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ФИЗИКО-ХИМИЧЕСКИЕ ИССЛЕДОВАНИЯ ШЛАКА ПРОИЗВОДСТВА НИЗКОУГЛЕРОДИСТОГО ФЕРРОХРОМА – КОМПОНЕНТА ЖАРОСТОЙКОГО ВЯЖУЩЕГО МАТЕРИАЛА

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Целью работы явились физико-химические исследования химико-минералогического состава шлака производства низкоуглеродистого феррохрома, предлагаемого к использованию в качестве компонента жаростойкого вяжущего материала. В результате исследований установлено, что основной кристаллической фазой в шлаке является ортосиликат кальция в виде модификации γ-Ca_2SiO_4, который также частично присутствует в виде α-Ca_2SiO_4, и магнезиальные силикаты в виде форстерита Mg_2SiO_4. Основные свойства, такие как плотность, удельная поверхность, дисперсность, тугоплавкость шлака, определяются свойствами доминирующего минерала ортосиликата кальция. В условиях медленного остывания шлакового расплава основной минерал в составе шлака, ортосиликат кальция β-Ca_2SiO_4, переходит в модификацию γ-Ca_2SiO_4 с увеличением объема кристаллической решетки на ~12%, что приводит к саморазрушению и переходу шлака в пылевидное состояние. Результаты исследований удельной поверхности, определения среднего размера частиц, результаты ситового анализа показали, что исследуемый шлак представляет собой тонкодисперсный порошок серого цвета со следующими характеристиками: удельная поверхность 2955 см^2/г, средний размер частиц 6,8 мкм, истинная плотность - 3,01 г/см^3, насыпная плотность - 739 кг/м^3. При применении тонкодисперсного шлака производства низкоуглеродистого феррохрома в качестве компонента композиционных вяжущих может быть исключен энергозатратный процесс его тонкого помола. В настоящее время шлак производства низкоуглеродистого феррохрома практически не используется в качестве вторичного минерального сырья, однако физико-химические свойства составляющих шлак минералов позволяют рекомендовать его в качестве компонента для изготовления жаростойких материалов. Результаты физико-химических исследований могут быть использованы для разработки эффективных технологий комплексной переработки шлаков низкоуглеродистого феррохрома.

Ключевые слова: шлак производства низкоуглеродистого феррохрома, морфология поверхности, элементный состав, удельная поверхность, ортосиликат кальция, ортосиликат магния, полиморфные модификации

PHYSICAL AND CHEMICAL STUDIES OF SLAG OF PRODUCTION OF LOW-CARBON FERROCHROME - COMPONENT OF HEAT-RESISTANT BINDER MATERIAL

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The aim of the work is physicochemical studies of chemical and mineralogical composition of the slag from the production of low-carbon ferrochrome proposed to use as a component of heat-resistant binder. As a result of studies, it was found that the main crystal phase in the slag is calcium orthosilicate in the form of γ-Ca_2SiO_4 modification, which is also partially in the form of α-Ca_2SiO_4, and magnesian silicates in the form of forsterite Mg_2SiO_4. The main properties of the slag such as density, specific surface, dispersiveness, refractoriness are determined by the properties of the dominant mineral- calcium orthosilicate. Under conditions of slow cooling of the slag melt, the main mineral in the slag composition, i.e. calcium orthosilicate β-Ca_2SiO_4 passes into γ-Ca_2SiO_4 modification with increase in the volume of crystal lattice by ~ 12%, which leads to self-destruction and transition of the slag to a dust state. The results of studies of the specific surface, determination of the average particle size, the results of sieve analysis showed that the slag from the production of low-carbon ferrochrome is a finely dispersed gray powder with the following characteristics: the specific surface is 2955 cm^2/g, the average particle size is 6.8 μm, the true density is 3.01 g/cm^3, the bulk density is 739 kg/m^3. When using the finely dispersed slag from the production of low-carbon ferrochrome as a component of composite binders, the energy-consuming process of its fine grinding can be eliminated. Currently, the slag from the production of low-carbon ferrochrome is practically not used as a secondary mineral raw material. However, the physicochemical properties of the minerals making up the slag allow to recommend it as a component for the manufacture of heat-resistant materials. The results of physical and chemical studies can be used to develop effective technologies for integrated processing of low-carbon ferrochrome slags.

