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Применение порошковых материалов и функциональных покрытий

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Prospects for the use of graphite-containing sludge for the production of composite coatings

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Abstract. Modern technologies must meet the criteria of sustainable development, taking into account economic, environmental, and social indicators. In this study, the potential use of graphite-containing sludge from the gas purification aspiration system during cryptocrystalline graphite production was investigated for its inclusion in composite anti-burn coatings for cast iron casting. The graphite-containing sludge consists of carbon, sulfur, sodium, aluminum, and silicon, with a phase composition that includes graphite, calcite, pyrite, quartz, halite, and others. The sludge is a dispersed material with an average particle size of 3.64 μm, a total surface area of 36,506 cm^2/cm^3, and a main fraction size of 1–8 μm. Sludge particles exhibit various shapes, ranging from irregular to isometric. Larger isometric particles can reach sizes of 1 mm or more. On the surfaces of larger particles, smaller dispersed particles are present. The structural parameters of the sludge correspond to those of hexagonal graphite. The analysis of the composition and properties of graphite-containing sludge suggests its suitability for use in composite anti-burn coating formulations. However, due to the presence of large graphite aggregates and acicular impurities in the sludge, sieving is required before use. Complete replacement of natural graphite with sludge increases the coating density from 1220 to 1750 kg/m^3, viscosity from 34 to 105 s, and abrasion resistance from 175 to 245 g/mm. Due to its high dispersity, the sludge-based coating nearly completely penetrates the pores of the sand-resin mixture mold without forming a cover layer. This does not ensure consistent reduction of burn-on defects on casting surfaces. Therefore, the full substitution of graphite with graphite-containing sludge in composite coating formulations is not recommended.

Keywords: self-drying coating, graphite-containing sludge, cast iron, burn-on, density, viscosity, thickness of the coating layer, depth of the penetrating layer, abrasion resistance

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Перспективы применения графитсодержащего шлама для изготовления композиционных покрытий

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Аннотация. Современные технологии должны соответствовать критериям устойчивого развития, учитывающим экономические, экологические и социальные показатели. Поэтому в работе исследовали возможность вовлечения графитсодержащего шлама из аспирационной системы очистки газов при производстве скрытокристаллического графита в состав композиционных противопригарных покрытий для чугунного литья. Графитсодержащий шлам представлен углеродом, серой, натрием, алюминием и кремнием, фазовый состав – графитом, кальцитом, пиритом, кварцем, галитом и др. Шлам – дисперсный материал со средним размером частиц 3,64 мкм, общей поверхностью 36 506 см^2/см^3 и основной фракцией 1–8 мкм. Для частиц шлама характерны различные формы – от неправильных до изометрических. Размеры крупных частиц изометрической формы могут достигать 1 мм и более. На поверхности крупных частиц присутствуют более мелкие дисперсные частицы. Параметры структуры шлама соответствуют параметрам гексагональной формы графита. Анализ состава и свойств графитсодержащего шлама позволяет рекомендовать его применение в составах композиционных противопригарных покрытий. Однако из-за наличия в составе шлама крупных агрегатов графита и примесей игольчатого характера его перед использованием необходимо просеивать. Полная замена природного графита на шлам позволяет повысить плотность покрытия с 1220 до 1750 кг/м^3, вязкость – с 34 до 105 с и прочность к истиранию – с 175 до 245 г/мм. Из-за высокой дисперсности покрытие на основе шлама практически полностью проникает в поры формы из песчано-смоляной смеси, не образуя при этом покровного слоя. Это не обеспечивает стабильного снижения пригара на поверхности отливок. Поэтому полная замена графита на графитсодержащий шлам в составах композиционных покрытий не рекомендуется.

