Refractory materials of metallurgical furnaces with the addition of silicon production waste

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Currently, there are problems with the operation of metallurgical furnaces at high process temperatures. In the melting zone, the destruction of the side lining and the hearth slab lining in the contact zone of the melt is especially common due to the chemical aggressive environment from the processes of interaction and reactions between the components and mechanical wear as a result of the impact of the tool during maintenance. In most cases, refractory materials do not provide stable operation of the metallurgical unit, they have low operational characteristics. Of scientific and technical interest is the use as a modifying additive for a refractory mixture of technogenic microsilica — waste in the production of metallurgical silicon. Microsilica is a unique finely dispersed composite material with high strength, low density, and with a highly developed particle surface, which further gives refractory products improved strength and durability properties. As part of the study, a series of experiments were conducted with microsilica waste in the production technology of general-purpose fireclay products, as well as in the production technology of refractory concrete mix for monolithic concrete linings and the manufacture of refractory products. It has been established that the optimal value of the content of microsilica in the total mass of general-purpose fireclay products is in the range from 3 to 7%, and in the composition of a dry refractory concrete mixture from 1 to 2%, which does not lead to a decrease in the quality characteristics of the products. Tests of samples have shown that the use of microsilica in the production of refractories can significantly improve properties such as heat resistance and fire resistance, while reducing the cost of production. The obtained results of experiments conducted with the initial microsilica and its behavior in refractory mixtures indicate that the proposed fine composite material can be used in refractory products and introduced into technical standards.

Key words: refractory material, technogenic microsilica, metallurgical furnace, porosity, strength, refractory resistance, refractory mixture.

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Introduction

With the global production volume of metallurgical silicon, with the output of more than 500 million tons per year [1], due to the imperfection of the technological process and the unsatisfactory operation of gas treatment facilities, more than 200 million tons of fine technogenic waste are generated annually. As a rule, these man-made wastes are stored either in closed landfills or in special containers big bags in warehouses [2–3]. Analytical studies show that currently there are no technologies for large-scale processing of technogenic silica, which is SiO_2 microsilicon, with an admixture of carbon of various shapes and sizes, as well as metal compounds with a content of no more than 100 ppm [4–5]. According to experts, the total amount of accumulated waste from the production of silicon in the form of silicon dioxide is more than 1 million tons. Of the resulting waste, no more than 10–15% (of the total volume) is used [6]. Manufacturers are interested in organizing the processing of waste from various industries to obtain value-added products [7–9].

However, in general, microsilica can be attributed to composite materials that have their own specific advantages. Microsilica particles together with carbon have low density, high strength, low coefficient of thermal expansion and a highly developed surface [7, 10–12]. In addition, as a rule, microsilica particles have a stable average size. Taking into account the unique properties of microsilica, currently there are technologies and developments for the disposal and processing of technogenic waste [13–14]. To date, microsilica is mainly used on a large scale only in the production of high-strength concrete, light concrete, shotcrete and concrete with reduced water permeability [15]. In 2000, international standards for the use of microsilica in the construction industry appeared [16], and it is actively used as an additive in most industrialized countries [17–19]. Of scientific and technical interest is the development of technologies for processing microsilica for its subsequent use as a modifying additive in refractory and building mixes [7].

In metallurgical units and furnaces, the quality and characteristics of refractories ultimately determine the melting efficiency and energy efficiency of the entire

process [20]. The lining usually uses high—alumina refractory bricks, or refractory materials consisting of various oxides — SiO_2, Al_2O_3, MgO, CaO, ZrO_2, etc. [21]. The wide variety of refractory materials is explained by the desire to exclude the interaction between the molten metal and the lining material [22]. In addition to mechanical and thermal destruction, metallization occurs, which is accompanied by exchange reactions between oxides and lining [23–24]. For stable and long-term operation of refractories in furnaces, high indicators of chemical, mechanical and thermal resistance of refractory materials are required to improve the operational characteristics of furnaces [25].

Recently, there has been a tendency to create new types of refractory materials with increased wear resistance and high thermophysical characteristics, for example, during their intensive operation in rotating furnaces. Analysis of the literature data showed that during heating a number of exchange reactions take place in the mixture depending on the standard heat of formation, which affects the change in free energy and the degree of dissociation in the melt [26–27]. On the other hand, there are problems in understanding the mechanisms of lining destruction [28–29]. The lack of knowledge in this area leads to a decrease in technical and economic indicators and product quality, which is unacceptable in modern conditions, especially within the framework of resource conservation and energy conservation policies [30].

