

**PRODUCTION OF CARBON NANOTUBES
FROM AN AIR-PROPANE-BUTANE MIXTURE
IN A HIGH-VOLTAGE ATMOSPHERIC-PRESSURE
DISCHARGE WITH AN EXTERNAL MAGNETIC FIELD**

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The synthesis of nanostructures with carbon nanotubes in a plasma reactor with a high-voltage atmospheric-pressure discharge (voltage 1–4 kV and current to 200 mA) on exposure of the discharge to an external magnetic field with an induction of 0.03 T or higher has been investigated. The composition of the soot products has been studied. The discharge was initiated in a quartz channel between the electrodes from catalytic metals in an atmosphere of hydrocarbons mixed with air. The nanotube yield in the soot product amounted to 10–70%. On imposition of the magnetic field, the yield increased by 10–30%, all other things being equal.

Introduction. Investigations of the influence of stationary and nonstationary magnetic fields on the plasma electric-arc synthesis of carbon nanotubes have appeared recently within the framework of this (already traditional) line of synthesis [1]. There are a limited number of publications on this line; nonetheless, they are quite promising as far as their results are concerned. For example, the production of small numbers of multiwalled nanotubes with a yield of to 97% on exposure to a magnetic field is reported in the works of Japanese authors [2, 3]. According to the data of Korean researchers [4], in nonstationary fast-moving arc spots on graphite rotating electrodes, they have managed to attain a product yield of 80% in the case of high efficiency. This does not seem to be a limit, since controlling the dynamics of electrode processes and nonequilibrium plasma states with a magnetic field can be quite efficient. The application of magnetic fields, a nonequilibrium magnetic plasma, and the processes of nonstationary ablation of the electrodes in arc spots moving rapidly under the action of the magnetic field to nanotechnology remains to be adequately studied, on the whole, but interest in this area of investigations rapidly grows, judging by recent publications. Thus, e.g., the Meunier group at McGill University (Canada), which are known for their investigations of electrode processes and the erosion of the electrodes in arc discharges, have recently applied them to delivery of a catalyst to the reaction zone in producing nanotubes using an electric-arc plasmatron [5]. With allowance for the fairly good deal of experiment gained in the last 20–30 years in investigations of heat and mass transfer in the electrode region of arc discharges in a magnetic field and in a nonequilibrium plasma [6, 7–11], it is quite expedient to investigate the influence of the external magnetic field on the synthesis of carbon nanotubes.

Experimental Setup and Procedure of Investigation. For investigation in this line we have developed a special setup based on a plasma reactor with a high-voltage atmospheric-pressure discharge and an external magnetic field. The high-voltage plasma reactor was equipped with a magnetic system which made it possible to create a constant or rotating magnetic field. During this work, we carried out the run of experiments on studying the process of synthesis of nanomaterials at atmospheric pressure and control of the discharge using the magnetic field. The prototype structures of such a plasma generator without a magnetic field and the results of experiments on it have been described in [6, 12]. A diagram of the setup used in the work is presented in Fig. 1. The setup incorporates a plasma-chemical reactor consisting of two basic parts: a quartz tube 1 and the chamber of deposition of carbon nanomaterial 2 above it. The quartz tube and the deposition chamber are flange-jointed with fasteners (not shown on the diagram). The chamber 2 takes the flow of gases formed by the interaction of the propane-air mixture and the high-voltage discharge and

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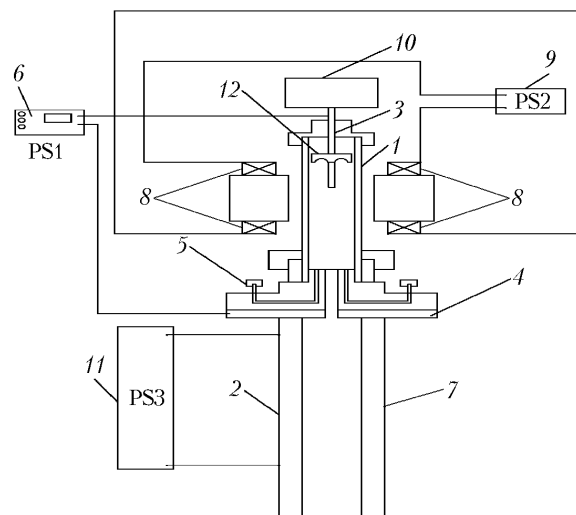