Ключевые слова: самовысыхающее покрытие, графитсодержащий шлам, чугун, пригар, плотность, вязкость, толщина покровного слоя, глубина проникающего слоя, прочность к истиранию

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Introduction

In the last decade, the criteria for product quality have changed, as modern technologies must meet the criteria of sustainable development, which take into account economic, environmental, and social indicators [1–4]. As a result, increasing attention is being paid to the potential for incorporating industrial powder waste into various sectors of industry [5–13].

The experience of using carbon-containing slags from various productions in the compositions of different products has been described in works [14–16]. The advantages of using carbon-containing waste not only enhance the quality of products based on them but also reduce the volume of sludge fields surrounding enterprises, thus contributing to environmental preservation. For example, the processing of graphite-containing dust from metallurgical production allows for the production of high-quality raw materials for a wide range of applications, while also generating additional profit by lowering production costs. On the other hand, the variable composition of the waste, which determines the nature of its thermal degradation, along with insufficient development of technologies, results in the majority of the waste currently being sent to landfills.

In the Krasnoyarsk region, there is a graphite processing enterprise working with the Kureiskoye deposit, which primarily produces cryptocrystalline graphite of the GLS grade, carburizer of the NSGK grade, and sorbent of the SGN-30 grade [17]. After graphite processing, sludge remains, which is stored at the enterprise and subsequently either disposed of or partially recycled.

The purpose of this study was to investigate the composition and properties of graphite-containing sludge and to develop graphite-based coatings for casting molds.

Research methodology

The graphite-containing sludge used in this research was sourced from the gas purification aspiration system during the production of cryptocrystalline graphite (Fig. 1).

The elemental and phase compositions of the sludge were assessed using an XRD-7000 X-ray diffractometer (Shimadzu, Japan) [18]. The elemental composition of the sludge particles was also determined through energy-dispersive microanalysis, using an energy-dispersive spectrometer from Oxford Instruments (UK), mounted on the column of a JSM-7001F scanning electron microscope (JEOL, Japan) [19]. The spectra of cha-

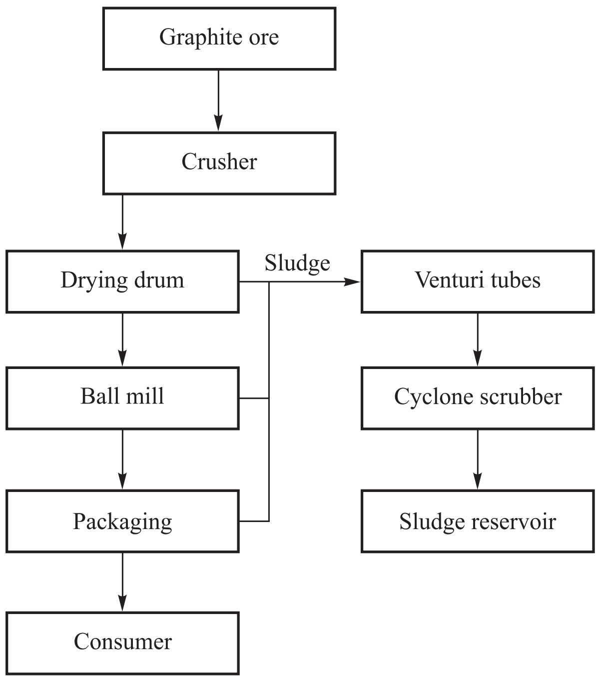


Fig. 1. Schematic of cryptocrystalline graphite production at JSC "Krasnoyarskgrafit"

Рис. 1. Схема производства скрытокристаллического графита на предприятии «АО Красноярскграфит» racteristic *X*-ray emissions from the chemical elements present in the powders were determined at specific points.

The size and total surface area of the sludge particles, as well as their size distribution, were analyzed using the light-scattering method with the Fritsch Analysette 22 MicroTec PLUS laser particle size analyzer (Germany).

The graphite-containing sludge was tested as part of a self-drying composite coating [20; 21].