The refractory industry is developing due to natural reserves of clay and other aluminosilicate mineral raw materials, which are used for the production of refractories with minimal use of additives [25]. In addition, scaling and the possibility of using microsilica formed during the production of technical (metallurgical) silicon creates environmental benefits by reducing the volume of waste stored in slurry fields; and the economic feasibility is to reduce the consumption of raw materials required per unit of manufactured commodity products with added value [31–33].

Materials and Methods

At the initial stage of the work, it was necessary to determine whether technogenic microsilica could be used in the production of refractory fireclay products for general purposes, or as additives in the production technology of refractory concrete mix for the manufacture of monolithic concrete lining furnaces. To do this, it is necessary to study the properties, composition and structure of microsilica, and justify the choice of its necessary content in the composition of refractory mixtures, for subsequent evaluation of the thermophysical and mechanical characteristics of the resulting products.

Technogenic microsilica (waste of silicon production of JSC "Kremniy") was used as the main object of research, as well as carbon-purified (separated) microsilica and microsilica of the MKU-95 brand (GOST R 58894–2020) for comparison.

At the 1st stage of the study, the chemical composition, humidity, size and distribution of particles in micro-volumes of silicon production waste were studied. This stage is necessary to predict the performance characteristics of the finished product, and determines the presence of impurities and inclusions in the samples that may affect their properties.

An *X*-ray multichannel spectrometer (CPM-25, Russia) was used to study the chemical composition of the samples **(Table 1)**. According to the results of energy dispersion analysis (EDA) of microsilica, the content of chemical elements such as S, C, etc. was determined in its composition.

Thus, the separated (purified) silica has fewer impurities compared to the original sample and the commercially used MKU-95, and there are no Fe_2O_3 and MgO impurities in it. The particle size distribution in a given volume was determined using the MicroSizer 201 laser particle analyzer (Russia). The correspondence of particle sizes (D, mkm) to the specified values of the weight fraction (P, %) are presented in **Tables 2, 3**.

The analysis of the granulometric composition shows that the average composition and size of the separated silica differs from the samples of technogenic silica and pure MKU-95 in the decreasing direction.

It was found that technogenic microsilica contains traces of S and C up to 100 ppm. Also, the analysis of calcination losses at pH 5% of the suspension with MKU-95

Table 1
Results of chemical analysis of a microsilica sample

Formula	Component content, %		
	Technogenic microsilica	Separated microsilica	MKU-95
Al_2O_3	0.36	0.17	0.21
Fe_2O_3	1.36	–	0.1
MgO	0.84	–	–
CaO	0.35	0.33	0.86
SiO_2	95.6	98.9	98.5
Na_2O+K_2O	1.19	0.29	0.33
S, C	+ chemical. el. according to EDA analysis		C – 1.87
ignition losses	2.05	2.09	2.11
pH	7.70	7.79	7.81
Humidity, %	1.2	0.8	0.8

Table 2
Correspondence of particle sizes (D, mkm) to the specified values of the weight fraction

Technogenic microsilica	2.50	4.03	6.31	8.96	12.3	15	17.5	20.4	24.6	50
Separated microsilica	1.52	2.59	3.28	3.91	4.98	6.17	7.71	9.69	12.78	50
MKU-95	1.56	2.61	3.34	4.11	5.07	6.24	7.76	9.78	12.9	50
P, %	10	20	30	40	50	60	70	80	90	100

Table 3
Weight fractions of particles (P, %) corresponding to the specified values of particle sizes

Technogenic microsilica	0	0.8	2.4	6.5	13.6	25.5	43.1	79.1	99.9	100
Separated microsilica	0	3.7	5.3	7.0	28.3	51.2	84.3	98.5	100	100
MKU-95	0	3.5	4.9	6.6	25.2	49.4	80.9	98	100	100
D, mkm	0.2	0.5	0.7	1.0	3.0	5.0	10.0	20	45	50

showed that the impurity content has identical values, as well as the moisture content of MKU-95 and silica, which have identical values.

At the second stage, *X*-ray phase and energy dispersion analysis of the studied samples of materials were carried out.

