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PLATINUM-CONTAINING CARBON NANOSTRUCTURES FOR THE CREATION OF ELECTRICALLY CONDUCTIVE CERAMICS USING 3D PRINTING OF CJP TECHNOLOGY

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Carbon nanostructures (CNS) were synthesized by the electric arc plasma chemical method during the evaporation of a high-quality graphite electrode of the brand “fine-grained dense graphite” (FGDG-7) filled with a catalyst (Pt), which was evaporated in a helium environment. In the synthesis process, the following were synthesized: multi-walled (MWCNT) and single-walled carbon nanotubes (SWCNT), fullerenes, graphene packets and nanocomposites. A deposit in the form of growth on the cathode electrode was also synthesized. All synthesis products were analyzed at the micro- and nanolevels, which made it possible to analyze the influence of platinum vapors on the formation of carbon nanomaterials (CNM). The non-uniform distribution of catalyst atoms (platinum) in the products of electrochemical synthesis in a gas medium using FGDG-7 graphite was investigated.

During the analysis, it was found that platinum is in the state of the face-centered cubic (FCC) lattice and is distributed in the synthesis products as follows: the core of the deposit is less than $< 0.001\%$, the shell of the deposit is less than $< 1\%$, the wall soot is more than $> 1\%$. The morphology and composition of the platinum deposit, which has a hexagonal graphite structure with an admixture of a rhombohedral graphite phase, was studied. In the studies, differential thermal analysis in air (TG, DTG, DTA) was carried out, which made it possible to identify the composition of the synthesis products. It is an established fact that the parts of the deposit with platinum are more heat-resistant compared to the deposit components that do not contain Pt. The resulting carbon nanotubes (CNTs) in diameter (5–25 nm) and length (1.5–2 μm) do not differ from those obtained without the participation of platinum, except for some anomalies.

When studying the suitability of platinum-containing carbon nanostructures for 3D printing of CJP (ceramic printing) technology, it was found that for the use of platinum-containing carbon black, it is necessary to carry out a preliminary short-term treatment, namely, grinding in special “ball mills” or rubbing through a fine sieve with minimal effort to create uniformity product. Previous studies have shown that such platinum-containing carbon nanostructures can already be used in 3D printing of CJP technology, or to create new composites for 3D printing technologies of FDM, SLA.

Keywords: carbon nanostructures, platinum (Pt), fullerenes, graphenes, deposit, plasma, electric arc synthesis, plasma chemical synthesis, 3D printing, CJP technology

INTRODUCTION

Today, the main problem of fuel cells using is the need for metals of the platinum group (mainly platinum and its alloys). The main efforts are to replace platinum or limit its quantity, which will

reduce the cost of fuel cells. We propose to create carbon nanostructures (CNSs) containing platinum (Pt) and use them to manufacture of fuel cells. This will make it possible to reduce the content of platinum in fuel cells to a minimum and significantly decrease their cost. The creation

of such a fuel cell by an automated system of CJP 3D printing technology will allow the production of cheap fuel elements for hydrogen energy.

Currently, carbon nanostructures can be used not only in the design of fuel cells, but also to solve the problem of efficient hydrogen storage [1–6]. Such ANS are capable to compete with existing materials for hydrogen storage [7–35]. It is possible that in the future the “fullerite – metal – hydrogen” system will become a modern advanced means of hydrogen storing – the most ecologically clean source of energy.

Today, the synthesis of carbon nanotubes (CNTs) and other CNSs is carried out by various methods: laser evaporation of metal-graphite electrodes; plasma chemical evaporation of graphite in the gas phase [36–37] and liquid environment in the presence of catalysts [38]; catalytic pyrolysis of carbohydrates [39–41]; in the presence of organometallic compounds (ferrocene, phthalocyanines); catalytic dismutation of carbon monoxide and others [39].

The method of plasma chemical vaporization of graphite in the gas phase is relatively widespread and quite effective, because it allows to obtain both soluble [42–52] and insoluble carbon nanomaterials (CNM) [53–56]. In addition, this method allows one to control synthesis modes, use different gas environments, and most importantly, to achieve a high yield of CNM with a given morphology and properties. Today, various CNMs are actively used in the creation of new composites and materials [57–65].

However, to date, the processes of oxidation of CNM deposit – a product that is formed on the cathode in the process of plasma chemical synthesis in the gas phase – remains insufficiently studied. Understanding of these processes would allow us to identify the forms of carbon. As will be shown, both CNTs and graphenes are present in the deposit.

In the work, it is planned not only to create carbon nanomaterials containing Pt, which are suitable for 3D printing, but also to study the morphology and structure of the obtained carbon nanostructures.

EXPERIMENTAL STUDIES

The method of plasma-chemical synthesis in a gas environment provides for the possibility of evaporation or spraying of the anode electrode, which allows to obtain various synthesis products.

Evaporation of the electrode is a mode of plasma-chemical synthesis, during which the anodic component passes into the atomic state, creating the conditions of the CNS synthesis zone. Fullerenes and fullerene-like structures are synthesized precisely during this evaporation of the electrode.

Electrode spraying is a mode of plasma-chemical synthesis in which the anodic component does not pass into the atomic state. As a rule, this process occurs at a high speed of the cathode electrode feeding. Under such conditions, the anodic component does not have time to transition into the atomic state, and the graphite component of the anode is loosened. Our studies of plasma-chemical synthesis products according to the given regime indicate the process of forming of graphene packets modified by the applied catalyst.

For the synthesis of CNS containing Pt atoms, by the plasma chemical method, the evaporation mode of a consumable anode electrode was used. The experimental equipment for the plasma-chemical synthesis consists of a vertical cylindrical reactor (Fig. 1), in the center of which there is a holder (Fig. 1 *b* (3)) of a consumable anode electrode (Fig. 1 *b* (9)), and inside from below – a holder Fig. 1 *b* (2)) of non-consumable cathode electrode (Fig. 1 *b* (7)). In the process of plasma-chemical synthesis, a plasma is formed between the electrodes (Fig. 1 *b* (1)) [66], in which the consumable anode electrode evaporates. The reactor chamber is filled with helium (Fig. 1 *b* (4)).