Fig. 1. Diagram of the experimental setup for synthesis of soot with carbon nanotubes; 1) quartz tube; 2) chamber of deposition of the carbon nanomaterial; 3) cathode; 4) grounded anode; 5) gas-supply system; 6) high-voltage electric-power supply; 7) thermocouple; 8) magnetic system; 9) electric-power supply of the magnetic system; 10) screw-type cathode-positioning mechanism; 11) source for heating the chamber of carbon-material deposition; 12) disk-type aerodynamic deflector; PS1, PS2, and PS3, power supplies.

by the chemical reactions between propane and oxygen and escaping from the quartz tube. Cathode 3 is arranged in the upper part of the quartz tube 1, whereas the grounded anode 4 manufactured in the form of a cylinder with an orifice on the axis and the gas-supply system 5 including the unit for feeding and monitoring hydrocarbon raw material and compressed air (not shown on the diagram) (they ensure the feed of the working gas mixture to the working gas mixture to the quartz tube 1 of the plasma-chemical reactor) are arranged in the lower part. A high-voltage electric-power supply 6 is connected to the cathode 3 and the anode 4. The cathode 3 is connected, in the upper part, to a screw-type cathode-positioning mechanism 10 intended for setting the distance between the anode 4 and the cathode 3; in the lower part, it is equipped with a disk-type aerodynamic deflector 12 intended for changing the flow direction and aerodynamics. The chamber of deposition of the carbon nanomaterial 2 is fitted with a source for heating the chamber of carbon-material deposition 11, i.e., an electric-heating system for maintaining the temperature in the synthesis zone optimum, and with thermocouple 7 installed on the outside of the chamber of deposition of the carbon nanomaterial for recording the temperature. The plasma-chemical reactor is fitted with a magnetic system 8 which makes it possible to create a constant or rotating magnetic field in the discharge zone. The discharge chamber (quartz tube 1) is arranged in the axis of the magnetic system 8 consisting of six pole pieces with windings and an electric-power supply of the magnetic system 9.

The plasmatron operated off a high-voltage single-phase (diode) rectifier with a no-load voltage of to 14 kV suitable for ensuring an inoperating discharge voltage to 10 kV. The plasmatron was connected to the rectifier via ballast resistors of the total value of 90 k Ω . The discharge current was set by shunting part of the resistors using a contactor. The rectifier operated off a 220 V/10,000 V transformer. A Γ -filter was used to diminish current and voltage pulsations. The supply voltage of the power transformer was set by an RNO 250-5 autotransformer. The values of current and voltage were recorded visually with pointer-type instruments. An OSU-20 low-voltage transformer for heating the chamber of nanoprodukt synthesis and deposition was used. The heating current was up to 600 A at a voltage of about 2 V. The temperature in the chamber was also controlled by the PNO 250-5 autotransformer from the readings of the pointer-type instrument connected to the Chromel-Alumel thermocouple on the chamber wall. The reactor's magnetic system ensured impositions of the external magnetic field on the discharge in the reactor.

High-Voltage Plasma Generator. This is a modernized version of the generator used in [6, 12]. The modernization involved optimizing the internal aerodynamics of the device to diminish the velocity loss with the aim of reveal-

ing better and comparing the effects of vortex and magnetic stabilization of the discharge. For this purpose we used the results of investigation of the aerodynamics of vortex devices of such a type using laser Doppler anemometry [13].

The high-voltage plasma generator consisted of the discharge chamber manufactured from a quartz tube of diameter 35 mm and the cathode made in the form of a bar from Kh18N10T stainless austenitic steel of diameter 6 mm and the anode in the form of a cylinder from the same steel of diameter 8 mm and with an orifice on the axis, which were arranged along the axis in it. Selection of the material was determined by its paramagnetic properties: the low content of ferrite, which enables us, even at a low temperature, to diminish the influence of the electrodes on the distribution and value of the magnetic field in the discharge zone. Furthermore, iron and nickel being the basic components of the alloy (forming the austenitic phase), are catalysts of the synthesis of carbon nanotubes. Catalyst particles can be delivered to the synthesis zone in the erosion of both electrodes under the action of an electric discharge. The application of a magnetic field can influence not only the electric characteristics of the discharge but also the erosion characteristics of the electrodes [5, 7, 14] delivering the catalytic material to the synthesis zone. Moreover, the catalyst can be injected into the reactor in the form of a vapor of organometallic compounds — ferrocene or cobaltocene.

