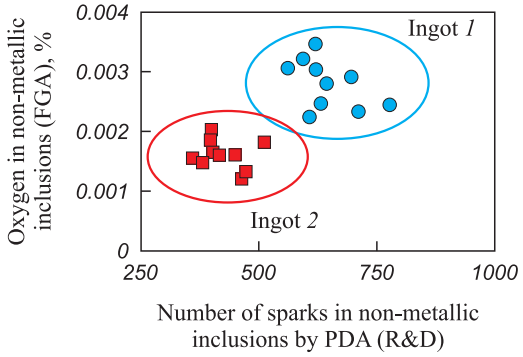

Fig. 13. Correlation between oxygen content in non-metallic inclusions obtained by FGA method and number of sparks in inclusions by PDA method (Plant Central Laboratory – PCL)

Рис. 13. Корреляция между содержанием кислорода в НВ, полученного методом ФГА, и количеством попаданий спарков во включения методом PDA (ЦЛК) для образцов разных слитков

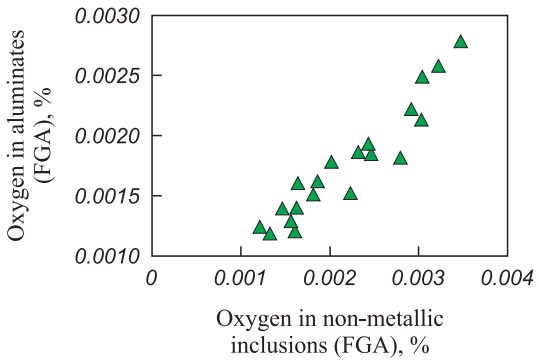

Fig. 14. Correlation between oxygen content in non-metallic inclusions and amount of oxygen in aluminates obtained by FGA method

Рис. 14. Корреляция между общим содержанием кислорода в НВ и количеством кислорода, содержащегося в алюминатах, полученных методом ФГА

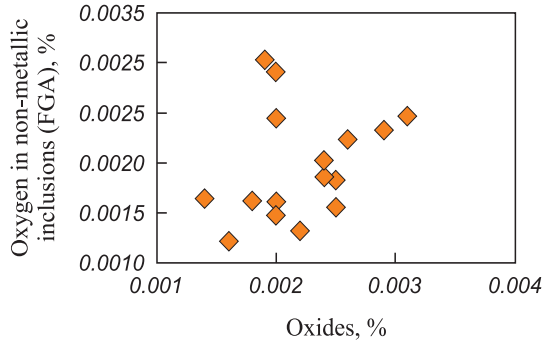

Fig. 15. Correlation between oxygen content in non-metallic inclusions obtained by FGA method and content of oxides obtained by ASTM method in Laboratory No. 17

Рис. 15. Корреляция между общим содержанием кислорода в НВ, полученного методом ФГА и содержанием оксидов, полученного методом ASTM в лаборатории № 17

An analysis of the spectral data array of metal samples on spectrometers was conducted. The files display the emission intensities of spectral wavelengths of various elements for each spark (I_{el}).

Based on the results of metal contamination assessment by NMIs according to ASTM E1245-03 standard, a correlation equation was found linking the area of NMIs with I_{el}.

Using the obtained equation, the volume fractions of NMIs for the studied samples were calculated.

All calculation variants were compared with the results of determining the volume fraction of NMIs using optical microscopy (VD-NMI – total volume fraction of NMIs based on spectral analysis data from two spectrometers at the plant) (Fig. 16).

Fig. 16 shows that the results of determining the volume fraction of NMIs in metal samples according to ASTM E1245-03 standard and the PDA method are consistent. The metal sample analyses show significant differences in NMI content in different parts of the slabs.

Conclusions

Studies of samples using quantitative optical metallography methods according to ASTM E1245-03 standard showed that in all samples, the percentage ratio of oxides to sulfides in the total volume fraction averaged 10 and 90 %, respectively. However, in samples 111 and 121, the oxide content was higher at 31 and 17 %, respectively, and these samples also exhibited uneven NMI distribution. In samples located closer to the small radius (111, 121), uneven NMI distribution was also observed. The upper zone of the samples showed minimal NMI content, with their size not exceeding 10 μm, and most inclusions were located below 1/3 of the sample height. Thus, the upper 1/3 of the samples were cleaner in terms of inclusions than the lower 2/3.

