FULLERENES AND FULLERENE-LIKE STRUCTURES: THE BASIS FOR PROMISING MATERIALS

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New data on the production and properties of fullerene-containing materials are given. Information on their properties and possible applications as nanostructural materials, superconductors, sorbents, hydrogen cells, optical devices, photosensitive elements, electronic devices, coatings for endoprostheses, and molecular bearings are generalized.

Introduction. New allotropic forms of carbon, i.e., fullerenes, were discovered in 1985 [1]; however scientific interest in studying fullerenes was expressed after the invention of the method of their production in macroscopic amounts in 1990 [2] and particularly after the awarding of the Nobel Prize in chemistry for the discovery of fullerenes in 1996. The interest in investigations of fullerenes is determined by the diversity of new physicochemical phenomena occurring with the participation of fullerenes and by the exceptional prospects for application of a new class of materials created on their basis [3–5].

Fullerenes and Fullerene-Like Structures. *Fullerenes* are carbon clusters with an even (more than 20) number of carbon atoms forming three bonds with each other [6]. The atoms in the molecules of fullerenes are located on the surface of a sphere or a spheroid in the vertices of hexagons and pentagons. Examples of fullerenes are given in Fig. 1. Fullerenes with a number of atoms greater than 70 (for example, C_{76}, C_{78}, and C_{84}) are called higher fullerenes.

A C_{60} molecule has the highest symmetry and stability among the fullerenes. Each carbon atom in the molecule is located in the vertices of two hexagons and one pentagon. The valence electrons of each atom are in sp^2-hybridized states similar to the states of the electrons in graphite [7].

Fullerenes are practically insoluble in polar alcohol-type solvents, in acetone, and in tetrahydrofuran; they are weakly soluble in normal alkanes (pentane, hexane, and decane). They are most soluble in liquids for which the ratio of the specific enthalpy of evaporation to the specific volume of the molecule of the solvent is close to the corresponding value for the C_{60} molecule (~100 cal/cm^3) [8], for example, in benzene and toluene. The behavior of fullerenes in solutions is of a complex character. For example, the solubility of fullerenes in normal decalin made up of *cis* and *trans* forms in the proportion 3:7 is noticeably higher than the solubilities in each of the forms taken separately.

Fullerene molecules are strong oxidizers since they possess high electronegativity and are capable of adding up to six free electrons.

Carbon nanotubes are graphene nets rolled into tubes, and they can be open or closed (Fig. 2), single-walled, double-walled, or multi-walled with a distance of about 0.35 nm between the walls. In addition to hexagonal cells characteristic of the structure of graphite there are pentagonal cells at the ends of closed nanotubes.

The band structure of single-walled carbon nanotubes is determined by their diameter and by the angle between the direction of rolling of the nanotube and the direction in which the neighboring hexagons
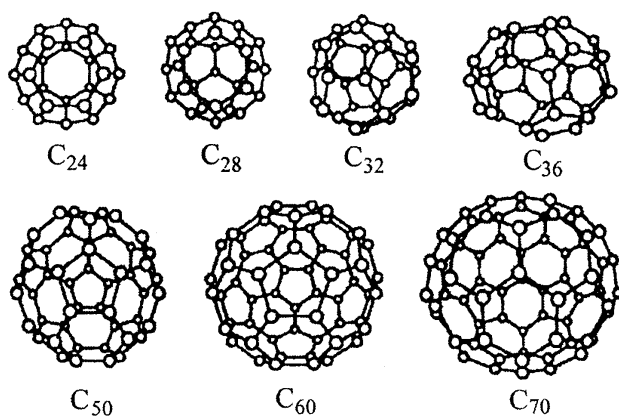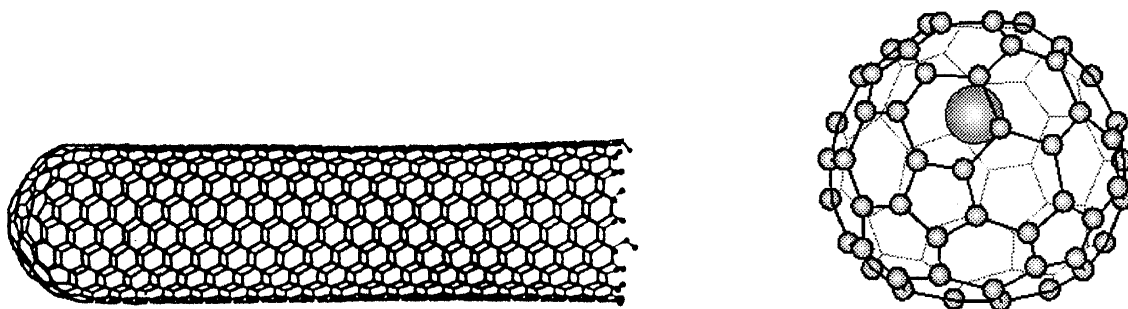
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Fig. 1. Examples of fullerenes.

