



## INTEGRATED TECHNOLOGIES FOR DESULFURIZATION OF CRYPTOCRYSTALLINE GRAPHITE

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### ABSTRACT

An increase in the carbon content in cast iron and steel during smelting is carried out by adding carbon-containing materials to the charge - carburizers (carburetors). For the fastest and most complete assimilation of carbon, the carburetor must have a minimum amount of impurities and a low ash content. As the ash content of the carburizer decreases, the amount of slag also decreases, which in turn increases the durability of the furnace lining. To prevent the cast iron from becoming unnecessarily saturated with sulfur, the carburetor must contain a minimum amount of sulfur. Currently, coals and natural graphites are widely used as carburetors. Compared to other carbon-containing materials, natural graphites are of limited use because of the high sulfur content, which is represented by pyrite (polysulfide form) or is in the sulfide form in the form of disseminations and aggregative accumulations  $2 \times 1$  mm in size. Therefore, the aim of the work is to develop graphite desulfurization technologies that allow reducing the sulfur content to 0.05%. In this work, mechanical activation, electroexplosive impulse activation, and oxidative leaching were used to desulfurize graphite. It is shown that each of the proposed methods intensifies all subsequent processes of sulfur extraction from graphite, more fully disclosing it from pyrite, pyrrhotite and chalcopyrite particles. Various combinations of these methods make it possible to obtain graphite of the required purity. Processing of graphite according to schemes, including electroexplosive-pulse processing followed by oxidative leaching and mechanical activation, followed by electroexplosive-pulse processing and oxidative leaching, makes it possible to reduce the sulfur content 1 to 0.16–0.21%. At the same time, the use of a combined scheme, including mechanical activation and oxidative leaching, reduces the sulfur content in the composition of graphite to 0.06–0.09%, replacing the oxidative leaching operation with steam treatment of graphite, to 0.05%.

**Keywords:** cryptocrystalline graphite, desulfurization, mechanical activation, electro-explosive impulse activation, oxidative leaching.

### INTRODUCTION

An increase in the carbon content in cast iron and steel during melting is obtained by means of adding carbonaceous materials—carburizers (carburizing agents) to furnace charge [1–5].

To optimize the fastest and complete carbon recovery a carburizer should have a minimal level of impurities and low-ash content. As the ash content of the carburizer decreases, the amount of bottom slag also decreases, which, in turn, increases the durability of the furnace lining. To avoid excessive sulfur saturation of cast iron, the carburizer must contain a minimum amount of sulfur [6, 7].

Currently, coals and natural graphites as carburizers find wide application [1–5].

In comparison with other carbonaceous materials, natural graphites are currently of limited use because of their high sulphur content, which is presented by pyrite (polysulfide form) or available in the sulfide form presented by inclusions and sinters of  $2 \times 1$  mm in size.

Due to the fact that the graphite raw material was formed of sapropelic coals, the mineral components are unevenly distributed within the bulk of the ore in the form of attachments to the main carbon material and micron-sized siltstone. This explains the position of the process men, based on the fact that graphite ore is difficult to enrich, including sulfur [8, 9].

The efficiency of removing sulfur compounds from graphite depends on numerous factors, the main ones being the physical and chemical characteristics of sulfides (crystal structure, stoichiometric composition, magnetic susceptibility, etc.); the total content and ratio of various types of sulfur in graphite; the nature and form of inclusions of sulfides in graphite; the distribution of sulfur compounds according to size classes; the degree of disclosure of iron sulfides attachments to graphite during grinding, crushing; the accuracy of separation in enrichment processes; applied equipment and desulfurization technology [10].

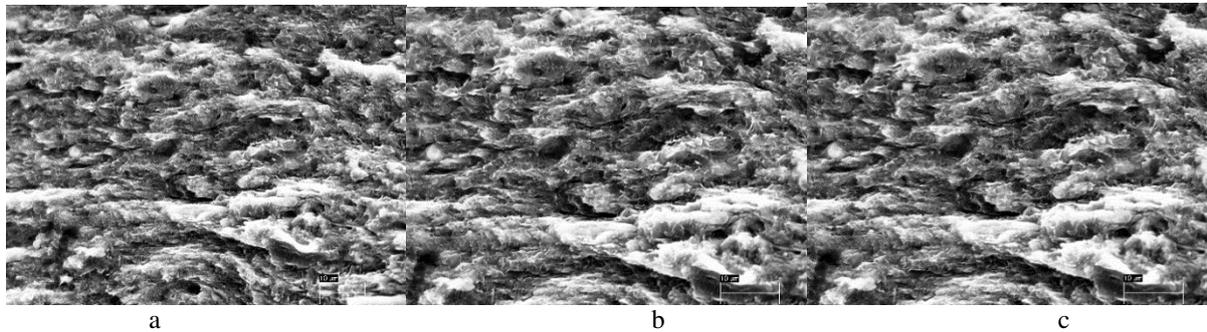


The objective of this paper is to develop technologies for graphite desulfurization, which allows reducing the sulfur content to 0.05 %.