An *X*-ray diffractometer Dron-8 (Russia) was used to perform *X*-ray phase analysis. The calculation of the content of crystalline phases was carried out by the Rietveld method (full-profile analysis, and normalization by 100%), while taking into account the composition of the amorphous phase **(Table 4)** [34].

Table 4
The content of crystalline phases in microsilica samples

Formula	Mineral name	Syngony	The amount of phase in the sample, %
			Microsilica
AlF_3–$3H_2O$	Rosenbergite	Tetragonal	–
SiC	Moissanite – 3C	Cubic	15.9
SiC	Moissanite – 6H	Hexagonal	2.97
SiO_2	Quartz	Hexagonal	6.68
SiO_2	Cristobalite	Tetragonal	3.33
Si	Silicon	Cubic	0.33
Amorphous phase			71

It has been established that morphological features, chemical composition of microsilicon, its quantity determine the selectivity of the action of impurities on colloidal chemical and structural-mechanical processes, the nature and kinetics of hydration of alumina and high alumina refractory mixtures, and the type and composition of neoplasms are factors that change the nature of the flow of physicochemical processes under temperature exposure [35]. The results of the study suggest that the amorphous highly active state of microsilica may lead to early accumulation of the liquid phase in a temporary aggregate state during heat treatment of the sample, and further sintering of the material in combination with compaction of the structure of materials. It is necessary to take into account the number of polymorphic water-soluble phases of SiO_2.

When analyzing the microstructure in **Fig. 1, *a*, *c***, an insignificant amorphous "halo" characteristic of noncrystalline carbon is observed in a sample of technogenic silica in the temperature range of 280–290 °C, which can act as a binder when heated in general combination with microsilica particles.

Table 5 shows the results of the energy dispersion analysis (EDA) of microsilica, which was carried out on a JEQL – 6380 LV electron microscope with an integrated INCA E-350 spectrometer, to explain possible phase transitions during the processing of mixtures.

Microstructure analysis indicates the appearance of agglomerates and areas of polymorphic structures accretion due to the highly developed microsilica surface and phase transitions during the formation of silicic acid in reaction with the water-soluble SiO_2 phase. **Fig. 2** shows that carbon particles are distributed throughout the sample

Table 5
Results of microanalysis of EDA for microsilica

Spectrum	C, %	Na, %	Mg, %	Al, %	Si, %	S, %	K, %	Ca, %	O, %	Total, %
Microsilica	20.85	0.02	0.06	0.06	10.61	0.08	0.12	0.23	67.98	100

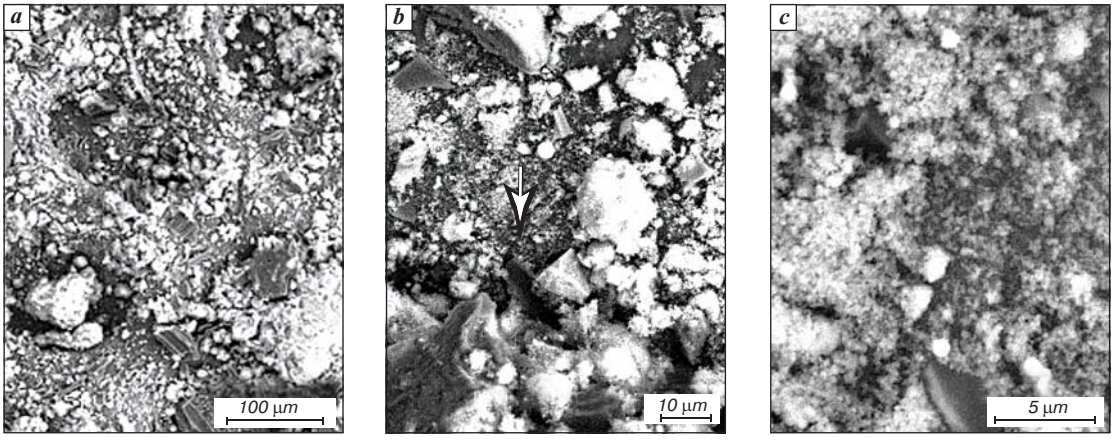

Fig. 1. Structure of technogenic microsilica (*a* – ×200; *b* - ×1000; *c* – ×5000)

volume, this can create conditions for uniform distribution of agglomerates (in the form of carbides), as well as create conditions for adsorption of particles on the surface of other components of refractory mixtures. Uneven distribution of potassium and magnesium (in the form of spindles) in micro-volumes can lead to the formation of cavities and cracks during further processing of mixtures, as shown in **Fig. 1, *a–c***.

