# SYNTHESIS OF CARBON NANOSTRUCTURES USING CHEAP GRADES OF GRAPHITE

**Ol.D. Zolotarenko[1,2], E.P. Rudakova[1,2], N.Y. Akhanova[3,4], M. Ualkhanova[4],**
**An.D. Zolotarenko[1,2], D.V. Shchur[2], M.T. Gabdullin[3], N.A. Gavrylyuk[1],**
**T.V. Myronenko[2], A.D. Zolotarenko[2], M.V. Chymbai[1,2], I.V. Zagorulko[5],**
**Yu.O. Tarasenko[1], O.O. Havryliuk[1]**

[1] *Chuiko Institute of Surface Chemistry, of the N.A.S. of Ukraine, 17General Naumov Str., 03164, Kyiv, Ukraine, a.d.zolotarenko@gmail.com*
[2] *Frantsevich Institute for Problems of Materials Science of N.A.S. of Ukraine, 3, Krzhizhanovskogo Str., 03142,Kyiv, Ukraine*
[3] *Kazakhstan-British Technical University, 59, Tole bi Str., 050000, Almaty, Kazakhstan*
[4] *National Nanotechnology Open Laboratory (NNOL), Al-Farabi Kazakh National University, Al-Farabi, 71, 050040, Almaty, Kazakhstan*
[5] *G.V. Kurdyumov Institute for Metal Physics of the N.A.S. of Ukraine, 36 Academician Vernadsky Blvd., 03142, Kyiv, Ukraine*

*In the work, carbon nanostructures (CNS) were synthesized on a plasma chemical plant using graphite electrodes SIGE (Special Impregnated Graphite Electrodes) and FGDG-7 (Fine-grained dense graphite) in a helium environment. In the experiments, it was established that graphite electrodes of the SIGE brand are suitable for the synthesis of CNS by the electric arc plasma chemical method. In addition, the experiments indicate that SIGE graphite in electric arc synthesis in a gas environment allows the creation of centimeter composite rods (deposits), where the core consists of graphene sheets rolled into nanotubes that can withstand extremely high temperatures (>4000 K). Studies using scanning microscopy have shown that the synthetic deposit of SIGE graphite can be divided into blocks, which is important for its use in high voltage stations because it is possible to prepare deposits of the required length without mechanical impact and without violating the integrity of its structure.*

*The structure of the synthesized carbon materials was studied by scanning and transmission electron microscopy and it was shown that carbon nanotubes are formed during the evaporation of SIGE brand graphite even without the use of a catalyst. Experiments have confirmed that the mass yield of wall fullerene-containing carbon black during the evaporation of SIGE grade graphite significantly exceeds the results obtained during the evaporation of FGDG-7 grade graphite electrodes. Such results make SIGE graphite more productive for the synthesis of expensive carbon nanoproducts (fullerenes and fullerene-like structures) by the electric arc method.*

*It was also recorded that during the synthesis of carbon nanostructures, single-walled carbon nanotubes are formed, which have a positive charge and are deposited in the form of a core on the surface of the cathode electrode under the action of an electromagnetic field.*

***Keywords:*** *nanotechnology, carbon nanostructures (CNS), fullerenes, nanocomposites, carbon nanotubes (CNT), graphene, deposit, plasma, electric arc synthesis, graphite grade «Special Impregnated Graphite Electrodes» (SIGE), graphite grade «Fine-grained dense graphite» (FGDG-7).*

## 1. Introduction

The method of electric arc evaporation of graphite in an inert gas environment is relatively common, productive and quite effective, as allows to obtain both soluble [1 – 11] and insoluble carbon nanostructures (CNS) [12 – 15]. In 1996, the authors of [16] received the Nobel Prize for the discovery of fullerenes, and in 2016 the work on the study of soluble carbon nanostructures (fullerenes) [17] was nominated for the Nobel Prize.

There are other no less perspective methods for the synthesis of carbon nanostructures [18 – 26], from which it is also possible to create new modern materials [27 – 32]. But only the electric arc evaporation of the anode can guarantee the synthesis of fullerene molecules in large quantities. The method allows you to easily change the modes of synthesis, use a gaseous medium of different chemical composition, and most importantly - to achieve a high percentage of carbon nanomaterials (CNM) of different chemical composition, structure and morphology.

Such materials can be used to hydrogen store [17, 33 – 36] and compete with existing materials for hydrogen storages [37 – 65]. The results of such research allow to create modern technological masterpieces [26, 28, 29, 30, 45, 43, 53, 57, 66 – 69].

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 production, 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 the FGDG -7 graphite brand.

However, for today, the carbon nanoproducts (CNPs)of electric arc synthesis in a gaseous environment of SIGE graphite brand remains unexplored. Therefore, to use graphite of this brand in nanotechnology, it is necessary to study and analyze the possibility of carbon nanomaterials synthesis 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-7graphite grades.

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

## 2. Research methods and equipment

In the process of electric arc synthesis in a gaseous environment, it is possible to evaporate or spray the anode electrode, which allows you to change the spectrum of synthesized products.