Samples (111, 112, 121, 122, 211, 212, 221, 222) closest to the small casting radius had the highest volume

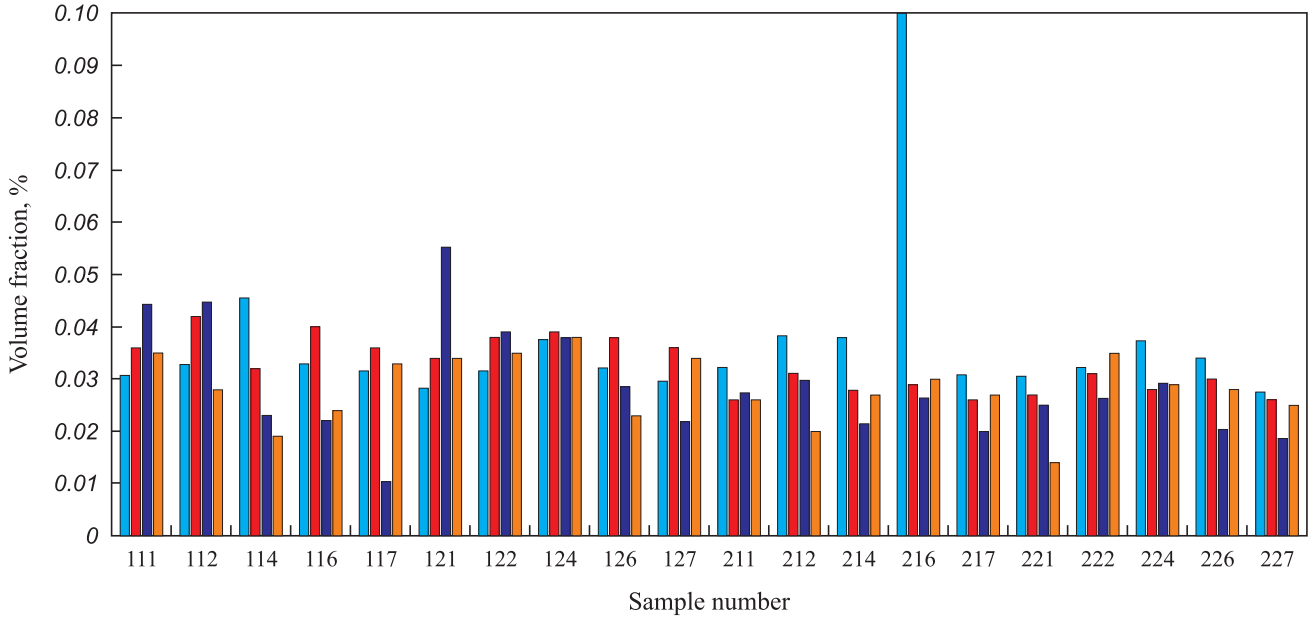

Fig. 16. Comparison of the results of determining the volume fraction of non-metallic inclusions according to ASTM E1245-03 standard (PCL – Laboratory No. 17, R&D) and by PDA method (total volume fraction of non-metallic inclusions, %):
– VF of NMI; – PCL; – Laboratory No. 17; – R&D

Рис. 16. Сравнение результатов определения объемной доли НВ по стандарту ASTM E1245-03 (ЦЛК, лаборатория № 17, R&D) и методом PDA (ОД-НВ, %):
– ОД НВ; – ЦЛК; – лаборатория № 17; – R&D

fraction of NMIs compared to other samples. The total volume fractions of NMIs in the samples and the separate volume fractions of oxide and sulfide inclusions were determined.

Comparison of the results of volume NMI fraction determination by quantitative optical metallography according to ASTM E1245-03 standard by PCL, R&D, and Laboratory No. 17 staff showed good measurement consistency.

To determine the content of major types of oxide NMIs in different parts of the slabs, fractional gas analysis was conducted on the selected metal samples. The analysis concluded that oxide NMIs, such as aluminates, predominantly occur in the metal samples from both ingots.

For ingot *1*, there was an increasing trend in spinel content from the central part of the columns (samples 114, 124) to the large radius of the ingot (samples 116, 117, 126, 127). In the samples from ingot *2*, unlike ingot *1*, spinel-type inclusions were practically absent. The average total oxygen content in ingot *1* was 0.0036 % for samples 11X and 0.0033 % for samples 12X. The average nitrogen content was 0.0041 and 0.0040 %, respectively. In ingot *2*, the average total oxygen content was 0.0021 % for samples 21X and 0.0020 % for samples 22X. The average nitrogen content was 0.0020 and 0.0022 %, respectively. Based on the average results for oxygen, nitrogen, and oxygen in NMIs content data, it can be concluded that ingot *2* is significantly cleaner in terms of oxide NMIs than ingot *1*.

A comparison of the results of oxygen in NMIs determined by FGA and the number of spark hits in inclusions during PDA optical-emission spectral analysis (data from PJSC "NLMK" R&D and PCL) was conducted. The analysis showed two distinct distributions of data points. The first distribution corresponds to ingot *2*, and the second to ingot *1*. The research results also showed a clear correlation between the oxygen content in NMIs and the oxygen content in aluminates obtained by FGA.

An analysis of the spectral data array of selected metal samples, obtained on the workshop spectrometer, was conducted. For the analysis, software was developed to determine the number of inclusions of various types and calculate their volume NMI fractions. The analysis showed good consistency between the results of the PDA optical-emission spectral analysis and the quantitative optical metallography methods according to ASTM E1245-03 standard.

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