We compared the synthesis of carbon nanotubes in the presence and absence of the magnetic field in the discharge zone during the experiments. Obtaining a stable discharge column of considerable length requires its stabilization with suppression of the inherent instabilities (of the electromagnetic and hydrodynamic nature) of electric discharges. Such stabilization is usually ensured by vortex flow of the working gas in the discharge chamber. The air-propane-butane mixture is fed, via two channels, to the annular channel between the tube and the anode, whence it flows out along tube walls via spiral channels, taking the tangential moment of momentum. In the basic volume of the vortex flow, the radial pressure gradient is balanced by centrifugal forces, which hinders direct flow from the periphery to the chamber axis but does not get in the way of axial flow from the site of injection of the gas under the action of the longitudinal pressure gradient; therefore, the bulk of the gas flows from the anode to the cathode along the walls. A deflector, i.e., the end wall with a toroidal recess, is installed on the cathode. The twist of the flow damps in the boundary layer on the end wall but the radial pressure gradient directed from the periphery to the axis in the vortex flow continues to act. Therefore, intense radial gas flow from the periphery to the center is formed at the end wall with simultaneous transfer of the moment of momentum to the axis. This produces a further increase in the tangential velocity on transition of the gas to smaller radii. A well-profiled end wall and an axisymmetric cathode of small diameter diminish the twist less and enhance vortex stabilization.

Thus, vortex stabilization plays a double role: it ensures spatial stabilization of the discharge column, making it possible to operate on longer discharges and to attain a higher power, but it also contributes to the thermalization of a nonequilibrium plasma in the discharge, bringing it closer to the thermal-equilibrium state. The reason is that the vortex stabilization of the discharge can be considered physically as ordinary thermal convection in a radial field of centrifugal forces much larger than gravitational forces. Therefore, the hot gas is blocked in the axial zone, hindering convective heat removal from the zone traversed by the current and reducing the electric-field strength in it. This effect may adversely affect the production of high-energy electrons in the discharge which, apparently, play an important role in exciting CO molecules by electron impact.

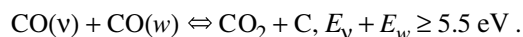
However, heat removal from the discharge column can be controlled using a magnetic field. In particular, it is well known that one can make the discharge precess about the axis of the magnetic system by imposition of a rotating magnetic field and thereby to deflect it from the geometric axis of the vortex [8–11]. This better distributes the discharge throughout the chamber volume and ensures its better interaction with the basic volume of the gas. One objective of the present work is to check this statement. Precession using a magnetic field can be created both in the direction of vortex rotation and opposite to it, which will have an effect on the burning of the discharge on the electrodes because of the change in the velocities of motion of the cathode and anode couplings on their surface. Furthermore, one can change the action of the rotating magnetic field on the discharge, creating either a homogeneous field in the discharge zone or a field with a radial gradient, when it is equal to zero on the axis and increases nearly linearly toward the chamber walls [8–11].

Chamber of Synthesis and Collection of Nanomaterials. This chamber is connected, at the bottom, to the discharge chamber and takes the gas flow escaping from it and formed in interaction of the propane-air mixture with the electric discharge and by the chemical reactions between propane and oxygen. The chamber is fitted with an electric-heating system for maintaining the temperature in the synthesis zone optimum. It is made from a segment of an

Kh18N10T-stainless-steel tube which is heated by passing an alternating current of strength to 600 A from a special OSU-20 low-voltage transformer directly through it. The exterior chamber surface is heat-insulated by asbestos cord and glass tape, which makes it possible to maintain a stationary temperature of to 1070 K in it during the work. The temperature is measured by a Chromel-Alumel thermocouple with a digital display device. The temperature is varied smoothly by a regulating the autotransformer according to the readings of this device.

