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Carbon Nanomaterials: Synthesis, Properties, and Applications

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Abstract

The development of scientific directions initiated by R. A. Buyanov, Corresponding Member of the Russian Academy of Sciences, laid the foundations of the methods to synthesize catalytic nanocarbon materials. As a continuation of his ideas, the studies in the area of thermocatalytic synthesis of carbon-mineral supports were carried out at the Institute of Combustion Problems (Almaty, Kazakhstan). A review of recent works on the formation of carbon nanotubes and nanocarbon sorbents is presented, the areas of their practical application are described. Results of the studies of the synthesis of carbon nanotubes on the catalysts obtained by means of solution combustion on glass cloth and the development of flexible heating elements on this basis are considered.

Keywords: carbon nanotubes, solution combustion method, carbonization, heating elements, smart textiles

INTRODUCTION

Studies in the area of nanotechnologies and nanomaterials are the most urgent directions of modern science. Manipulations with matter at the atomic and molecular levels allow researchers to create absolutely new materials possessing unique physicochemical properties. Solutions to many problems in materials science, electronics and technology in general are related to the development and achievements of nanotechnologies. Among the broad range of nanomaterials, the class of carbon nanotubes (CNT) occupies a special position due to the discovery of fullerenes and graphenes possessing a number of unique electric, physical, mechanical and optical properties [1].

Since the moment of obtaining CNT more than 25 years ago, attention to this class of carbon nanomaterials is permanently increasing, and the area of their practical applications broadens from one year to another. In this connection, the scientific community is extremely active in the area of obtaining, investigation and search for practical applications of unidimensional (1*D*) carbon materials including CNT. Attention of the scientists is attracted to the unique optical, electrophysical, thermophysical, mechanical and other properties of CNT, differing from the properties of bulk materials. Due to this, CNT are called the material of the future [2]. Investigations aimed at obtaining and developing practical applications of CNT are one of the major directions of modern science, which is confirmed by a large number of publications dealing with the subject, both in Russian and in foreign journals [1–3].

The foundations of the methods of synthesis of the catalytic nanocarbon materials were paved by Roman Alekseevich Buyanov, Corresponding Member of RAS. When he carried out investigation of the reasons of deactivation and destruction of industrial catalysts of hydrocarbon dehydrogenation, we developed their scien-

tific systematization and revealed the mechanism of carbide cycle during catalyst coking, which is a process connected with the formation of nanocarbon materials through the catalytic decomposition of hydrocarbons. The results of his works were generalized in the monograph [4]. In the course of investigations, R. A. Buyanov discovered the formation of catalytic carbon on the metals of the group of iron (Fe, Ni, Co) and described the mechanism of this process (the mechanism of carbide cycle) [5], which explains a principal difference of carbon formation on the metals of the VIII group and on their oxides.

The essence of the carbide mechanism of the formation of carbon deposits on the dispersed particles of metals may be described with a general scheme [6]:

$$\alpha\text{-}Fe_2O_3 \xrightarrow{C_nH_m} Fe \xrightleftarrows{C_nH_m} Fe_2C \longrightarrow [Fe] + C$$

According to this mechanism, the interaction of hydrocarbons with metal oxides involves the reduction of metals, then the metals interact with hydrocarbons forming carbides. Decomposition of carbides again leads to the formation of metals and to the formation of free carbon, on this basis the carbonaceous formation with different structures may be formed. During the decomposition of the carbide-like compound, the regions with the great supersaturation of the surface metal layer with the released carbon are formed on metal surface (two orders of magnitude higher than the concentration of the saturated solution of carbon in the metal), which causes the growth of carbon filament.

As a result, the united theory of the formation of carbon on the metals of the VIII group was developed, allowing purposeful regulation of the reaction rate and the properties of the formed reaction products.

This provided the possibility to explain the positive role of carbonization of industrial slime and natural clay in the presence of the salts of iron group metals. The above-stated fact became the basis for the search for new methods to obtain adsorbents for gas purification and for the extraction of valuable substances from gases and waste waters, and resulted in the synthesis of carbon and silicon containing adsorbents. Through modification of the surface of natural mineral sorbents with carbon due to the pyrolysis of propane-butane mixture, carbon-containing composites are obtained; they combine the main advantages of mineral sorbents and active coal: high mechanical strength and hydrophobicity.