The properties of the coatings were determined according to GOST 17022–78. The thickness of the cover layer and the depth of the penetrating layers were measured using an Observer.D1m microscope (Carl Zeiss, Germany), and the burn-on level was evaluated using the step test described in [22].

Results and discussion

The elemental composition of the sludge (wt. %) assessed using the XRD-7000 *X*-ray diffractometer is presented below:

O 26.00
C 45.85
H 0.24
Na 0.61
Cl 0.94
S 1.95
Si 13.10
Mg 4.51
Ca 5.10
Fe 1.70

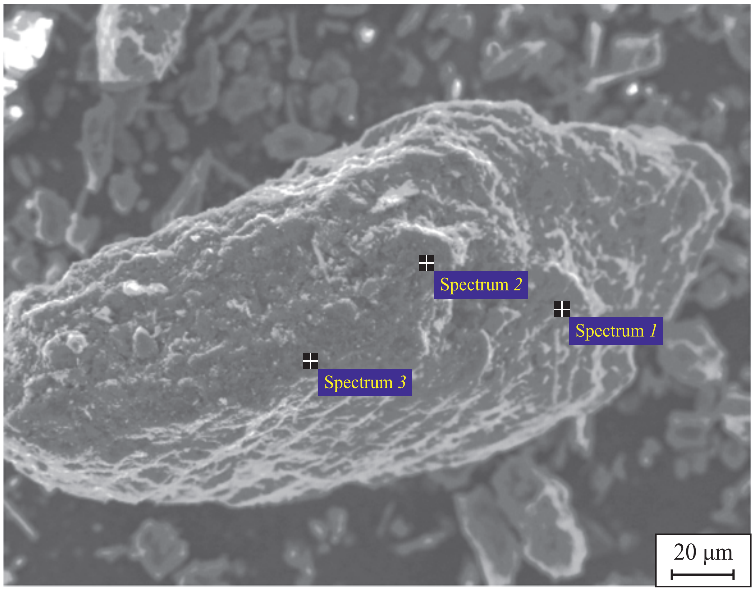
The elemental composition of the sludge particles was also determined using the JSM-7001F scanning electron microscope, and the results are shown in Fig. 2.

The obtained data indicate that the sludge is mainly composed of carbon, sulfur, sodium, aluminum, and silicon.

The phase composition of the sludge (Table 1) includes graphite (up to 47 %), calcite (up to 13 %), pyrite (up to 4 %), quartz (up to 21 %), halite (up to 2 %), with other impurities making up to 17 %.

The reduced pyrite content compared to natural graphite will help decrease burn-on defects on casting surfaces, as discussed in detail in [23]. Calcite, halite, and quartz are materials whose presence increases the coating's refractoriness (due to their high melting points) and prevents interaction between the melt and the molding mixture. Clay minerals (kaolinite, montmorillonite, etc.), present among other phases in the sludge, will act as binders, providing the coatings with higher strength.

The sludge is a dispersed material with an average particle size of 3.64 µm and a total surface area of 36,506 cm^2/cm^3. The study of particle size distribution showed that the majority of particles fall within the range of 1–8 µm (Fig. 3).



Spectrum	C	O	Na	Mg	Al	Si	Cl	K	Ca	Fe
1	80.82	8.77	2.27	0.19	0.65	0.84	4.41	0.63	0.96	0.46
2	75.27	15.45	1.21	0.57	1.09	1.72	2.10	0.36	0.56	1.22
3	85.47	6.70	0.72	–	1.92	2.30	1.32	0.25	1.32	–

Fig. 2. Elemental composition (wt. %) of sludge particles

Рис. 2. Элементный состав (мас. %) частиц шлама

***Table 1.* Phase composition of GLS-2 graphite and graphite-containing sludge**

***Таблица 1.* Фазовый состав графита ГЛС-2 и графитсодержащего шлама**

Phase	Phase content, wt. %	
	GLS-2 graphite	Graphite-containing sludge
Graphite	67.23	43.0–46.56
Quarz	9.99	20.12–21.05
Calcite	10.60	11.70–12.70
Pyrite	4.72	3.65–3.79
Halite	4.37	1.56–1.57
Other inclusions (kaolinite, montmorillonite, etc.)	3.09	17.10–17.20