Fig. 2. Carbon nanotube closed at one end.

Fig. 3. Molecule of an endofullerene.

have a common side. Carbon nanotubes can be conductors or semiconductors. The external magnetic field is capable of changing the forbidden gap of a semiconductor tube or even converting it into a conducting state. The conductivity of carbon nanotubes doped with potassium and boron is more than 30 times higher than the conductivity of pure nanotubes at 300 K [9]. Carbon tubes are extremely strong and elastic. Their Young modulus is 0.40–3.7 TPa [10].

Endofullerenes are molecules in which inside the fullerene molecule there is one or several noncarbon atoms (Fig. 3).

Ultradispersed carbon aggregates (UDCA) are associates of carbon clusters. Clusters can have different structures: chain, single-layer, multilayer ("onion-type"), etc.

Production of Fullerene-Containing Materials. *Synthesis of fullerenes and ultradispersed carbon aggregates.* The most efficient methods of production of fullerenes are technologies using electric-arc plants [2]: fullerenes are obtained as a product of thermal sputtering of a graphite anode in a helium atmosphere at a pressure of 100–150 torr.

In the Republic of Belarus, an automated technological complex for the production of fullerenes that allows the production of tens of grams of fullerenes a shift has been created [11]. The creation of the complex required the development of new technical solutions throughout the entire technological chain — from the selection of raw material, the production of fullerene-containing soot, and the separation of fullerenes and ultradispersed carbon aggregates to the packing, storage, and diagnostics of the products obtained (Fig. 4) [12–15]. Optimization of the technology at the stage of production of fullerene-containing soot is ensured by the selection and automatic stabilization of the arc-discharge regime, the distance between the electrodes, and also the pressure of the inert gas and its circulation regime. The fullerene output depends on the synthesis temperature, the frequency of collisions of small carbon clusters, and the conditions of cooling of synthesized molecules.
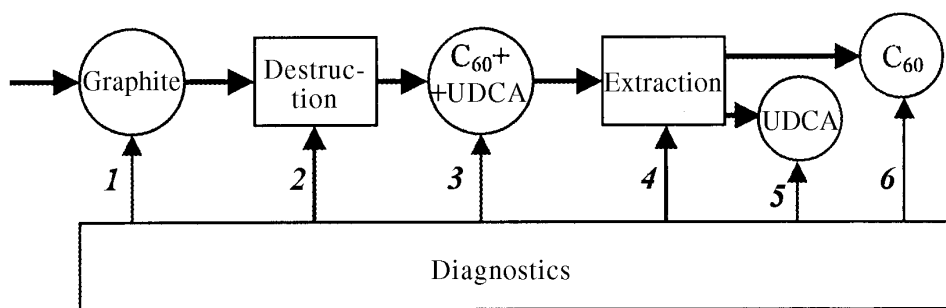
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Fig. 4. Structural scheme of a technological complex for production of fullerenes: 1, 3, 5, and 6) operations of monitoring of the quality of materials; 2 and 4) operations of monitoring of technological regimes.