Catalytic synthesis methods are most frequently used to obtain CNT. A catalyst is often a complex of the matrix and the active phase [7]. Catalysts based on transition metals (composed of the particles of metals or their compounds, such as salts and oxides) are the most efficient ones in the synthesis of CNT. The choice of a matrix for the catalysts, its structure predetermine the properties of the final product. The development of new catalytic systems with different compositions of active phases and matrices allows obtaining CNT with different morphologies and properties.

In the present paper, we report some results of the studies of carbonization by means of electron microscopy (EM) and electron paramagnetic resonance (EPR) of chromite slime and natural clays of Kazakhstan, as well as SO_2 adsorption of carbonized clay. We also describe our achievements in obtaining electroconductive glass cloth based on carbon nanomaterials synthesized on metal oxides that were obtained by means of liquid-phase combustion.

CARBON NANOTUBES ON THE SURFACE OF CARBONIZED CLAY AND THEIR APPLICATION OF PURIFICATION OF GASES AND LIQUIDS

Carbonized natural clay as a sorbent for SO_2

Sulphur compounds (SO_2, SO_3, *etc.*) are present in many industrial emissions. These compounds have negative effects on living organisms and are poisonous for the catalysts of gas purification, decreasing their activity and operational lifetime.

It was demonstrated in [8–10] that carbonization of natural clay may improve its adsorption characteristics with respect to SO_2. An improvement of the adsorption properties of clay is explained by the formation of catalytically active carbon during carbonization. This carbon acts as a catalytically active centre. The mechanism of carbon formation on catalysts during thermal pyrolysis was studied by R. A. Buyanov with colleagues [6, 7].

In [11], samples of Narynkol clay were studied. The catalysts were prepared by clay impregnation with the solution of nickel acetate. The samples were molded in cylinders (d = 1–2 and 5–6 mm), dried at the ambient temperature (12 h), and then annealed at 150 (2 h) and 350 °C (3 h). Thermocatalytic synthesis of carbon-mineral catalysts and the sorbent was carried out through pyrolysis of propane-butane mixture at a flow rate of 80 cm^3/min within temperature range 400–800 °C for 10–30 min. After the pro-

cess was complete, argon was admitted into the reactor, and the samples were cooled to ambient temperature. Adsorption of SO_2 on the carbonized sample was studied in a flow reactor by means of temperature programmable desorption within temperature range 25–500 °C with the rate of 17 °C/min, and then chromatographic analysis of desorption products was carried out.

Interactions between SO_2 (0.08–0.1 vol. %) and carbonized samples (0.5 g) treated preliminarily in vacuum at $3 \cdot 10^{-5}$ Pa and then subjected to a temperature of 400 °C for 2 h continued at 150 °C in helium flow (40 cm^3/min). The effect of SO_2 on carbon filaments of the carbonized samples of clay and nickel-containing catalysts was studied by means of EM. The electron microscopic studies were carried out using an EM-125 K microscope (Elektron NPP, Urkaine) at the magnification of 120 000×.

The dependences of the specific surface area (S_{sp}) and the diameter (D) of carbon filaments on carbonization temperature (T) are shown in Table 1. One can see that the diameter of carbon fibres increases with an increase in temperature. Specific surface at first increases almost by a factor of 2 with an increase in temperature to 550 °C, but at higher carbonization temperature it decreases.

The effect of SO_2 on the filamentary carbon was studied through SO_2 adsorption at 150 °C on the carbonized samples of clay and nickel-containing catalysts containing filamentary carbon formed as a result of carbonization at 450 °C. After SO_2 adsorption, temperature programmed desorption was carried out within a temperature range from room temperature to 800 °C.

The microscopic images (Fig. 1, *a*) provide evidence that clay carbonization is accompanied by the formation of filamentary carbon nanostructure; its diameter depends on carbonization

TABLE 1

Effect of carbonization temperature (T) on specific surface area (S_{sp}) of clay and the diameter of carbon filaments (D) [12]

T, °C	D, nm	S_{sp}, m^2/g
–	–	4.6
400	8–20	4.9
450	20–22	6.3
500	22–30	8.0
600	30–50	9.2
700	60–70	8.4
800	80–100	4.9

temperature. Diameters increase with an increase in temperature to 600 °C (see Table 1). Moreover, it follows from EM images (see Fig. 1, *b*) that metal atoms are mainly located at the ends of nanotubes [12].