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

Electrode spraying is a mode of electric arc synthesis, where the anode component does not turn into an 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 this electric arc synthesis mode indicates to the process of forming 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 consists of a vertical cylindrical reactor (Fig. 1), in the center of which is a collet (Fig. 1b (3)), which holds the consumable anode electrode (Fig. 1b (9)). At the above is a collet (Fig. 1b (2)), which holds a non-consumable cathode electrode (Fig. 1b (7)). In the process of electric arc synthesis between the electrodes, plasma is generated (Fig. 1b (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. 1b (4)).
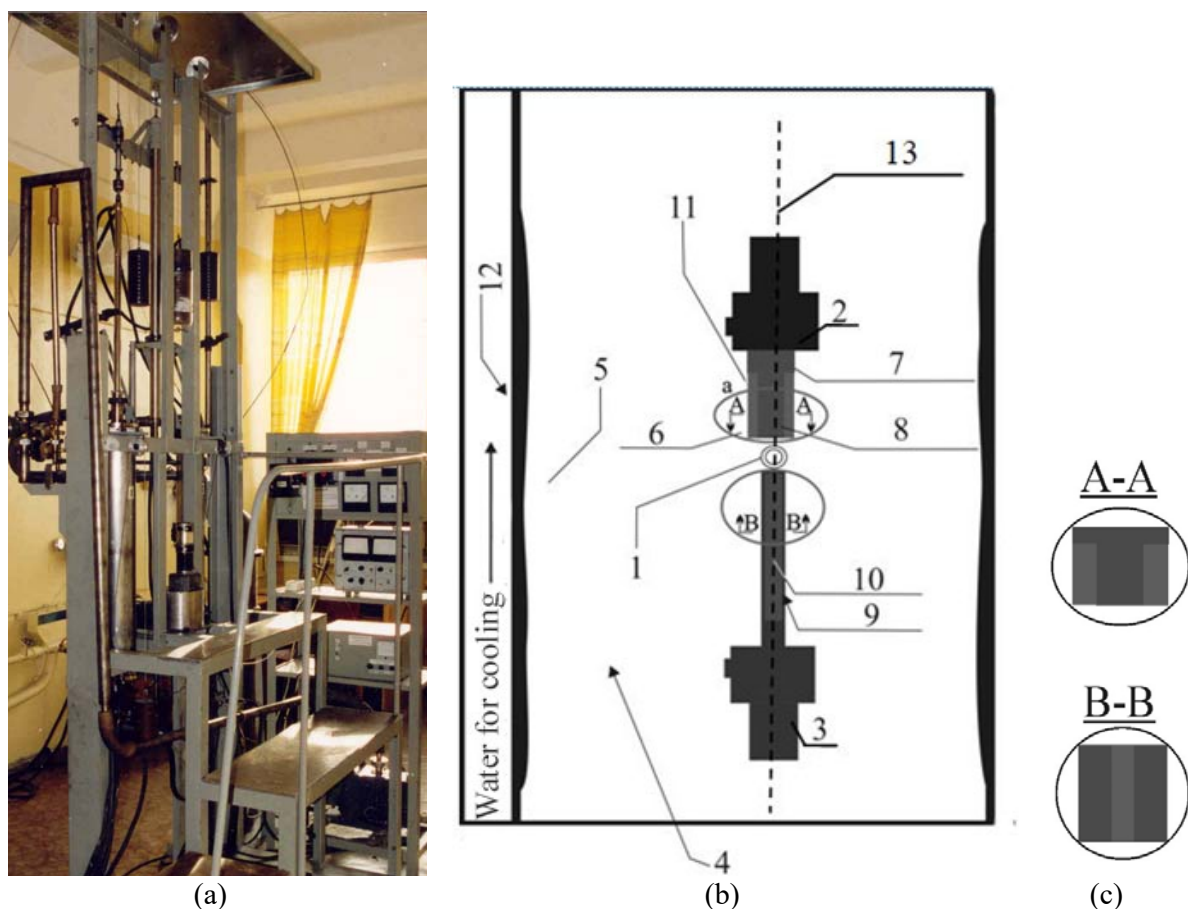


**Fig. 1.** The back view of the electric arc plasma-chemical installation (a), and scheme of the cylindrical reactor for the CNS synthesis in the gas environment (b): 1 – plasma; 2 – collet, which holds a non-consumable cathode electrode; 3 – collet for fixing the consumable anode electrode; 4 – helium 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. (c) – schematic representation of rind and core of the approved deposit

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 for research.

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

An empty graphite anode located along the reactor axis (Fig. 1b (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 fixing it in the anode cavity with pressed graphite dust (Fig. 1b (10)).

Anodes of different geometric sizes were used (0,9×0,9×48 cm; 1,5×1,5×48 cm; 2×2×48 cm; 2,5×2,5×48 cm).

In this work, the study of morphology and structure at the micro- and nanoscale materials formed by electric arc evaporation in a gaseous environment of graphite grades SIGE and FGDG-7, and studied the effect of vapors of chemical impurities in graphite (Si, Fe, Al, Mg, Cu, B, Mn) for the formation of carbon nanostructures, were carried out.

Comparison of the content of some elements - impurities in the graphite grades SIGE (produced by "Ukrgrafit") and FGDG-7 are given in table 1. Comparisons of other characteristics of graphite grades SIGE and FGDG-7 are given in table. 2.

