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COMPARATIVE ANALYSIS OF PRODUCTS OF ELECTRIC ARC SYNTHESIS USING GRAPHITE OF DIFFERENT GRADES

Carbon nanostructures (CNSs) of different types (carbon nanotubes, fullerenes, and fullerene-like structures) are obtained by the method of electric arc evaporation of SIGE and FGDG-7 graphites in an inert gas (He). A comparative analysis of the characteristics of synthesized CNSs is performed. The optimal technological conditions for the synthesis of CNSs from graphite anode electrodes of comparable grades (SIGE and FGDG-7) are determined. Deposits of the plasma chemical synthesis have been studied. The structure of the synthesized carbon materials is studied by scanning and transmission electron microscopies, and it is shown that carbon nanotubes are formed during the evaporation of SIGE brand graphite even without the use of a catalyst. Differential-thermal, thermogravimetric and differential thermogravimetric analyzes are carried out, and the temperatures of the beginning of the interaction of the formed CNSs with oxygen in air are established. According to data of the photospectral analysis of the synthesis products, it is shown that the fullerene component obtained by the evaporation of SIGE brand graphite contains 10–12% of C_{60} and C_{70} fullerenes, which is not inferior to similar indicators of MPG-7 brand graphite. In view of the cheapness of SIGE brand graphite as compared to FGDG-7 graphite brand, it can be argued that carbon nanostructures synthesized from SIGE brand graphite have a lower cost. This fact is important for the synthesis of carbon nanostructures used as fillers in modern composites. In addition, the synthesis of low-cost fullerene and fullerene-like molecules is a great advantage for their study and use in modern materials, because new modern advanced nanotechnologies on the basis of fullerenes are beginning today to be created.

Keywords: nanotechnology, carbon nanostructures (CNSs), fullerenes, nanocomposites, carbon nanotubes (CNTs), graphene, plasma, electric arc synthesis, graphite grade MPG-7, graphite grade SIGE.

1. Introduction

The method of electric arc evaporation of graphite in the inert gas environment is relatively common, productive, and quite effective, since it allows one to obtain both soluble [1–7] and insoluble carbon nanostructures (CNSs) [8–12]. In 1996, the authors of [13] received the Nobel Prize for the discovery of

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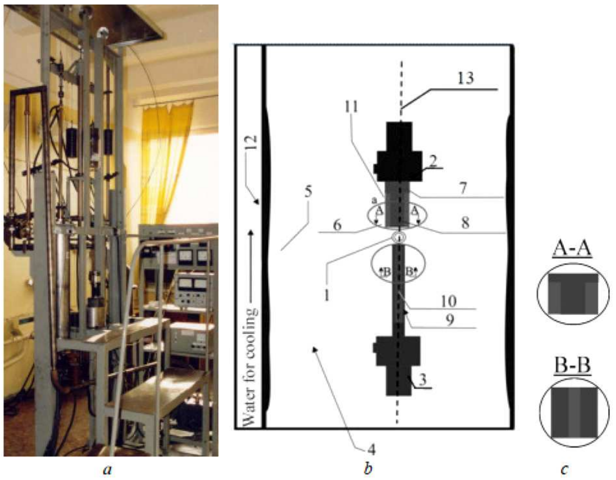


Fig. 1. The back view of the electric arc plasma-chemical installation (*a*) and the scheme of a cylindrical reactor for the CNS synthesis in the gas environment (*b*): *1* – plasma; *2* – collet, which holds a non-consumable cathode electrode; *3* – collet for fixing the consumable anode electrode; *4* – helium-containing environment; *5* – wall soot; *6* – deposit; *7* – non-consumable electrode (cathode); *8* – deposit core; *9* – consumable anode; *10* – catalyst in the electrodes; *11* – deposit rind; *12* – wall of the reactor, *13* – axis of the reactor for the CNS synthesis. Schematic representation of the rind and core of the approved deposit (*c*)

fullerenes, and, in 2016, the work on the study of soluble carbon nanostructures (fullerenes) [14] was nominated for the Nobel Prize.

There are other no less perspective methods for the synthesis of carbon nanostructures [15–22], whithin which it is also possible to create new modern materials [23–30]. But only the electric arc evaporation of the anode can guarantee the synthesis of fullerene molecules in large quantities. The method allows one to easily change the modes of synthesis, use a gaseous medium of different chemical compositions, and, which is most important, to achieve a high percentage of carbon nanomaterials (CNMs) of different chemical compositions, structures, and morphologies.

Such materials can be used to store hydrogen [14, 31, 32] and compete with ainailable materials for the hydrogen storage [33–47].

For the synthesis of new carbon nanomaterials, FGDG-7 graphite is usually used. The name of the mark is deciphered as “Fine-grained dense graphite”, and the number 7 at the end of the abbreviation indicates the density of graphite, which is equal to 1700 kg/m^3.

The mass production of SIGE graphite brand, which deciphered as “Special Impregnated Graphite Electrodes”, allows it to be used not only in industry, but also to create new productions based on it. For example, such graphite can be the basis for the synthesis of new carbon nanomaterials. In addition, the price of SIGE graphite is much lower than that of the FGDG-7 graphite brand.

However, for today, the carbon nanoproducts of the electric arc synthesis of SIGE graphite brand in a gaseous environment remains unexplored. Therefore, to use graphite of this brand in a nanotechnology, it is necessary to study and analyze the possibility of the synthesis of carbon nanomaterials from it.