So, it was discovered, with the help of EM, that carbon is formed as a result of carbonization both on the surface of clay and on the surface of the catalyst. This carbon is the centre of SO_2 adsorption, and thus it promotes enhancement of the sorption characteristics of natural clay with respect to SO_2 [11]. The obtained results allow developing efficient sorbents for the removal of hazardous substances from industrial emissions.

Adsorbents based on clay and slime for water purification

The problem of obtaining pure water is one of the essential ecological problems worldwide and especially in Kazakhstan. Modification of natural clay and wastes from mining industry (slimes) with carbon is at present one of the main routes to broaden the assortment of adsorbents for waste water purification.

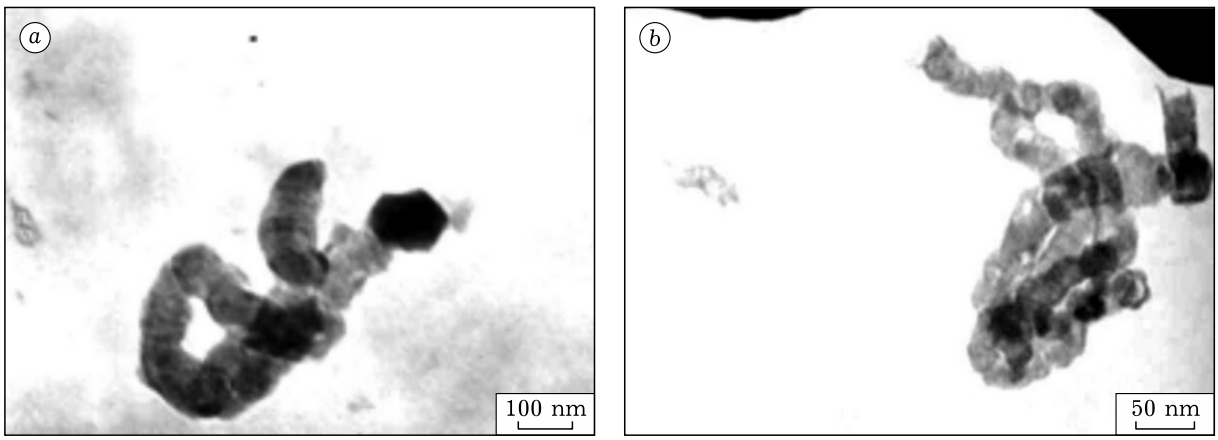

Fig. 1. EM images of clay samples carbonized at 600 °C: diameter of carbon filaments is 70 (*a*) and 40 nm (*b*) [12].

For the purpose of obtaining new adsorbents for water purification from organic compounds and the ions of heavy metals, bentonites modified with catalytic carbon were obtained and investigated.

Carbonization was carried out under jet conditions in a quartz reactor (d = 30 mm) using a propane-butane mixture. Investigation of carbonization of clay and the mixtures of clay with slimes was performed depending on temperature (450–900 °C), carbonization time (5–30 min) and the flow rate of gas admission (5–80 cm^3/min) [9, 10].

During carbonization, catalytic carbon and carbonaceous deposits were formed on the surface of slimes and clay, which promoted an increase in specific surface and mechanical strength of the material.

Of great interest are the works on carbonization of natural clay and slimes with the addition of oxides of iron group metals [6, 8], which are used as sorbents and refractory materials.

The EM image of carbonized chromite slime obtained in 1992 is shown in Fig. 2. One can clearly see the structure of chromite slime and carbon fibres, due to which the sorption capacity of the samples may increase. The image shows the formation of several carbon tubes from one centre, with metal atoms brought to the ends – the octopus effect [13].

Catalyst coking and the formation of carbonaceous deposits of different morphological kinds have been intensively studied [8, 11, 12], and definite advances have been made in this direction.

