

FORMATION AND CONVERSION OF CARBON NANOSTRUCTURES UNDER RADIATION

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Consideration has been given to the current status of research on the use of radiation techniques for synthesizing new carbon nanomaterials based on fullerenes, carbon nanotubes, and graphene sheets and their derivatives, which are promising for practical application due to their unique properties. An analysis has been made of the existing experimental and theoretical works on studying different processes induced by the action of radiation on carbon nanostructures (melting of cross-linking carbon nanotubes and formation of molecular junctions between them, polymerization of fullerene layers, formation of new structures, radiation chemistry of fullerenes, and others).

Keywords: carbon nanostructures, radiation, fullerenes, carbon nanotubes, graphene sheets, electron beam, ion beams.

Introduction. Nanotechnology is one of the most actively developing branches of science and technology [1]. Emerging at the interface of electronics, physics, biology, and materials science, it studies the possibility of creating new functional materials, devices, and systems of nanometric size. Considerable progress has been made at present in obtaining and investigating different nanoobjects and nanostructures, which is primarily due to the development of tunnel electron and scanning microscopy, atomic-force microscopy, x-ray and optical methods with the use of synchrotron radiation, optical laser spectroscopy, radio-frequency spectroscopy, Mössbauer spectroscopy, etc. [2].

Carbon nanostructures (CNSs), such as fullerenes, carbon nanotubes (CNTs), and graphene sheets and their derivatives occupy a highly important place among nanomaterials due to their unique properties that are promising for practical application [3–5]. The reason for the fairly wide diversity of the structures in question is the unique capability of carbon for forming electron configurations intermediate between sp^2 and sp^3 and very similar energetically [6]. The fractional degree of hybridization is conditioned by the curvature of the structural skeleton (cage) and the formation of a closed shell.

Fullerenes first synthesized in laser evaporation of graphite [7] represent hollow closed shells of a spherical or more intricate 3D shape which are constructed from hexa- and pentagonal carbon cells (see Fig. 1). Their most widespread representative is fullerene C_{60} , which is usually formed in larger quantities than other fullerenes. Fullerenes enter into chemical reactions with other substances, forming many derivative compounds [8, 9]. They, in particular, include endohedral fullerenes containing atoms or molecules inside the carbon skeleton [10], substitution fullerenes in which one or a few atoms of the carbon lattice are replaced by the atoms of other chemical elements [11], fullerenes incorporated into polymer chains [12], fillerites representing fullerene-based solid-state structures [13], and others. Also, hybrid carbon nanomaterials, including fullerenes, nanotubes, and graphene sheets, have been synthesized [14–16].

The class of carbon nanotubes is much superior to the class of fullerenes in diversity of structures and physicochemical characteristics [17]. An ideal nanotube can be considered as a graphene sheet rolled into a cylinder, forming no seam in gluing, and terminating in hemispherical peaks. Unlike fullerenes representing the molecular form of carbon, CNTs combine the properties of molecules and a solid and can be considered as a state of the substance intermediate between the molecular state and the condensed one. Depending on formation conditions, they can be single-layer and multilayer [18], T-, X-, and Y-shaped [19], contain various fillers inside [20], be part of different polymer and composite materials [21], etc. Nanotubes possess an abnormally high tensile and bending strength (in multilayer CNTs, the ultimate tensile strength attains 150 GPa [22]). This substantially improves the mechanical characteristics of polymer composites on introduction of even a small number of CNTs into them.

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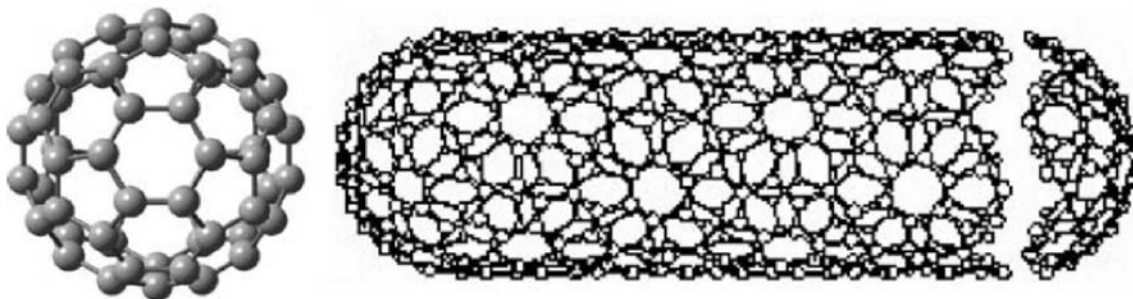


Fig. 1. Structure of a fullerene molecule and a single-layer carbon nanotube.