Synthesis of nanotubes. The most wide spread method is that of production of carbon nanotubes in an arc-discharge plasma, burning in a helium atmosphere, at technological plants for production of fullerenes [12, 16, 17]. However, to produce nanotubes use is made of other arcing regimes: low densities of the arc-discharge current, a higher pressure of helium (~500 torr), and larger-diameter cathodes. An increase in the output of nanotubes in sputtering products can be attained by introducing a catalyst into the graphite rod and also by changing the pressure of the inert gas and the regime of sputtering. Mixtures of metals of the iron group make efficient catalysts [18]. In the cathode deposit, the content of nanotubes is as high as 60%. The 40-μm-long nanotubes formed grow from the cathode perpendicularly to its surface and combine into cylindrical bundles about 50 μm in diameter.

Production of metal-fullerene films in vacuum. Metal-fullerene films are usually produced by the method of thermal sputtering in vacuum [19]. Since fullerenes begin to sublimate at temperatures below 700 K and the temperature of evaporation of metals is much higher, two evaporators are used to produce the films.

The concentration of the fullerenes in films is determined by the velocities of arrival of the components (atoms and molecules) that are regulated by the temperature of the evaporators and their location with regard to the substrate. The temperatures of the evaporators are selected from experimental dependences of the evaporation rate on the temperature.

Electrochemical deposition of metal-fullerene films. To produce thick metal films with a low (less than 1%) content of fullerenes one can use the method of electrochemical deposition in which fullerite powder or a fullerene solution is mixed with an electrolyte [20, 21]. To increase the homogeneity of the electrolyte an ultrasonic vibrator is used. The technological parameters are the composition of the electrolyte, the density and the regime of the current, the power, the pulse length, and the frequency of the attendant laser radiation.

Production of polymer-fullerene materials. Polymer-fullerene materials are produced by the following methods:

(1) joint sputtering and deposition of components;

(2) mixing of the fullerite powder with a polymer melt and subsequent cooling of the mixture obtained;

(3) mixing of the solution of fullerenes with the solution of polymers and subsequent drying [22, 23].

Depending on the temperature, the type of solvent, the ratio of the amounts of polymer, fullerene, and solvent, and the degree of mixing, materials of different types can be formed. The porosity, internal mechanical stresses, adhesion, and also the size of fullerene associates and sites of their attachment in polymer chains depend on the regime of drying.

Production of endofullerenes. Endofullerenes can be formed in several ways:

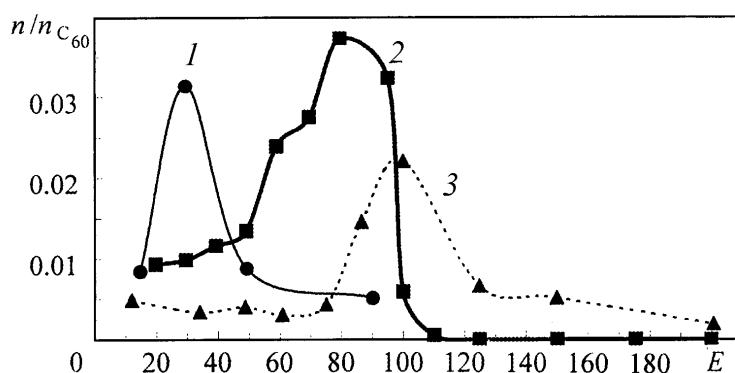(1) by introduction of ions of foreign atoms in ion implantation;

Fig. 5. Ratios of the number of endofullerenes to the number of hollow fullerenes vs. energies of the ions used for bombardment of a fullerene film: 1) Li@C$_{60}$; 2) K@C$_{60}$; 3)Rb@C$_{60}$. E, eV.

(2) penetration of small particles (protons, deutrons) inside fullerenes with subsequent addition of electrons;

(3) capture of foreign ions or atoms by fullerenes for large amplitudes of oscillations of the atoms of the fullerene molecule.