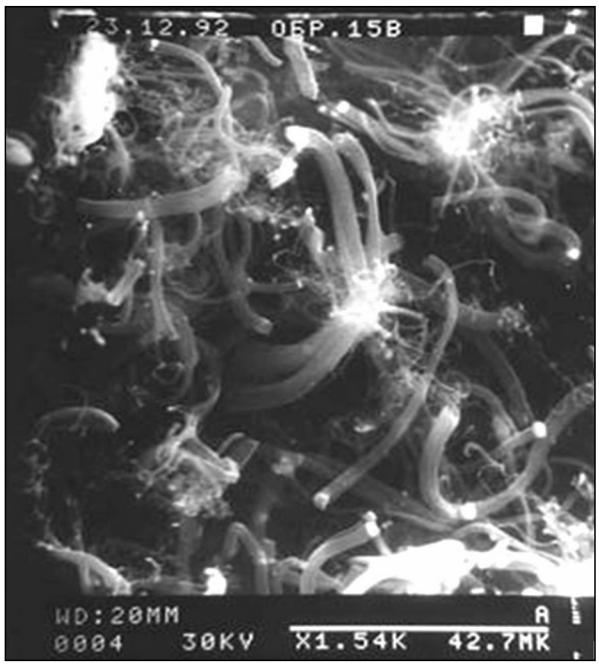

Fig. 2. EM image of chromite slime [13].

In [8], some results of EPR investigation of the processes of carbonization of a mixture of Turgay slime and Chilik clay (SC) are reported.

The Turgay slime is the waste product of aluminium industry. The samples of SC were prepared for carbonization as follows. A mixture of slime and clay at a ratio of 1 : 1 (SC-1:1) was thoroughly mixed, then water was added. The resulting thick mass was pressed through a sieve with 3 mm mesh, and dried. Thus obtained samples were carbonized. Carbonization of SC-1:1 samples was carried out for 30 min at 450, 550, 650, 750 and 900 °C. EPR spectra were recorded at a temperature of ~20 °C with the radiospectrometer RE-1301 (Smolensk Instrument Engineering Plant, Russia) [8].

In the EPR spectrum of the initial SC sample, the prevailing lines (Fig. 3) are those related to Fe^{3+} ions (ΔH = 840 E, g = 2.11 and ΔH = 70 E, g = 4.3).

The authors suppose that the EPR line with g = 4.3 is connected with isolated iron complexes, which interact with each other only weakly. These iron complexes are also in the strong field of low symmetry, conditioned by the rhombic distortions of the coordination note. The line with g = 2.0 appears due to the interaction of these iron complexes with the weak distortions of octahedral or tetrahedral symmetry of the coordination node (Table 2).

The authors state that sample carbonization at 450 °C leads to an increase in the line width by 33 %, while the intensity increases more than by a factor of 4.

CARBON NANOTUBES ON THE SURFACE OG GLASS CLOTH FOR THE DEVELOPMENT OF HEATING ELEMENTS

Catalyst nanoparticles on glass cloth, synthesized by means of solution combustion

The essence of solution combustion is in the reaction proceeding with the formation of metal oxide as the final product. Initial reagents are metal salts and organic compounds. Mixing is performed at the molecular level by preparing a solution of initial components. Then the mixture is subjected to high temperature at which self-ignition of the system occurs. This method allows obtaining a catalyst in the form of powder when the process is carried out in a liquid, or nanoparticles may be synthesized directly on the surface of a definite material (matrix) by means of impregnation or wetting the matrix material with the solutions of initial components [14].

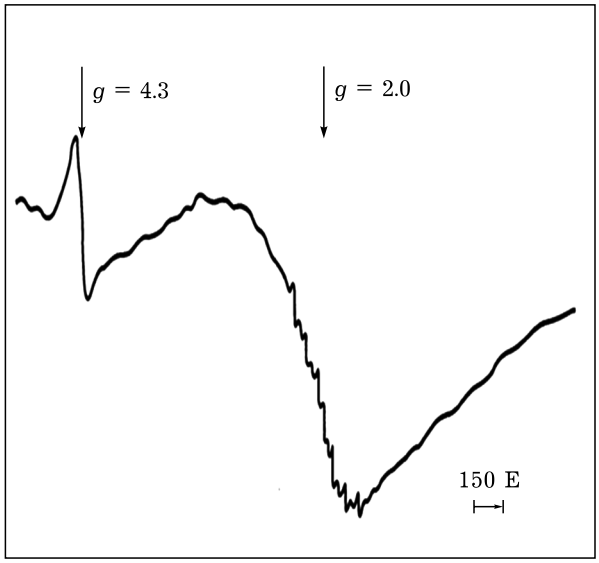

Fig. 3. EPR spectrum of SC-1:1 sample [8].