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

| Impurities, % | Graphite grade SIGE* | Graphite grade FGDG-7 |
|---|---|---|
| Silicon (Si) | $4,6 \times 10^{-3}$ | – |
| Iron (Fe) | $9,0 \times 10^{-2}$ | $1 \times 10^{-3}$ |
| Aluminum (Al) | $6,4 \times 10^{-4}$ | $1 \times 10^{-3}$ |
| Magnesium (Mg) | $4,1 \times 10^{-3}$ | – |
| Copper (Cu) | $0,14 \times 10^{-4}$ | $1 \times 10^{-4}$ |
| Boron (B) | $0,31 \times 10^{-4}$ | $3 \times 10^{-4}$ |
| Manganese (Mn) | $0,14 \times 10^{-4}$ | $5 \times 10^{-5}$ |
| Calcium (Ca) | $2,4 \times 10^{-2}$ | – |
| Titanium (Ti) | $4,8 \times 10^{-4}$ | – |
| Sodium (Na) | $3,1 \times 10^{-4}$ | – |
| The total content of impurities, % | 0,3 | – |

* the content of only some of the impurities is given.

**Table 2**. CharacteristicsofgraphitegradesFGDG-7 and SIGE

| Characteristic | Graphite grade SIGE | Graphite grade FGDG-7 |
|---|---|---|
| Density, g/cm$^3$, not less | 1,55 | 1,72 |
| Compressive strength, MPa, not less | – | 65 |
| Bending strength, MPa, not less | 6,5 | 34 |
| Specific resistance, µOhm·m, not more | 11 | 18 |
| Ash content,%, not more | 0,2 | 0,25 |

**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 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, overall dimensions. In addition, increasing the cross-sectional area of the consumable electrodes allowed to increase the quantitative output of fullerene-containing ash in the one synthesis cycle.

The duration of arc synthesis of CNS from graphite of SIGE brand was not more than 3 hours with a small overall size of the electrode (0.9×0.9×48 cm), while the evaporation of the electrode from graphite brand FGDG-7 of the same size lasted in twice as long (6 hours) (Table 3). Also, the evaporation of the graphite electrode of SIGE brand was accompanied by fluctuations in electric current from 120 to 180 A at a voltage of 14 - 42 V, and evaporation of the graphite electrode of FGDG-7 brand is accompanied by an electric current of 185 - 200 A at a voltage of 30 - 33 V (Table 3).In the process of evaporation of SIGE graphite electrodes in the size of 0.9×0.9×48 cm was calculated that the required power of the electric arc, the average value of which is 3.98 kW, which is twice less than 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 the gaseous environment on the walls of the reactor (Fig. 1b (12)) wall soot is formed (Fig. 1b (5)), and on the cathode electrode the deposit is synthesized (Fig. 1b (6)), which is a by-product of synthesis.

The wall soot is the main product of electric arc synthesis. It contains soluble and insoluble carbon nanostructures, the nucleation of which occurs in the synthesis zone. Soluble carbon nanostructures contained in the ash, 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. 1b (6)). It was found that the deposit consists of the core and rind of the deposit. Physicochemical studies of the deposit morphology indicate that the structure of the core and rind of the deposit depends on the catalyst present in the electric arc synthesis zone.

The deposit core is a component of the deposit formation (Fig. 1b (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 it is easily separated from the rind.

The deposit rind is a component of the deposit formation and is its outer layer (Fig. 1b (11)). The deposit rind has its own structure and consists of graphene, graphene packaging and CNTs, which in 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 (0.9×0.9×48 cm), the mass of the deposit (14 – 16 g) exceeds the mass of fullerene-containing wall soot (6 – 10 g). Therefore, a mode evaporation of graphite electrode of SIGE brand was developed, in which it is necessary to spend 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 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 CNS by 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 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.

(a)  (b)

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

**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 s/n | Conditions | | | | | | | | | Results | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Cathode length, cm | Anode length, cm | The anode mass before annealing, g | The mass of the anode after annealing, g | Current strength, A | Voltage, V | Vacuum meter, atm | Reactor wall temperature, °C | Synthesis environment | Soot, g | Deposit mass, g | Deposit length, cm | Anode length after synthesis, cm | Mass of anode residue, g | Electrode chips, g | Synthesis time | Power, kW |
| №1 | 7.5 | 40.5 | 53 | 48 | 120 - 140 | 24 - 36 | 0.73 | 25 | | 6 | 15.95 | 7.8 | 21 | 25.9 | 2 | 1:15 | 2.9 - 5 |
| №2 | 7.5 | 40.5 | 52 | 47 | 160 | 14 - 18 | 0.71 | 27 | Helium | 10 | 13.95 | 5.8 | 19 | 23.9 | 3 | 3:15 | 2.2 - 2,9 |
| №3 | 7.5 | 40.5 | 53 | 48 | 180 | 20 | 0.71 | 25 | | 20.8 | 7 | 2.5 | 11 | 15.5 | 5.5 | 3:10 | 3.6 |
| №4 | 7.5 | 40.5 | 50 | 45 | 180 | 23 | 0.72 | 26 | | 21.8 | 7 | 2.5 | 13 | 17.5 | 4.5 | 3:10 | 4.14 |
| №5 | 7.5 | 40.5 | 53 | 48 | 170 | 42 | 0.75 | 25 | | 25 | 8 | 2.5 | 11 | 19.5 | 6.5 | 3:05 | 7.14 |