The main purpose of this work is realization of comparative studies of products and technological conditions for the synthesis of carbon nanomaterials from SIGE and FGDG-7 graphite grades.

Such an analysis can reveal a number of advantages of SIGE graphite grade in the production of various CNPs and can open the additional opportunities for its use in the electric arc synthesis.

2. Research Methods and Equipment

In the process of electric arc synthesis in a gaseous environment at the installation for the production of fullerene carbon black “VEGA-2”, it is possible to evaporate or spray the anode electrode, which allows one to change the spectrum of synthesized products.

The electrode evaporation is a mode of electric arc synthesis, where the anode component turns into the atomic state, creating conditions for the CNS synthesis. During this evaporation of the electrode, the fullerenes and fullerene-like structures are synthesized.

The electrode spraying is a mode of electric arc synthesis, where the anode component does not turn into the atomic state. As a rule, such process takes place at a high feed rate of the cathode electrode, where the anode does not have time to evaporate, and its graphite component turns into a deflated state. Our results of studies of the electric arc synthesis mode indicates the process of formation of graphene packets modified by the used catalyst.

Evaporation mode of the consumable anode electrode was used for the synthesis of CNS by the electric arc method. The experimental electric arc plasma-chemical synthesis unit includes a vertical cylindrical reactor (Fig. 1). At its center, there is a collet (Fig. 1, *б* (3)), which holds the consumable anode electrode (Fig. 1, *б* (9)). Above it, we see a collet (Fig. 1, *б* (2)), which holds a non-consumable cathode electrode (Fig. 1, *б* (7)). In the process of electric arc synthesis between the electrodes, plasma is generated (Fig. 1, *б* (1)). The consumable anode electrode evaporates in this plasma. The environment of the working chamber of the CNS synthesis reactor is filled with helium (Fig. 1, *б* (4)).

Non-expendable cathodes made of high-quality graphite brand FGDG-7, as well as consumable anode electrodes made of graphite brand SIGE (State Standard ISO 9001-2015) and graphite brand FGDG-7 (State Standard 26132-84) were used in the research.

During the electric arc synthesis of CNSs, two types of electrodes are used: a consumable anode electrode (Fig. 1, *б* (9)) and a non-expendable cathode electrode (Fig. 1, *б* (7)). Near the vertical reactor (Fig. 1), a non-expendable cathode electrode (Fig. 1, *б* (7)) moves by bridging the reactor axis (Fig. 1, *б* (13)), when evaporating a fixed consumable anode electrode in plasma (Fig. 1, *б* (1)) at a pressure of 0.02–0.09 MPa in the helium environment (Fig. 1, *б* (4)). In this manner, the identical growth of soot (Fig. 1, *б* (5)) can be ensured from all of the reactor walls, and the same thermodynamic means for the synthesis of all CNMs and condensation can be reached.

An empty graphite anode located along the reactor axis (Fig. 1, *б* (13)) was used to introduce the catalyst into the electric arc synthesis zone. The placed catalyst was in the form of a wire or powder, and it was fixed in the anode cavity with pressed graphite dust (Fig. 1, *б* (10)).

Anodes of different geometric sizes were used ($0.9 \times 0.9 \times 48$ cm; $1.5 \times 1.5 \times 48$ cm; $2 \times 2 \times 48$ cm; $2.5 \times 2.5 \times 48$ cm). In this work, we will study the morphology and structure of the micro- and nanoscale materials formed by the electric arc evaporation in a gaseous environment of graphite grades SIGE and FGDG-7. Moreover, we will consider the effect of vapors of chemical impurities in graphite (Si, Fe, Al, Mg, Cu, B, and Mn) on the formation of carbon nanostructures.

The comparison of the contents of some elements-impurities in the graphite grades SIGE (produced by "Ukrgrafit") and FGDG-7 is given in Table 1, and some other characteristics are compared in Table 2.

3. Research Results and Discussion

Graphite electrodes of SIGE brand with a size of not more than $0.9 \times 0.9 \times 48$ cm in the process of evaporation acquired a high fragility. Therefore, it was concluded that, for the CNS synthesis, it is necessary to evaporate electrodes larger than $0.9 \times 0.9 \times 48$ cm in size. In addition, increasing the cross-sectional area of the consumable electrodes allowed one to increase the quantitative output of fullerene-containing ash in the one synthesis cycle.

The duration of the arc synthesis of CNS from graphite of SIGE brand was not more than 3 hours with a small overall size of the electrode ($0.9 \times 0.9 \times 48$ cm), whereas the evaporation of the electrode from graphite brand FGDG-7 of the same size lasted in twice as long (6 hours) (Table 3). In addition,

Table 1. **The content of some elements in graphite grades FGDG-7 (State Standard 26132-84) and brand SIGE (State Standard ISO 9001-2015)**

Impurities, %	Graphite grade SIGE [a]	Graphite grade FGDG-7
Silicon (Si)	4.6×10^{-3}	–
Iron (Fe)	9.0×10^{-2}	1×10^{-3}
Aluminum (Al)	6.4×10^{-4}	1×10^{-3}
Magnesium (Mg)	4.1×10^{-3}	–
Copper (Cu)	0.14×10^{-4}	1×10^{-4}
Boron (B)	0.31×10^{-4}	3×10^{-4}
Manganese (Mn)	0.14×10^{-4}	5×10^{-5}
Calcium (Ca)	2.4×10^{-2}	–
Titanium (Ti)	4.8×10^{-4}	–
Sodium (Na)	3.1×10^{-4}	–
The total content of impurities, %	0.3	–

[a] The content of only some of the impurities is given.