Two types of electrodes are used in plasma chemical synthesis: a consumable anode electrode (Fig. 1 *b* (9)) and a non-consumable cathode electrode (Fig. 1 *b* (7)). The non-consumable cathode electrode moves along the axis of the reactor (Fig. 1 *b* (13)), and the consumable anode electrode evaporates in the plasma (Fig. 1 *b* (1)) [66], under a pressure of 0.02–0.09 MPa in a helium gas environment (Fig. 1 *b* (4)). Thus, uniform distribution of wall soot (Fig. 1 *b* (5)) on the entire wall of the reactor is ensured and almost the same thermodynamic conditions are achieved for the synthesis of all CNMs and their condensation.

Electrodes made of high-quality fine-grained dense graphite (FGDG-7) were used for the synthesis of CNM. To introduce the catalyst into the zone of plasma chemical synthesis, a hollow graphite anode was used, into which the catalyst

(platinum) was placed in the form of a wire, which was fixed in the anode cavity with pressed graphite dust (Fig. 1 *b* (10)). The cavity of the anode electrode is located along the axis of the reactor (Fig. 1 *b* (12), 2).

On the cold walls of the reactor (Fig. 1 *b* (12)) in the process of plasma chemical synthesis in a gaseous environment, the wall soot (Fig. 1 *b* (5)) which is the main product of the synthesis, is formed, and on the cathode electrode a deposit (Fig. 1 *b* (6)) is synthesized which is a byproduct.

Wall soot contains soluble and insoluble carbon nanostructures, the nucleation of which occurs in the synthesis zone. Soluble carbon nanostructures include fullerenes, endofullerenes, and fullerene-like products. Insoluble carbon nanostructures include carbon nanotubes, graphene and graphene packets.

The deposit (Fig. 1 *b* (6)) is a by-product of synthesis, which is formed on the cathode electrode in the form of an outgrowth, always

along the of the anode electrode axis. As our research shows, the deposit consists of a core and a rind. Physico-chemical studies of the morphology of the deposit indicate that the structure of the core and the rind depends on the catalyst present in the zone of plasma-chemical synthesis.

The core of the deposit (Fig. 1 *b* (8)) is a component of the deposit formation, which has its own structure and consists of CNTs, and is firmly connected to the rind of the deposit. But, as the experiments show, if a catalyst is used in the process of plasma chemical synthesis, the core of the deposit is formed as an independent structural component and is easily separated from the rind.

The rind of the deposit (Fig. 1 *b* (11)) is the outer layer of the deposit formation component. It has its own structure and consists of graphenes, graphene packets and CNTs, and is also strongly connected to the deposit core during catalyst-free synthesis.

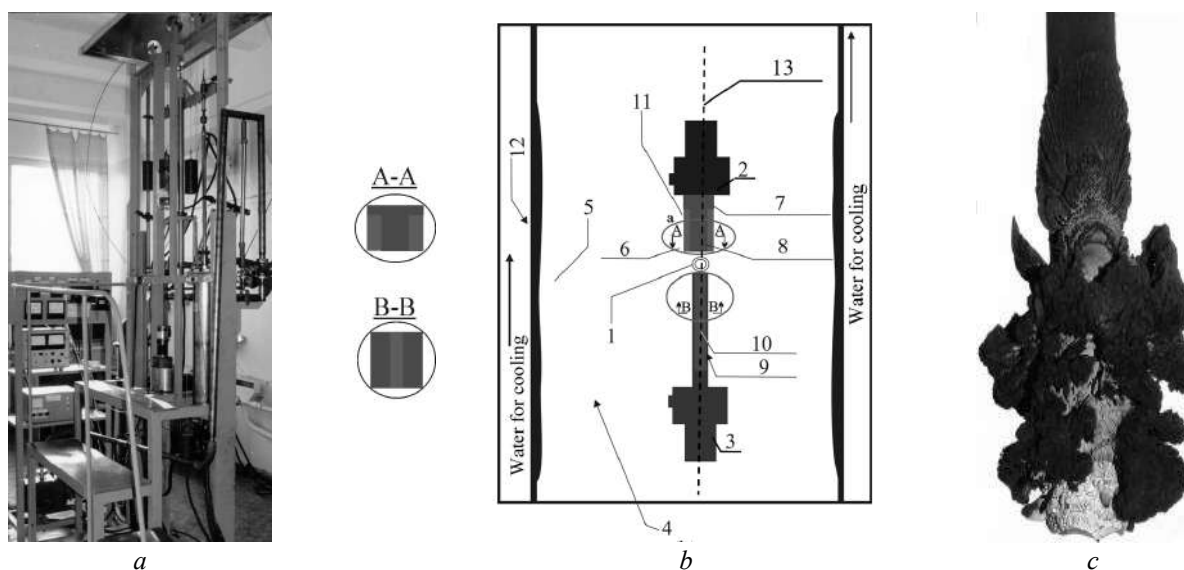


Fig. 1. Plasma-chemical installation of synthesis in a gaseous environment. *a* – scheme of a cylindrical reactor for plasma-chemical synthesis of CNS in a gaseous medium: 1 – plasma; 2 – cathode holder; 3 – anode holder; 4 – helium-containing environment; 5 – platinum-containing wall soot; 6 – deposit; 7 – non-consumable electrode (cathode); 8 – the deposit core; 9 – consumable electrode; 10 – wire catalyst fixed with graphite dust; 11 – the deposit rind; 12 – reactor wall; 13 – axis of the CNS synthesis reactor; *b* – general view; *c* – appearance of Pt-containing deposit on the cathode

During plasma chemical synthesis, the evaporation of the anode made of pure graphite (FGDG-7) and of the anode doped with a platinum catalyst, occurs (Fig. 1 *b* (9), Table 1). The process of plasma chemical synthesis in the presence of a Pt catalyst lasts 40 min longer,

despite the fact that the length of the platinum-doped anode is shorter than that of the graphite anode without the catalyst (18 and 21.1 cm, respectively). Evaporation of a platinum-containing electrode is accompanied by fluctuations in current (175–225 A) and voltage

(35–37 V), while evaporation of a graphite anode occurs at a current of 185–200 A and a voltage of 30–33 V. This can be explained by the higher evaporation temperature of the Pt-containing anode.