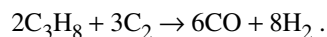
Materials and Raw Materials Utilized. In the work, we used SPBT commercial propane, All-Union State Standard 20448-90, as a raw material. It was fed to the plasma generator from cylinders corresponding to the All-Union State Standard 15860-84 (type 3, capacity 50 dm³, gas volume 12,000 dm³, working pressure to 1.6 MPa, and gas mass 22 kg) with a cylinder gas reducer of the BPO-5 type size for DPP-1-65 propane-butane, All-Union State Standard 6268-79 (maximum gas flow rate 5 m³/h, inlet gas pressure no higher than 2.5 MPa, outlet pressure 0.01–0.3 MPa, and mass 2.6 kg) via an RS-5 flowmeter or an RM rotameter, All-Union State Standard 13045-67 through I VN propane-type rubber hoses of diameter 12 mm, All-Union State Standard 9356-80, to 5 m long. The content of propane in the propane-butane raw material was 71.6 vol. %. The average molar weight of the hydrocarbon raw material was 47.2 g/mole.

Experimental Procedure. The high-voltage plasmatron in this reactor was the generator of reaction gases from which nanomaterials including carbon nanotubes had to be synthesized in the reaction chamber. The relation of the propane and air flow rates in the gas feeding the plasmatron was selected so as to obtain, at its exit, the maximum content of carbon oxide CO which was considered as the raw material for obtaining atomic carbon as a result of the disproportionation (Boudoir) reaction [6, 12]:

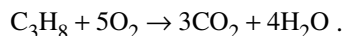


Vibrationally excited carbon-oxide molecules under strongly nonequilibrium conditions and at relatively low temperatures and atmospheric pressure are involved in this reaction. An excess of 5.5 eV over the reaction's activation barrier occurs because of the excitation of the upper vibrational levels of a CO molecule by electron impact in the plasma of a high-voltage discharge. The maximum rate of deposition of nanomaterials requires an optimum temperature in the range 550–750°C. This temperature was maintained in the reaction chamber where the gases from the plasmatron arrived using a special heating system. Since the synthesis of nanotubes required catalysts (this purpose is usually served by metals of the iron group), the reaction-chamber walls were made from Kh18N10T stainless steel. The cathode of the plasma generator, which supplied catalytic nano- and microparticles of the metal as a result of erosion under the action of the cathode spot, was made from the same material.

The optimum composition of the reaction gases and the maximum amount of CO in the ideal case require the reaction



The ratio of the contents of the fuel and the oxidant in this reaction is equal to 2/3. The actual composition of the gases is conventionally described by the equivalence factor γ which characterizes the composition compared to the reaction of complete oxidation of the raw material:



The ratio of the contents of the fuel and the oxidant in this reaction is equal to 1/5. The equivalence factor [15] is equal to the reciprocal of the ratio of the number of oxygen moles per fuel mole in the reaction mixture of a prescribed composition to the number of oxygen (oxidant) moles per fuel mole in the case of the complete oxidation of this hydrocarbon raw material (fuel). It would be ideal to maintain an equivalence factor of 3.2 in the reaction mixture used to attain the maximum concentration of CO in the reaction gases.

The reaction products containing nanomaterials were collected from the reaction-chamber walls and were analyzed on a transmission electron microscope. The conclusions on the efficiency of one operating regime or another were drawn from the results of this analysis.

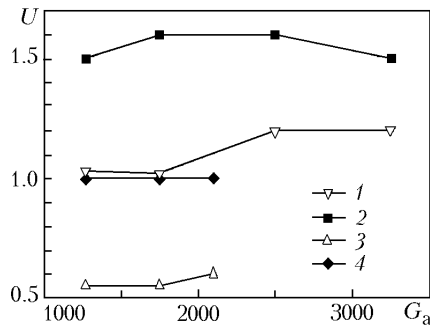


Fig. 2. Voltages on the high-voltage atmosphere-pressure discharge in the vortex air flow in the absence of the magnetic field (1 and 3) and on imposition of a rotating magnetic field of 32 mT (2 and 4): 1 and 3) $I = 100$ mA; 3 and 4) 200 mA. The rotation of the vortex and the magnetic field is cocurrent. U , kV; G_a , liters/h.

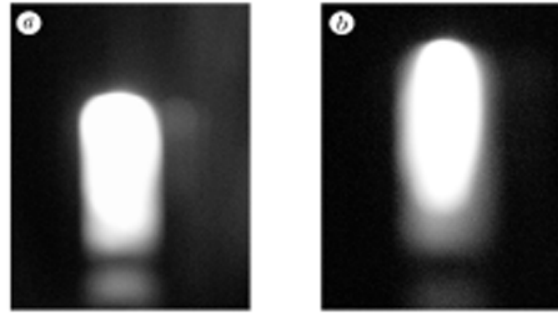


Fig. 3. View of the discharge in air in the rotating magnetic field (a) and without a magnetic field (b): $I = 150$ mA, $U = 1.2$ kV (a) and 0.8 kV (b), and $G_a = 1000$ liters/h.