Key words: low-carbon ferrochrome production slag, surface morphology, elemental composition, specific surface, calcium orthosilicate, magnesium orthosilicate, polymorphous modifications

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INTRODUCTION

At present, the huge volumes of industrial wastes accumulated in the dumps of enterprises create serious problems both in the environmental aspect polluting the surrounding environment and in the economic aspect, since considerable financial resources are spent to the conservation and maintenance of the dumps.

Large-tonnage mineral wastes of metallurgical industry under certain conditions of their preparation can become full-fledged raw materials for production of various types of composite binders [1, 2].

At the same time, when applying various activation methods, the efficiency of using technogenic raw materials in the technology of composite binders increases significantly [3-5].

In the Russian Federation, in the dumps of Chelyabinsk Electrometallurgical Plant alone, more than 8.2 million t of low-carbon ferrochrome slags have been accumulated [6].

In Kazakhstan, at Aktobe Ferroalloy Plant of Kazchrome Transnational Company JSC, when producing the low-carbon ferrochrome, more than 200000 t of slag are generated annually, which are sent to the company' disposal area. Kazchrome Transnational Company JSC is the world leader in the quality and volume of chrome raw materials production, ranked second in the global ferroalloy market.

Over 15 million t of industrial wastes are accumulated in the company's slag disposal area, of which about 8-9 million t are slag of low-carbon ferrochrome production. The costs for the slag wastes disposal and the slag disposal area maintenance are the major economic and environmental problem facing the company.

In world practice in the production of ferroalloys, importance is attached to the integrated use of large-tonnage slag wastes according to environmentally friendly technologies.

The European Union provides for a comprehensive audit of any technology for registration, evaluation, authorization and restriction of chemicals in accordance with the European Union Legislation on Registration, Evaluation, Authorization and Restriction of Chemicals – REACH [7].

In the processing of slags from the production of ferrochrome, the problem of ecological safety is no less important, since chromium is recognized as a dangerous carcinogen [8, 9].

According to the chemical and mineralogical characteristics, slags from the production of low-carbon ferrochrome [10-12], characterized by a tendency to self-decomposition and transformation into fine powder [13], are among the wastes of ferroalloy production.

A distinctive feature of the slags from the production of ferrochrome is their refractory and heat-resistant properties [14, 15].

The published works analysis showed a small amount of research and proposed directions for using the slag of low-carbon ferrochrome production in agriculture for soil liming, in foundry for making self-hardening mixture, in road construction as a mineral filler for preparing asphalt concrete, as a raw material for cement production [16, 17].

The analysis of the published works showed that there is a small number of works on the use of slags from the production of low-carbon ferrochrome in the technology of heat-resistant binders and concretes.

According to the authors of [18], ferroalloy slags can be a promising raw material for heat-resistant binders and concretes with a temperature of application from 800 to 1800 °C. It is shown that the replacement of sodium silicofluoride with ferrochrome slag as a hardener in heat-resistant concretes on liquid glass made it possible to increase the temperature of their use.

The possibility of using metallurgical ferroalloy slags as components for the manufacture of heat-resistant concretes was also shown in [19].

The rationale for the use of self-decomposing ferrochrome slag of Chelyabinsk Electrometallurgical Plant for the manufacture of heat-resistant concretes is given in [20].

As a result of studies in [21], based on binders using the waste of a ferroalloy plant, slag-alkali heat-resistant concretes with a temperature of application of 1100 °C were obtained.

The authors' work [22] presents the results of developing a technology for processing stabilized slag of low-carbon ferrochrome, and its composition and structure were investigated.

It should also be noted that the authors of [23-25] examined the current state of the theory and practice of heat-resistant binders and concretes, generalized theoretical provisions on the possibility of obtaining heat-resistant compositions using technogenic raw materials and various binders.

Currently, the slag of low-carbon ferrochrome production is not used as a secondary material resource for building materials production, which is associated with insufficient research on the slag properties, as a mineral raw material, and insufficient research on the formation of structure and properties of building materials and products based on it.