During the synthesis, when the SIGE graphite anode is evaporates, a deposit constantly formed on the non-consumable cathode electrode (Figs. 3, 4 (a)). It could be formed coaxially (Fig. 3 (a)) or not coaxially (Fig. 3 (b)) relative to the cathode electrode, but it 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 multi-walled carbon

nanotubes (MCNTs). Schematic representation of the deposit growth in section along its axis is shown in Fig. 1(c) in the footnote of cross section A-A, which illustrated the rind and core of the formed deposit.
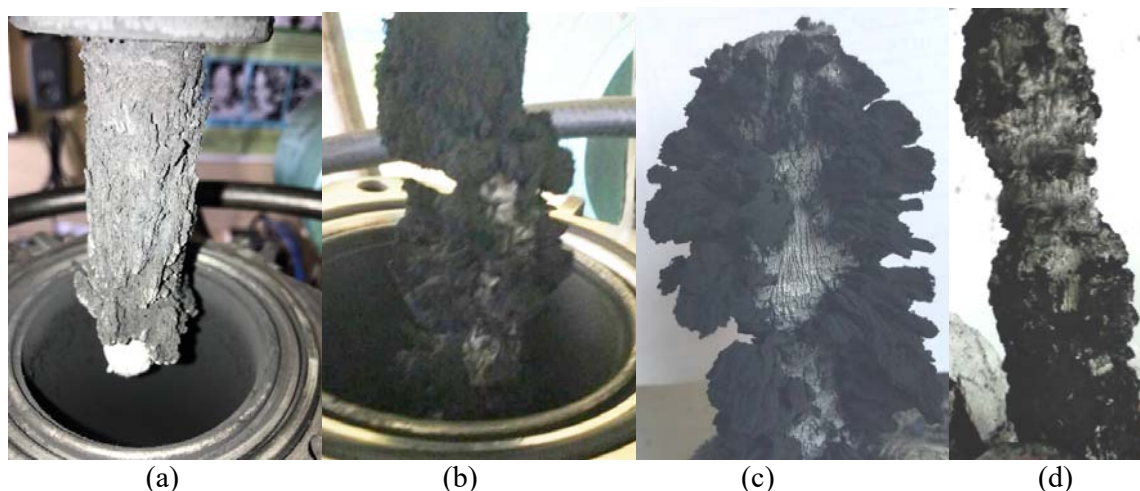


|  | (a) | (b) | (c) | (d) |

**Fig. 3.** Appearance of SIGE brand graphite deposit (growth on the cathode): (a) - coaxial cathode deposit; (b) - misaligned cathode deposit; (c) - with carbon nanostructures on the surface, (d) - the surface of the deposit without carbon nanostructures on the surface

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

|  |  | FGDG-7 graphite | SIGE graphite |
|---|---|---|---|
| Conditions | Anode material | FGDG-7 graphite | SIGE graphite |
|  | 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, ºC | 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 |

In compliance with the specially developed evaporation regime of graphite of SIGE brand, when the maximum amount of fullerene-containing soot synthesizes at an electric arc power of 6.64 kW, and a deposit of 2.5 cm long and 8 g weight is formed. Such characteristics do not exceed the parameters of the deposit obtained by 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 evaporation of

the consumable anode electrode of SIGE brand with an average electric arc power of 3.98 kW (at 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).
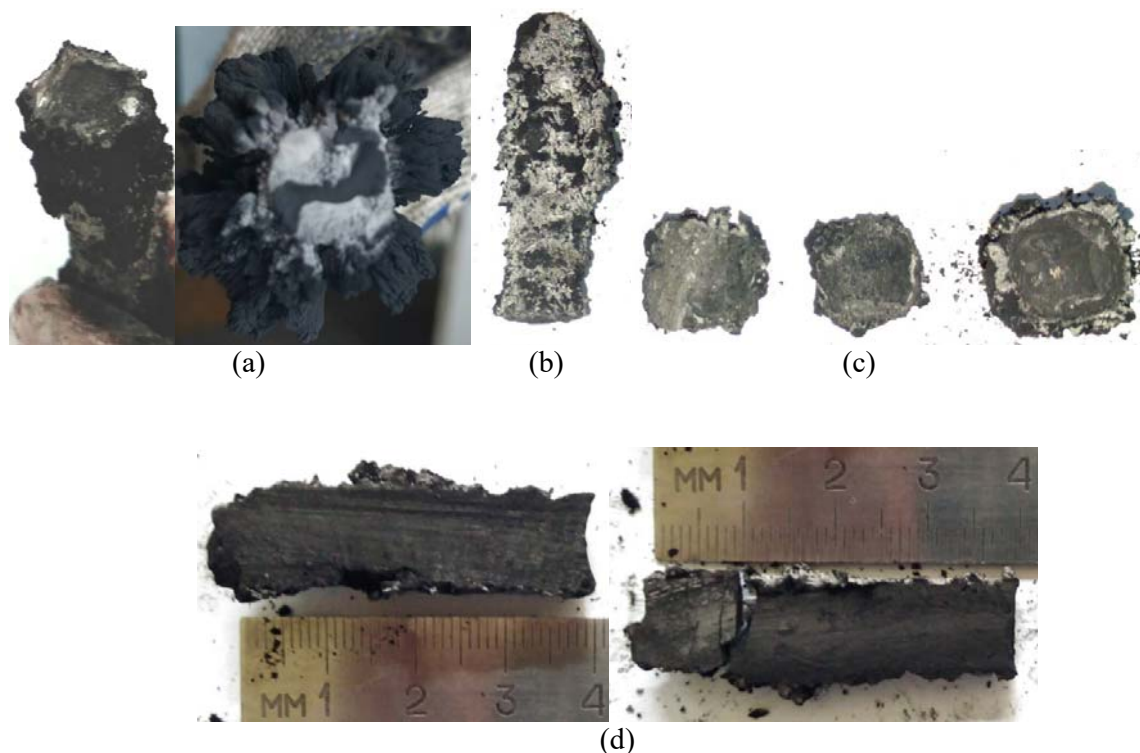


**Fig. 4.** General view of the deposit formed when using SIGE brand graphite: (a) - top view; (b) side view; (c) - cross section of the deposit, (d) - longitudinal section of the deposit (section along its axis)

The general view of the deposit formed at using of graphite of the SIGE brand is presented in fig. 4. The figure shows the form of deposit from above (a), side (b), 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. 4 (c, d)), 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. 4 c, d).