After the evaporation process of the platinum-containing electrode, the mass of the synthesis product (deposit and wall soot) is much higher than the similar parameter of the product obtained by evaporation of the graphite anode without a catalyst. It was found that a significant mass of synthesized CNS is located in the wall soot. This allows us to assume that in the process of plasma chemical synthesis, a large number of platinum atoms move to the zone of lower pressures and temperatures, namely, to the periphery of the CNS synthesis zone, until they cool and condense on the reactor wall (Fig. 1 *b* (12)).

The length (5 cm) and mass (44.99 g) of the Pt-containing deposit significantly exceed the parameters of the deposit obtained during evaporation of the graphite electrode (length 2.1 cm; mass 7.5 g) (Table 1). This allows us to assume that in the process of synthesis, platinum atoms reorganize carbon atoms, which allows for the formation of CNS on the cathode electrode in the form of a deposit. Pt-containing deposits are resistant to plasma precursor temperatures, as they are formed under such conditions, and are also characterized by a sufficient level of electrical conductivity due to the fact that an electric arc is formed from the closer core of the deposit to the anode electrode. Such an electric arc creates the necessary conditions for the formation of a deposit coaxial to the anode electrode.

Table 1. Conditions and results of plasma-chemical synthesis

| | C | C – Pt | |
|-------------------|---|-----------------|--------------------|
| Conditions | Composition | Pure graphite | Graphite and Pt |
| | Cathode length | 8.4 cm | 8.4 cm |
| | Anode length | 21.1 cm | 18 cm |
| | Catalyst (length×width) | – | 11×0.1 cm |
| | Current | 185–200 A | 175–225 A |
| | Voltage | 30–33 V | 35–37 V |
| | Vacuum meter | 0.190–0.174 atm | 0.70–0.13–0.33 atm |
| | The temperature of the reactor cooling jacket | 31 °C | 25 °C |
| Results | Synthesis environment | Helium | Helium |
| | Mass of wall soot | 10.400 g | 24.690 g |
| | Mass of the deposit | 7.500 g | 44.990 g |
| | Mass of chips of graphite anodes | 4.700 g | 8.495 g |
| | The remainder of the anode | 6.6 cm | 1.8 cm |
| | Deposit length | 2.1 cm | 5 cm |
| | Synthesis time | 2 h | 2 h 40 min |

Deposit. During the synthesis, when the Pt-containing anode evaporates, an outgrowth called a deposit is formed on the non-consumable electrode (cathode) (Fig. 1 *c*). The deposit consists of a loose core formed by multi-walled carbon nanotubes (MWCNTs) with a minimal number of structural defects (Fig. 2), as well as a strong rind formed by layered graphite-like structures containing a smaller amount of MWCNTs.

When a graphite electrode doped with platinum is evaporated, a much denser core is formed than the core obtained by the non-catalytic method of synthesis. It has a columnar structure oriented parallel to the deposit axis. The synthesized columnar structures have a diameter of 100–150 μm (Fig. 3 *a, b, c*) and, in turn, are formed from conglomerates of bundle-like MWCNTs with a diameter of 4–25 nm (Fig. 4 *c*).

During the plasma-chemical synthesis of Pt-containing CNS, not only empty CNTs are formed, but also tubes with various structural anomalies. They have a more developed outer surface and some inclusions. X-ray microanalysis data indicate a small content of platinum in the

core (up to a hundredth of a percent), and the presence of hexagonal graphite with an admixture of the rhombohedral phase of graphite was recorded in all parts of the deposit (core and shell).

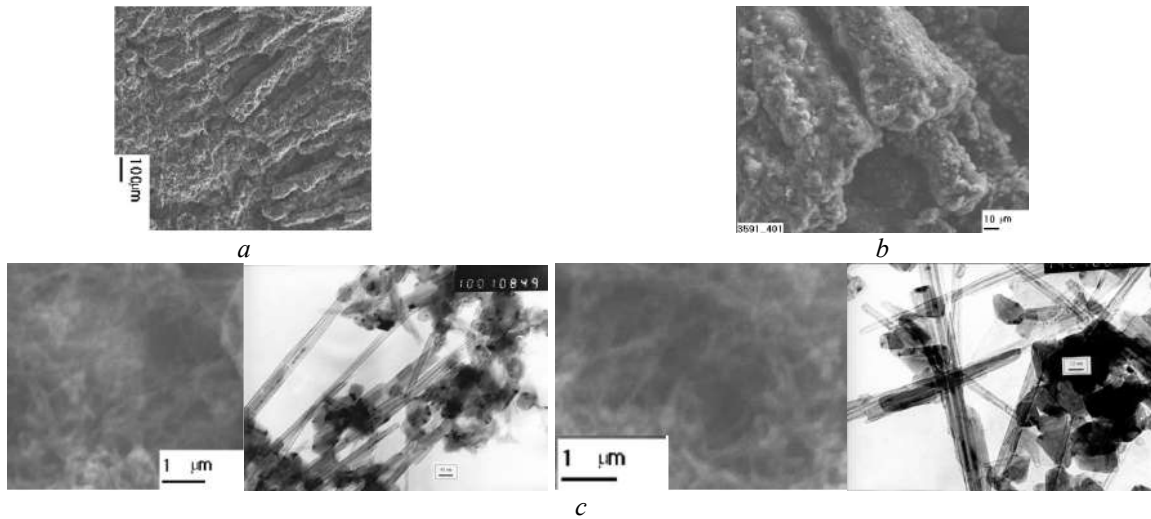


Fig. 2. Loose core of the deposit: *a* – general view of the core conglomerates; *b* – conglomerate of MWCNT beams; *c* – beams MWCNT – the components of the conglomerate