Table 2. **Characteristics of graphite grades FGDG-7 and SIGE**

Characteristics	Graphite grade SIGE	Graphite grade FGDG-7
Density, not less, g/cm^3	1.5	1.72
Compressive strength, not less, MPa	–	65
Bending strength, not less, MPa	6.5	34
Specific resistance, not more, μOhm $\cdot$ m	11	18
Ash content, not more, %	0.2	0.25

Table 3. **Conditions and results of synthesis of fullerene-containing soot from graphite electrodes of brands FGDG-7 and SIGE (0.9 × 0.9 × 48 cm)**

Experiments with graphite of the SIGE brand	No 1	No 2	No 3	No 4	No 5
Conditions					
Cathode length, cm	7.5	7.5	7.5	7.5	7.5
Anode length, cm	40.5	40.5	40.5	40.5	40.5
The anode mass before annealing, g	53	52	53	50	53
The mass of the anode after annealing, g	48	47	48	45	48
Current strength, A	120–140	160	180	180	170
Voltage, V	24–36	14–18	20	23	42
Vacuum meter, atm	0.73	0.71	0.71	0.72	0.75
Reactor wall temperature, °C	25	27	25	26	25
Synthesis environment	Helium	Helium	Helium	Helium	Helium
Results					
Soot, g	6	10	20.8	21.8	25
Deposit mass, g	15.95	13.95	7	7	8
Deposit length, cm	7.8	5.8	2.5	2.5	2.5
Anode length after synthesis, cm	21	19	11	13	11
Mass of anode residue, g	25.9	23.9	15.5	17.5	19.5
Electrode chips, g	2	3	5.5	4.5	6.5
Synthesis time, hours	1 : 15	3 : 15	3 : 10	3 : 10	3 : 05
Power, kW	2.9–5	2.2–2.9	3.6	4.14	7.14

the evaporation of the graphite electrode of SIGE brand was accompanied by fluctuations in the electric current from 120 to 180 A at a voltage of 14–42 V, whereas the evaporation of the graphite electrode of FGDG-7 brand occurred at an electric current of 185–200 A at a voltage of 30–33 V (Table 3). As for the process of evaporation of SIGE graphite electrodes 0.9 × 0.9 × 48 cm in size, we calculated that the average required power of the electric arc is 3.98 kW, which is twice less than for the evaporation of graphite electrode of FGDG-7 brand (6.65 kW) (Table 3).

Such difference in the evaporation rates of graphite electrodes of various brands can be explained by the low density of graphite of SIGE brand (Table 2).

In the process of electric arc synthesis in a gaseous environment on the walls of the reactor (Fig. 1, *b* (12)), *wall soot* is formed (Fig. 1, *b* (5)), and, on the cathode electrode, the *deposit* is synthesized (Fig. 1, *b* (6)), which is a by-product of the synthesis.

The *wall soot* is the main product of the electric arc synthesis. It contains soluble and insoluble carbon nanostructures, the nucleation of which occurs in the synthesis zone. Soluble carbon nanostructures, which are contained in theash, include fullerenes, endofullerenes, and fullerene-like products. Insoluble carbon nanostructures include carbon nanotubes (CNTs), graphenes, and graphene packets.

The *deposit* is a by-product of the synthesis formed on the cathode electrode in the form of an outgrowth always located along the electrode reactor axis (Fig. 1, *b* (6)). It was found that the deposit consists of the *core* and *rind.* Physicochemical studies of the deposit morphology indicate that the structures of the core and rind of the deposit depend on the catalyst present in the electric arc synthesis zone.

The *deposit core* is a component of the deposit formation (Fig. 1, *b* (8)), which has its own structure and consists of CNTs, which are firmly connected with the rind of the deposit. But, it has been experimentally proven that, when using a catalyst in the electric arc synthesis process, the deposit core is formed as an independent structure in the deposit and is easily separated from the rind. The *deposit rind* is a component of the deposit formation and is its outer layer (Fig. 1, *b* (11)). The deposit rind has its own structure and consists of graphene, graphene packaging, and CNTs, which, in the non-catalytic synthesis, are also firmly connected with the core of the deposit.

At the end of the evaporation of the graphite electrode of SIGE brand ($0.9 \times 0.9 \times 48$ cm), the mass of the deposit (14–16 g) exceeds the mass of fullerene-containing wall soot (6–10 g). Therefore, a mode of evaporation of the graphite electrode of SIGE brand was developed, in which it is necessary to spend a more power for the electric arc (6.64 kW), and the output of wall soot is more than 25 grams from one electrode (Table 3). Such high output of near-wall soot in the case of electric arc evaporation of graphite electrodes of the SIGE brand significantly exceeds the similar parameter of the evaporation of graphite electrodes of the FGDG-7 brand of the same size. This means that SIGE brand of graphite is a more productive for the synthesis of CNSs by the electric-arc method. The general view of the consumable anode electrode after its partial evaporation is presented in Fig. 2.

Thus, it can be argued that, in the process of CNS synthesis during the evaporation of the graphite anode of the SIGE brand, a smaller part of the chemical component forms a deposit, and the main part of the electrode is spent on the formation of near-wall soot. Wall soot is formed by moving the active component of the gaseous environment in the zone of lower pressure and temperature to the periphery until it cools on the reactor wall.