Upon detailed study of the deposit, you can see that in the synthesis it takes the form of an anode. That is, if you look at the deposit from above, the core represented a square insert in the deposit (Fig. 4 (c)). Therefore, if the evaporating anode has a rectangular shape, then the core of the deposit in the synthesis will acquire a rectangular shape (Fig. 4). This is important for the creation of a controlled shape composite (carbon nanotubes and graphene packs) in the form of a deposit out growth that can be resistant to high electrical loads (10 kW) and plasma temperatures (> 4,000 K).

Analysis of the mechanical strength of the synthesized deposits revealed that the deposit consists of hemispherical blocks (Fig. 5), enclosed in a single structure, fastened by a shell (Fig. 4 (d)). Studies have shown that the length of one block of the formed deposit is equal to the step of feeding the electrode at electric arc synthesis.

The inner (upper) part of the deposit block is visually resembles a hemisphere (Fig. 5 (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. Separation of deposit blocks is possible by simple mechanical manipulation.

The lower part of the hemispherical block of the deposit is concave from the anode electrode to the cathode electrode (Fig. 5 (b)). To visually identify the hemispherical state of the deposit block at the bottom of its lower part a red paper indicator wasplaced(Fig. 5b). At the bottom of the formed hemispherical deposit block you can see the formation of a new deposit block in the process of electric arc synthesis from graphite of the SIGE grade (Fig. 5 (b, c)).
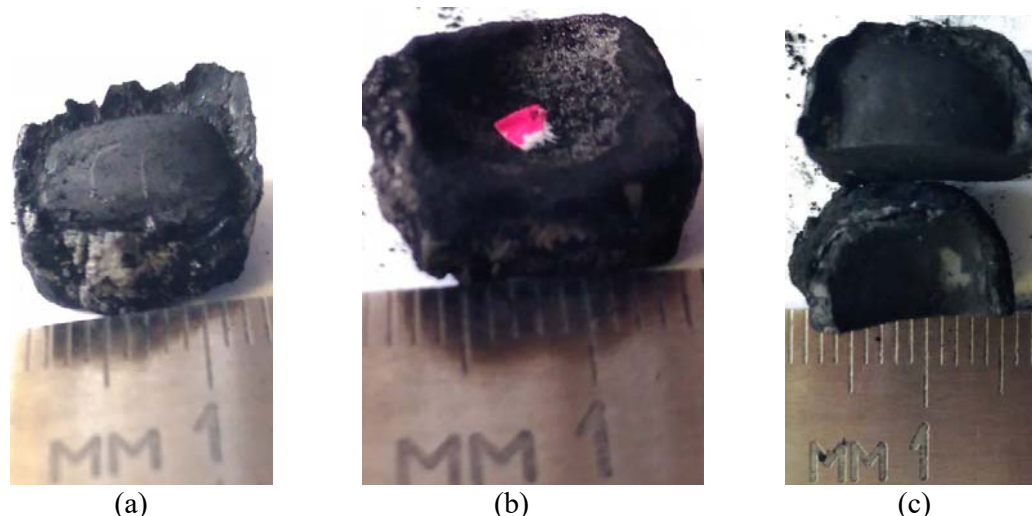


|        (a)        |        (b)        |        (c)        |

**Fig. 5.** Appearance of one of the blocks of SIGE brand graphite deposit: (a) - inner (upper) part of the hemispherical block; (b) - the lower part of the hemispherical block; (c) is a deposit block cut along its axis

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

According to the results of scanning electron microscopy (SEM) analysis, it was found, that the shell (rind) of deposits formed by evaporation of graphite electrodes of SIGE and FGDG-7 brands, consists from layered structures located perpendicular to the deposit axis and firmly enclosed in a single rigid construction. The results of such studies are shown in Fig. 6.

The deposit core is rigid and monolithic. It consists of graphene sheets rolled into nanotubes (Fig. 4 (c, d)). When the graphite electrode of the SIGE brand evaporates, a core is formed. It has the structure of same density as in the deposit obtained from the FGDG-7 graphite brand.

However, studies of SIGE graphite deposits have shown that its core is very poorly separated from the deposit shell by mechanical means and cannot exist as a single synthesis product (rod) 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 [70]).