TABLE 2

Parameters of the magnetic resonance spectra of SC-1:1 samples at different carbonization temperatures [8]

Sample carbonization temperature	ΔH (g = 2.0), E	I (g = 2.2), r. u.
20	840 (70)*	0.6
450	1120 (70)*	2.7
550	1190	196
650	1480	28
750	1280	12.8
900	970	14

* The value of ΔH (g = 4.3) is shown in parentheses.

The glass fibre of KT-11 grade was used as the base for the catalytic system. To prepare impregnation solutions for catalyst deposition on the surface of glass cloth, cobalt nitrate ($Co(NO_3)_3 \cdot 6H_2O$) or ferric chloride ($FeCl_3 \cdot 6H_2O$) and glycine ($C_2H_5NO_2$) were used (chemically pure grade, without additional purification); distilled water was used as the solvent.

To prepare catalysts, a sample of glass cloth with the area of 5 cm^2 was preliminarily washed in 5 mL of ethanol, then dried at 100 °C in a muffle furnace. After that, the glass fibre sample was impregnated with the calculated amounts of aqueous solutions of cobalt nitrate (or ferric chloride) and glycine. The temperature of sample treatment in the air was 500–600 °C, which was sufficient to initiate the exothermal reaction of glycine decomposition proceeding with a temperature rise to 1200 °C. As a result of the reaction, ultrafine metal oxide particles are formed on the surface of glass cloth. The sequence of stages used to prepare a catalyst based on glass cloth is shown in Fig. 4.

Gaseous products (CO_2, N_2 and H_2O) that are formed during thermal treatment of the samples in the air play the part of dispersing agents and prevent catalyst sintering. The formed particles of the oxides of metals (cobalt, iron) are the catalysts for the growth of carbon nanotubes and nanofibres on the surface of glass cloth.

Synthesis and properties of carbon nanotubes on glass cloth

Chemical vapour-phase deposition from propane-butane-argon mixture was used for the synthesis of CNT.

The catalyst was placed into the reactor, and the quartz tube was blown with argon. When the necessary temperature was reached, a propane-butane mixture was supplied to the reactor. After synthesis was over, the flow of the propane-butane mixture was switched off. To cool the reactor to room temperature, argon was admitted into it. Gas flow rates were: Ar – 150 cm^3/min;

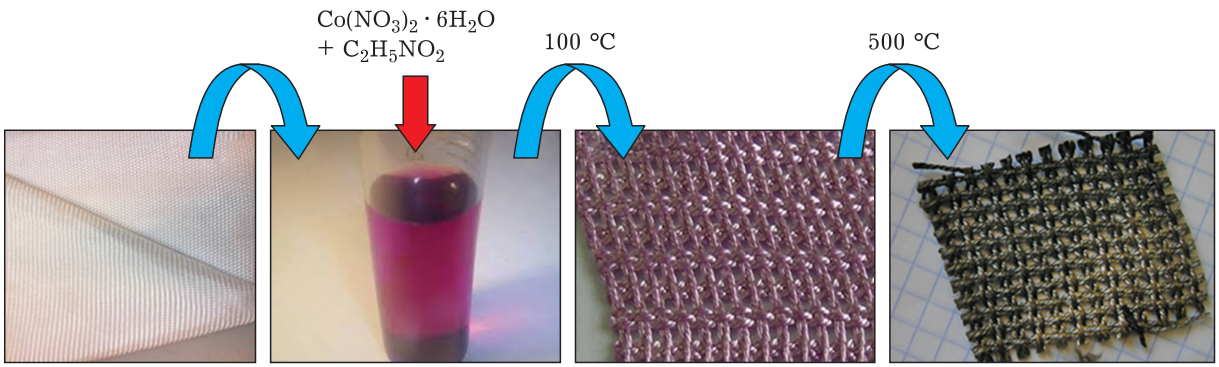

Fig. 4. Sequence of the stages of synthesis of metal oxide nanoparticles on glass cloth by means of solution combustion [15].