To create of efficient and safe technologies for integrated processing of low-carbon ferrochrome slags requires physicochemical and technological research combined with a technical and economic assessment of the effectiveness of the technologies developed.

EXPERIMENTAL PART

The physical and chemical studies of the slag sample microstructure of low-carbon ferrochrome production were carried out using JSM-6490LV scanning electron microscope of JEOL company (Japan), which also determined the elemental chemical composition by the energy dispersion method.

The X-ray phase analysis of the slag was carried out using DRON-3 diffractometer with CuK_α radiation with a nickel filter. The goniometer counter speed was 8 gon/min. The tube voltage is 40 kV, the tube current is 20 mA.

The slag's specific surface and the average particle size were determined by the dispersed material air permeability method using PSKh-K (ПСХ-К) computer multifunctional device produced by Ruspribor LLC (Russia).

The sieve analysis of the slag was carried out using Analysette 3 vibration sieve device of FRITSCH company (Germany).

The results of the study of the slag sample surface morphology and the energy dispersion analysis are shown in Fig. 1. The surface of the studied slag sample is polydisperse in nature, while there are both dusty particles and larger dense particles having a detrital and fragment form.

The studies of the elemental chemical composition (Table 1) showed that the slag composition has a significant content of such elements as Ca, Si, Mg, Cr, Al, which characterizes the mineralogical slag composition.

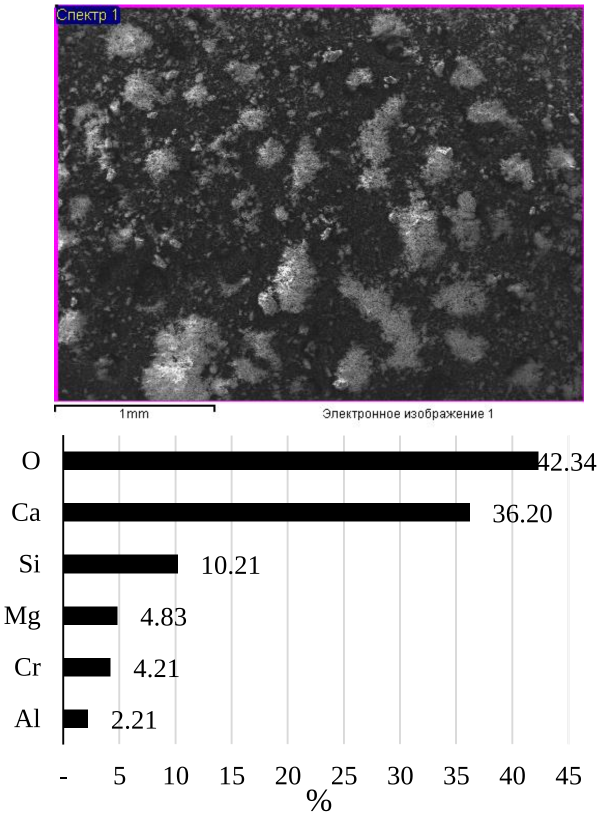

Fig. 1. The surface morphology and the elemental composition when scanning the slag sample of low-carbon ferrochrome production

Рис. 1. Морфология поверхности и элементный состав при сканировании образца шлака производства низкоуглеродистого феррохрома

Table 1

The elemental chemical composition of the slag sample (mass %)

***Таблица 1.* Элементный химический состав образца шлака (мас. %)**

O	Mg	Al	Si	Ca	Cr
42.34	4.83	2.21	10.21	36.20	4.21

The chemical slag composition (mass percent): SiO_2 – 24.12%; Al_2O_3 – 4.69%; CaO – 55.40%; MgO – 8.92%; Cr_2O_3 – 6.86%.

The X-ray investigation of the slag showed that the main crystalline phase in it is γ-Ca_2SiO_4, there are also α-Ca_2SiO_4, and magnesian silicates, possibly, in the form of magnesium orthosilicate – forsterite Mg_2SiO_4.

On the slag diffractogram (Fig. 2), the most intense diffraction reflections with d = 3.81; 2.79; 2.74; 1.88; 1.62Å correspond to the phase γ-Ca_2SiO_4.