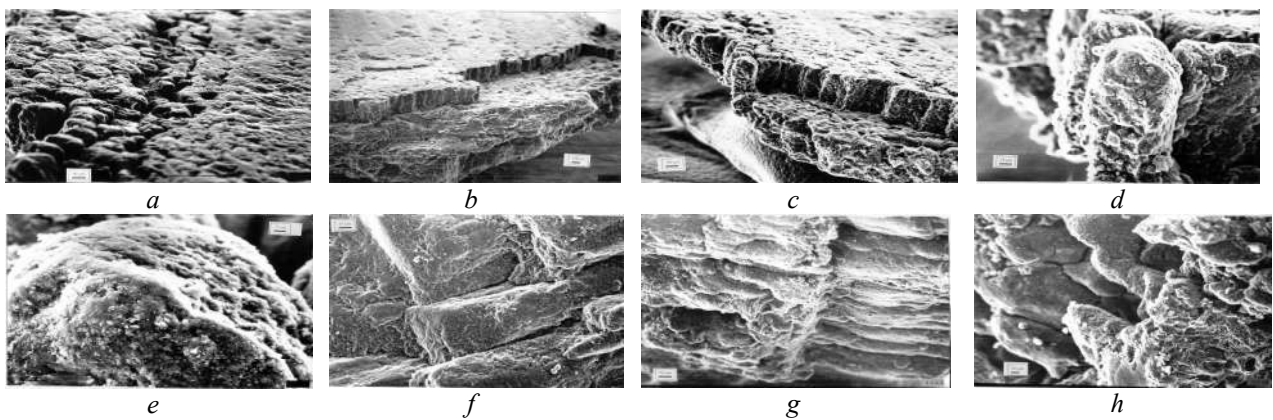


Fig. 3. Morphology of the core of Pt-containing deposit (PEM), formed by the joint evaporation of a graphite electrode doped with platinum: *a, b, c, d* – core blocks; *e* – morphology of the block; *f, g* – the boundary between the shell and the core; *h* – the structure of the boundary loose layer

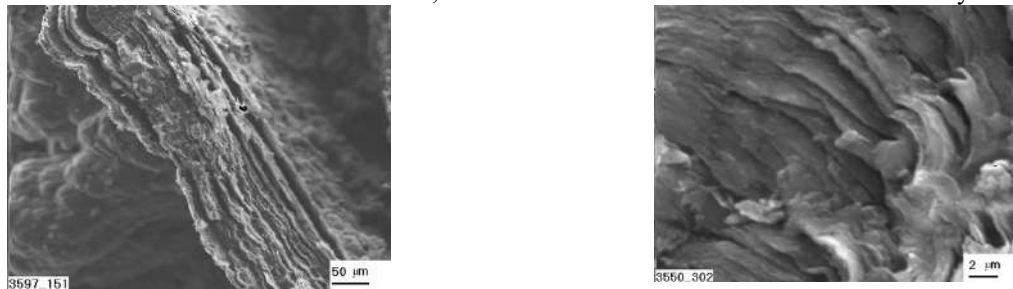


Fig. 4. Dense deposit bark formed by layered graphite-like structures

Thus, the Pt-catalyst during synthesis in a gaseous environment allows to form of almost

centimeter rods from bundle-like CNTs that can withstand temperatures up to 12 000 K [66].

The possibility of separating the deposit core, consisting of CNT bundles, from the deposit rind allows considering the deposit parts as independent products of plasma-chemical synthesis. The separation of deposited CNTs from the graphitic-like rind increases the purity of the deposited CNTs, and this reduces their cost, since previously the deposits were crushed to separate the CNTs from the graphitic component. At the same time, a Pt-containing graphene-like rind (shell) can be used to obtain platinum-enriched graphene.

The final products of the processing of the Pt-containing deposit (Pt-CNT and Pt-graphene-like structures) can become the key to the creation of cheap fuel cell electrodes on which hydrogen recovery takes place.

Thus, during the evaporation of a platinum-doped graphite electrode in an electric arc in a helium environment, the catalyst takes part in all plasma-chemical reactions and is redistributed among various synthesis products. Most of the platinum with the flow of condensate moves to the walls of the reactor. According to the data of the emission spectral analysis, the largest amount of platinum (~ 1 % by mass) accumulates in the wall soot. Part of the platinum (less than < 1 wt. %) in the cationic state together with the carbon vapor under the influence of a strong electric field moves to the cathode, forming a Pt-containing deposit. It was found that platinum is present in the deposit, despite the high temperature of the deposit formation zone (up to 11 727 °C) [66], which exceeds the boiling point of platinum (3800 °C).

As a result of studying the chemical composition of the constituent parts of the Pt-containing deposit, it was determined that Pt

atoms are unevenly distributed and concentrated in the deposit rind. We believe that when an electric arc occurs, the flow of electrons that passes through the forming deposit and heats it, affects not only the processes of formation of the carbon structure of the deposit, but also the quantitative content of platinum atoms in various constituent parts of the deposit. Platinum atoms settle mainly in the low-temperature part of the reactor, where there is a deposit rind.