Due to its layered structure, the deposit shell can be used for graphene obtaining. Differential thermal (DTA), thermogravimetric (TG) and differential thermogravimetric (DTG) analyzes showed that the temperature interaction of the deposit shell with air oxygen starts at 575 ºC (Table 5, item 1) and does not exceed the interaction temperature of the deposit core (Table 5, item 2) (Fig. 9 (a, b)).
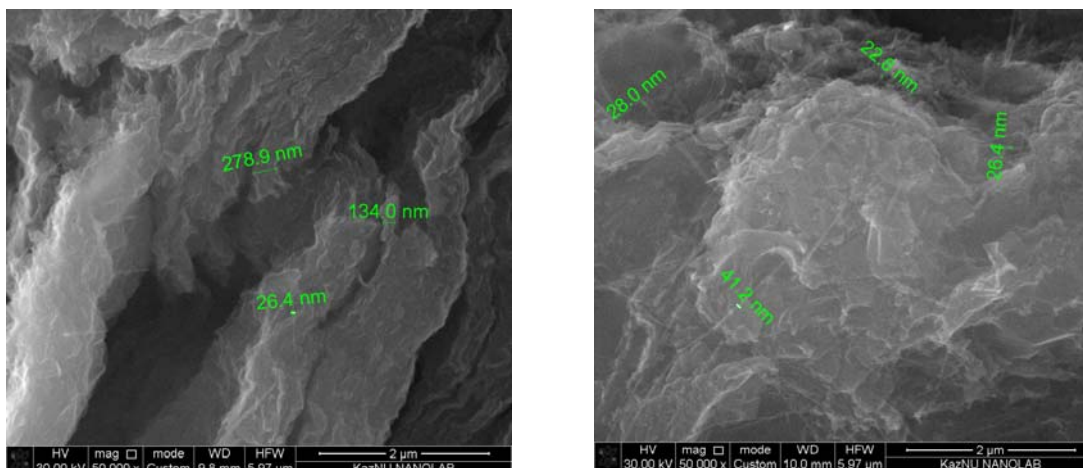
**Fig. 6.** Deposition rind for med by layered graphite-like structures (SEM)

The DTA curve (Fig. 7 (b)) indicates the two-phase of core of the deposit obtained by 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 by 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, 4 (a)). According to the results of differential thermal (DTA), thermogravimetric (TG) and differential thermogravimetric (DTG) analyzes, it was found that the temperature of the beginning of the interaction of the formed CNS with oxygen is 910 ºC (Table 5, item 4). This indicates that the deposit surface contains single-walled carbon nanotubes (SCNT). The data are given in Table. 5, item 4.
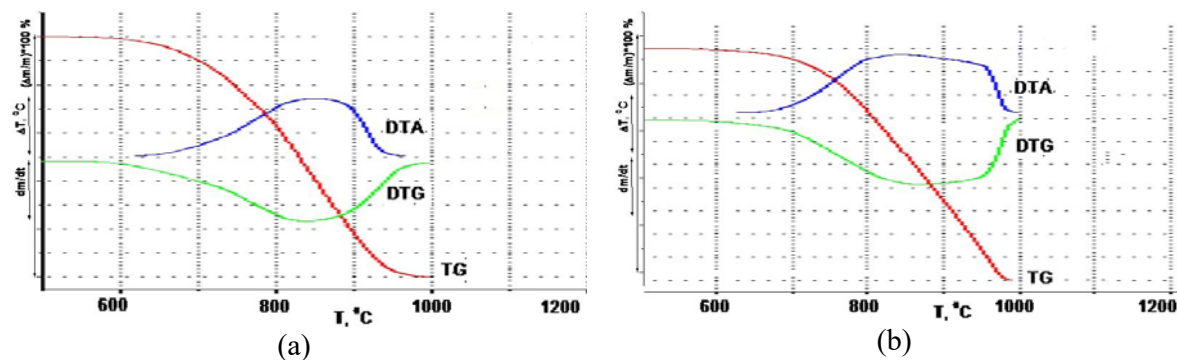


(a)                                                    (b)

**Fig.7.** Heat resistance of the deposit obtained by evaporation of the electrode of SIGE brand: (a) – the shell of the deposit; (b) - the ore of the deposit

That is, 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 the cathode non-consumable electrode in the form of a deposit (Fig. 3, 4 (a)).

By the method of transmission electron microscopy (TEM) it was found that the core of the deposit obtained by evaporation of the graphite anode electrode of SIGE brand, consists of multi-walled carbon nanotubes (MCNT) (Fig. 8).

That is, 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.

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

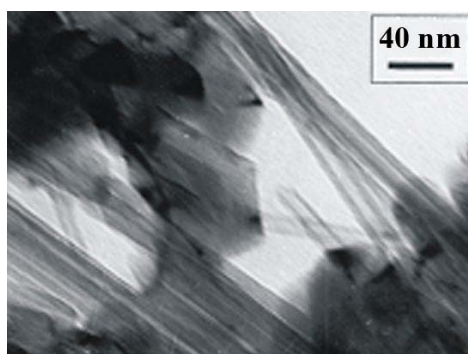| s/n № | Material | Interaction interval, ºC | DTG | DTA | |
|-------|----------|--------------------------|-----|-----|---|
| | | | $T_{1max}$, ºC | $T_{1max}$, ºC | $T_{2max}$, ºC |
| 1 | Deposit shell | 575 – 980 | 840 | 840 | - |
| 2 | Deposit core | 575 – 990 | 870 | 850 | 960 |
| 3 | Wall soot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



**Fig. 8.** Multiwall carbon nanotubes that are components of the core of the deposit obtained by evaporation of the graphite anode electrode of SIGE brand (TEM)
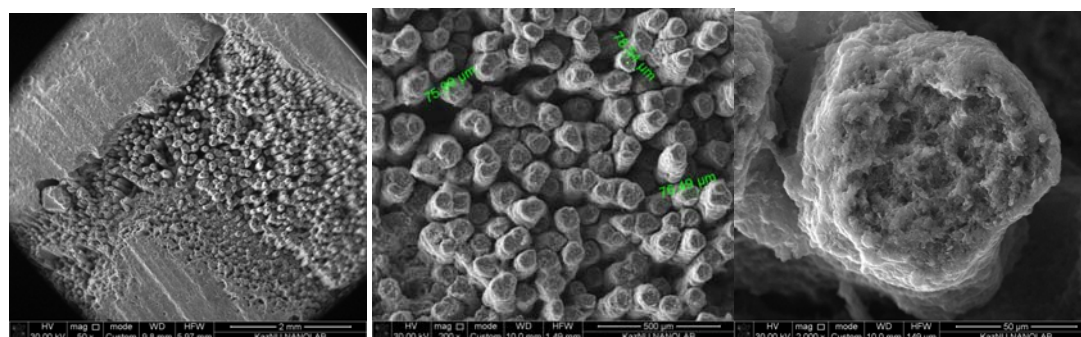