At the third stage, the results of changes in the properties of technogenic material selected directly from the production of silicon were studied using thermogravimetric and differential thermal analyses **(Fig. 3)**.

After conducting experiments on the heat treatment of samples, it was found that successive physico-chemical processes occur in different temperature intervals.

When analyzing the results of heating and heat treatment of samples of technogenic microsilica (waste), it was revealed that:

Up to a temperature of 252 °C, adsorption moisture and internal moisture are removed with a decrease in sample weight by 1.2–1.4% with several successive endothermic reactions in the interaction of calcium and carbon, with a maximum at 50.5 °C. The inertness of the behavior of sulfur compounds was found, which indicates the sulfate resistance of the samples.

Two consecutive peaks of evaporation of organic substances with maxima at 500.36 and 577.25 °C correspond to a 19.1% decrease in the mass of the sample, and are in the temperature range of 254–794 °C. These data correspond to phase transitions in the interaction of carbon and calcium, and are characteristic, among other things, of standard technologies for heating carbon or coke.

The analysis also showed that microsilica particles have a low coefficient of thermal expansion, which inevitably affects the thermal conductivity and fire resistance of the samples.

The studied properties and behavior of microsilica samples during heat treatment suggest that this composite additive can positively affect the properties of refractory materials, improving their qualitative characteristics, such as strength, permeability, chemical resistance, sulfate resistance, wear resistance, etc., which allows them to be exposed to a chemically aggressive high-temperature medium — melt for a long time. It can be concluded that microsilica has the properties of highly reactive pozzolan, which causes the effect of hardening of the hardening system, binding lime from the mixture more intensively than other mineral additives.