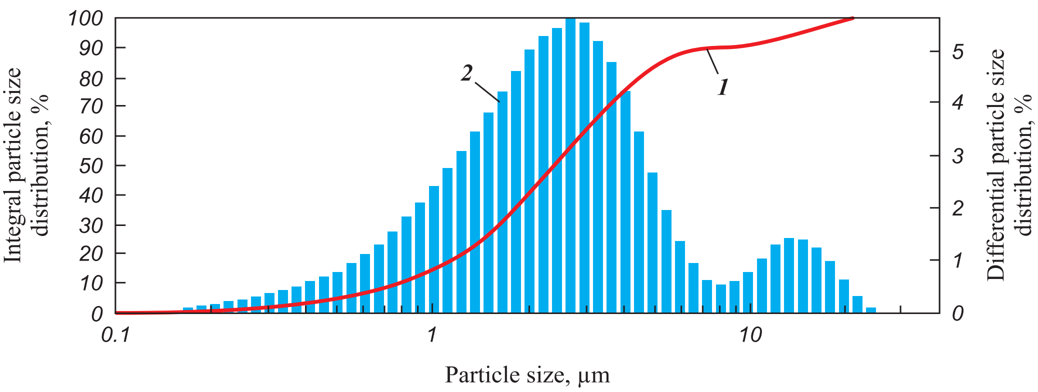


Fig. 3. Fractional composition of the sludge

1 – integral particle distribution; *2* – differential particle distribution

Рис. 3. Фракционный состав шлама

1 – интегральное распределение частиц; *2* – дифференциальное

Morphological analysis of the particles (Fig. 4) revealed that the sludge contains particles of various sizes and shapes, ranging from irregular to isometric.

The majority of the material is uniform, but there are large graphite aggregates and cylindrical inclusions. Smaller dispersed particles are observed on the surface of larger particles. Large inclusions can reach sizes of ≥1 mm. The cylindrical inclusions are impurity phases.

The analysis of the sludge structure showed that its parameters correspond to the hexagonal form of graphite. This suggests that the sludge can be recommended