**Fig. 9.** The surface of the wall soot of the graphite electrode of SIGE brand at tersonication in ethyl alcohol (SEM)
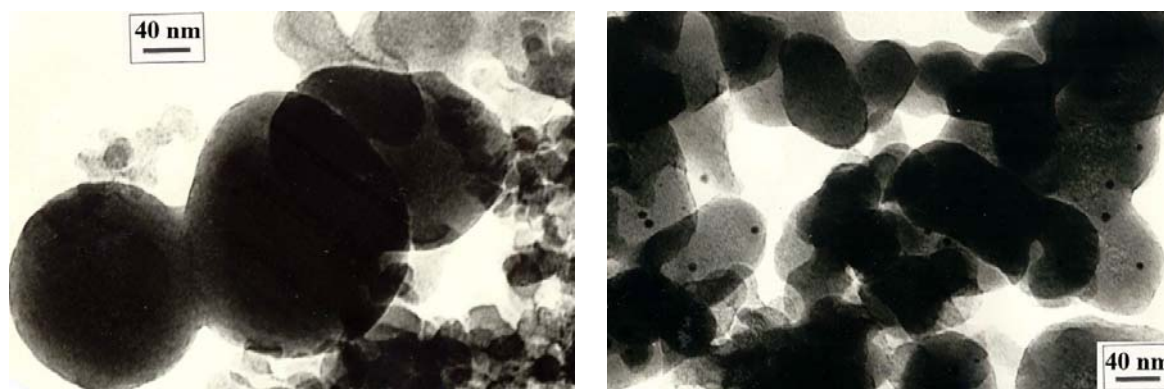


**Fig. 10.** Wall soot, which contains carbon nanosized particles (TEM)

**Fig. 11.** Express analysis of the content of the soluble component of soot fractions $C_{60}$ and $C_{70}$: (a) - does not confirm the presence of fullerenes $C_{60}$ and $C_{70}$ in the first three samples (Table 3, item 1,2,3); (b) - confirms the presence of fullerenes $C_{60}$ and $C_{70}$ in the last two samples (Table 3, item 4,5)

(a)            (b)

The condensate mixture of the graphite electrode vapor of the SIGE brand is formed 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 micro level was determined by scanning electron microscopy (Fig. 9). Layers of wall soot were previously sonicated in alcohol. Carbon nanostructures that are part of the wall soot have different geometric shapes and structures (Fig. 10).

Thermal analysis of near-wall soot synthesized from SIGE graphite showed that there are two phases that react with oxygen at temperatures of 800 ºC and 910 ºC (Table 4, item 3), as well as in wall soot obtained at 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 and hydrocarbon clusters, fuel oils and other components.

Using express analysis, we established that the wall soot obtained from the graphite consumable anode electrode of the FGDG brand (0.9×0.9×48 cm) also contains the soluble component fraction $C_{60}$ and $C_{70}$ (Fig. 11) in the last two samples from five (Table 3, items 4, 5). Therefore, fullerenes and fullerene-like products were not formed in the first three samples of wall soot (Table 3, items 1, 2, 3) due to the low power of the electric arc (3.98 kW). Thus, it is necessary to withstand the electric current power of 6.64 kW for the formation of fullerenes and fullerene-like nanoproducts from SIGE graphite, which does not exceed the synthesis parameters during evaporation of FGDG-7 graphite.

The soluble constituent fraction of wall soot can be determined spectrophotometrically. This method allows not only to identify fullerenes $C_{60}$ and $C_{70}$, but also to show the presence of higher and lower fullerenes in soot. This method of analysis confirmed the presence of fullerenes $C_{60}$ and $C_{70}$ in fullerene-containing soot (Table 3, items 4,5), obtained by different modes of electric arc synthesis in the process of evaporation of the graphite electrode of SIGE brand (Fig. 11-12).

Calculations based on photospectral analysis data showed that the fullerene component in the obtained two samples of soot (Table 3, items 4, 5) from graphite of SIGE brand, contains from 10% to 12% of fullerenes. This content exceeds the concentration of the fullerene component in the soot obtained by electric arc evaporation of the graphite electrode of FGDG-7grade.
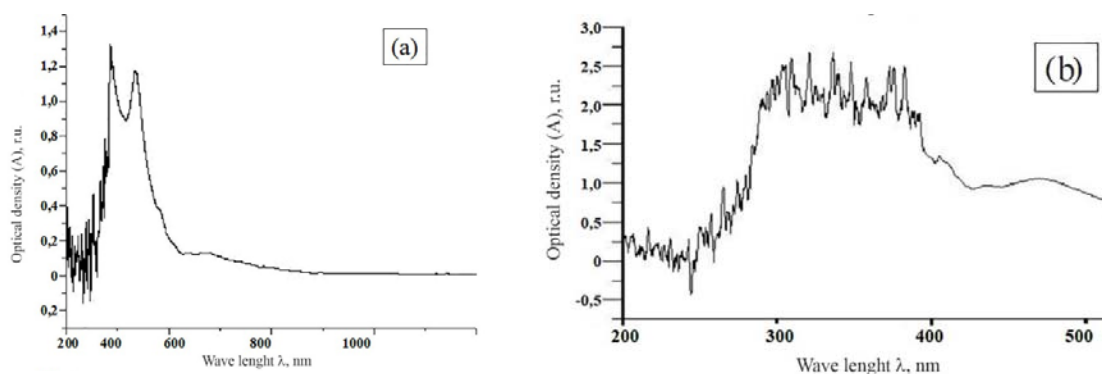
124

