Research Article

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Electric discharge method of synthesis of carbon and metal–carbon nanomaterials

<https://doi.org/10.1515/htmp-2020-0078>

received June 07, 2019; accepted November 02, 2019

Abstract: The current state and prospective of electric discharge methods of synthesis of carbon and metal–carbon nanomaterials (CNMs) in different areas of science, technology and industry are considered in the present paper. It was shown that as a result of electric discharge treatment of liquid hydrocarbons, the productivity of CNMs (namely, onion-like carbon, carbon nanotubes, carbon nanofibers and carbon thin films) in terms of the percentage of carbon, released from initial liquid as a nanomaterial, was 6.1% for pentane, 10.2% for hexane, 8.7% for cyclopentane, 14.4% for cyclohexane, 12.0% for benzene and 16.8% for kerosene. Component and phase composition of obtained products depends on the electrode material and composition of initial hydrocarbon liquid.

Keywords: high-voltage electric discharge, carbon nanomaterial, metal–carbon nanomaterial, organic liquids, metal powders

1 Introduction

Unique electric, strength, optical and magnetic properties of such carbon nanomaterials (CNMs), as fullerenes, onion-like carbon (OLC), carbon nanotubes (CNTs), carbon nanofibers (CNFs) and carbon thin films allow their usage in different areas of science, technology and industry. Recently new nanoforms of carbon as well as

the so-called carbon–carbon nanocomposites, which have their own atomic structures, sizes and morphology and display a large variety of physical and chemical properties, were predicted and synthesized [1–6].

The main areas of CNM applications are electronics, medicine, chemistry, pharmaceuticals and biology. Nevertheless, the issue of the selection method of obtaining CNMs becomes very important while considering the prospective of different applications of CNMs. The following criteria for evaluating the methods of different CNM synthesis can be highlighted, namely, low cost of CNMs, the efficiency of processing raw materials and the controllability of the synthesis process. All these criteria are met in the application of electric discharge methods, which are based on plasmochemical and thermodynamic processes of synthesis of carbon nanostructures from the plasma that contains carbon.

Compared to other known methods of obtaining CNMs (electric arc, chemical vapor deposition (CVD), detonation, etc.), the electric discharge method is characterized by the easiness of electric discharge parameter variation, which allows changing the synthesis conditions in a wide range as well as by the fact that there is no need to create special conditions in the reactors (vacuum, usage of inert gases, etc.). It is worth noting that the creation of effective technologies of OLC is of high practical interest. different methods of OLC formation are known, namely, physical vapor deposition, nanodiamond annealing, carbon black irradiation by electrons, implantation of carbon ions in the metallic matrix and explosion. However, all of them either have low productivity or have high cost of obtained OLC.

For example, according to [7], the productivity of CNM synthesis using CVD method varies from 0.45 to 100 g/h depending on the peculiarities of the used technology, catalysts, precursors and equipment. As the power consumption of CVD devices varies from 300 W to 4 kW, the energy cost of CNM synthesized by CVD varies ranging from 1 MJ/kg to 32 GJ/kg.

The possibility of the usage of high-voltage electric discharge treatment (EDT) method of metal powders in organic liquids aimed at their dispersion and change of their phase composition, namely, the synthesis of metal carbides, was shown in [8–10]. The characteristic feature

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Figure 1: The schematic of the experimental setup for the synthesis of CNM: 1 – container with organic liquid; 2 – discharge chamber for treatment of liquid phase; 3 – filter for filtering liquid phase; 4 – discharge chamber for treatment of gaseous phase; 5 – filter for filtering gaseous phase; 6 – pyrolysis reactor.

of this method is the multifactoriality of treatment [8], which includes thermal impact of low-temperature plasma of discharge channel and hydrodynamic impact on processed medium. Energy cost of CNM synthesis using the EDT method is ~ 10 MJ/kg, which is less than

the energy cost of most CNM synthesized by CVD. Moreover, the EDT method uses less costly equipment and reagents and does not require catalyzers, making it even more economically effective compared to CVD in all known cases [7,11].

The tasks of process control aimed at the increase in productivity and the increase in raw material processing efficiency are urgent for the development of technology of electric discharge synthesis of carbon and metal-CNMs. The solution of these tasks is possible by the development of ideas on the mechanisms of electric discharge synthesis of different nanomaterials as well as by conduction of studies of the regularities of production of synthesized materials based on the selected organic raw materials and the technological conditions of synthesis.

The goal of the present work is to analyze the possibility of aimed synthesis of carbon and metal-CNMs of a given phase composition as a result of EDT of organic liquids, metal powders and formed gases.