The methods of producing fullerenes and carbon nanotubes are quite diverse. The main ones among them include arc discharge with the use of graphite electrodes [23, 24], laser irradiation of the graphite surface [25], thermochemical decomposition of hydrocarbons on a metal-based-catalyst surface (CVD method) [26], and combustion of aliphatic and aromatic hydrocarbons at large excess-fuel coefficients [27]. The methods of physical action on the gas, which give rise to fullerenes, can also include ionizing radiation [28, 29]. The production of fullerenes and CNTs in large quantities [30, 31] has opened up the way for their practical application. They are currently used in optical and electronic devices [32, 33], CNS-based polymers [12, 34], composite materials [35], hydrogen-storage systems [20], biosensors [36], devices of focusing of ion beams [37], etc.

Graphene represents a single layer of carbon atoms packed into a dense hexagonal crystalline structure which can be considered as an ideal realization of a two-dimensional material [38]. The recent success in the technology of separation of graphite samples into single-crystalline films [39–41] has made it possible to investigate this exotic system experimentally. As the measurements show, individual graphene sheets have extraordinary electron transport properties, which makes it possible to use this material for replacement of today's silicon transistors in microelectronics [42, 43].

The use of radiation techniques is of considerable importance in investigating and producing nanostructured systems [44, 45]. Methods based on the radiation action of the beams of high-energy charged particles (electrons and ions) and of x and γ rays on the starting material are widely used in manufacturing ion-track membranes [45], 3D nanostructures [46], nanostructured glasses [47], metallic nanoparticles [48], nanocomposites [49], heat-resistant polymers [50], etc.

In the present work, we give a review of the current status of research on the use of radiation techniques for production and transformation of carbon nanostructures. The available experimental and theoretical data on the interaction between radiation and CNSs that leads to their modification and destruction or to the formation of new structures are analyzed. Most of the works in question investigate the action of an electron beam on different carbon materials in an electron microscope, which makes it possible to observe *in situ* dynamic processes at the atomic level.

Formation of Fullerenes and CNTs under Radiation. The radiation method of CNS production compared to other methods has not gained wide acceptance because of the technical difficulties in its implementation and low efficiency of the process. The latter is attributed to the relatively low temperatures induced by the exposure of the initial carbon samples to radiation [51, 52] and accordingly low rates of the chemical reactions responsible for the formation of the structures in question. Therefore, the available investigations in this line are few in number and are mainly methodological in character.

As the experiments show, the radiation action of a high-energy-electron beam on the carbon surface (soot, graphite, carbon films, etc.) gives rise to different single- and multilayer hollow structures corresponding to fullerenes or onions in shape and size [53–57]. This process can be represented as the scaling of graphene fragments off the surface of a carbon sample due to the breaking of bonds between carbon atoms and to the formation, by radiation, of defects in the carbon structure followed by the turning of these fragments into small spherical shells, as is shown in Fig. 2. The intensity of the process increases with electron energy in the beam. The threshold energy below which no structural changes are observed in the sample lies in the interval 40–80 keV [53].

The formation of closed carbon shells corresponding to fullerenes is a rather complex and not clearly understood process [58]. There is a great number of models at present that offer versions of assembly of fullerenes from

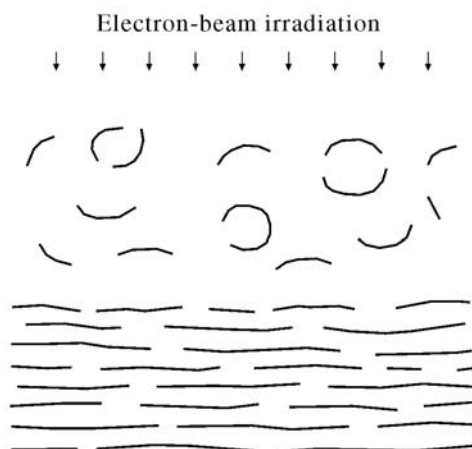


Fig. 2. Formation of fullerene-like shells above the graphite surface under electron-beam irradiation.