The diffraction maxima with d = 2.71; 2.66, 1.72Å, are apparently due to the presence of α-Ca_2SiO_4 in the slag. The presence of this phase in the slag indicates that $\alpha \rightarrow \alpha'_H \rightarrow \alpha'_L \rightarrow \beta \rightarrow \gamma$ modification transformations did not occur completely, and a small amount of α-Ca_2SiO_4 is present in the system.

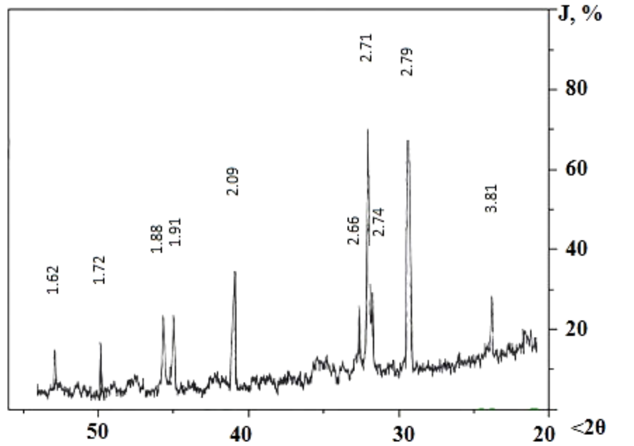

Fig. 2. The diffractogram of the slag of low-carbon ferrochrome production

Рис. 2. Дифрактограмма шлака производства низкоуглеродистого феррохрома

The reflections with d = 3.81; 2.74; 2.66 indicate the presence of forsterite Mg_2SiO_4.

The results of studies of specific surface, determining the average particle size, the results of sieve analysis showed that the slag production of low-carbon ferrochrome is a fine gray powder with the following characteristics: the specific surface is 2955 cm^2/g, the average particle size is 6.8 μm, the true density is 3.01 g/cm^3, the bulk density is 739 kg/m^3. By the chemical composition, the slag can be referred to the main with the lime factor M_O = 2.16, determined by the percentage (%CaO + %MgO)/(%SiO_2+%Al_2O_3), with low activity factor Ma = 0.18, determined by the percentage of Al_2O_3 and SiO_2 content in the slag.

RESULTS AND DISCUSSION

The dominant mineral of slag from the production of low-carbon ferrochrome is calcium orthosilicate Ca_2SiO_4 (2CaO·SiO_2); therefore, its physical and chemical properties are determined by the basic properties of slag. Complex polymorphism and associated volume changes in the structure leading to self-destruction of the mineral are inherent in calcium orthosilicate Ca_2SiO_4.

When producing the low-carbon ferrochrome under conditions of slow slag melt cooling down, the main mineral in the slag composition, the calcium orthosilicate Ca_2SiO_4 passes into γ-Ca_2SiO_4 modification with increase in the crystal lattice volume by ~12%, which leads to the slag self-destruction and transition to a dusty state. Due to the polymorphous transformations and high dispersion, the slag of low-carbon ferrochrome production was also called “self-destructing”.

Magnesium orthosilicate forsterite Mg_2SiO_4 is known only in the form of one modification; therefore, forsterite present in the slag does not undergo volumetric structural changes upon slow cooling of the slag. Petrographic studies showed that in the polydisperse slag medium, forsterite Mg_2SiO_4 is present in the form of larger dense particles of a detrital and fragment form.

Unlike the so-called “belite” mineral, i.e. β-modification of calcium orthosilicate β-Ca_2SiO_4, which is the mineralogical component of Portland cement clinker, formed in the slag of low-carbon ferrochrome production, γ-Ca_2SiO_4 modification has practically no hydraulic activity and binding properties.

The scheme of modification transformations in the system CaO – SiO_2, according to which calcium orthosilicate Ca_2SiO_4 exists in four polymorphous modifications α, α', β, γ, is presented in [23].

According to other authors, for calcium orthosilicate Ca_2SiO_4, there are five polymorphous varieties: α, α', β, γ, taking into account the fact that for α'-modification there are high-temperature α'_H and low-temperature α'_L, modifications [24]. Fig. 3 shows the scheme of polymorphous transformations of calcium orthosilicate Ca_2SiO_4 modifications, described in detail and summarized in [25].