The presence of platinum explains the higher heat resistance of the rind of the platinum deposit. Differential thermal (DTA), thermogravimetric (TG) and differential thermogravimetric (DTG) analyzes support these conclusions. Thus, the temperature of the beginning of the interaction of the Pt-containing rind with oxygen (680 °C) (Table 2, item 2) exceeds the similar parameter of the pure carbon rind (ignition temperature is 575 °C) (Table 2, item 1) (Fig. 5 *a, b*). The DTA curve (Fig. 5 *b*) indicates a two-phase nature (CNT and graphene-like structures) of deposit rind, which is to replace platinum. In addition to the graphite-like component, this sample contains MWCNTs, the increase in the content of which is initiated by the presence of platinum in the rind.

In the deposit core, as it is composed of bundle-like MWCNT, it was revealed low content of platinum, and it was found that it is characterized by a higher thermal stability (640 °C), then deposit core, obtained by catalytic plasma-chemical synthesis from the helium environment) (Fig. 6 *a, b*).

The data of the thermal analysis show that in the composition of the deposit core synthesized in the presence of the Pt catalyst, there are two phases that react with air at 800 °C and 910 °C (Table 2).

Table 2. Heat resistance of components of Pt-containing deposits after plasma-chemical synthesis in gaseous environment

| № n/s | Material | Process temperature, °C | DTG | | DTA | |
|----------|--------------------------|-------------------------|----------------|----------------|----------------|--|
| | | | $T_{1max}, °C$ | $T_{1max}, °C$ | $T_{2max}, °C$ | |
| 1 | Shell without catalyst | 575–980 | 840 | 840 | | |
| 2 | Shell with catalyst (Pt) | 680→1000 | 865 | 825 | 990 | |
| 3 | Core without catalyst | 575–990 | 870 | 850 | 960 | |
| 4 | Core with catalyst (Pt) | 640–965 | 865 | 800 | 910 | |

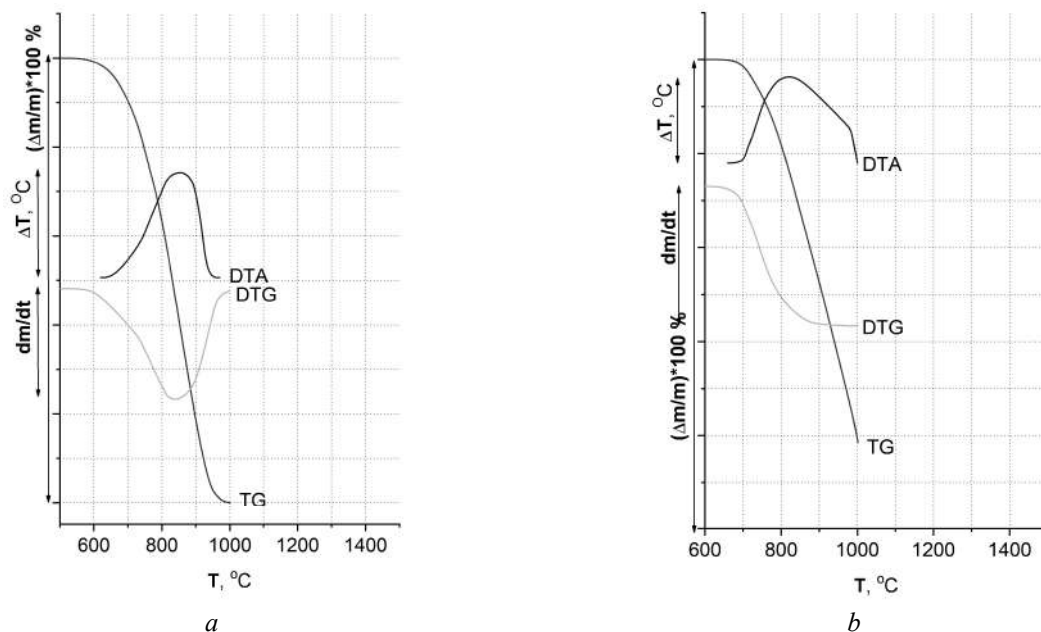


Fig. 5. Investigation of the heat resistance of the deposits shell after plasma-chemical synthesis in a gaseous environment: *a* – the bark of the deposit obtained by non-catalytic synthesis; *b* – deposit bark containing Pt

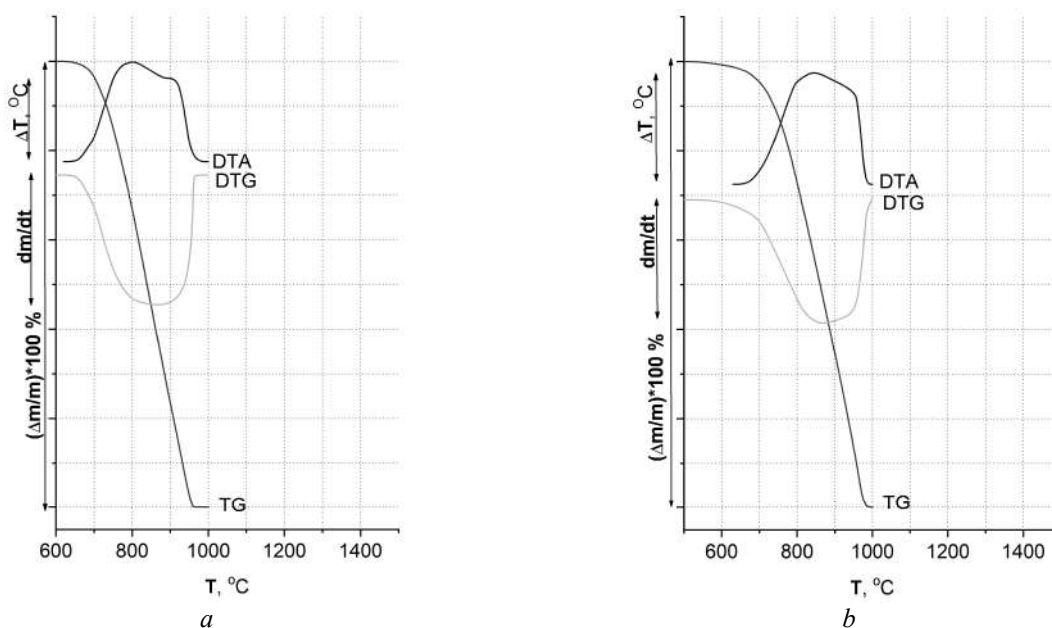


Fig. 6. Investigation of the heat resistance of the core of plasma chemical synthesis deposits in a gaseous environment: *a* – Pt-containing deposit core; *b* – the core of the non-catalytic deposit