The method of ion implantation is efficient in the production of the endofullerenes N@C$_{60}$, Li@C$_{60}$, K@C$_{60}$, Rb@C$_{60}$, Na@C$_{60}$, and La@C$_{60}$ [24–26]. To obtain ions, a standard ion source or a glow discharge is used. The dependences of the ratio of the number of endofullerenes to the number of hollow fullerenes on the energy of the ion beam are given in Fig. 5 and have a pronounced maximum with a certain energy of the ions, which indicates the existence of an "energy window" for the formation of endofullerenes. The existence of the maximum can easily be explained proceeding from simple physical considerations. With low energies ions are unable to overcome the energy barrier preventing their penetration into the fullerene. When the energies are very high the collision of an ion with a fullerene molecule leads to the destruction of the latter. For ions with a larger radius the energy required for penetration into the molecule is higher; therefore, the energy that corresponds to the maximum of the output of fullerenes is also higher.

Hydrogenation of fullerenes. Hydrogenation and deuteration of crystalline fullerene can be performed on a standard high-pressure plant at a pressure of 1.0–2.5 MPa and a temperature of about 673 K [27]. Preliminary degassing of the fullerene is carried out by heating it to 500 K in vacuum (~1 Pa). To attain a more complete hydrogenation and produce specimens with a homogeneous distribution of the components, the process is conducted in a cyclic thermal regime, i.e., the specimen is heated to 673 K under hydrogen pressure and is kept at this temperature, then the reaction mixture is cooled down to room temperature, and the cycle is repeated several times.

Prospects for the Application of Fullerene-Containing Materials. *Nanostructural materials.* The interim position occupied by nanoparticles in transition from crystals to individual molecules and atoms predetermines their special properties in comparison to crystals, molecules, and atoms. Nanostructural materials possess unique physical properties, which has led to a highly promising applied-research direction in investigations by physicists and chemists in recent years [3, 28, 29].

One type of nanostructural materials is represented by metal-fullerene films deposited in vacuum [30, 31]. Already with low (~1%) concentrations of fullerenes in titanium–fullerene films structure-forming particles ("grains") have a round shape and a size of 15 to 40 nm. Figure 6 shows the structure of the surface of copper–fullerene and titanium–fullerene films obtained using an atom-force microscope. The decrease in the size of the grains as the fullerene concentration increases is attributed to the low mobility of C$_{60}$ molecules on the surface of the growing film and to their limiting the migration of the metal atoms. Thus, the addition of fullerenes to melts can be used as a method of creation of nanostructural materials.
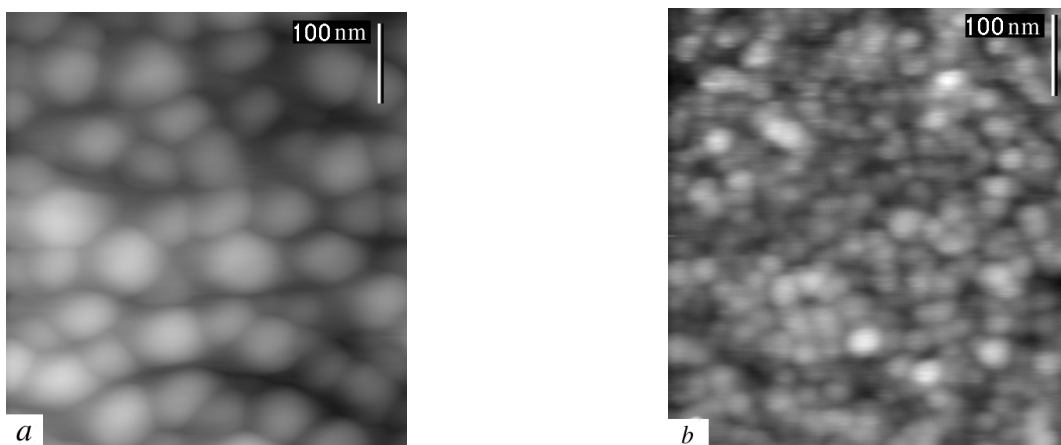
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Fig. 6. Pictures of the surfaces of metal–fullerene films obtained using an atomic force microscope: a) copper–fullerene; b) titanium–fullerene.
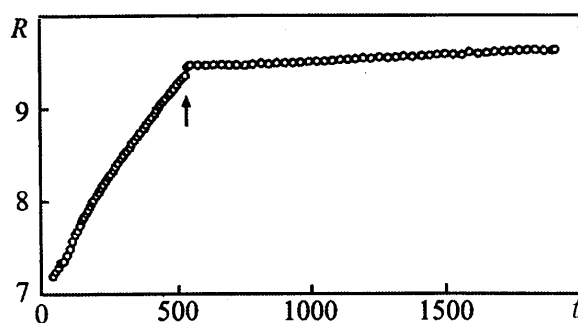