C_3H_8, C_4H_{10} – 150 cm^3/min. Synthesis temperature was 770–780 °C, synthesis time was 20 min.

Glass clothes with the nanoparticles of metal oxides were studied using various physicochemical methods. To determine the structure and composition of metal oxides on glass cloth, X-ray phase analysis was carried out. It was established that the reaction on the surface of glass fibres leads to the formation of Co_3O_4 and Fe_2O_3 particles 30 to 100 nm in size [16].

Catalysts based on glass cloth with an active component in the form of metal oxide nanoparticles were applied to the synthesis of CNT. Carbon nanotubes on glass cloth with the nanoparticles of cobalt and iron oxides were studied by means of SEM and TEM to determine the structure and morphology of unidimensional nanomaterials synthesized on glass cloth surface.

SEM and TEM images of CNT grown on the surface of glass cloth with the nanoparticles of cobalt oxide (Co_3O_4) are shown in Fig. 5.

One can see that many ultrafine cobalt oxide particles are formed on the surface of glass fibres with Co_3O_4. These particles are precursors of the active centres of CNT growth. Nanofibres and nanotubes are 23–25 nm in diameter and form a three-dimensional disordered structure. The obtained unidimensional carbon nanomaterials are strongly fixed on the glass fibre surface due to a strong adhesion of catalyst particles with the subsurface layer of glass fibres. Carbon nanotubes inosculate with each other and form a strong film on the surface of glass fibres (Fig. 6).

On the surface of glass fibres with Fe_2O_3, a large amount of ultrafine iron oxide particles are formed; they are precursors of the active centres of CNT growth. The data from TEM showed that CNT 15–17 nm in diameter were obtained as a result of synthesis. The amorphous carbon phase is also present in the sample in small amounts. For coiled nanotubes, the diameter is about 14 nm [18].

To reveal electroconductive characteristics of the obtained material, resistance was measured, and voltage-current characteristics were recorded (Fig. 7, *a*). Pure (non-modified) glass cloth is dielectric and does not conduct electric current. For glass cloth sample with the coating composed of CNT on Co_3O_4 with the area of 4.95 cm^2, resistance R is equal to 1.7 Ohm, the corresponding specific resistance R_{sp} is equal to 0.3636 Ohm · cm^2. The heating value of the glass cloth with CNT was also investigated (see Fig. 7, *b*).

Application of smart textile

On the basis of the obtained electrically conducting smart textile, a vest for a maquette of a soldier was manufactured. For this purpose, a sample of glass cloth with CNT, 24 cm^2 in area, was equipped with two electrodes made of copper wire and connected to the power supply unit, which was a galvanic element. To render an esthetic appearance and shape, a case was sewn from thermally stable material. The photographs of a soldier maquette with the vest based on the obtained electrically conducting smart textile are shown in Fig. 8 [18].

After maquette preparation, the material was tested for the heat value at decreased temperature, which was to simulate the properties of the obtained material under critical conditions. For this purpose, the sample was cooled to 0 °C with-