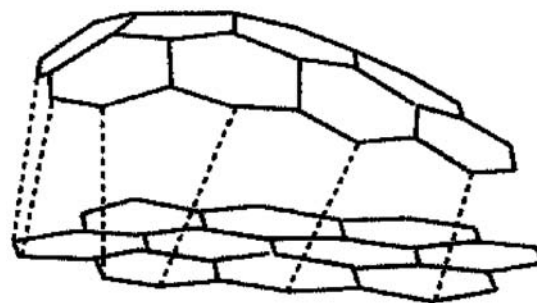


Fig. 3. *Zipper* variant of the coagulation mechanism of formation of fullerenes from two aromatic fragments.

aromatic predecessor clusters of every kind, which are often no different from each other, in practice. Quite detailed reviews in which different mechanisms of formation of fullerenes and different methods of their modeling are analyzed are given in [59–61].

The process of formation of fullerenes in hydrocarbon combustion includes the nucleation of aromatic molecules, further buildup in their structure due to the addition of acetylene molecules, and coagulation in which fullerenes are assembled from different aromatic fragments [62, 63]. Analogous stages are characteristic of the case of laser evaporation of graphite where closed carbon structures are formed from a hot carbon vapor [61]. Since the reaction of addition of an acetylene molecule to an aromatic particle has an activation energy of 42.2 kJ/mole, which is nearly two orders of magnitude higher than the corresponding quantity for the reaction of coagulation [63], it is the coagulation mechanism that becomes dominant at low temperatures of the process, which are characteristic of the radiation action on carbon samples. In the irreversible *zipper* variant of this mechanism, the process begins with the formation of a sandwich-like configuration of two aromatic fragments to establish C–C bonds forming hexa- and pentagonal cells between them and to localize them in positions most favorable energetically [64] (see Fig. 3).

Fullerenes obtained from the electron-beam irradiation of a graphite surface possess a higher radiation stability than C_{60} molecules inside fullerite crystals and films on a silicone surface. The process of formation of fullerenes in irradiation of the graphite surface was observed in a high-resolution electron microscope with an electron energy of 1.25 MeV and a current density of 200 A/cm^2 [53]. On the other hand, destruction of C_{60} crystals on the silicon surface was recorded even at an electron energy of 80 keV [65]. The stability of fullerenes attached to the graphite surface by van der Waals forces is attributable to the occupation of vacancies (which are formed in the fullerene's carbon lattice by the action of fast electrons) by carbon atoms captured from the damaged graphite surface. The rapid rotation of fullerenes contributes to the process.

The formation of fullerene molecules is also recorded in the wake of high-energy ions transmitted by the irradiated carbon sample [66–68]. A simplified theoretical model of the process which is based on the ion-track mechanism of formation of fullerenes in a condensed carbon material considers a short thin core in the wake of an ion; the core consists of strongly ionized carbon representing a dense nonideal gas. Nucleation and further growth of the aromatic structures, finally giving rise to fullerene molecules, occur on the core's exterior surface.

Onions of a unique structure in the form of hollow concentric shells enclosed in each other can be separated into an individual group in the family of fullerenes [69]. The formation of onions under the radiation action of an electron beam on different carbon materials (soot, amorphous carbon, and graphite) has been observed in the experiments of [53–55, 70]. The radiation dose required for total conversion of amorphous carbon to onions decreases with growth in the electron energy. The time of the process is nearly 45 min for an electron energy of 400 keV and a current density of 180 A/cm^2 in the beam [54]. Onions are also formed by the action of a high-energy-ion beam [71].