### EXPERIMENTAL METHODS

In this study, we have suggested cryptocrystalline graphite which is finely ground, even-grained material, particles of which can reach 0.15 mm, the predominant particle size is less than 0.05 mm. Dust content in the

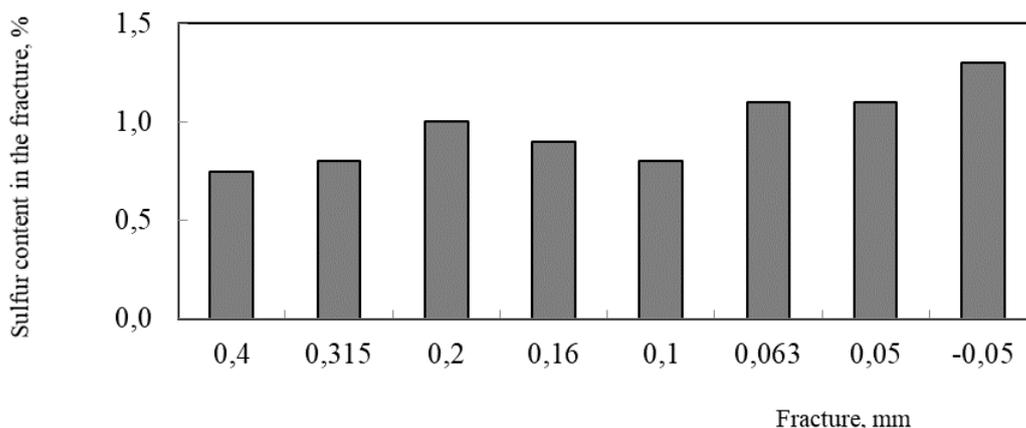
graphite may reach 15 %. A characteristic feature of cryptocrystalline graphite is that the graphite flakes have the form of its attachments to non-metallic minerals making up the ore. The distribution of non-metallic minerals in the ore may be uneven (from 5 to 45 %) and presented by scattered inclusions (particle size may be from 0.05 to 0.001 mm), separate particles or sinters crosscutting the ore in all directions. In this case the size of large inclusions reaches 0.2 cm (Figure-1) [8, 9, 11].



**Figure-1.** The surface of GLS-2 (K) graphite [11]. Zooming, times: *a* – 3 000; *b* – 4 000; *c* – 5 000.

The sulfur content in graphite corresponds to 1 %, and the maximum content of sulfur is observed in small fractions (Figure-2). In cryptocrystalline graphite, sulfide sulfur is mainly available (2-3%), presented by pyrite, pyrrhotine and chalcopyrite. Sulfides form inclusions and

sinters in the ore in the form of lenses and nests with a size of 2×1 mm. Sulfides are most widely developed in the veins in the form of rims at the contact of the ore with the host rock [8, 9, 11].



**Figure-2.** Sulfur content in cryptocrystalline graphite.

The methods of enrichment of cryptocrystalline graphite developed by the authors previously do not allow reducing the sulfur content less than ... % [12-21].

Therefore, in order to reduce the total sulfur content, mechanical, electro-explosive impulse activation, oxidative leaching and steam-air activation were tested.

Mechanical activation of cryptocrystalline graphite was carried out in a planetary ball-bearing mill [22].

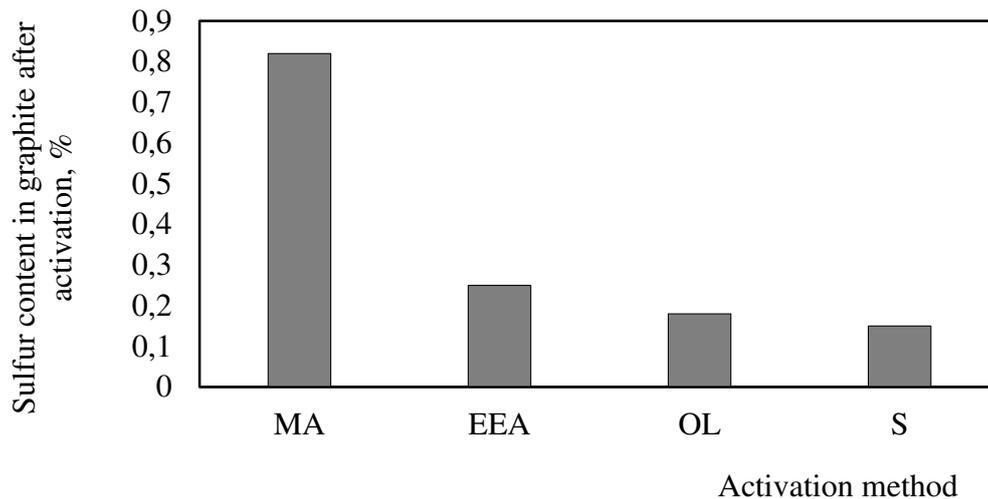
The installation described in the works was used for electro-explosive impulse activation [23, 24].