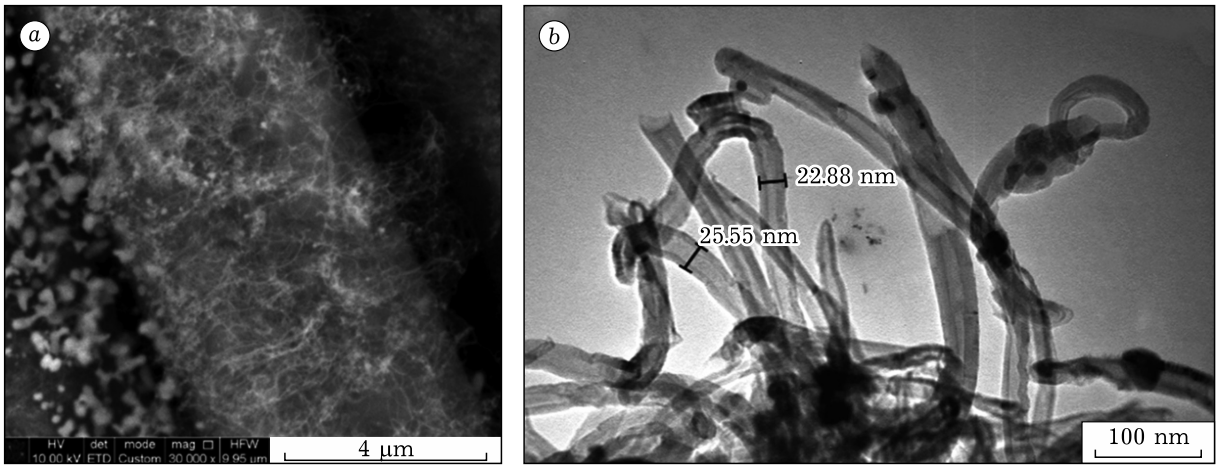

Fig. 5. Images of carbon nanotubes grown on glass cloth surface with the nanoparticles of cobalt oxide (Co_3O_4): SEM [17] (*a*) and TEM [18] (*b*).

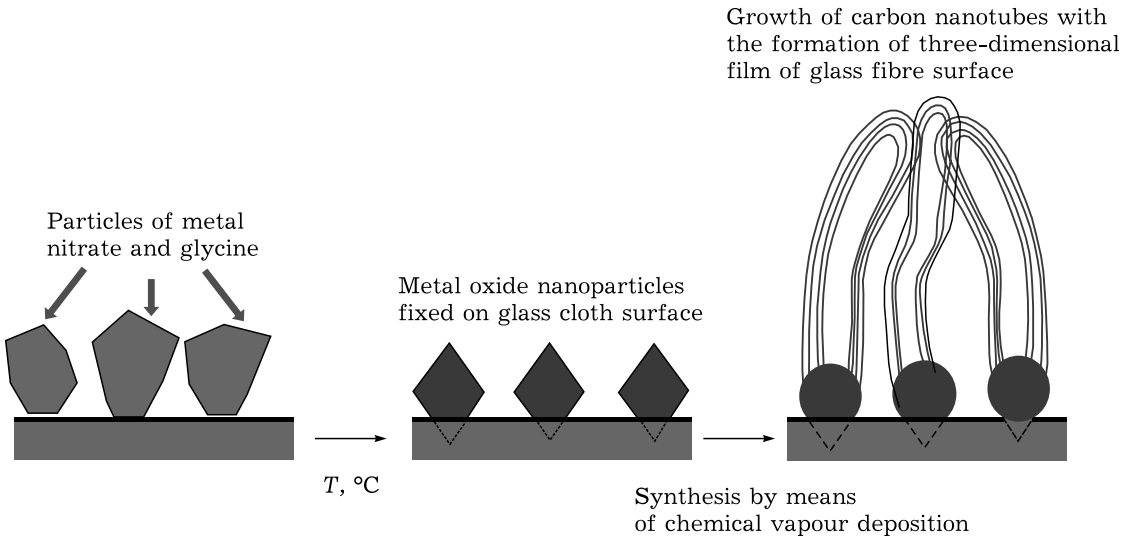

Fig. 6. Scheme of the formation of metal oxide nanoparticles on glass cloth surface and the growth of carbon nanotubes [19].

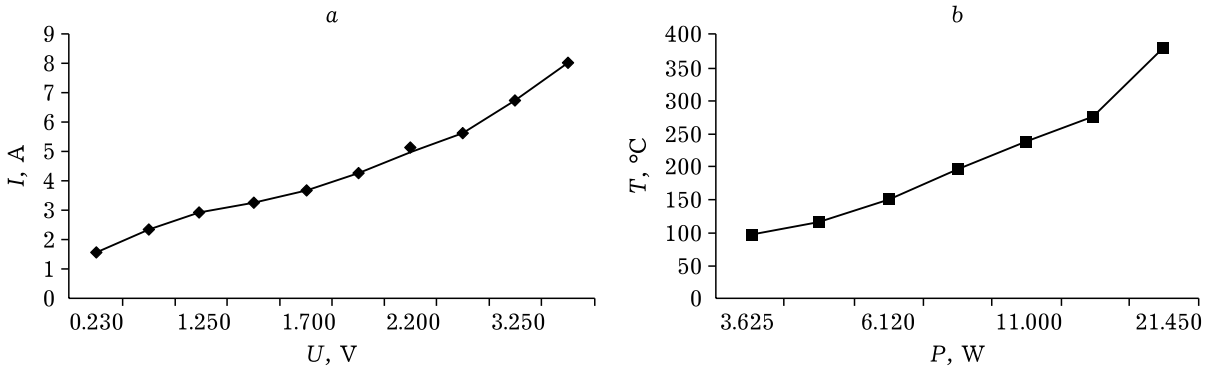

Fig. 7. Voltage-current characteristics of glass cloth with the coating made of CNT on Co_3O_4 (*a*); changes of the temperature of glass cloth sample with CNT depending on electric power *P* (*b*) [18].