During the synthesis, when the SIGE graphite anode is evaporates, a deposit is constantly formed on the non-consumable cathode electrode (Fig. 3). It could be formed coaxially (Fig. 3, *a*) or not coaxially (Fig. 3, *b*) relative to the cathode electrode, but it is necessarily formed coaxially with the consumable anode electrode.

The SIGE graphite deposit also consisted of two parts: a core and a rind, which was formed by layered graphite-like structures containing a number of multiwalled carbon nanotubes (MCNTs). The schematic representation of the deposit growth in a section along its axis is shown in Fig. 1, *c* in the footnote of cross-section A-A, which illustrates the rind and core of the formed deposit.

In compliance with the specially developed evaporation regime of graphite of SIGE brand, when the maximum amount of fullerene-containing soot is synthesized at an electric arc power of 6.64 kW, and a deposit of 2.5 cm long and 8g weight is formed. Such characteristics do not exceed the parameters of the deposit obtained by the evaporation of the graphite electrode of FGDG-7 brand, which had an average length of 5 cm and a weight of 14.5 g (Table 4). At the evaporation of the consumable anode electrode of SIGE brand with an average electric arc power of 3.98 kW (under standard conditions) forms a deposit with a length of 7.8 cm and a weight of 15.95 g. Such indicators exceed the values of similar parameters of synthesis products from graphite of FGDG-7 brand (Table 4).

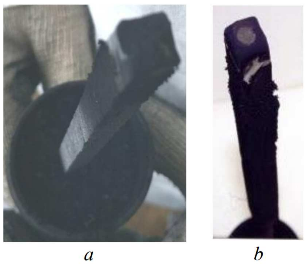


Fig. 2. General view of the consumable anode electrode after its partial evaporation: SIGE graphite brand (*a*); FGDG-7 graphite brand (*b*)

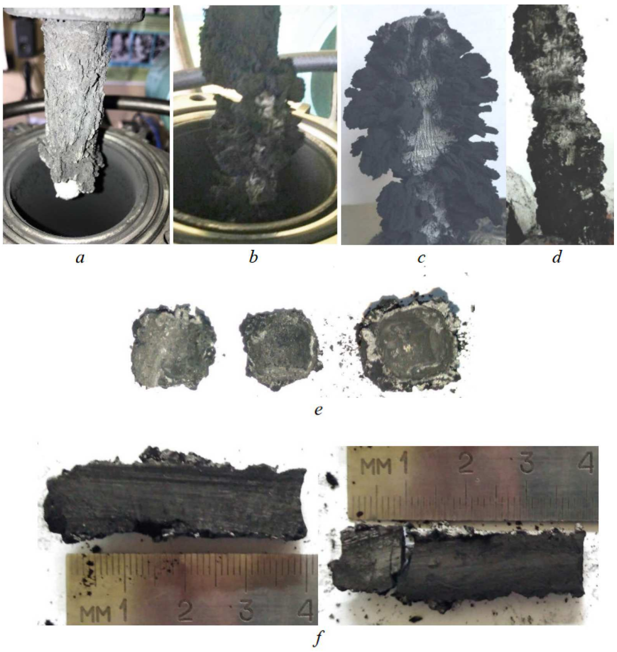
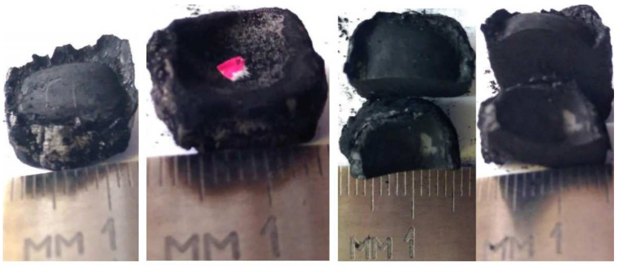


Fig. 3. General view of the deposit formed when using SIGE brand graphite: coaxial cathode deposit (*a*); misaligned cathode deposit (*b*); with carbon nanostructures on the surface (*c*), the surface of the deposit without carbon nanostructures on the surface (*d*); cross-section of the deposit (*e*), longitudinal section of the deposit (section along its axis) (*f*)



a *b* *c*

Fig. 4. Appearance of one of the blocks of SIGE brand graphite deposit: inner (upper) part of the hemispherical block (*a*); the lower part of the hemispherical block (*b*); a deposit block is cut along its axis (*c*)

Table 4. **Comparison of conditions and results of the synthesis of fullerene-containing soot from graphite electrodes of brands FGDG-7 and SIGE**

Anode material	FGDG-7 graphite	SIGE graphite
Conditions		
Cathode length, cm	8.4	7.5
Anode length, cm	40.5	40.5
Current strength, A	185–200	120–180
Voltage, V	30–33	14–42
Pressure, atm.	0.190–0.174	0.70
Reactor cooling jacket temperature, °C	31	25.6
Synthesis environment	Helium	Helium
Results		
Soot mass,g	20.4	25
Deposit mass, g	14.5	8
Chips mass, g	9.400	6.5
Anode length after synthesis, cm	4.5	11
Deposit length, cm	5	2.5
Average discharge power, kW	6.65	6.64
Synthesis time, hours	6	3