For oxidative leaching we applied an installation consisting of a glass vessel with a device for mixing and

heating the solution; for steam-air oxidation a flow - type installation was used. The settings are described in the studies [23, 24].

### THE EXPERIMENTAL PROCEDURE

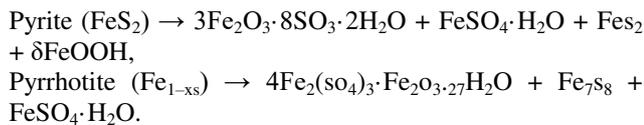
Previous studies have shown that the use of separate desulfurization technologies (mechanical activation (MA), electro-explosive impulse activation (EEA), oxidative leaching (OL) and steam-air activation (S)) does not allow reducing the sulfur content in graphite less than 0.15 % (Figure-3).



**Figure-3.** Sulfur content in graphite activated by various methods: MA-mechanical activation; EEA-electro-explosive impulse activation; OL-oxidative leaching; S-steam-air activation.

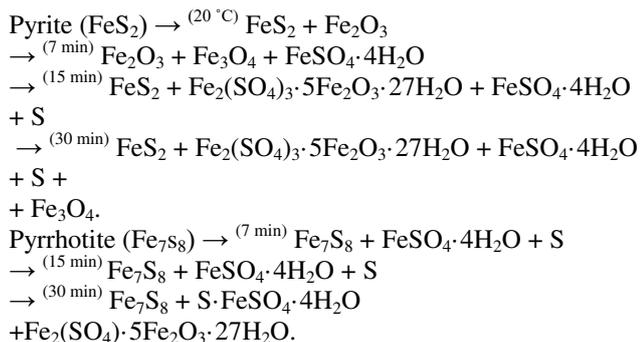
The results of the study of chemical analysis of graphite showed that mechanical activation contributes to an increase in the sulfur content from 1.0 (in natural graphite) to 0.82 %. This is probably due to a more complete disclosure of sulfur-containing minerals, the development of their reactivity, and a more complete transition of sulfur to a controlled gaseous form in terms of chemical analysis.

The following chemical reactions proceed in terms of *mechanical activation* of iron sulfides in the air environment for 2–10 min [25, 26]:



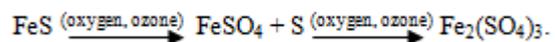
Disruptions of the crystal lattice of these minerals under mechanical action facilitate the course of redox reactions, as a result of which sulfur atoms are able to form water-soluble compounds [27-32].

The grinding of sulfide minerals in an aqueous medium can be presented by the following reactions:



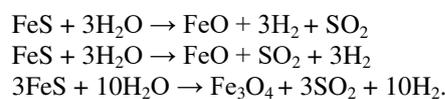
In terms of *electro-explosive impulse activation*, the released oxygen and ozone oxidize FeS<sub>2</sub> to sulfates. Pyrite oxidation proceeds through the formation of iron

(II) sulfide with free sulfur release and its subsequent oxidation to sulfates [32–34, 35], according to the scheme:

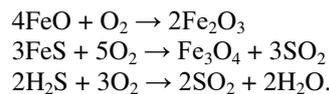


The chemistry of *oxidative leaching* (OL) depends on the oxidant, which can be used as ozone, KMnO<sub>4</sub>, H<sub>2</sub>O<sub>2</sub>, Na<sub>2</sub>O<sub>2</sub> [36], K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, NaNO<sub>3</sub> [37], technical bleach powder Ca (OCl)<sub>2</sub> [38]. In this case, the sulfide sulfur of the solid phase (S<sup>2-</sup>) passes into the soluble form-sulfate (SO<sub>4</sub><sup>2-</sup>).