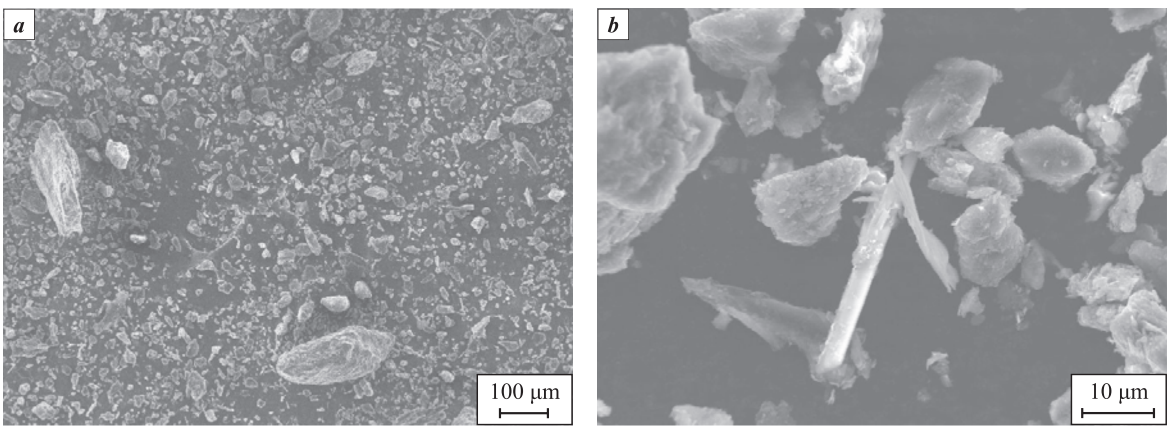


Fig. 4. Sludge images from the JSM-7001F microscope

Рис. 4. Съемки шлама на микроскопе JSM-7001F

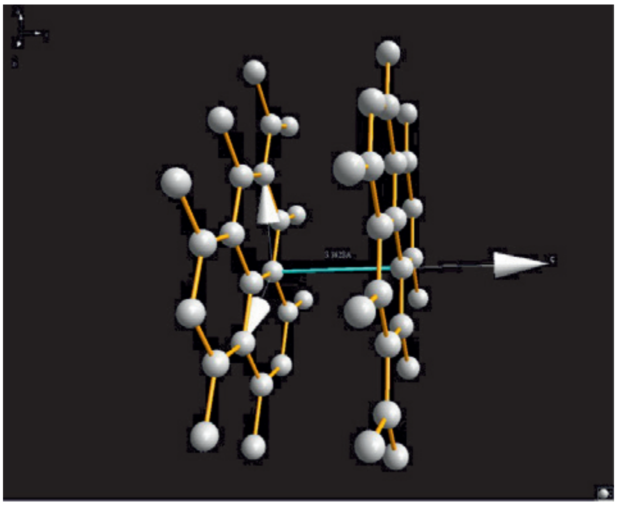


Fig. 5. Simulated graphite structure in the TOPAS 3 program

Рис. 5. Смоделированная структура графита в программе TOPAS 3

for use in composite coatings, provided the large graphite aggregates and cylindrical impurities are removed by sieving. Fig. 5 presents a 3D model of the graphite structure, simulated in the TOPAS 3 program.

The study examined the feasibility of using graphite-containing sludge in the composition of anti-burn coatings for cast iron casting.

To study the thickness of the covering and depth of the penetrating layers of the coating, a cold-setting mixture with the following composition (wt. %) was used: 97.2 – quartz sand $2K_1O_3 03$; 2.4 – alphabond resin; 0.4 – T-01 hardener. The working life of this mixture is 3.2 min, and the tensile strength (after 1 h) is 0.19 MPa.

The properties of composite anti-burn coatings based on graphite-containing sludge are shown in Fig. 6.

The composite coating based on graphite-containing sludge has a higher density and requires addi-

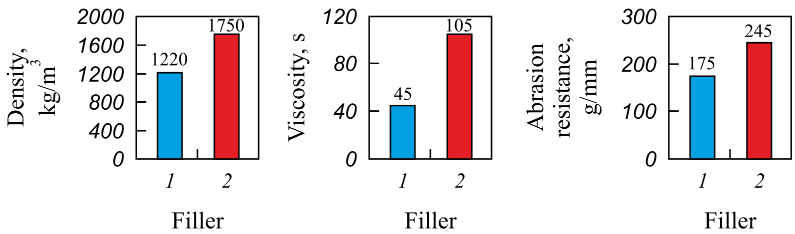


Fig. 6. Properties of anti-burn coatings based on GLS-2 graphite (*1*) and graphite-containing sludge (*2*)

Рис. 6. Свойства противопригарных покрытий на основе графита ГЛС-2 (*1*) и графитсодержащего шлама (*2*)