2 Experimental

Organic liquids, which belong to aromatic compounds (benzene), cycloalkanes (cyclopentane and cyclohexane), alkanes (pentane and hexane) as well as the mixture of aromatic compounds, alkanes and cycloalkanes (kerosene), were selected as a raw material for these studies.

Figure 2: Typical high-resolution microphotographs of the products of hydrocarbons EDT: a – benzene; b – cyclohexane.

Figure 3: The impact of microprotrusions on electrode surface on the electric field intensity: (a) microphotograph of thin section of electrode and (b) the dependence of electric field intensity on the distance from microprotrusion.

Figure 4: Microphotograph of nanocarbon–aluminum composite material.

The energy of σ bond in C–C molecules of cycloalkanes is few times higher than the energy of C–H bond; thus, the process of dehydrogenation (the removal of hydrogen) is more possible as a result of EDT, while the destruction of molecules (breaking of C–C bonds) leads to the formation of alkanes. Alkanes break apart at the temperature of 450–700°C due to the breakage of C=C π bonds, which leads to the formation of alkanes and alkenes with fewer carbon atoms. EDT of alkanes leads to the formation of large variety of gaseous hydrocarbons. Higher temperature (over 1,000°C) leads to not only the breakage of the C=C π bonds but the breakage of more durable C–H bonds as well.

Dissociation of hydrocarbon molecules with the formation of charged clusters and hydrogen ions occurs in strong electric fields, while the thermal impact of electric

discharge currents leads to changes in orbital hybridization of carbon atoms from sp^3 to sp^2 . Part of C–C σ bonds can remain after the EDT of cycloalkanes, which can lead to the formation of sp^2/sp^3 nanocomposites.

Results of gas chromatography and the measurement of refractive indexes of treated alkanes and aromatic compounds show the presence of intermediate compounds. They can be arranged in following sequences by order of their concentration decrease:

hexane (C_6H_{14}) $\rightarrow$ toluene (C_7H_8) $\rightarrow$ phenylacetylene (C_8H_6) $\rightarrow$ styrene (C_8H_8) $\rightarrow$ ethylbenzene (C_8H_{10}) $\rightarrow$ benzene (C_6H_6) $\rightarrow$ naphthalene ($C_{10}H_8$);

benzene (C_6H_6) $\rightarrow$ biphenyl ($C_{12}H_{10}$) $\rightarrow$ 1,4-biethynyl-benzene ($C_{10}H_6$) $\rightarrow$ fluorene ($C_{13}H_{10}$) $\rightarrow$ fluoranthene ($C_{16}H_{10}$) [10].

The structures of the molecules of such intermediate EDT products, as naphthalene and anthracene, contain benzene rings, while the atom bonds in molecules of fluorene and fluoranthene form pentagons and hexagons that are similar to fullerenes structure. Their subsequent dehydrogenation and enlargement of similar

Table 1: Chemical composition (mass%) of areas noted in Figure 4

Memo	C	Al	Total
20	53.83	46.17	100
21	32.43	67.57	100
22	26.16	73.84	100
23	41.15	58.85	100
24	43.70	56.30	100
25	40.34	59.66	100
26	58.40	41.60	100
27	51.68	48.32	100
28	42.48	57.52	100

structures during EDT lead to the formation of graphite-like or fullerene-like CNM, respectively.

Thus, EDT of hydrocarbon liquids leads to the series of chemical transformations, which include destruction, dehydrogenation and polymerization (formation of new C–C bonds). Qualitative and quantitative composition of all EDT products (gaseous, insoluble solid CNM and compounds, dissolved in initial liquid) can vary in wide range and is largely dependent on the type of raw materials.

As the EDT of liquid hydrocarbons results in the formation of lower acyclic gaseous hydrocarbons mixture, in order to achieve the higher utilization of raw materials, simultaneous treatment of liquid and gaseous hydrocarbons in two discharge chambers, connected with venting pipeline, was realized (see Figure 1).

Organic liquid was transferred from the container (see Figure 1, pos. 1) to the discharge chamber (see Figure 1, pos. 2), where the series of electric discharges with fixed frequency of pulses was realized. Gaseous phase, which formed in this chamber during treatment, was transferred to the second discharge chamber (see Figure 1, pos. 4), where EDT of gaseous phase was performed with the same frequency of pulses. Carbon materials formed in both chambers during the treatment were deposited on filters (see Figure 1, pos. 3 and 5). Depleted gaseous phase was transferred to the pyrolysis reactor (see Figure 1, pos. 6).