The general view of the deposit formed at the use of graphite of the SIGE brand is presented in Fig. 3. The figure shows the form of the deposit from side (Fig. 3, *a*–*d*), in cross *c* and longitudinal *d* sections, where the interfacial boundary between the core and the rind (shell) is clearly expressed. The main part of the deposit obtained from graphite of SIGE brand, is the core (Fig. 3, *e, f*), as well as in the deposit of graphite of FGDG-7 brand. Thin crusty shell of the deposit obtained from SIGE graphite is rigidly bonded to the core (Fig. 3, *e, f*). Also shown is the surface of the deposit without carbon nanostructures on the surface (Fig. 3, *d*). Carbon nanostructures on the surface of the deposit turned out to be bundles of single-walled carbon nanotubes. This is explained by the fact that, during the formation of single-walled carbon nanotubes, they receive a positive charge, which directs their motion vector to the cathode electrode, enhanced by the turbulent flows of the synthesis zone.

Upon a detailed study of the deposit, we see that, in the synthesis, it takes the form of an anode. In other words, if we look at the deposit from above, the core represents a square insert in the deposit (Fig. 3, *e*). Therefore, if the evaporating anode has a rectangular shape, then the core of the deposit in the synthesis will acquire a rectangular shape (Fig. 3). This is important for the creation of a controlled shape of the composite (carbon nanotubes and graphene packs) in the form of a deposit outgrowth that can be resistant to high electrical loads (10 kW) and plasma temperatures (>4000 K).

The analysis of the mechanical strength of synthesized deposits revealed that the deposit consists of hemispherical blocks (Fig. 4), enclosed in a single structure, fastened by a shell (Fig. 3, *f*). Studies have shown that the length of one block of a formed deposit is equal to the step of feeding the electrode at the electric arc synthesis.

The inner (upper) part of the deposit block is visually resembles a hemisphere (Fig. 4, *a*). It is adjacent to the lower part of the hemispherical block of the deposit, which was formed earlier in the process of electric arc synthesis from SIGE graphite. The separation of deposit blocks is possible by a simple mechanical manipulation.

The lower part of the hemispherical block of the deposit is concave from the anode electrode to the cathode electrode (Fig. 4, *b*). To visually identify the hemispherical state of the deposit block at the bottom of its lower part, a red paper indicator was placed (Fig. 4, *b*). At the bottom of the formed hemispherical deposit block, we see the formation of a new deposit block in the process of electric arc synthesis from graphite of the SIGE grade (Fig. 4, *b, c*).

The phenomenon of the deposit structure, which consists from the blocks, is not observed in the deposits synthesized from graphite of FGDG-7 grade. Information about the natural differentiation of the

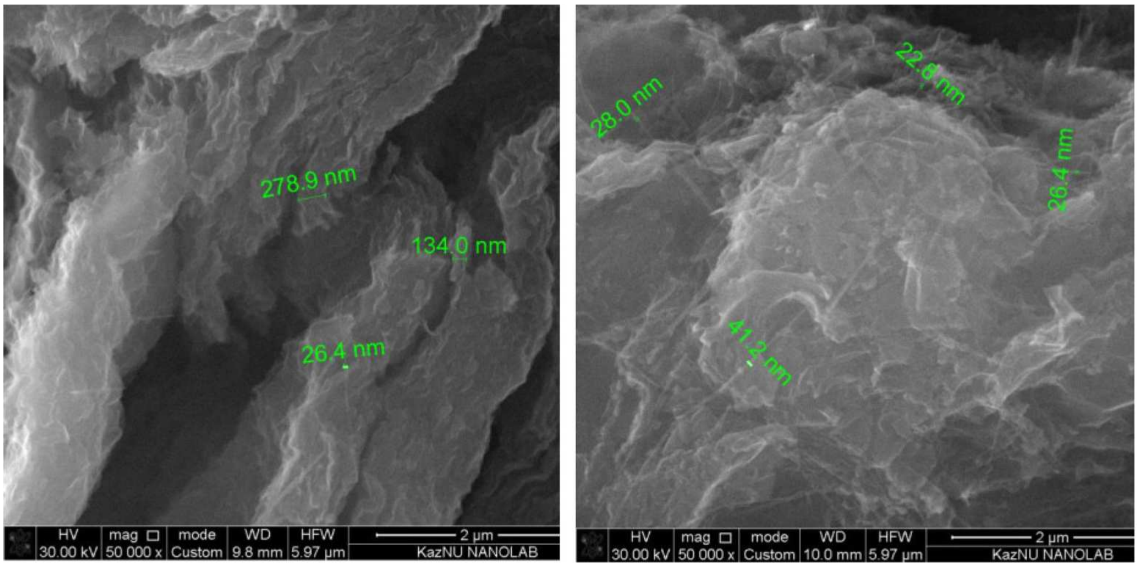


Fig. 5. Deposition rind formed by layered graphite-like structures (SEM)

deposit into blocks is important for its use in high-voltage blocks. This allows one to prepare deposits of the required length without mechanical influence and disruption of its integrity.

According to the results of scanning electron microscopy (SEM) obtained on an electron microscope JEM-100CX, it was found that the shell (rind) of deposits formed by the evaporation of graphite electrodes of SIGE and FGDG-7 brands, consists from layered structures located perpendicularly to the deposit axis and firmly enclosed in a single rigid construction. The results of such studies are shown in Fig. 5.