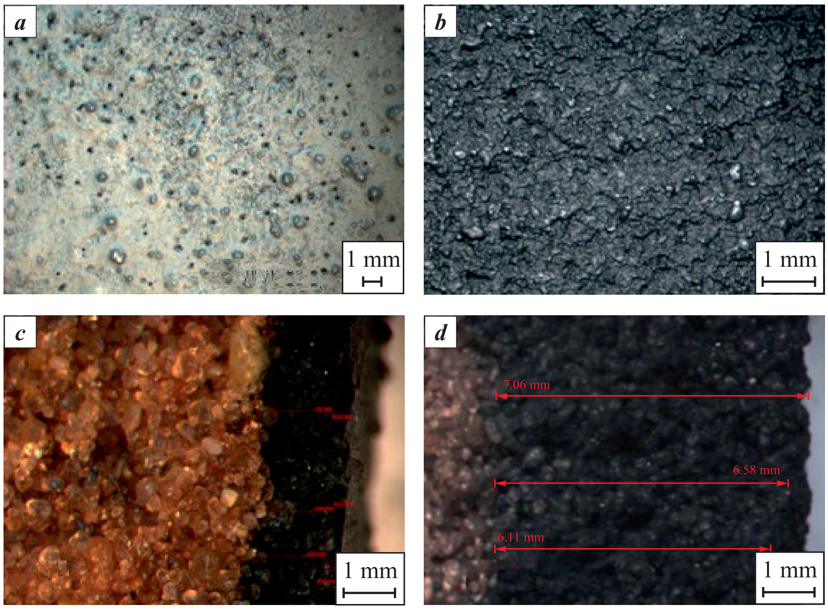


Fig. 7. Covering (***a***, ***b***) and penetrating (***c***, ***d***) layers of the coating
a, ***c*** – natural GLS-2graphite; ***b***, ***d*** – graphite-containing sludge

Рис. 7. Покровный (***a***, ***b***) и проникающий (***c***, ***d***) слои покрытия
a, ***c*** – природный графит ГЛС-2; ***b***, ***d*** – графитсодержащий шлам

tional dilution with PVB lacquer, which may reduce the sludge content.

The thickness of the covering and the depth of the penetrating layers of the coating are shown in Figs. 7 and 8.

The composite coating based on graphite-containing sludge does not form a covering layer, while the depth of the penetrating layer can reach 6.0–6.5 mm. This is because the sludge particles (~3.6 μm) are significantly smaller than the pores of the mold (~230 μm, Fig. 9).

In the study of burn-on magnitude on casting surfaces, a step sample was used. A cold-setting mixture was selected for the research.

During the tests at a temperature of 1400 °C, gray cast iron of the SCH20 grade was poured, with the following composition (wt. %):

C 2.90–2.97
Mn 0.92–0.93
P 0.019–0.021
S 0.042–0.044
Cr 0.10–0.11
Si 2.41–2.60
Ni 0.084–0.086
Cu 0.12–0.13
V 0.049–0.052
Ti 0.021–0.024
Sn . . . . <0.01

The effect of casting wall thickness on burn-on magnitude is shown in Tables 2 and 3.

When graphite is replaced with graphite-containing sludge in the coating, the average burn-on thickness on the casting surfaces decreases. This is due to changes in the phase composition and the smaller particle size of the sludge, which oxidizes more quickly during the heating of the mold surface layers, creating a reducing atmosphere.

However, the variation in burn-on values is significantly higher in the case of graphite-containing sludge. This is due to the higher sulfur content in the form of pyrite, which contributes to an increase in burn-on thickness.

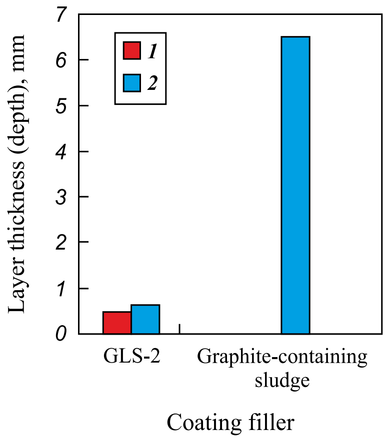


Fig. 8. Thickness of the covering (*1*) and depth of the penetrating (*2*) layers of anti-burn coating

Рис. 8. Толщина покровного (*1*) и глубина проникающего (*2*) слоев противопригарного покрытия