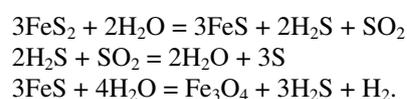
The oxidation of iron (II) sulfide by water steam can be described by means of the following equations [32, 33, 39–42]:



One of the components of the steam-gas mixture is air oxygen, which is also a part of the oxidation processes:



Oxidation of pyrite (FeS<sub>2</sub>) with water steam starts at a temperature of 200 °C and proceeds according to the following reactions [39]:



The analysis of the sulfur content in desulfurization products shows that the use of the



considered methods does not lead to a sulfur content decrease in graphite less than 0.15 %.

Thus, the obtained data indicate that the MA method promotes the release of sulfide minerals from cryptocrystalline graphite, but does not lead to a sulfur decrease in graphite and can be only used in combination with other activation methods. At the same time, the methods of EEA, OL and S make it possible to convert sulfide-containing compounds into water-soluble or gaseous ones, but due to the close fusion of sulfur-containing compounds and graphite particles, the efficiency of their removal is quite low and does not allow reducing the sulfur content in graphite less than 0.15 %.

Therefore, the following different combination of these methods was tested (Tables 1, 2):

- mechanical activation + oxidative leaching (MA + OL);
- mechanical activation + steam-air oxidation (MA + S);
- mechanical activation + oxidative leaching + steam-air oxidation (MA + OL + S);
- electro-explosive impulse activation + oxidative leaching (EEA + OL);
- mechanical activation + electro-explosive impulse activation + oxidative leaching (MA + EEA + OL).

Treatment of cryptocrystalline graphite by a complex method-mechanical activation followed by oxidative leaching allows reducing the sulfur content in graphite to the values approximate to the required ones (0.1–0.05 %).

The combination of mechanical activation with further oxidation of impurity components by water steam reduces the sulfur content in conditioned graphite to 0.05 %.

Due to the impact on graphite samples caused by electric explosion combined with chemical treatment reduces the sulfur impurity by 30 %.

Processing graphite according to the combination of MA + EEA + OL leads to a decrease in the sulfur content by 50 %. When studying the chemical composition of graphite, it was noted that along with a decrease in the sulfur content, the ash content of graphite samples decreases.

The obtained data indicate that pre-activation intensifies all subsequent processes of sulfur extraction from graphite significantly, revealing it to more extend from pyrite, pyrrhotite and chalcopyrite particles. Complex methods of graphite desulfurization (MA + S; MA + S + OL; EEA + OL; MA + EEA + OL) can reduce the sulfur content to 0.05 %.

**Table-1.** Sulfur content in graphite according to the parameters of mechanical and chemical activation.

Method of desulfurization	Activation						The sulfur content, %
	Mechanical (MA)		Steam-Air (S) at temperature, °C	Oxidative leaching (OL)			
	Activator type	Processing environment		The concentration of the oxidant, %	Temperature, °C	Leaching time, h	
Natural graphite	–						1,00
MA + S	Planetary Ball-bearing Mill	Air	800	–	–	–	0,05
MA + OL			–	10	40	2	0,21
MA + OL			–	20	40	2	0,11
MA + OL			–	30	40	2	0,09
MA + OL			–	10	60	2	0,10
MA + OL			–	10	75	2	0,06
MA + OL	Water	Water	–	10	–	0,5	0,16
MA + S + OL			800	10	–	2	0,105



**Table-2.** Sulfur content in graphite according to the parameters of electro-explosive impulse, mechanical and chemical activation.

Method of desulfurization	Activation				The sulfur content, %
	Mechanical (MA)		Electro-explosive impulse (EEA)	Chemical (OL)	
	Activator type	Environment	Number of explosions	Oxidant concentration, %	
Natural graphite	–				1,00
EEA + OL	–	–	2	–	0,21
MA + EEA + OL	Planetary Ball-bearing Mill	Air		10	0,16

## CONCLUSIONS

Combined technological schemes of graphite desulfurization have been developed, which allow choosing the optimal method according to the required graphite purity. The obtained data make it possible to conclude that pre-activation intensifies all subsequent processes of sulfur extraction from graphite significantly, revealing it from pyrite, pyrrhotite and chalcopyrite particles to more extend. According to studies outcomes, the content of sulfur in graphite decreases from 1 % to 0.16 % in terms of processing based on the following scheme: electro-explosive impulse treatment followed by oxidative leaching. Mechanical activation followed by electro-explosive impulse treatment and oxidative leaching leads to decrease of sulfur content from 1 % to 0.21 %. The use of a combined scheme, including mechanical activation and oxidative leaching, reduces the sulfur content in the graphite composition to 0.06–0.09%, replacing the oxidative leaching operation with water steam treatment of graphite reaches to values up to 0.05 %.

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