Thus, differential thermal analysis in air using the TG, DTG, DTA methods allows to detect slight differences in the heat resistance and oxidation temperatures of various CNS, so thanks to this, the results can be used for their identification.

Wall soot. The mixture of condensate that forms on the walls of the reactor and in the gas

phase, forms the wall soot. It contains both soluble (fullerenes and fullerene-like structures) and insoluble fractions (nanocomposites, CNTs, graphenes) in liquid hydrocarbons.

Studies of the carbon products structures were carried out using scanning (JSM-T20) and transmission (JEM 100 CXII) electron microscopes (SEM and TEM), which show that

nanostructured components have different geometric shapes and structure (Figs. 7, 8).

At the micro level, the wall soot layer has the structure shown in Fig. 7. The layer of wall soot was previously subjected to ultrasound treatment in an alcohol environment.

Extraction of wall soot is carried out by opening the upper and lower reactor covers (flanges), after which the sediment is squeezed out with a rubber piston from top to bottom. The wall soot falls into the storage container, which is installed after opening the reactor covers.

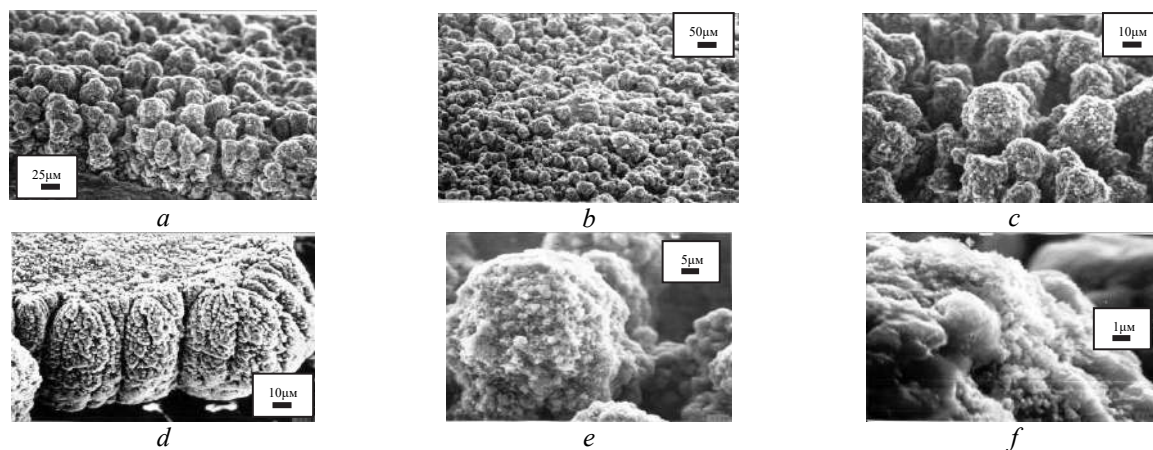


Fig. 7. Surface morphology of the wall soot after its sonication in ethanol: *a, b, c, d, e, f* – SEM photo of the wall soot at different magnifications

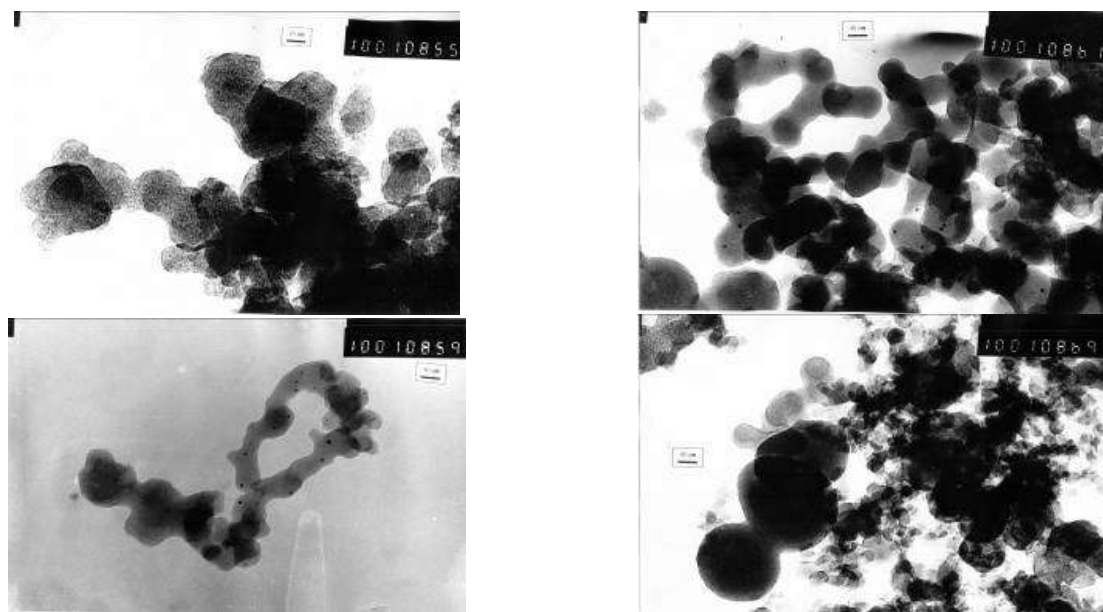


Fig. 8. TEM photographs of nanosized particles that make up Pt-containing wall soot

The soluble component of wall soot – fullerenes and fullerene-like structures – was obtained by the extraction method.