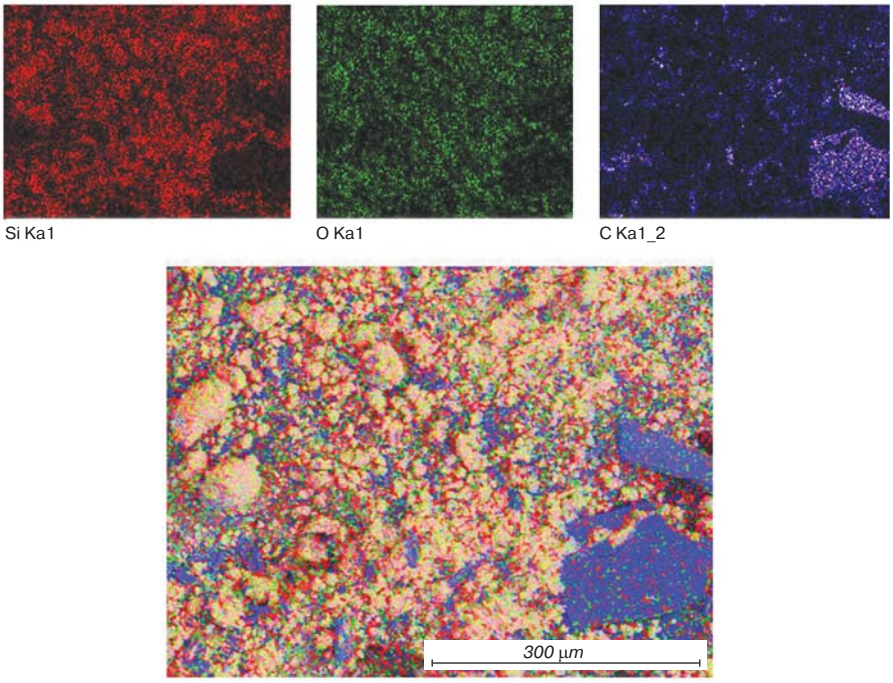

Fig. 2. Distribution map of elements of technogenic microsilica

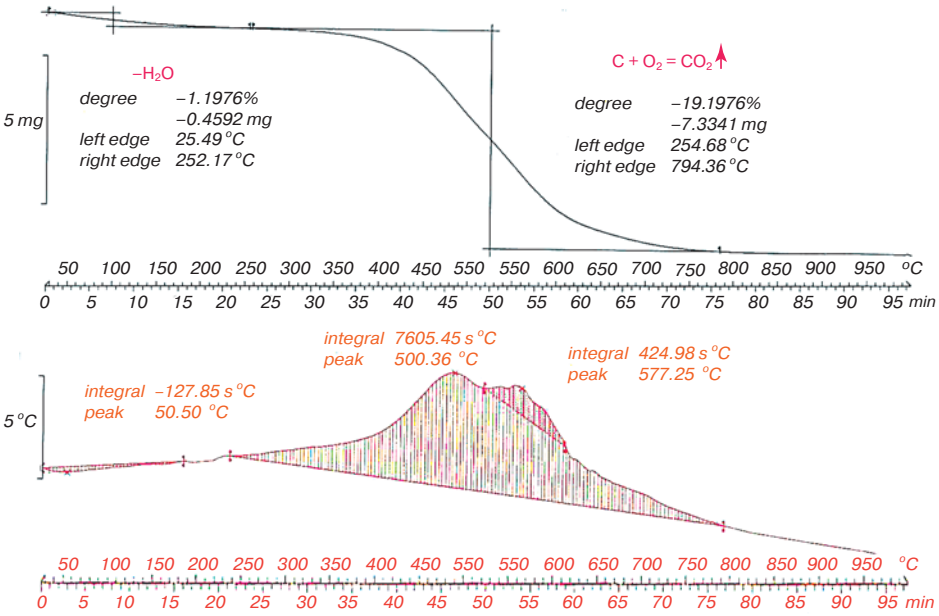

Fig. 3. Thermogravimetric heating curve of technogenic microsilica

Results and Discussion

At the next stage, the issue of the use of microsilica in the production technology of general-purpose fireclay products, as well as in the production technology of refractory concrete mix for monolithic concrete linings, as well as other refractory products for metallurgical furnaces, was studied.

To substantiate the use of technogenic microsilica (Tec. Mic.) in the production technology of general-purpose fireclay products, the following materials were prepared:

– ordinary brand chamotte;

– a mixture of clay and kaolin in a ratio of 1:1.

The preparation of the molding mass was carried out in "laboratory runners", and the weight of the sample was 5 kg (2 batches each). The material compositions of the mixtures, in wt.%, are given in **Table 6.**

Composition No. 1 was a control (C.C.) for comparing the indicators of the remaining mixtures. Pressing of samples with a rib size of 50 mm was carried out on a laboratory press with a force of 5 MPa. After pressing, the apparent density, linear dimensions and mass of the raw (unburned) refractory sample were determined.

Additionally, samples were tested for compliance with the requirements of the international standard ISO-2000 for acid-resistant bricks of the KP class A grade in terms of water absorption and acid resistance.

As a result of the experiments, the qualitative indicators of the samples were determined in accordance with the requirements of ISO-2000 for general-purpose fireclay products (**Table 7**). The dependence of the tensile strength on the content of technogenic microsilica in the composition of the product is shown in **Fig. 4.**

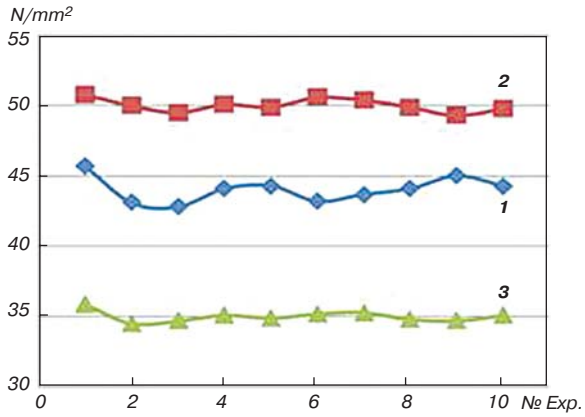

Fig. 4. Dependence of the tensile strength on the content of microsilica in the composition of the product:
1 — C.C; *2* — 5% Tec. Mic.; *3* — 10% Tec. Mic.