The efficient regimes for EDT of organic liquids in the flow through mode (continuous flow of the liquid at a rate ranging from 1 to 3 dm³/s, voltage ranging from 40 to 50 kV,

pulses passing frequency ranging from 4 to 15 Hz and stored energy of single pulse ranging from 0.02 to 0.2 kJ), as well as for EDT of produced gases (voltage ranging from 30 to 40 kV, pulses passing frequency ranging from 10 to 25 Hz and stored energy of single pulse ranging from 0.05 to 0.1 kJ), are found [12].

3 Results and discussion

3.1 EDT of organic liquids

CNMs with developed surface ($S_{\text{BET}} \sim 50\text{--}300 \text{ m}^2/\text{g}$) were synthesized as a result of liquid hydrocarbons EDT on this setup. Individual particles had a spherical shape and sizes ranging from 10 to 20 nm. High-definition electron microscopy indicates complex morphology of individual particles, which depends on the carbon source type. For example, microphotographs of CNM, obtained from the EDT of benzene, clearly show layered structure with characteristic distance between separate layers of $\sim 3.5 \text{ nm}$ (see Figure 2a). However, in case of using cyclohexane, the morphology of particles becomes noticeably more complicated. It can be clearly seen in Figure 2b that in the structure consists of a core with a size of $\sim 5 \text{ nm}$, which is surrounded by shell that consists of ~ 5 layers with a distance of $(0.332 \pm 0.001) \text{ nm}$ between them (graphite interlayer distance is 0.3354 nm). Such a “core-shell” structure is characteristic of OLC [13].

Figure 5: X-ray diffraction pattern of the products of cyclohexane EDT while using aluminum-made electrodes.

Figure 6: Microphotographs of powder obtained by EDT of cyclohexane using niobium-made electrodes with different magnification. (a) Microstructure, (b) nanostructure.

Table 2: Chemical composition (mass%) of areas noted in Figure 6a

Memo	C	Nb	Total
36	68.43	31.57	100
37	66.86	33.14	100
38	73.30	26.70	100
39	92.82	7.18	100

Electric discharges are inevitably accompanied by electrode erosion and, as a result, by inclusion of electrode material metal particles into the products used for synthesis.

Analysis of the microphotographs of thin sections of electrodes (see Figure 3a), the roughness of surface of which is caused by microprotrusions ($d_m \sim 160 \mu\text{m}$), was performed in work [14]. When the voltage is applied to positive electrode, the presence of microprotrusion leads to significant local increase in electric field intensity E (see Figure 3b). It was found that at the voltage of $U_0 = 50 \text{ kV}$, the electric intensity near the positive electrode reaches the value of $E > 5 \times 10^7 \text{ V/m}$, which ensures the development of plasma channels from microprotrusions that lead to a discharge. Release of heat energy in the discharge channel and the rise of temperature

Figure 7: X-ray diffraction pattern of the products of cyclohexane EDT while using niobium-made electrodes.

Figure 8: X-ray diffraction pattern of the products of cyclohexane EDT while using electrodes made from aluminum and titanium.

($T > 10,000$ K) during the time of few microseconds lead to melting and explosive evaporation of microprotrusions.

In order to study the impact of erosion on the composition of the products of cyclohexane EDT (stored energy of $W = 28.8$ J), electrodes, made from different materials (titanium, aluminum, cuprum, niobium), were used.

SEM photographs and composition of composite materials obtained by cyclohexane EDT using electrodes made of aluminum are shown in Figure 4. It can be seen

that these specimens are relatively homogeneous (see Figure 4 and Table 1) due to their elementary composition and are the disperse system of “solid sol” type with nanosized carbon matrix and polydisperse metal phase. Maximal size of erosion products is not higher than $10\text{ }\mu\text{m}$.

Microphotographs and chemical composition of the obtained nanomaterials indicate the significant content of particles with shape, close to spherical, which are formed as a result of electrode erosion. Results of X-ray diffraction phase analysis (see Figure 5) indicate the

Figure 9: X-ray diffraction pattern of the products of cyclohexane EDT while using electrodes made from cuprum and titanium.

Figure 10: Electron microphotograph of CNT obtained from gases that are synthesized during cyclohexane EDT.

Figure 11: Electron microphotograph of CNT obtained from gases that are synthesized during kerosene EDT.

phase composition of nanomaterial obtained while using aluminum-made electrodes – amorphous carbon, Al and its Al_4C_3 carbide.