Fig. 7. Electrical resistance of a C_{60} film vs. time with increase in the pressure from 10^{-4} Pa to atmospheric and with subsequent pumping (instant of time marked by an arrow) to 10^{-4} Pa. R, kΩ; t, sec.

Superconductors. Fullerides of alkaline metals A_3C_{60} (A = K, Rb, Cs) possess superconducting properties. The temperature of the phase transition depends on the lattice constant of the fulleride. The temperature of the superconducting transition for the system Rb–Tl–C_{60} is rather high — it exceeds 40 K; for the system Cu–C_{60} it exceeds the boiling point of nitrogen [32]. Thus, metallofullerenes with simple composition are the most high-temperature superconductors apart from ceramics with complex composition. Unlike complex copper oxides, they are isotropic superconductors, i.e., their superconducting properties are the same in all crystallographic directions. The problem of theoretical description of the superconductivity of both metallofullerenes and traditional high-temperature superconductors based on copper oxides has not yet been solved.

Sorbents. Fullerenes may act as sorbents since they possess a high sorption power. This is indicated by changes in their properties in different gas media. The resistance of fullerene films produced in vacuum increases when they are exposed to air (Fig. 7) [33]. The physical bases of the evaluation of sorption properties of carbon materials and the identification of the presence of fullerene-like structures in them have been developed in [34].

Accumulators of hydrogen. Progress in the mass-scale use of hydrogen as an environmentally safe source of energy depends in many ways on the solution of the problem of an efficient method of its storage and transportation. According to the requirements of the International Energy Agency, storage systems must contain at least 5 wt.% of hydrogen and release it at a temperature no higher than 373 K.

Carbon materials appear at present to be the most promising for use as hydrogen-accumulating matrices, especially in connection with the discovery of fullerenes which could contain up to 7.7 wt.% of hydrogen

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TABLE 1. Characteristics of the Sorption of Hydrogen by Carbon Nanomaterials

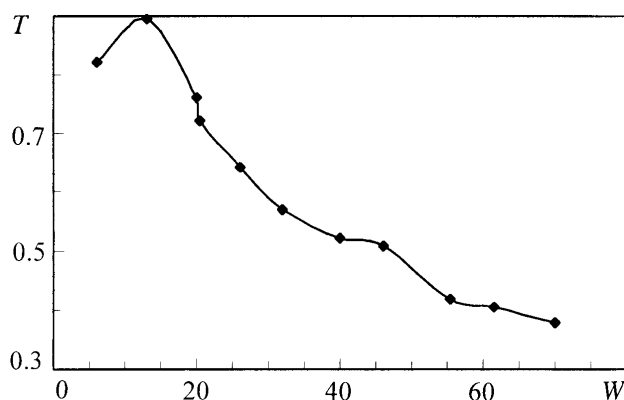| Material	Maximum capacity, wt.%	Temperature, K	Hydrogen pressure, MPa	Reference
Single-walled nanotubes	8.25	80	7.18	[36]
	5–10	133	0.04	[37]
	4.2	300	10–12	[38]
	3.5	77–300	5–10	[39]
	6.5–7	300	0.1	[39]
Graphite nanofiber	11–66	300	11	[40]
	10–12	373	11	[41]
Graphite nanofiber + K	14	473–673	0.1	[42]
Graphite nanofiber + Li	20	473–673	0.1	[42]

Fig. 8. Transmission of light by C_{84}-tetrahydronaphthalene at a wavelength of 1.064 μm vs. input energy. W, μJ.