**Fig. 12.** Optical density UF-Vis spectroscopy, which detects the presence of $C_{60}$ and $C_{70}$ in solutions of wall soot of two samples (Table 3, items 4,5): (a) - the spectrum of a solution of fullerene-containing soot of the 4th sample (Table 3, item 4); (b) - the spectrum of a solution of fullerene-containing soot of the 5th sample (Table 3, item 5)

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 4,5), 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 electric arc method in a gaseous environment.

It has been experimentally proven that SIGE graphite in electric arc synthesis in gaseous environmental lows to create centimeter composite rods (deposits), the core of which consists of graphene sheets rolled into nanotubes that can withstand ultra-high temperatures (> 4000 K). This product is very important for creating high voltage equipment.

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

Itis shown that the overall magnification in the consumable anode electrode allows 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 evaporation of SIGE graphite significantly exceeds the results obtained by 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 non-catalytic synthesis of SCNTs significantly reduces their cost.

Calculation and experimental results showed 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 FGDG-7 graphite.

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# СИНТЕЗ ВУГЛЕЦЕВИХ НАНОСТРУКТУР ПРИ ВИКОРИСТАННІ ДЕШЕВИХ МАРОК ГРАФІТУ

**Ол.Д. Золотаренко[1,2], О.П. Рудакова[1,2], Н.Е. Аханова[3,4], М. Уалханова[4], Ан.Д. Золотаренко[1,2], Д.В. Щур[2], М.Т. Габдуллин[3], Н.А. Гаврилюк[1], Т.В. Мироненко[2], О.Д. Золотаренко[2], М.В. Чимбай[1,2], І.В. Загорулько[5], Ю.О. Тарасенко[1], О.О. Гаврилюк[1]**

[1]*Інститут хімії поверхні ім. О.О. Чуйка НАН України, вул. Генерала Наумова, 17, 03164 Київ, Україна, a.d.zolotarenko@gmail.com*
[2]*Інститут проблем матеріалознавства ім. І.М. Францевича НАН України, вул. Кржижановського, 3, 03142 Київ, Україна*
[3]*Казахсько-британський технічний університет (КБТУ), Толі бі 59, пр. Аль-Фарабі, 050040 Алмати, Казахстан*
[4]*Національна нанотехнологічна лабораторія відкритого типу (NNLOT), Казахський національний університет ім. Аль-Фарабі, д. 71, пр. Аль-Фарабі, 050040 Алмати, Казахстан*
[5]*Інститут металофізики ім. Г.В. Курдюмова НАН України, бульв. Акад. Вернадського, 36, 03142 Київ, Україна*

У роботі синтезовані вуглецеві наноструктури (ВНС) на плазмохімічній установці при використанні графітових електродів ЕГСП (Спеціально імпрегновані графітові електроди) та МПГ-7 (Дрібно зернистий щільний графіт) в середовищі гелію. В експериментах встановлено, що графітові електроди марки ЕГСП придатні для синтезу ВНС електродуговим плазмохімічним методом. Крім того, експерименти вказують на те, що графіт марки ЕГСП в електродуговому синтезі в газовому середовищі дозволяє створити сантиметрові композитні стрижні (депозити), де серцевина складається з графенових листів, згорнутих в нанотрубки, які витримують надвисокі температури (>4000 К). Дослідження за допомогою скануючої мікроскопії показали, що синтезований депозит з графіту марки ЕГСП може розділятися на блоки, що важливо для його використання в станціях високого напруження тому, що можна готувати депозити необхідної довжини без механічного впливу і не порушуючи цілісність його конструкції.

Дослідження за допомогою просвітлюванної мікроскопії показали, що в процесі електродугового випаровування електрода марки ЕГСП формуються безкаталітичні вуглецеві нанотрубки). Експерименти підтвердили, що вихід по масі пристінної фуллеренвмісної сажі при випаровуванні графіту марки ЕГСП значно перевищує результати, отримані при випаровуванні графітових електродів марки МПГ-7. Такі результати роблять графіт марки ЕГСП більш продуктивним для синтезу дорогих вуглецевих нанопродуктів (фуллеренів та фуллереноподібних структур) електродуговим методом.

Також зафіксовано, що в процесі синтезу вуглецевих наноструктур формуються одностінні вуглецеві нанотрубки, які мають позитивний заряд і під дією електромагнітного поля осаджуються у вигляді серцевини на поверхні катодного електрода.

**Ключові слова:** нанотехнології, вуглецеві наноструктури (ВНС), фуллерени, нанокомпозити, вуглецеві нанотрубки (ВНТ), графен, осадження, плазма, електродуговий синтез, графіт марки (SIGE) «Спеціально імпрегновані графітові електроди», графіт марки (FGDG-7) «Дрібно зернистий щільний графіт».