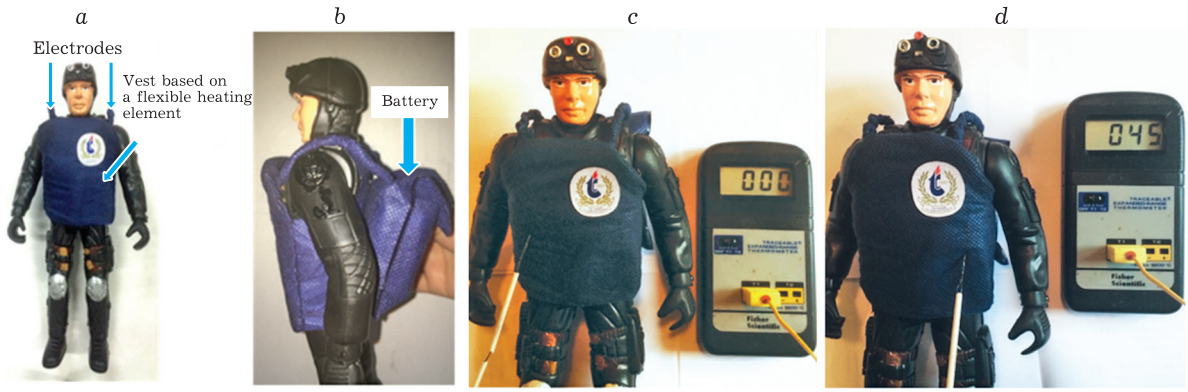

Fig. 8. Photographs of the maquette of a soldier (*a*, *b*) and the dynamics of changes of the vest temperature before switching it to the power supply (*c*) and after switching (*d*) [18].

out switching to the power supply, and then it was connected to the power supply unit. Sample temperature was measured with a chromel-alumel thermocouple with the help of a digital temperature sensor. Photographs of the maquette with the dynamics of vest temperature variation before and after its connection to the power supply unit are shown in Fig. 8, *c*, *d*. After the vest based on glass cloth with CNT was switched to the power supply unit, its temperature smoothly increased from 0 °C to the prescribed temperature. So, the test of the model of heated vest was successful. Thus obtained smart textile may be used as the basis to manufacture flexible heating elements and products to equip people under the temperature conditions that are critical for human organism: military men, mountain climbers, sportsmen, *etc*. [18].

CONCLUSION

A review of publications on the methods of obtaining and the areas of application of carbon nanotubes, their mechanical, optical, electric, and adsorption characteristics is presented.

Examples of carbonization of clay and the mixtures of clay with slimes are described. The studies demonstrated the efficiency of solution combustion method for the deposition of the oxides of transition metals (cobalt, iron) on glass fibre surface. The particles of metal oxides exhibited good catalytic activity during the synthesis of CNT by means of chemical vapour deposition from the propane-butane mixture.

It is demonstrated using physicochemical methods that the best CNT samples were obtained with cobalt oxide as the active component of the catalyst (CNT diameter was 23–25 nm); CNT diameter on glass cloth with iron oxide was 15–17 nm. The catalysts of this class are simple and convenient; their manufacture does not require much expense and effort. On the basis of glass cloth with coating composed of CNT, a maquette of flexible heating element was manufactured. As a result of these investigations, a simple and inexpensive technology for the production of electrically conducting cloth was developed. This smart textile may be used as the basis to manufacture flexible heating elements and products made of them.

So, as a result of the development of an integrated united theory of carbon formation on the metals of the VIII group, allowing purposeful regulation of process rate and the properties of reaction products, it turned out to be possible by present to synthesize a range of carbon and carbon-mineral composite materials with specific useful properties.

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