The deposit core is rigid and monolithic. It consists of graphene sheets rolled into nanotubes (Fig. 3, *e, f*). When the graphite electrode of the SIGE brand evaporates, a core is formed. It has the structure of the same density as in the deposit obtained from the FGDG-7 graphite brand.

However, the studies of SIGE graphite deposits have shown that their cores are very poorly separated from a deposit shell by mechanical means and cannot exist as individual synthesis products (rods) without mechanical intervention.

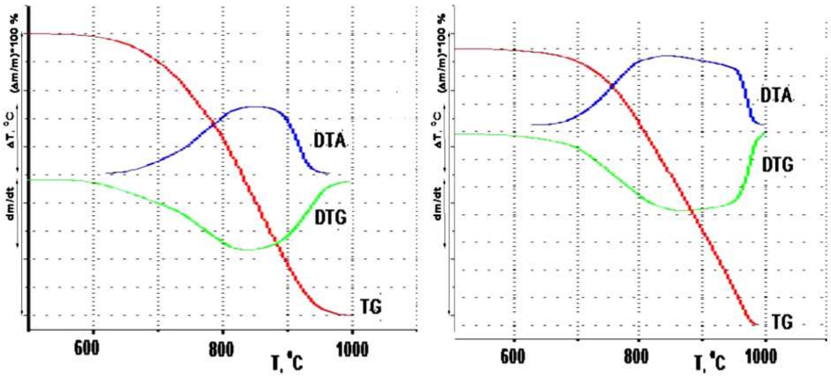
Thus, in the case of electric arc synthesis in a gaseous environment from graphite of the SIGE brand, it is possible to create centimeter rods resistant to ultra-high temperatures (>4000 K [48]).

Due to its layered structure, the deposit shell can be used to obtain graphene.

Differential thermal (DTA), thermogravimetric (TG), and differential thermogravimetric (DTG) analyzes on a derivatograph Q-1500D showed that the temperature-sependent interaction of the deposit shell with oxygen of air starts at 575 °C (Table 5, item 1), which does not exceed the interaction temperature of the deposit core (Table 5, item 2) (Fig. 6, *a, b*).

The DTA curve (Fig. 6, *b*) indicates two phases of the deposit core obtained by the evaporation of the SIGE brand electrode. This evidences that, in addition to the graphite-like component, the core contained the multi-walled carbon nanotubes, the growth of which was initiated at a low electric arc power (3.98 kW).

In the process of electric arc synthesis, the CNS is formed on the surface of the deposit of graphite of SIGE brand (Fig. 3). According to the results of differential thermal (DTA), thermogravimetric (TG), and differential thermogravimetric (DTG) analyses, it was found that the temperature of the beginning of the interaction of the formed CNS with oxygen is 910 °C (Table 5, item 4). This indicates that the deposit surface contains single-walled carbon nanotubes (SCNTs). The data are given in Table 5, item 4.



a *b*

Fig. 6. Heat resistance of the deposit obtained by the evaporation of the electrode of SIEGE brand: the shell of the deposit (*a*); the ore of the deposit (*b*)

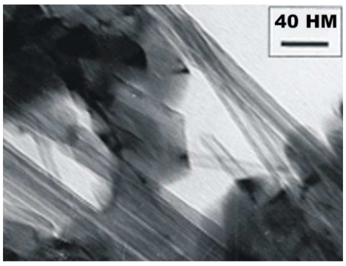


Fig. 7. Multi-wall carbon nanotubes that are components of the core of a deposit obtained by the evaporation of the graphite anode electrode of SIGE brand (TEM)

Table 5. **Heat resistance of the deposit parts after the evaporation by electric arc synthesis in a gaseous environment of a graphite electrode of SIGE brand**

S/n No.	Material	Interaction interval, °C	DTG	DTA	
			$T_{1\max}$, °C	$T_{2\max}$, °C	$T_{2\max}$, °C
1	Deposit shell	575–980	840	840	–
2	Deposit core	575–990	870	850	960
3	Wall soot	575–990	800	800	910
4	CNT	575–650	650	550	650

Thus, in the process of electric arc synthesis of carbon nanostructures, the carbon nanotubes are formed, which have a positive charge and, under the action of an electromagnetic field, are deposited on the surface of a non-consumable cathode in the form of a deposit (Fig. 3).

By the method of transmission electron microscopy (TEM), it is found that the core of the deposit obtained by the evaporation of the graphite anode electrode of SIGE brand, consists of multi-walled carbon nanotubes (MCNTs) (Fig. 7).

Thus, in the process of electric arc evaporation of the SIGE brand electrode, the non-catalytic carbon nanotubes (CNTs) are formed. Obtaining CNTs without the use of a catalyst can reduce their cost.

Wall soot. The condensate mixture of the graphite electrode vapor of the SIGE brand is produced in the gas phase, forming a near-wall soot. It contains both soluble and insoluble components of the fraction. The insoluble fraction of wall soot includes CNTs and similar structures.

The structure of the wall soot layer at the microlevel was determined by scanning electron microscopy (Fig. 8). Layers of wall soot were previously sonicated in alcohol. Carbon nanostructures that are a part of the wall soot have different geometric shapes and structures.

Thermal analysis of near-wall soot synthesized from SIGE graphite showed that there are two phases that react with oxygen at temperatures of 800 °C and 910 °C (Table 4, item 3), as well as in wall soot obtained at the evaporation of the graphite electrode of graphite brand FGDG-7.