The results of the analysis of wall soot by the Raman scattering method (Raman spectroscopy) are shown in Fig. 9, where the two-peak structure of the G-maximum indicates the presence of two types of nanotubes in the studied

samples: the maximum near the frequency of 1570 cm^{-1} is associated with “armchair” type nanotubes (conductor), and the maximum near the frequency of 1590 cm^{-1} – with nanotubes “zigzag” (semiconductor). The dotted line (Fig. 9) represents the classical reference spectrum of multi-walled carbon nanotubes according to the Hirsch reference book.

Quantitative and qualitative analysis of fullerene solutions was carried out by the spectrophotometric method of UV-Vis spectroscopy (Fig. 10), where fullerenes C₆₀ and C₇₀ are recorded in extraction solutions of wall soot. Peak maxima $\lambda_{\max} = 335.6$ and 407 are characteristic of C₆₀ fullerenes, and $\lambda_{\max} = 334.6$ and 472.8 are characteristic of C₇₀ fullerenes.

As a result of the study of the chemical composition of the constituent parts of the

Pt-containing components of the synthesis, it was determined that the Pt atoms are unevenly distributed and concentrated in the wall soot. We believe that when an electric arc occurs, the distribution of Pt in the catalyst is not uniform in the synthesis products. Platinum atoms settle mainly in the low-temperature part of the reactor, where there is wall soot.

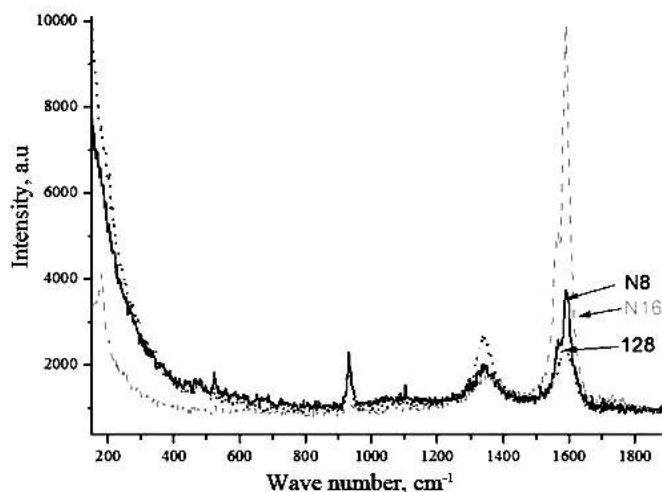


Fig. 9. Raman spectrum of wall soot

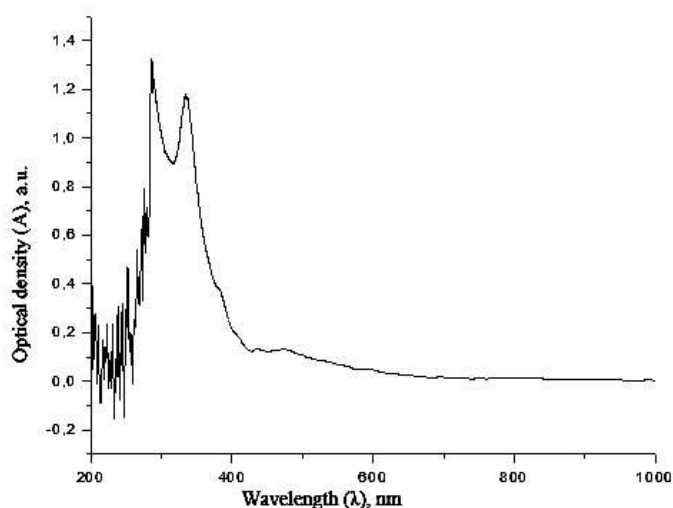


Fig. 10. Optical density spectrum of UV-Vis spectroscopy of solution of fullerene of electric arc synthesis

The formed hollow CNTs in diameter (5–25 nm) and length (1.5–2 μm) do not differ from those obtained without using of platinum, except for some anomalies. It was also found that the deposit has a hexagonal graphite

structure with an admixture of rhombohedral graphite phase.

Platinum with a face-centered cubic lattice (FCC) in synthesis products is distributed as follows: deposit core – less than < 0.001 %, deposit shell – less than < 1 %, wall soot – more

than > 1 %. The components of the deposit with platinum are more heat-resistant, compared to the deposit components that do not contain Pt.

Platinum-containing wall soot is pre-treated for a short time, namely grinding in special “ball mills” or rubbed through a fine sieve with minimal effort to create a homogenous product. Previous studies have shown that such platinum-containing and other carbon nanostructures [67] can already be used in 3D printing technology CJP (ceramic printing) [59, 68], or to create new composites for 3D printing technology FDM, SLA [69–72]. To create electrically conductive ceramics, platinum-containing wall soot, purified from fullerenes and fullerene-like nanostructures, was used as a fuel cell electrode. For this, the process of extracting fullerenes from wall soot with hydrocarbon solvents was carried out, which increased the electrical conductivity of Pt-containing carbon structures in several times.

Using of the deposit and the components of its structure in the creation of a fuel cell did not yield significant results. We believe that this is due to the low content of platinum (Pt) catalyst in the deposit. In addition, the process of grinding in a special “ball mill” or rubbing through a fine sieve required considerable effort and work time to create a homogeneous state of the product.

Today, the research results of this work made it possible to create not only ceramic electrodes, but also fuel cells for a hydrogen cycle fuel element without applying a layer of platinum (Pt) catalyst during usage of CJP 3D printing technology. And this is the first step towards the creation of cheap fuel elements for hydrogen energy.