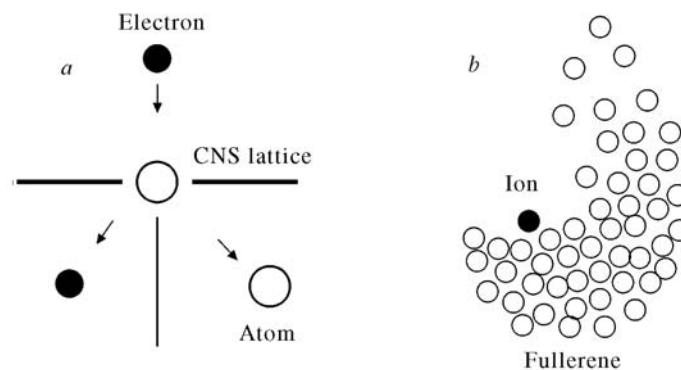


Fig. 4. Basic mechanisms of destruction of carbon nanostructures under irradiation: a) *knock-on* mechanism; b) *excitation* mechanism.

In the experiments of [72, 73], the formation of CNTs in the electron irradiation of a carbon material containing carbene ($(-\text{C} \equiv \text{C}-)_n$) has been investigated. Samples of the material applied to the surface of epoxy resin using electrochemical reduction were heated to temperatures of 870–1070 K and were irradiated in a transmission electron microscope with an electron energy of 100 keV and a current density of 1 A/cm^2 . As the observations have shown, the process follows a two-step course: rapid growth of carbon nanorods on the sample's surface and then a slow formation of hollows inside the rods followed by the graphitization of the walls. The reaction rate is dependent on temperature: at 1070 K, the duration of the process is no longer than 1 min; at 870 K, it increases to 30–60 min. At a temperature higher than 1070 K, the whole amount of carbon evaporates from the sample's surface, whereas at a temperature lower than 870 K, the reaction is frozen. The formed nanotubes have nearly the same length (about $1 \mu\text{m}$), diameter (10 to 50 nm), and number of graphene layers (about 5) whatever the temperature is. Continuation of irradiation leads to a deformation of the nanotubes due to the formation of cross-linking bonds between the layers.

Carbon nanotubes have also been synthesized in the irradiation of carbon samples with ion beams [74, 75]. In certain cases one observes the formation of carbon nanowires, nanocapsules, and coral-like structures condensing on different surfaces [76–78]. The appearance of the formed structures is strongly dependent on the parameters of the process such as temperature, pressure, and type of substrate. Irradiation of thin films of different hydrocarbons, applied to a titanium or glass surface, with a beam of N^+ ions with an energy of 200 keV leads to their restructuring and to the formation of conducting carbon nanostructures [79].

Development of direct methods of numerical modeling of the processes under study occupies a highly important place in the effort to explain the mechanisms of formation of CNTs and related structures. There is a large number of models at present that describe the nucleation and growth of CNTs and are based on the Monte Carlo method and the method of classical molecular dynamics and on solution of diffusion equations, quantum-mechanical calculations, etc. A rather comprehensive review of the methods and approaches to modeling of CNT formation under different conditions of the process can be found in [80–82].

Formation of Defects in the CNS Lattice and Its Destruction. The interaction of high-energy particles with CNSs produces different radiation effects (electron excitation or ionization of individual atoms, collective electron excitation, breaking of bonds between atoms, displacement of atoms from the lattice into the environment, and others) in them [51]. These effects determine possible mechanisms of destruction of the CNS structure under radiation. The most important of them are direct transfer of the energy of incident particles to the inner motion of lattice atoms (*knock-on* mechanism) and excitation of the CNS electron structure followed by the redistribution of excitation energy among the internal vibrational degrees of freedom of a macromolecule (*excitation* mechanism) [83]. The first mechanism is realized in irradiation of a CNS with a fast-electron beam, when the elastic scattering of the electrons on the lattice atoms causes the latter to shift beyond the lattice. The second mechanism is responsible for the decay of CNSs on exposure to high-energy ions [84]. The interaction between high-energy particles and CNSs is schematized in Fig. 4.

Numerous experiments in which the destruction of CNSs under electron-beam irradiation is investigated have been carried out (see, e.g., [83, 85–90]). The process begins with the formation of individual defects in the CNS lattice in accordance with the *knock-on* mechanism; these defects gradually spread over the entire surface of the lattice,



Fig. 5. Welding of single-layer carbon nanotubes by a beam of high-energy charged particles [19].

finally leading to its collapse. One of the prime objectives of these investigations is determination of the threshold electron energy at which carbon atoms begin to shift beyond the lattice. As the experiments show, destruction of fullerite films occurs at an electron energy higher than 80 keV [51]. Simultaneously with the decomposition of the fullerite crystalline structure, one observes the destruction of individual C_{60} molecules that are part of the crystal. In the first step of the process, the C_{60} molecules coalesce, forming large irregularly shaped cells; on further irradiation, we have their graphitization and the formation of onion-like clusters.