Wall soot also contains a soluble component fraction in liquid hydrocarbons. The soluble fraction contains fullerene-like products, hydrocarbon clusters, fuel oils, and other components.

An express analysis of the quantitative content of the solution component of soot was carried out and

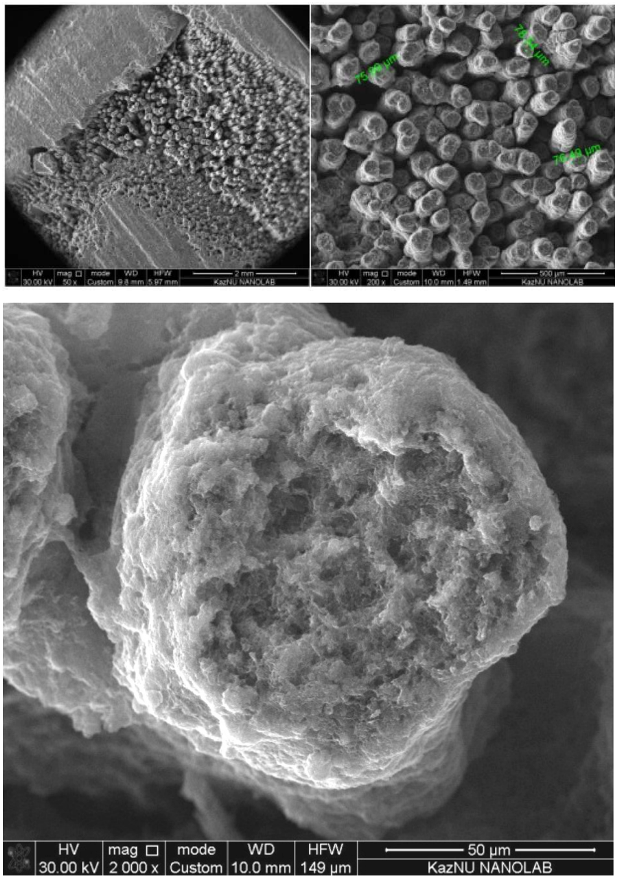


Fig. 8. Surface carbon deposits obtained with the SIGE graphite electrode and sonicated in bromine-containing ethyl alcohol (SEM)

did not confirm the presence of C_{60} and C_{70} fullerene fractions (Fig. 9, *a*) in the first three samples (Table 3, items 1, 2, and 3). But, in the last two samples (Table 3, items 4 and 5), a change in the color decoration of a solution of purified toluene (Fig. 9, *b*), confirming the presence of a mixture of C_{60}/C_{70} fullerenes, was recorded. The change in the color of the solvent is an express indicator testifying to the process of extraction of C_{60}/C_{70} fullerenes from wall soot [49–51].

Spectrophotometric analysis on a spectrophotometer SF-2000. The soluble constituent fraction of wall soot can be determined spectrophotometrically. This method allows one not only to identify fullerenes C_{60} and C_{70}, but also to show the presence of higher and lower fullerenes in soot. The obtained peaks on the wavelength spectra (λ, nm) correspond to the absorbing solutions of C_{60} and C_{70} fullerenes [55, Table 1],

Fig. 9. Express analysis of the content of the soluble component of soot fractions C_{60} and C_{70}: does not confirm the presence of fullerenes C_{60} and C_{70} in the first three samples (Table 3, items 1, 2, and 3) (*a*); confirms the presence of fullerenes C_{60} and C_{70} in the last two samples (Table 3, items 4 and 5) (*b*)

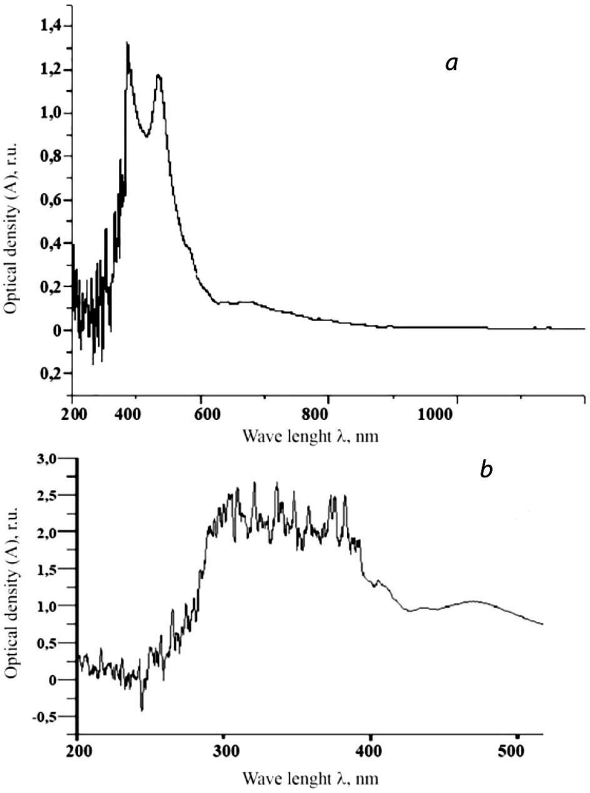


Fig. 10. Optical density UV-Vis spectroscopy, which detects the presence of C_{60} and C_{70} in solutions of wall soot of two samples (Table 3, items 4 and 5): the spectrum of a solution of fullerene-containing soot of the 4th sample (Table 3, item 4) (*a*); the spectrum of a solution of fullerene-containing soot of the 5th sample (Table 3, item 5) (*b*)

which confirmed the presence of fullerenes C_{60} and C_{70} in fullerene-containing soot (Table 3, items 4 and 5), obtained by different modes of the electric arc synthesis in the process of evaporation of the graphite electrode of SIGE brand (Fig. 10) [50, 51].