In [26], the results of Ca_2SiO_4 phase polymorphous transitions parameters (Table 2) and the characteristics of calcium orthosilicate Ca_2SiO_4 individual polymorphous modifications (Table 3) are presented.

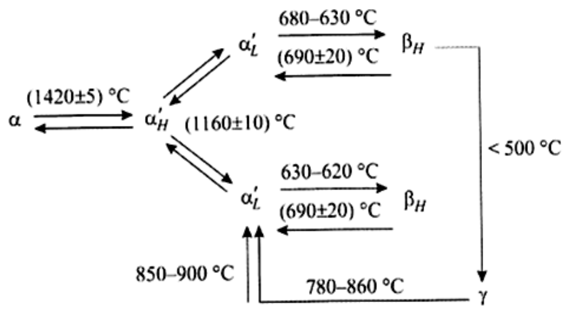

Fig. 3. The scheme of polymorphous transformations of dicalcium silicate Ca_2SiO_4

Рис. 3. Схема полиморфных превращений модификаций двухкальциевого силиката Ca_2SiO_4

Table 2

Ca_2SiO_4 phase polymorphous transitions parameters

***Таблица 2*. Параметры фазовых полиморфных переходов Ca_2SiO_4**

	Phase polymorphous transitions of Ca_2SiO_4 modifications					
	$\alpha'_H \leftrightarrow \alpha$	$\alpha'_L \leftrightarrow \alpha'_H$	$\alpha'_M \leftrightarrow \alpha'_L$	$\alpha'_M \leftrightarrow \beta$	$\beta \rightarrow \gamma$	$\gamma \rightarrow \alpha'_M$
Temperature, °C	1440	1160	979	650-690	<510	711
$\Delta V_{modifications}$, %	-8.3	+0.3	+1.6	+1.2	+11.5	+5.1

Table 3

The characteristics of Ca_2SiO_4 polymorphous modifications

***Таблица 3*. Характеристики полиморфных модификаций Ca_2SiO_4**

Parameters	Ca_2SiO_4 polymorphous modifications					
	γ	α'_M	α'_L	α'_H	α	β
Symmetry	Rhombic	Monoclinic	Rhombic	Rhombic	Hexagonal	Monoclinic
Density, kg/m^3	2970	3240	3230	3300	3070	3310

Such minerals of the slag of low-carbon ferrochrome production as calcium orthosilicate Ca_2SiO_4, and forsterite Mg_2SiO_4 have high refractoriness. Thus, larnite mineral Ca_2SiO_4 is the most refractory slag mineral, it melts congruently at 2130 °C, and refractoriness of forsterite mineral Mg_2SiO_4 is reached at 1900 °C [27, 28]. Due to these properties, the slag of low-carbon ferrochrome production can be used as a component of mixture in the heat-resistant materials production.

CONCLUSIONS

As the result of conducted studies, the chemical and mineralogical composition and main physical and technical properties of the slag from the production of low-carbon ferrochromium were determined. As the result of physical and chemical studies, it was established that the main mineral of the slag of low-carbon ferrochrome production is calcium orthosilicate Ca_2SiO_4, magnesium orthosilicate in the form of forsterite Mg_2SiO_4 is also present in the slag.

The slag from the production of low-carbon ferrochromium is characterized by high dispersiveness, conditioned by changes in modification changes of the dominant material – slag of calcium orthosilicate Ca_2SiO_4. As a result of thermal conversion under the conditions of slow slag melt cooling down, the calcium orthosilicate Ca_2SiO_4 passes into γ-Ca_2SiO_4 modification with increase in the crystal lattice volume by ~12%, which leads to the slag self-destruction and transition to the finely dispersed state.

The slag under study represents finely dispersed powder of grey colour with the following characteristics: specific surface 2955 cm^2/g, regular particle 6,8 μm, real density – 3000 kg/m^3, packed density – 739 kg/m^3.

When using the finely dispersed slag of low-carbon ferrochrome production as a component of composite binders, the expensive process of its fine grinding in mills can be eliminated.

The results of present physical and chemical studies will contribute to solving the issues of rational and efficient use of the slag of low-carbon ferrochrome production in technology of building materials, including as a component of heat-resistant binders.

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