The results of experiments conducted with the starting materials and the study of their behavior in mixtures and in general-purpose fireclay products indicate that technogenic microsilica can be used in fireclay products. However, for further studies of the use of this material in fireclay products, it is necessary to consider the effect of the presence of carbon on the quality indicators of products. For this purpose, the use of separated (purified) microsilica (Sep. Mic.) in the production technology of fireclay products was studied. The experiments followed the same pattern as with technogenic microsilica. The results of the experiments are presented in **Table 8.** The dependence of the tensile strength on the content of separated microsilica in the composition of the product is shown in **Fig. 5.**

Table 6.
Material composition of mixtures, wt. %

	Composition No. 1	Composition No. 2	Composition No. 3
Chamotte	60%	55%	50%
Mixture of clay and kaolin	40%	40%	40%
Technogenic microsilica	–	5%	10%

Table 7
Characteristics of the obtained mixture samples

Material composition, %			Apparent density of raw, g/cm^3	Apparent density, g/cm^3	Open porosity,%	Water absorption, %	Compressive strength N/mm^2	Tem. of the beginning of softening, °C	Refracto-riness, °C	Mass fraction Al_2O_3
Chamotte	C + K	Tec. Mic.								
60	40	–	2.23-2.4	2.13	18.9	8.9	45.7	1370	1690	34.2
				2.13	18.9	8.9	43.1			
				2.13	18.8	8.8	42.8			
55	40	5	2.18-2.21	2.08	19.6	9.4	50.8	1370	1660	32.9
				2.08	19.3	9.3	50			
				2.08	19.5	9.4	49.5			
50	40	10	2.11-2.14	1.99	22.7	11.4	35.9	1350	1650	31.4
				2	22.4	11.2	34.5			
				1.98	22.9	11.6	34.7			
ISO-2000 SHA grades (I subgroup)				–	≤24	–	≥20	≥1300	≥1690	≥30
ISO-2000 — acid-resistant brick of the KP kl. A brand				–	–	≤6	≥55	–	–	–

Table 8
Quality indicators of products with the addition of separated microsilica

Material composition, %			Apparent density of raw, g/cm^3	Apparent density, g/cm^3	Open porosity, %	Water absorption %	Compressive strength N/mm^2	Tem. of the beginning of softening, °C	Refracto-riness, °C	Mass raction Al_2O_3
Cha-motte	C + K	Sep. Mic.								
60	40	–	2.23–2.4	2.13	18.9	8.9	45.7	1370	1690	34.2
				2.13	18.9	8.9	43.1			
				2.13	18.8	8.8	42.8			
57	40	3	2.21–2.22	2.12	18.2	8.8	50.8	1380	1690	34.0
				2.12	18.1	8.9	50.2			
				2.12	18.2	8.9	49.9			
55	40	5	2.20–2.21	2.10	18.3	9.2	52.8	1390	1700	33.7
				2.10	18.2	9.1	51.7			
				2.09	18.3	9.2	52.4			
53	40	7	2.18–2.20	2.08	18.6	9.4	56.3	1390	1710	32.9
				2.08	18.5	9.3	55.8			
				2.08	18.6	9.4	57.1			
50	40	10	2.15–2.17	2.02	22.1	11.1	49.3	1320	1660	30.4
				2.01	21.5	10.2	48.6			
				2.01	22.0	11.0	48.9			
ISO-2000 SHA grades (I subgroup)				–	≤24	–	≥20	≥1300	≥1690	≥30

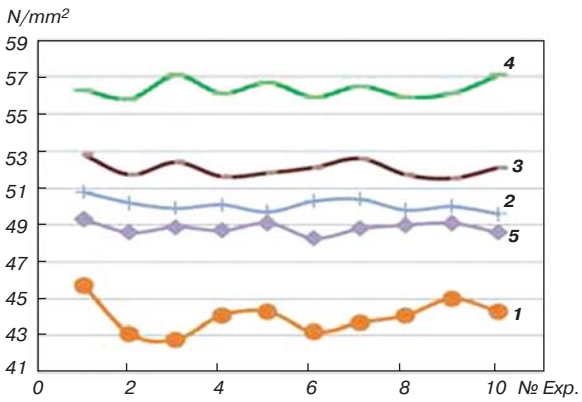

Fig. 5. Dependence of the tensile strength on the content of microsilica in the composition of the product:
1 — C.C; *2* — 3% Sep. Mic.; *3* — 5% Sep. Mic.; *4* — 7% Sep. Mic.; *5* — 10% Sep. Mic.

An increase in strength can be observed when replacing chamotte with microsilica from 3 to 10%; however, with a microsilica content of more than 7%, a significant decrease in the strength of the samples is observed. This is explained by the fact that at this stage there is an intermolecular interaction of Van der Waals forces with the formation of dipoles due to bridging bonds between microsilica molecules, carbon residues and particles of the main refractory mass. With an increase in the silica content of more than 7%, these bonds are layered, leading to softening of the sample. This is confirmed by the microstructural study data [36–37].