Photographs of nanocomposite material, obtained by EDT of cyclohexane using Niobium-made electrodes, are shown in Figure 6. Chemical composition of the obtained powder are given in Table 2.

X-ray diffraction pattern of products of cyclohexane EDT (see Figure 7), obtained while using niobium-made electrodes, indicates the synthesis of amorphous carbon and niobium carbide.

While using electrodes made of aluminum and titanium, the X-ray diffraction phase analysis (see Figure 8) indicates that the electric discharge processes in cyclohexane lead to the synthesis of aluminum and titanium carbides. Phase composition of the obtained nanomaterials are amorphous carbon, Al, Al_4C_3 and TiC carbides.

According to results of X-ray diffraction phase analysis, the use of electrodes made of cuprum and titanium electric discharge processes in cyclohexane only lead to the synthesis of titanium carbide, as cuprum is not one of carbide-forming elements (see Figure 9). Phase composition of obtained nanomaterial are amorphous carbon, Cu, TiC and titanium carbide.

Thus, component and phase compositions of EDT products largely depend on electrodes material, the content of which in treatment products depends on stored energy. Synthesis of metal–carbon materials in the process of EDT of liquid hydrocarbons, which is connected to electrode erosion, shows the possibility of chemical reactions between carbide-forming metals and carbon clusters.

3.2 EDT of formed gases

C_k carbon clusters are the products of gases EDT, and agglomeration of which in discharge chamber volume leads to the synthesis of amorphous carbon. Different structures of CNM (namely CNF, CNT or thin films), which are synthesized from the mixture of formed gases on the substrates, are obtained from different materials. Catalytically active nickel nanoparticles were deposited by electric explosion of conductors on brass mesh of 0.1 mm thickness and cell size of 1×1 mm in order to synthesize CNTs. Silicon and quartz were used as a catalytic surface for the synthesis and deposition of carbon thin films [15].

CNMs, synthesized on nickel-made catalyzer, consist of CNTs and CNFs (see Figures 10 and 11), which fully cover the surface of the catalyzer. The size of the obtained nanotubes and nanofibers depends on the dispersity of the catalyzer.

Synthesis of nanocarbon from gases that are formed during EDT of liquid alkanes and cycloalkanes also occurs on quartz surface; nanocarbon is synthesized as films with a thickness of up to 1 mm (see Figure 12). External (darker) surface of the film (see Figure 12) only contains carbon, while internal (lighter) surface also contains silicon (from 10 to 20%), which means that

Figure 12: Electron microphotograph of CNT obtained from gases that are synthesized during hexane EDT.

growth of obtained films occurs by carbidization of quartz.

Films with thickness ranging from 5 to 10 μm can fold up, forming scroll-like structure, whose internal surface is covered by globules with size of up to 0.1 mm, while their external surface is covered by crystallites with size up to 1 μm . Chemical composition of the area, marked in Figure 12a, is as follows – 96 mass% C and 4 mass% O. In macroscopic area (see Figure 12b and c), the scrolls of film have column-layered structure.

Thus, EDT of liquid and gaseous hydrocarbons leads to the formation of carbon clusters C_k and hydrogen according to the following scheme:

The molar masses of raw materials $M_r = 12n + k$, as well as of obtained product $M_p = 12n$, were calculated in order to theoretically evaluate maximal mass m_{th} of CNM, which can be synthesized as a result of the plasmocatalytic reaction by scheme (1) as a result of EDT of raw materials of m_r mass. Then

$$m_{th} = m_r M_p / M_r = m_r \times 12n / (12n + k). \quad (2)$$

The practical CNM productivity $\gamma = \gamma_1 + \gamma_2$ was calculated by the experimental measurement of mass m_p^1 and productivity γ_1 of CNM obtained by full EDT of organic liquid, as well as of the mass m_p^2 and productivity γ_2 of CNM, obtained by PDT of the mixture of gases, given that carbon was released from all the volumes of raw materials without any leftovers

$$\gamma_{1,2} = m_p^{1,2} / m_{th} = (1 + k/12n)(m_p^{1,2} / m_r). \quad (3)$$

It was shown that as a result of EDT of liquid hydrocarbons, the productivity of CNM γ_1 was 6.1% for pentane, 10.2% for hexane, 8.7% for cyclopentane, 14.4% for cyclohexane, 12.0% for benzene and 16.8% for kerosene. Gases are formed from the rest of the liquid hydrocarbons. The productivity of CNM γ_2 as a result of EDT of gases was up to 10% without catalyzer and over 50% when the Fe group catalyzers were used.