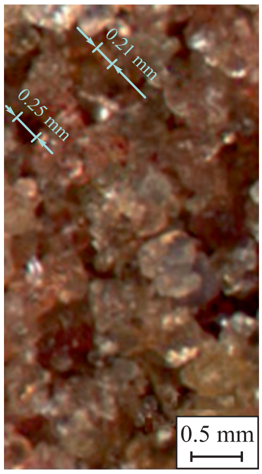


Fig. 9. Pores of the mold

Рис. 9. Поры формы

Thus, the results indicate that it is not possible to completely replace natural graphite in the composite coating with graphite-containing sludge. Therefore,

***Table 2.* Burn-on thickness on the surface of castings obtained using coatings based on GLS-2 graphite**

***Таблица 2.* Значение толщины пригара на поверхности отливок, полученных с применением покрытий на основе графита ГЛС-2**

Casting wall thickness, mm	Casting side	Burn-on thickness, μm			
		minimum	maximum	average	standard deviation
25	Side	40.77	66.31	53.54	9.09
	Bottom	39.88	171.71	105.80	42.55
50	Side	60.77	170.81	115.79	49.98
	Bottom	64.71	265.97	165.34	70.04
75	Side	78.77	223.83	151.30	55.44
	Bottom	82.56	276.84	179.70	82.07

Table 3. **Burn-on thickness on the surface of castings obtained using coatings based on graphite-containing sludge**

Таблица 3. **Значение толщины пригара на поверхности отливок, полученных с применением покрытий на основе графитсодержащего шлама**

Casting wall thickness, mm	Casting side	Burn-on thickness, μm			
		Minimum	Maximum	Average	Standard deviation
25	Side	1.20	69.10	15.98	26.28
	Bottom	10.37	180.03	66.80	51.85
50	Side	4.02	190.72	49.83	66.21
	Bottom	13.02	270.01	64.97	91.03
75	Side	15.46	353.83	95.36	118.63
	Bottom	32.58	420.25	128.94	132.17

further research is required to explore the partial replacement of natural graphite with graphite-containing sludge.

Conclusion

This study has demonstrated the potential for using graphite-containing sludge from the gas purification aspiration system in cryptocrystalline graphite production as part of composite anti-burn coatings. The results of the sludge's elemental composition analysis show that it consists of carbon, sulfur, sodium, aluminum, and silicon. The phase composition of the sludge includes graphite (up to 47 %), calcite (up to 13 %), pyrite (up to 4 %), quartz (up to 21 %), halite (up to 2 %), and other impurities (up to 17 %). The sludge is a dispersed material with an average particle size of 3.64 μm (total surface area – 36,506 cm^2/cm^3); most particles range from 1 to 8 μm. The sludge contains particles of various shapes: the bulk of them measure between 1 and 8 μm, with shapes ranging from irregular to isometric. Larger isometric particles can reach sizes of 1 mm or more. Smaller dispersed particles are present on the surfaces of larger particles. Acicular impurity phases are also present in the sludge. Analysis of the sludge's parameters indicates that it can be used in composite coatings, provided that large graphite aggregates and cylindrical impurities are removed by sieving. In composite anti-burn coatings where natural graphite is replaced with sludge, it is possible to increase the density from 1220 to 1750 kg/m^3, viscosity from 34 to 105 s, and relative strength from 175 to 245 g/mm. However, due to the high dispersity of the sludge, the coating completely penetrates the pores of the sand-resin mold mixture without forming a surface layer. With complete replacement of graphite with graphite-containing sludge in the coating, the average burn-on thickness on the surface of the castings decreases, depending on the wall thickness, from 53.54–151.30 to 15.98–95.36 μm (on the side surface of the castings) and from 105.80–179.70 to 66.80–128.94 μm (on the bottom surface of the castings). However, the variation in burn-on thickness is significantly higher, which is attributed to the higher sulfur content in the graphite-containing sludge, contributing to increased burn-on thickness. Therefore, further research should focus on the partial replacement of graphite with graphite-containing sludge, which would reduce the cost of the coatings and improve the quality of cast iron products.

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