The threshold electron energy for single-layer CNTs is nearly equal to the corresponding quantity for C_{60} molecules. Observation of the behavior of CNTs in a transmission electron microscope shows that their structure begins to change at electron energies lower than 100 keV [90]. Theoretical evaluation shows that for an isolated nanotube, surface portions that are normal to the direction of the beam are the most vulnerable [85]. The minimum incident-electron energy necessary for displacing a carbon atom is equal to 86 keV under these conditions. At energies higher than 139 keV, the atoms of all surface portions of the nanotube can get a pulse allowing them to leave the lattice.

The formation of vacancies by the action of fast electrons on the carbon lattice is also observed in graphene membranes. As the experiments show, graphene membranes possess a high radiation stability, withstanding irradiation with electrons with an energy of 100 keV and a beam density of 7 A/cm^2 for a few hours [88]. For the sake of comparison, single-layer CNTs are strongly deformed under the same conditions [85, 87], which is attributable to the nanotubes' cylindrical geometry, making it possible to attenuate defects formed in the lattice due to the surface reconstruction. Vacancies in graphene membranes can be filled due to the interaction with molecules absorbed on the membrane surface from the environment [88].

Irradiation of different CNSs with high-energy ion beams leads to their destruction, too (see, e.g., [91–97]). In particular, the collision of C_{60} fullerene molecules uniformly distributed in the gaseous phase with ions leads to their fragmentation [84]. The dynamics of the process is described by the *excitation* mechanism, when the electromagnetic field of the incident ion causes collective electron excitation of the fullerene (formation of a plasmon). Subsequent transfer of the electron excitation to the vibrational degrees of freedom with the corresponding redistribution of the excitation energy among vibrational modes finally leads to a disintegration of the fullerene structure (see Fig. 4). From a computational viewpoint, the colliding ion–fullerene system is an ideal model system for studying the mechanisms of electron and vibrational excitation, including the fundamental process of electron-vibrational pairing in the atomic system with a large number of degrees of freedom.

Using the *excitation* mechanism one can explain not only the fragmentation of individual C_{60} molecules by high-energy ions but also destruction of ultrathin fullerene films (1–4 layers) grown on hydrogen-passivated silicon substrates upon their irradiation with an electron beam with a relatively low electron energy of the order of 3 keV [83]. As in the previous case, the incident electrons induce multiple energy transitions with the excitation of vibrational degrees of freedom in the structure of the fullerene film. The formation of a highly excited vibrational state is equivalent to the local heating of an individual C_{60} molecule. The process of subsequent molecular decay can include both successive emission of C_2 dimers and complete disintegration of the carbon shell. Irradiation with low-energy electrons also leads to destruction of single-layer CNTs due to the formation of defects in excitation of their electron structure [98].



Fig. 6. Formation of molecular junctions in the bundle of single-layer carbon nanotubes in irradiating it with a beam of Ar^+ ions [114].

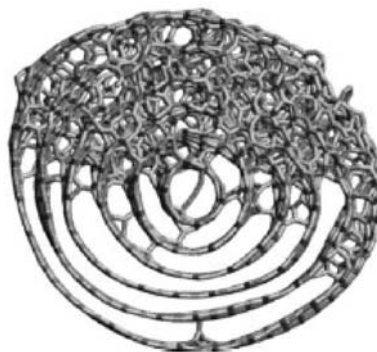


Fig. 7. Modification of a multilayer carbon nanotube in irradiating it with a beam of Ar^+ ions [99].

Modification of CNSs under Radiation. The use of radiation techniques for production of new carbon nanomaterials is one of the most promising trends in nanotechnology. Modification of CNSs under radiation includes welding of cross-linking CNTs [99], polymerization of layers from C_{60} molecules [100], formation of new nanosize structures [101], radiation chemistry of CNSs [102], etc. The irradiation of a CNS can be considered as the initiating step that leads to a formation of vacancies and other defects in its lattice. Further restructuring is dependent on both its internal structure and the position relative to other nanoobjects.