Obtained results should be taken into account in order to implement targeted synthesis of CNM and, accordingly, to optimize the technological parameters of EDT process.

Figure 13: Integral microphotographs of electric discharges in “kerosene–nanocarbon–Ti powder” disperse system (stored energy $W_1 = 90 \text{ J}$): (a) 5th pulse; (b) 10th pulse; (c) 50th pulse; (d) 100th pulse.

Figure 14: X-ray diffraction patterns of the products of Ti powder EDT in kerosene: (a) $W_s = 5$ MJ/kg; (b) $W_s = 10$ MJ/kg; (c) $W_s = 20$ MJ/kg.

3.3 EDT of metal powders in kerosene

Theoretical study of inhomogeneity of electric field in the volume of discharge chamber, caused by the presence of metal powder particles in liquid, was performed in order to evaluate the conditions of discharge development in disperse medium. Analysis of electric field distribution around metal particle of 100 μm diameter has shown that local values of electric

field intensity near the particle can be higher than $E = 10^7$ V/m; and thus, the processes of dissociation, ionization and plasma formation are possible in this area. Thermal impact of plasma leads to melting and evaporation of particles on the surface layer, ejection of metal vapor into the liquid, which contains carbon clusters, as well as to exothermal reactions between metal and carbon ions. In turn, as a result of the rapid expansion of the discharge channel, processed medium

is impacted by compression–decompression waves and hydro flows, which intensively mix the treated powders.

Thus, during EDT of metal powders in organic liquids, simultaneous grinding and mixing of powders can be achieved, as well as the synthesis of nanosized metal carbides. However, further development of the method is possible only after solving a number of issues related to the control of percentage of components of the resulting product and ensuring the stability of the process [8,16], which is possible only after studying the physical processes that occur during EDT of disperse systems.

Video registration of physical processes that occur during EDT of Ti powder of $d_m = 100\ \mu\text{m}$ dispersity was performed in order to evaluate the processes of EDT in “kerosene–nanocarbon–Ti powder” disperse system (see Figure 13) [16].

Integral photographs of high-voltage electric discharges show the distribution of plasma in discharge chamber volume. At the initial stage of treatment, plasma formations were only located in the areas situated near the electrode (see Figure 13a and b), which leads to the increase in the distance between the powder layer and positive electrode. Also, on the photograph of 10th pulse (see Figure 13b), the relative increase can be noted in the diameter of plasma formation near the positive electrode, which indicates the increase in the fraction of energy, released at this area, and as a result, the increase in hydrodynamic impact of gas–vapor cavity on the medium.

We should also pay attention to the position of the powder relative to the electrode system (see Figure 13c and d). Cyclic formation of gas–vapor cavity in near-electrode area leads to ejection of powder from the central part of the chamber, which leads to intensification of hydrodynamic impact on the medium. However, the intensification of hydrodynamic impact leads to the decrease in the destruction efficiency of electric erosion particles, as the concentration of solid phases in the central part of discharge chamber decreases. Therefore, the frequency of pulse passage was selected in such a way that it is equal to the inverse evaluated time of particle sedimentation, which are constantly located in the treatment as a suspension [17].

As it can be seen from the photographs of 50th and 100th pulses (see Figure 13c and d respectively), maintaining such a frequency promotes the formation of suspended particles of titanium in kerosene and provides mixing of the powder during processing. Also the increase in kerosene opacity should be noted in this two photographs, which indicates the synthesis of nanocarbon and nanosized titanium carbide.

Physical modeling allowed obtaining qualitative description of a set of physical processes that take place during the EDT of “kerosene–nanocarbon–titanium powder” disperse system. Experimental proof was found for the hypothesis of the impact of the complex factors on “kerosene–nanocarbon–titanium powder” disperse system, namely, electric erosion and hydrodynamic impact.

Experimental studies have shown that EDT of Ti powder of $60\ \mu\text{m}$ initial mean diameter allows obtaining the Ti–TiC mixture powders in the range of submicrometer and nanosize [18,19]. Results of X-ray diffraction analysis of the obtained powders indicate the increase in titanium carbide content from 22 to 60% when specific treatment energy is increased from 5 to 20 MJ/kg (see Figure 14).

4 Conclusions

Electric discharge synthesis of carbon and metal–carbon materials has firmly occupied its niche among other methods. Realization of different technological schemes of treatment of organic liquids, powders and formed gases allows increasing the efficiency of processing raw materials as well as combining few different technological operations and achieving the aimed synthesis of CNMs (OLC, CNT, CNF or carbon thin films) and of metal–carbon materials of given phase composition.

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