(0.92 m^3 of H_2 per kg of $C_{60}H_{60}$) after the hydrogenation of all double bonds [35]. However, no one has so for managed to obtain such a compound. The produced fullerene hydrides $C_{60}H_x$ (the maximum composition is $C_{60}H_{36}$) contain up to 6 wt.% of hydrogen. The characteristics of the sorption of hydrogen with nanotubes and nanofibers are still higher (see Table 1).

Optical devices. The effect of nonlinear transparency of fullerene-containing solutions and compounds [43, 44] opens up opportunities for their use as a basis for optical locks — limiters of the intensity of laser radiation of the visible and near-infrared ranges. The threshold intensity that characterizes an optical lock based on fullerenes is several times lower than the corresponding value for materials traditionally used for such purposes (indantron, chloroaluminum phthalocyanine, and others). For C_{60} the region of optical limitation lies in the range of wavelengths of 400–700 nm. The regions of optical limitation of higher fullerenes (C_{70}, C_{78}, C_{84}) lie in the visible and near-infrared regions. Figure 8 gives the dependence of the transmission of light by a fullerene-containing polymer film on the input energy [45]. An important property of fullerene-containing optical limiters is their quick operation — from several femtoseconds.

The production of dynamic holograms on the basis of fullerenes showed their prospects for use in devices for processing of optical information and inversion of the wave front [46]. The high degree of non-linearity of a medium with fullerenes can be used for the compression of an optical pulse in the nanosecond region of duration as a function of the input energy [47]. Fullerene-containing materials can be used in de-vices for image reconstruction.

Photosensitive elements. Fullerites possess photoconductivity in the spectral range that is optimum for creation of solar cells [48]. The spectrum of photoabsorption of fullerite films lies in the range of wave-
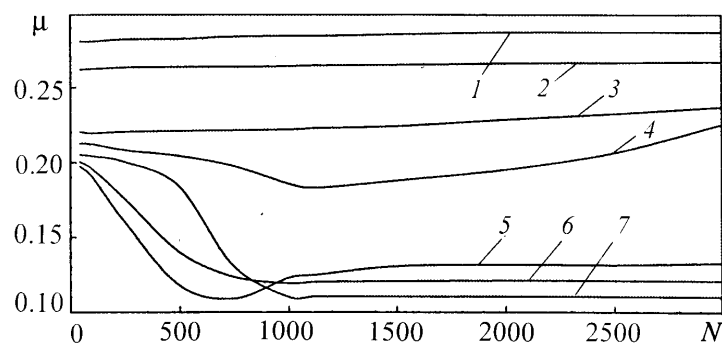
Fig. 9. Coefficients of friction of the titanium–glass couple with different boundary lubrications vs. number of friction cycles: 1) graphite powder in toluene; 2) graphite powder; 3) without a lubrication; 4) toluene; 5) toluene solution of fullerenes, 0.72 mg/ml; 6) the same, 1.08 mg/ml; 7) the same, 2.15 mg/ml.

lengths of 280–680 nm, and the quantum yield which represents the probability of formation of an electron-ion pair during the absorption of one photon is 0.9.

Electronic devices. The polymerization of fullerenes under the action of luminous radiation and the formation of a film insoluble in organic solvents make it possible to use fullerenes as a new material for photoresistive masks. Using a mask of polymerized C_{60} film, high resolution (approximately 20 nm) has been attained in etching of silicon with an electron beam [49].