Experimental Results and Their Discussion. *Influence of the External Magnetic Field on the Parameters of the High-Voltage Electric Discharge.* We have carried out experiments on studying the character of the influence of a constant and rotating magnetic field on the parameters of the high-voltage electric discharge used for synthesis of nanomaterials. The experiments were carried out with a switched-on and switched-off magnetic field without changing the remaining controlled parameters (flow rate and type of the gas, current strength, and sense of rotation of the magnetic field and the gas vortex). We conducted the experiments for codirected and opposing rotations of the magnetic field and the gas vortex and when a constant magnetic field with like poles oriented to the magnetic system's axis, just as in the experiments of [2], was imposed. These experiments to reveal the qualitative influence of the magnetic field were carried out with the use of air as a plasma-generating gas. According to [8–11], the linear velocity of motion of the discharge on the radius of its precession about the magnetic system's axis under the action of the rotating magnetic field is dependent on the balance of the Ampère driving force and the aerodynamic-drag force of the discharge channel in its transverse blowing-in motion relative to the ambient gas in the same manner as in rotation of the discharge between coaxial electrodes in an axial magnetic field [11]. The equation of balance of the resultant forces on the surface of a current-conducting cylinder which can model the conducting cord moving in the perpendicular magnetic flux has the form

$$\mathbf{I} \times \mathbf{B} = C_x \rho v^2 d.$$

In this case the characteristics of the discharge (primarily voltage drop across the discharge) determined by its transverse blowing are only dependent on the velocity of discharge motion relative to the gas and are independent of the velocity of motion relative to the stationary walls of the discharge chamber and the electrodes. This has been confirmed by the experiments, but the stability of burning of the discharge is better and the pulsations of its parameters are smaller in the motion of the discharge in the sense of rotation of the gas vortex.

Figure 2 compares the voltage drops across the discharge in its burning in the vortex air flow for two currents — 100 and 200 mA — without a magnetic field and in precession co-motion in the direction of rotation of the vortex. The voltage was measured in cyclic switching-on and switching-off of the magnetic field, all other parameters being constant. It is seen that the imposition of the magnetic field produced an increase of 40–50% in the discharge voltage.

The imposition of a constant magnetic field of to 100 mT on the discharge exerted no pronounced influence on its characteristics.

Figure 3a gives the photograph of the air-blown discharge with imposition of a rotating magnetic field of about 35 mT, and Fig. 3b gives the photograph of the discharge without a field. Visually the zone of existence of the

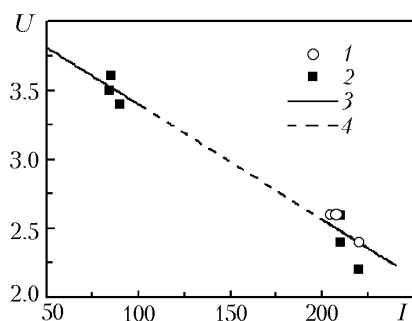


Fig. 4. Volt-ampere characteristic of the discharge in the mixture of air and propane-butane: 1) without a magnetic field; 2) with a magnetic field; 3) cocurrent magnetic field; 4) magnetic counterfield. $G_a = 0.24$ g/sec and $\gamma = 2.8$. U , kV; I , mA.

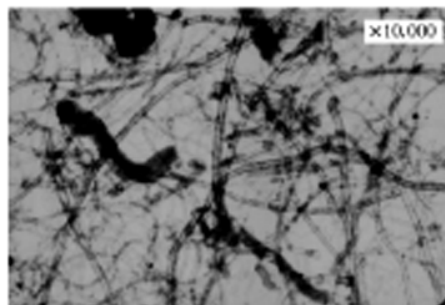


Fig. 5. Transmission electron micrograph of the sample of soot with a 65% content of carbon nanotubes, obtained on the setup with a high-voltage atmospheric-pressure discharge with imposition of the cocurrent rotating magnetic field on the mixture of air and propane-butane (zone II).

discharge extends radially, enveloping, as it were, the larger volume of the discharge chamber, on imposition of the rotating magnetic field.