Electron-beam irradiation of single-layer CNTs induces structural defects, which leads to a welding of the tubes to form stable junctions of different geometry between their surfaces [103, 104] (see Fig. 5). The coalescence of several tubes into one of a larger diameter on exposure to 1.25 MeV electrons at a temperature of the process of the order of 1100 K is recorded in the experiments of [105]. On the other hand, the action of an electron beam on a CNT bundle leads to its exfoliation [106]. Selective electron-beam irradiation of individual CNTs can be used for welding of tubes on a metal surface, which improves electric contact between the tube and the surface [107]. The welding and formation of specific carbon nanostructures of the spindle, dumbbell, rod, and other types were observed in electron-beam irradiation of multilayer CNTs [108–110]. Electron-beam irradiation of a bundle of single-layer fluorinated CNTs gives rise to multilayer nanotubes [111], which is attributable to the fluorination-generated local stresses. Structural changes in single-layer CNTs, which lead to their modification, occur under x rays, too [112].

As in the case of electron-beam irradiation, the action of ion beams on bundles of single-layer and multilayer CNTs modifies their structure to form molecular junctions between the tubes (see Fig. 6) and between individual layers in the multilayer tubes [113–119]. This process is accompanied, as a rule, by the distortion of the tube surface due to the formation of numerous defects in the carbon lattice (see Fig. 7). On exposure to ion beams, multilayer CNTs can change to amorphous carbon nanowires and diamond nanorods [120–122]. Proton irradiation of CNTs gives rise to C–H bonds on their surface, which are capable of participating in chemical reactions that follows [123]. Ion beams are capable of welding CNTs on metal and graphite surfaces due to the formation of chemical bonds between the tubes and the substrate atoms near the formed defects [124]. Proton-beam irradiation of composite polymer films containing nanotubes causes the nanotubes to reach their surface, which forms a new structure [125].

Recently, researchers have paid increasingly greater attention to the polymerization of films consisting of C_{60} molecules, since this new form of carbon material exhibits the physical and chemical properties of both graphite and diamond [100] (see Fig. 8). In the case of radiation-induced polymerization the integration of fullerene molecules into the crystal lattice is accompanied by the destruction of the fullerene structure, which makes the process of polymerization a rather complex phenomenon [126–131]. Restructuring of C_{60} films induced by electron-beam irradiation represents their polymerization at an electron energy of 0.5 keV and fragmentation at an energy of 3.3 keV [83]. Sufficiently long irradiation of a fullerite film with electrons having an energy of 1.5 keV leads to its gradual transformation to an amorphous structure [132]. Amorphization of fullerenes is also observed in films of an Ag– C_{60} nanocomposite irradiated with fast heavy ions [133]. The polymerization and formation of giant fullerenes in the films of C_{60} molecules crystallized on the surface of different substrates in injection of electrons from the probing tip of a

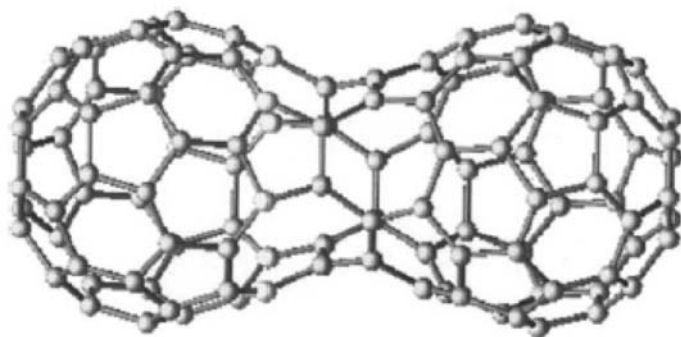


Fig. 8. Diagrammatic sketch of a C_{120} dimer formed by the electron-beam irradiation of a fullerene film [100].