Carbon nanotubes seem to be very promising for devices of electronics and nanomechanics [50]. On a single nanotube, one can manufacture a field-effect transistor [51], while systems of nanotubes can be used as the cold cathodes of cathode-ray tubes [52].

Molecular bearing. The investigations of the coefficient of friction between the titanium needle–glass surface couple carried out by us have shown a decrease in the coefficient of friction when the toluene solution of fullerenes was used as the boundary lubrication. Figure 9 gives the dependences of the coefficient of friction of a titanium–glass couple with different boundary lubrications (toluene, graphite powder, a toluene solution of fullerenes, a mixture of graphite powder and toluene) on the number of friction cycles. The decrease in the coefficient of friction is related to the presence of the molecules of fullerene in toluene (an unsaturated solution of C_{60} was used). Thus, the molecules of fullerene act as a molecular bearing. This conclusion is indirectly confirmed by general data on the properties of C_{60}, namely: high elasticity and strength, low surface energy, weak intermolecular interactions, and a quasispherical shape.

Fullerene C_{60} has been investigated in the form of a solid-lubrication coating [53] and in the form of admixtures to liquid lubricants [54]. The addition of fullerene-containing soot to industrial oil leads to a decrease in the coefficient of friction to 0.02 [55]. The composite materials UDCA-polytetrafluoroethylene have a low coefficient of friction (0.1–0.2). The investigation results point to the prospects for application of C60 and UDCA to solve different tribological problems.

Coatings for endoprostheses. For manufacture of endoprostheses wide use is currently made of stainless steel, alloys of cobalt with chromium, and also titanium and its alloys. A particularly common titanium alloy is Ti-6Al-4V, which is most frequently used in the manufacture of implants subjected to mechanical loads (for example, in thigh and knee joints). However, the difference of the prosthesis material and the bone in rigidity (hardness, elasticity) leads to a change in the tension of the skeleton, which causes the resorption of the implant and its failure. Furthermore, the prosthesis material undergoes not only mechanical loads. It is also prone to corrosive damage in a rather aggressive medium. The corrosion rate of prosthesis materials is very low; however in contact with tissues of the organism, even small amounts of corrosion products can be

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hazardous since they can lead to the initiation of pathological immune processes, the growth of connective tissue, and infection [56, 57].

Carbon is the main element in living organisms and is widely used for medical-biological purposes without causing substantial negative reactions. Investigations *in vitro* with tissues of mice and tests *in vivo* on sheep [58] have shown a very good biocompatibility of carbon films [59, 60]. In interaction of carbon clusters with living tissue and blood, unlike metals, active ions do not penetrate into the organism. No harmful side reactions of the immune system occur even when rather large carbon particles break away from the implant.

The results of the investigations performed on the structure, phase composition [61, 62], and chemical stability of composite titanium-fullerene coatings in acidic and alkaline media and also the structural-morphological characteristics of blood cells in their interaction with fullerenes [63, 64] indicate that fullerene-containing materials can be used in biomedicine, in particular, titanium–fullerene films can be used for coatings of endoprostheses.

Conclusions. The development of methods of production of fullerenes and fullerene-containing structures and the study of their properties are being continued, as is the search for ways of using them in electronics, biology, medicine, and other fields of human activity. Fullerene materials science has only taken its first steps. The properties of the already produced fullerene-containing materials instill hopes for their wide use and the appearance of still newer materials.

NOTATION

UDCA, ultradispersed carbon aggregates; $N@C_{60}$, $Li@C_{60}$, $K@C_{60}$, $Rb@C_{60}$, $Na@C_{60}$, and $La@C_{60}$, molecules of endofullerenes; E, ion energy, eV; T, transmission of light; W, energy, J; $n/n_{C_{60}}$, ratio of the number of endofullerenes to the number of hollow fullerenes; R, electrical resistance, kΩ; t, time, sec; μ, coefficient of friction; N, number of friction cycles.

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