No special experiments on determining the volt-ampere characteristics (VACs) of the discharge in the mixture of air and propane-butane were carried out; therefore, there are no data on the entire range of variation in the current and voltage. However the approximate behavior of VACs can be reconstructed from technological experimental data. A typical form of the VAC of a high-voltage discharge in the mixture of air and propane-butane without the imposition of a rotating magnetic field and with a magnetic field of 30 mT is shown in Fig. 4. It can be stated that the VAC is dropping, and no dependence of it on the magnetic field has been noted. It is likely that the discharge zone in this case is determined predominantly by the process of combustion of the hydrocarbon-air mixture, not by discharge transfer, as in air.

As a result of the technological experimental investigations, we performed 126 experiments (part of them were adjustment-type). Because of the extremely time-consuming treatment of samples with the aim of determining the content of nanomaterials in the end product, the samples were analyzed selectively. The experiments were performed for the same value of the cocurrent rotating magnetic field or the field counter-type in relation to the twisting by the gas flow; this value was 30 mT. The chamber of synthesis and collection of nanomaterials was arbitrarily subdivided into three zones: the upper (I), the middle (II), and the lower (III) zones. Sampling and an analysis of the samples were carried out separately in each zone.

Influence of the Rotating Magnetic Field on the Synthesis of Nanotubes. It has been established from experimental data that, all other things being equal ($\gamma = 2.6$ and the wall temperature in the chamber of deposition of the soot product, differing little), there is a tendency for increasing the yield of nanomaterials on imposition of a cocurrent magnetic field. In zone III, this increase amounted to 10–20%. The same conclusion can also be drawn from a comparison of the data in Figs. 5 and 6. Here the increase in the yield in zone II was even more significant (by 20–30%); however, the power of the discharge with a field was nearly 1.7 times higher. It is likely that the influence of the two factors simultaneously was combined in this case. As far as the difference in the nanomaterial yield in the cocurrent magnetic field and the counterfield is concerned, we did not observe it within the framework of experimental error. In both cases the yield of nanomaterials in zone II amounted to 20–30%, although the powers and voltages across the discharge were somewhat different.

The value of the nanomaterial yield in the experimental regimes with different levels of the equivalence factor γ can be judged by Fig. 7, according to which the yield remains constant within $\gamma = 2.5$ –3.4, all other things being equal. A point referring to the high $\gamma \approx 17$, where $m \approx 10\%$, is marked in the figure. This point has been obtained for lower discharge power and voltage; however, it may indicate the tendency for decreasing m with increase in γ .

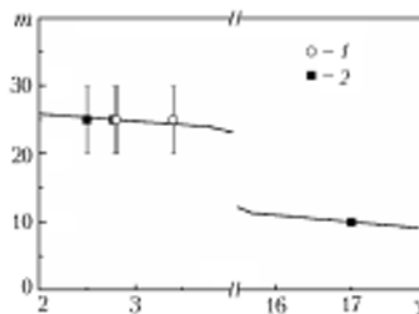
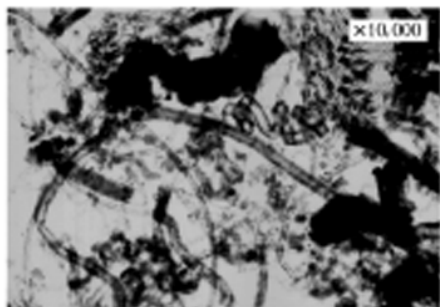


Fig. 6. Transmission electron microphotograph of the sample of soot with a 40% content of nanotubes, obtained on the setup with a high-voltage atmospheric-pressure discharge without imposition of the magnetic field on the mixture of air and propane-butane (zone II).

Fig. 7. Nanomaterial yield m with variation in the equivalence factor γ : 1) without a magnetic field; 2) with a magnetic field. m , %.