CONCLUSIONS

In the process of scientific research work the following was done:

1. The distribution of atoms of the catalyst (platinum) in the products of electrochemical synthesis in a gas medium using Fine-grained dense graphite (FGDG-7) was studied.

2. It has been found that Pt atoms in the structure of the deposit and in the synthesis products are unevenly distributed. This is confirmed by the analysis of the chemical composition of the platinum-containing deposit and wall soot.

3. It has been found that the central part of the deposit (core) contains only trace amounts of platinum, and the main part of Pt atoms is concentrated in the shell of the deposit (rind). This phenomenon is explained by the influence of the flow of electrons from the electric arc, that occurs during synthesis and passes through the deposit, heating it, not only on the process of forming the carbon part, but also on the content of platinum in different parts of the deposit.

4. It has been proven that the atoms of the catalyst, in our case platinum, affect the synthesis process and contribute to the formation of a deposit in which the core, containing platinum-containing CNT bundles, is easily separated from the shell and can then be used as a separate rod.

5. It was found that platinum-containing deposit components (core and rind) can be considered as independent products of plasma chemical synthesis.

6. It was found that the differential thermal analysis of CNM in air by TG, DTG, DTA methods allows us to detect even slight differences in the thermal resistance of various CNMs, due to which these data can be used to identify the composition and type of nanomaterials (CNTs, graphenes).

7. It was found that Pt atoms are concentrated in wall soot. We believe that when an electric arc occurs, the distribution of Pt in the catalyst is uneven in the synthesis products. Platinum atoms settle mainly in the low-temperature part of the reactor, where there is wall soot.

8. It was found that the formed hollow CNTs do not differ in diameter (5–25 nm) and length (1.5–2 μm) from those obtained without the participation of platinum, with the exception of some anomalies.

9. It was found that the deposit has a hexagonal graphite structure with an admixture of a rhombohedral graphite phase.

10. Platinum with a face-centered cubic lattice (fcc) in synthesis products is distributed as follows: deposit core – less than 0.001 %, deposit shell – less than 1 %, wall soot – more than 1 %.

11. Components of a deposit with platinum are more heat resistant compared to deposit components that do not contain Pt.

To study the suitability of platinum-containing carbon nanostructures for CJP

(Ceramic Printing) 3D printing, the following was done:

1. It has been found that for the use of platinum-containing wall soot, it is necessary to carry out preliminary short-term processing, namely, grinding in special “ball mills” or rubbing it through a fine sieve with minimal effort to create product homogeneity. Previous studies have shown that such platinum-containing carbon nanostructures can already be used in CJP 3D printing technology, or to create new composites for FDM, SLA 3D printing technology.

2. It was found that using of the deposit and its structural components in the creation of a fuel

cell did not yield significant results, unlike wall soot. We believe that this is due to the low content of platinum (Pt) catalyst in the deposit.

3. It was found that the process of grinding the deposit in a special “ball mill” or rubbing it through a fine sieve required considerable effort and work time to create a homogeneous state of the product.

4. It was found that the investigations of this research work made it possible to create not only ceramic electrodes, but also fuel cells for a hydrogen cycle fuel element without applying a layer of platinum (Pt) catalyst when using CJP 3D printing technology.

Платиновмісні вуглецеві наноструктури для створення електропровідної кераміки при використанні 3D друку технології CJP

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Вуглецеві наноструктури (ВНС) синтезувались електродуговим плазмохімічним методом при випаровуванні графітового електрода високої якості марки «дрібнозернистого щільного графіту» (МПП-7) заповненого каталізатором (Pt), який випаровувався в середовищі гелію. В процесі синтезу були синтезовані: багатостінні (БВНТ) та одностінні вуглецеві нанотрубки (ОВНТ), фуллерени, графенові пакети та нанокомпозити. Також був синтезований депозит у вигляді наросту на катодному електроді. Всі продукти синтезу були проаналізовані на мікро- та нанорівнях, що дало змогу проаналізувати вплив парів платини на формування вуглецевих наноматеріалів (ВНМ). Досліджено нерівномірний розподіл атомів каталізатора (платини) в продуктах електрохімічного синтезу в газовому середовищі при використанні графіту марки МПП-7.

При аналізі встановлено, що платина знаходиться в стані гранецентрованої кубічної (ГЦК) ґратки та розподіляється у продуктах синтезу наступним чином: серцевина депозиту – < 0.001 %, оболонка депозиту – < 1 %, пристінна сажа – > 1 %. Вивчені морфологія та склад платинового депозиту, що має гексагональну графітову структуру з домішкою ромбоедричної графітової фази. В дослідженнях проведений диференціально-термічний аналіз на повітрі (TG, DTG, DTA), що дозволило ідентифікувати склад продуктів синтезу. Встановлено, що частини депозиту з платиною більш термостійкі порівняно з депозитними складовими, що не містять Pt. Вуглецеві нанотрубки (ВНТ), що утворюються, по діаметру (5–25 нм) і довжині (1.5–2 мкм) не відрізняються від таких, що отримані без участі платини, за винятком деяких аномалій.

При вивченні придатності платиновмісних вуглецевих наноструктур для 3D друку технології СJP (друк керамікою) встановлено, що для використання платиновмісної пристінної сажі необхідно проводити попередню недовгу обробку, а саме – подрібнювати у спеціальних «кульових млинах» або протирати крізь дрібне сито з мінімальними зусиллями для створення однорідності продукту. Попередні дослідження показали, що такі платиновмісні вуглецеві наноструктури вже можна використовувати у 3D друку технології СJP або для створення нових композитів для технології 3D друку FDM, SLA.

Ключові слова: вуглецеві наноструктури (BHC), платина (Pt), фуллерени, графени, депозит, плазма, електродуговий синтез, плазмохімічний синтез, 3D друк, технологія СJP

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