At the next stage, a series of experiments was conducted on the use of microsilica in the technology of production of refractory concrete mix for the implementation of monolithic concrete linings, and the manufacture of refractory products. For this purpose, separated microsilica was used in all the technologies presented below, since it has a cleaner chemical composition in comparison with technogenic microsilica.

To prove the possibility of using microsilica in the production technology of refractory concrete mix for monolithic concrete linings of metallurgical furnaces, the following 3 compositions were prepared:

Composition No. 1 was a control for comparing the indicators of other mixtures

Composition No. 2 with the addition of 1% microsilica

Composition No. 3 with the addition of 2% microsilica

To identify the nature and degree of interaction of the developed compositions of samples in the melt, additional studies of the corrosion resistance of samples from the mixture were carried out in the laboratory. The tests were carried out by the crucible method. The samples were formed in cubic shapes of 70×70×70 mm. It should be noted that during the pouring of concrete samples, no changes in rheological properties were observed. The test results are presented in **Tables 9, 10**.

The analysis showed that under the given conditions and temperatures of heat treatment, the necessary indicators of apparent density, porosity and fire resistance of the obtained products corresponding to ISO-2000 of the SHA brand (I subgroup) are provided.

The results of experiments conducted with the use of separated microsilica and its behavior in general-purpose fireclay products indicate that carbon-free microsilica can improve the quality of the product.

Table 9
Test results of the corrosion resistance of samples

Resistance to aggressive environments, %	Refractory concrete mixture		
	Composition No. 1	Composition No. 2	Composition No. 3
Area of corrosion (Steel), %	1.3 ; 2.1	1.1 ; 1.6	1.0 ; 1.5
Area of intensive interaction with corrosion (Steel), %	14.3-17.1	14.9 ; 14.1	14.3 ; 13.9
Area of corrosion (Highlybasic slag), %	14.9 ; 14.1	2.9 ; 3.4	2.7 ; 3.1
Area of intensive interaction with corrosion (Highlybasic slag), %	35.9 ; 20.8	22.2 ; 15.8	21.4 ; 16.8

Table 10
Test results of refractory mixture samples

The name of the indicator	Refractory concrete mixture		
	Composition No. 1	Composition No. 2	Composition No. 3
Chemical composition, %			
Al_2O_3	93.6	93.0	92.8
Fe_2O_3	0.20	0.25	0.25
MgO	2.77	3.17	3.17
CaO	0.89	1.33	1.33
SiO_2	1.29	1.32	1.39
K_2O+Na_2O	0.49	0.50	0.50
Compressive strength after firing at a temperature of 1300 °C, N/mm^2 (not less than 45)	109.9; 113.1; 110.6; 111.0	112.3; 113.7; 111.6; 110.5	117.2; 118.5; 118.7; 119.1
Apparent density after firing at 1300 °C, g/cm^3 (not less than 2.7)	3.21; 3.20; 3.20; 3.21	3.18; 3.18; 3.19; 3.17	3.19; 3.18; 3.19; 3.19
TBS (the temperature of the beginning of softening), °C	1690	1690	1690

Conclusions

The behavior of microsilica in general-purpose fireclay products indicates that this additive makes it possible to improve the quality of the product, such as compressive strength and fire resistance. It is established that the most optimal value of the use of microsilica in the composition of the mass is from 3 to 7%.

Microsilica, as a composite, has the properties of highly reactive pozzolan, which causes the effect of hardening of the hardening system, binding lime from the mixture more intensively than other mineral additives.

It is established that under the given conditions and temperatures of heat treatment, the necessary indicators of apparent density, porosity and fire resistance of the obtained products corresponding to ISO-2000 of the SHA brand are provided.

It is proved that microsilica can be used in the production technology of refractory concrete mix for the execution of monolithic concrete linings and the manufacture of refractory products. The optimal value of the use of silica in the composition of the mass is its content from 1 to 2%. Tests of samples have shown that the use of microsilica can improve such indicators of the mixture as compressive strength and resistance to aggressive media.

This study was carried out based on the results of the scientific work carried out in the preparation of the final article on the candidate's thesis.

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