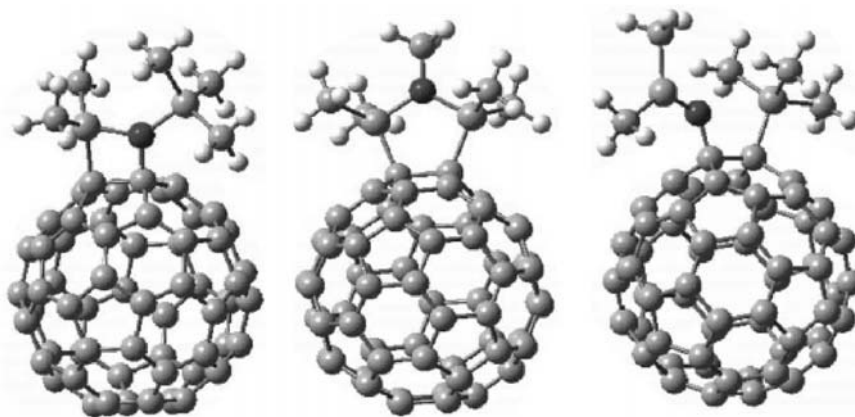


Fig. 9. Polyfunctional fullerenes formed in radiolysis of a C_{60} -molecule-containing medium [151].

scanning tunnel microscope has been recorded in [134–136]. Irradiation of graphite samples including interlayers of C_{60} molecules with 200 keV electrons leads to their conversion to diamond without using high pressures and temperatures [137].

The process of formation of new carbon materials is closely associated with the previous consideration. In particular, the action of an electron beam on a solid layer of C_{60} molecules crystallized on a graphene sheet forms a new nanostructure, which has great promise for use in nanoelectronics [101]. The experiments with single-layer CNTs uniformly filled with $ZrCl_4$ using the capillary method show the formation of clusters under electron irradiation [138]. This phenomenon can become a basis for synthesizing a one-dimensional set of quantum points inside CNTs filled with semiconductor or metal components. An analogous structural evolution of single-layer fullerene-filled CNTs has been observed in [139]. Ion-beam irradiation of single-layer CNTs leads to a substitution of carbon atoms on the tube surface [140–142], which is an alternative method for doping of the carbon lattice with different impurities in addition to the chemical and gas-discharge methods. The implantation of an atomic ion into the shell of a fullerene under its ion-beam irradiation gives rise to an endohedral fullerene [143].

To model the restructuring of CNTs under radiation, one basically uses the molecular-dynamics method [19, 87, 104, 114, 144–146]. Practical use of the method requires that the carbon lattice be identified, which is attained by assigning the corresponding interatomic-interaction potential (see, e.g., [147, 148]). These potentials are classical in nature, as a rule, and disregard quantum-mechanical effects. An alternative approximation is the incorporation of the directional effects of covalent bonds into the system's Hamiltonian (*tight-binding* method), which makes it possible to naturally allow for the nature of covalent bonds in the interatomic potential [149, 150].

The radiation chemistry of fullerenes [102, 151], which considers chemical reactions between fullerenes and active components generated in a medium in its radiolysis [28, 152] to form different fullerene derivatives (see Fig. 9), plays a significant role in investigating and producing CNSs. Potential participation of water-soluble fullerenes

in different biological applications has initiated a study of the formation, in the radiolysis, of its derivatives such as polyfunctional fullerenes containing hydroxyl, carboxyl, amine, and sulfate groups [153, 154], negatively charged fullerene clusters [155], etc. Certain applications of the radiation chemistry of fullerenes are associated with the utilization of other solvents. In particular, the radiolysis of C₆₀ in chlorinated methane gives rise to such adducts as C₆₀-CHCl₄ and C₆₀-CCl₃ [156, 157]. The radiation-induced synthesis of a C₆₀-Si hybrid nanomaterial in the toluene solution is considered in [158].

CONCLUSIONS

1. Because of the unique electron, mechanical, and chemical properties, CNSs have a significant potential for practical application (optical and electronic devices, composite materials, biosensors, devices of focusing of charged-particle beams, etc.).

2. The radiation method of production of fullerenes and CNTs has not gained wide acceptance compared to other methods because of the technical difficulties in its implementation and low efficiency of the process, which is attributed to the relatively low temperatures induced by the action of radiation on the starting carbon samples.

3. The irradiation of a CNS can be considered as the initiating step that leads to a formation of vacancies and other defects in its lattice. This causes its destruction, on the one hand, and the formation of new nanostructures, on the other, which is dependent on the irradiation intensity and on the internal structure of the CNS in question and on its position relative to other nanoobjects.

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