Calculations based on photospectral analysis data showed that the fullerene component in the obtained two samples of soot (Table 3, items 4 and 5) from graphite of SIGE brand, contains from 10% to 12% of fullerenes. This content exceeds the concentration of the fullerene component in soot obtained by the electric arc evaporation of the graphite electrode of FGDG-7 grade.

In addition, the calculations according to photospectral analysis data confirmed the presence of fullerenes C_{60} and C_{70} in a ratio of 74 : 25% in 2 samples of wall soot extract (Table 3, items 4 abd 5), obtained from graphite of SIGE brand.

4. Conclusions

• Studies have shown that graphite electrodes of SIGE brand are suitable for the synthesis of carbon nanomaterials by the electric arc method in a gaseous environment.

• It has been experimentally proven that SIGE graphite in the electric arc synthesis in a gaseous environment allows one to create centimeter composite rods (deposits), whose cores consist of graphene sheets rolled into nanotubes that can withstand ultrahigh temperatures (>4000 K). This product is very important for creating the high-voltage equipment.

• Scanning microscopy studies have shown that the deposit synthesized from SIGE graphite can be divided into blocks, which is of importance for its use in high-voltage stations. Indeed, it is possible to prepare deposits of a required length without mechanical impact and without contravention of the integrity of their construction.

• Transmission electron microscopy studies have shown that non-catalytic carbon nanotubes (CNTs) are formed during the electric arc evaporation of the SIGE graphite electrode. Obtaining CNTs without the use of a catalyst can reduce their cost.

• It is shown that the overall magnification in the consumable anode electrode allows one to increase the quantitative output of fullerene-containing soot.

• Experimental studies have confirmed that the output by weight of near-wall fullerene-containing soot by the evaporation of SIGE graphite significantly exceeds the results obtained by the evaporation of FGDG-7 graphite electrodes. These results make SIGE graphite more productive for the synthesis of expensive carbon nanoproducts (fullerenes and fullerene-like structures) by the electric arc method.

• It is determined that, in the process of synthesis of carbon nanostructures, the single-walled carbon nanotubes are formed. They have a positive charge and are deposited in the form of a core on the cathode electrode surface under the action of an electromagnetic field.

• Today, the single-walled carbon nanotubes (SCNTs) are the most expensive type of carbon nanotubes, and the non-catalytic synthesis of SCNTs significantly reduces their cost.

• Calculations and experimental results show that the fullerene component in the two obtained samples of fullerene-containing soot from graphite of SIGE brand is from 10 to 12 wt.%, which is not worse and sometimes better than for FGDG-7 graphite.

We also believe that the qualitative and quantitative analyses of the content of fullerenes in soot from SIGE graphite will better help in the future, when creating the industrial production of fullerenes.

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ПОРІВНЯЛЬНИЙ АНАЛІЗ ПРОДУКТІВ ЕЛЕКТРОДУГОВОГО СИНТЕЗУ ПРИ ВИКОРИСТАННІ ГРАФІТУ РІЗНИХ МАРОК

Методом електродугового випаровування графіту марок ЕГСП та МПГ-7 в інертному газі (He) отримано вуглецеві наноструктури (ВНС) різних типів (вуглецеві нанотрубки, фулерени та фулереноподібні структури). Проведено порівняльний аналіз синтезованих характеристик ВНС. Визначено оптимальні технологічні умови для синтезу ВНС з графітових анодних електродів нюваних марок (ЕГСП та МПГ-7). Досліджено метод плазмохімічного синтезу. За допомогою скануючої та просвічуючої електронної мікроскопії досліджено структуру синтезованих вуглецевих матеріалів і показано, що вуглецеві нанотрубки утворюються при випаровуванні графіту марки ЕГСП навіть без використання каталізатора. Проведено диференційно-термічний, термогравіметричний та диференційно-термогравіметричний аналізи, за результатами яких встановлено температури початку взаємодії утвореної ВНС з киснем повітря. За даними фотоспектрального аналізу продуктів синтезу проведено розрахунки і показано, що фулереновий компонент, отриманий випаровуванням графіту марки ЕГСП, містить 10–12% фулеренів C_{60} і C_{70}, що не поступається аналогічним показникам графіту марки МПГ-7. Беручи до уваги дешевину графіту марки ЕГСП порівняно з маркою графіту марки МПГ-7, можна стверджувати, що вуглецеві наноструктури, синтезовані з графіту марки ЕГСП, мають меншу вартість. Цей факт є важливим для синтезу вуглецевих наноструктур як наповнювача для сучасних композитів. Крім того, синтез значно дешевших фулеренів і фулереноподібних молекул є великою перевагою для їх вивчення та використання в сучасних матеріалах, адже сьогодні починають створюватися нові передові нанотехнології на основі фулеренів.

Ключові слова: нанотехнологія, вуглецеві наноструктури (ВНС), фулерени, нанокомпозити, вуглецеві нанотрубки (ВНТ), графени, плазма, електродуговий синтез, графіт марки МПГ-7, графіт марки ЕГСП.