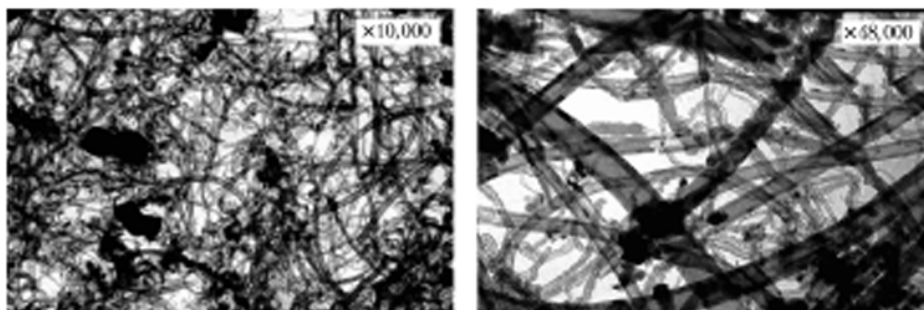


Fig. 8. Transmission electron microphotograph of the sample of soot with a 50% content of carbon nanotubes, obtained on the setup with a high-voltage atmospheric-pressure discharge with imposition of a rotating magnetic field on the mixture of air and propane-butane (zone II).

Figures 5, 6, and 8 give photographs of typical samples obtained by the method of transmission electron microscopy on a JEM 100-CX microscope, Japan, as examples of the morphology and dimensions of synthesized nanotubes (these measurements have been carried out in cooperation with A. S. Egorov at the Institute of Physiology, National Academy of Sciences of Belarus).

Thus, as a result of the experiments we have revealed that the discharge zone expands in the radial direction on exposure to a rotating magnetic field with an induction to 32 mT, whereas voltage and power grow 1.4–1.5 times in burning of the discharge in air. The yield of carbon nanotubes grows, too, attaining 70 wt. % in certain regimes. The data of the electron-microscopy analysis were confirmed by thermogravimetric measurements according to the well-known procedure that was based on the thermooxidation of unstructured carbon phases in the synthesized soot product [1, 6]. It has been established that the imposition of a magnetic field cocurrent with the vortex air flow produced an increase of 40–50% in the discharge voltage. At the same time, no dependences of the VACs in the mixture of air and propane-butane on the magnetic field with induction $B = 30$ mT have been noted. It is likely that the discharge zone in this situation is predominantly determined by the process of partial oxidation and burning of the carbon-air mixture, not by discharge transfer, as in air.

With variation in the equivalence factor γ the yield of nanomaterials m remains constant within $\gamma = 2.5$ – 3.4 and amounts to ~ 20 – 30% . As γ increases to 17, there is a tendency for m to decrease by 10%. It has been established that, all other things being equal (equivalence factor $\gamma = 2.6$ and the wall temperature not differing much), there is a tendency for the nanomaterial yield to increase by 30% on imposition of the external cocurrent magnetic field. As far as the difference in the nanomaterial yield in the cocurrent magnetic field and the counterfield is concerned, we do not observe it within the experimental error.

CONCLUSIONS

1. We have developed a new high-voltage plasma reactor consisting of a high-voltage plasmatron arranged on the axis of a magnetic system which forms a constant or rotating magnetic field and a chamber for the synthesis and deposition of nanomaterials with electric heating in order to maintain a prescribed temperature in the synthesis zone.

2. We have carried out experiments on the synthesis of nanomaterials from a mixture of air and propane-butane in a high-voltage atmospheric-pressure arc discharge with control of the discharge using a magnetic field. The parameters of the discharge were as follows: current strength 87–240 mA, voltage 0.55–3.6 kV, air flow rate 0.24–0.65 g/sec, propane-butane flow rate 0.04–0.275 g/sec, and induction of the rotating magnetic field 30 mT. The nanomaterial yield amounted to 10 to 70% of the total mass of the resulting product in the form of multiwalled carbon nanotubes with a thickness of 10 to 90 nm and with a length to 5–10 μm . The soot products have been analyzed by electron-microscopy and thermogravimetry methods.

3. It has been shown that in regimes with the imposition of an external magnetic field one can increase the carbon-nanotube content in the soot by 10–20%, all other things being equal (equivalence factor $\gamma = 2.6$ and close wall temperatures). Thus, the external magnetic field can be used in the type of reactors investigated to ensure increased yield of multiwalled carbon nanotubes in the soot in synthesis.

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NOTATION

B , magnetic-field induction, T; C_x , coefficient of aerodynamic drag of the discharge channel in its transverse blowing; d , doubled radius of precession of the discharge about the magnetic system's axis, m; E_v and E_w , energies of an excited CO molecule in vibrational quantum states v and w , eV; G_a , air flow rate, g/liter; I , current strength, A; m , mass, g; U , voltage drop across the discharge, V; ρ , gas density, kg/m^3 ; v , velocity of motion of the discharge relative to the gas, m/sec; γ , equivalence factor. Subscript: a, air.

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