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JAIRI ISSN 3069-762X JOURNAL OF ARTIFICIAL INTELLIGENCE RESEARCH AND INNOVATION

Research Article

Methods for Manufacturing Nanotubes – State, Parameters, Analysis, Development, and Application

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Submitted: December 02, 2025
Accepted: December 15, 2025
Published: December 16, 2025

Citation: Kartunov S. Methods for Manufacturing Nanotubes – State, Parameters, Analysis, Development, and Application. J Artif Intell Res Innov. 2025; 1(1): 094-099. Available from: https://dx.doi.org/10.29328/journal.jairi.1001011

Keywords: Methods; Nanotubes; Superlattice

Abstract

The article analyzes key technologies of the 21st century - methods for producing graphene nanotubes (CNTs) and superlattices. Parameters, analysis, development, and application of nanotube production methods are analyzed.

Section theoretical setting

In nanotechnology, nanoobjects (nanoelements) and nanomaterials are distinguished, both by nanotechnology and by functional parameters, but often this division has a dual nature. The parameters include quantum size 6 - 13 nm × 2.5 - 20 μm, coherence length, internal organization/ orientation, strong lateral interactions, and a large area-to-volume ratio. The quantum size of small systems is given by the de Broglie wavelength (about 1 nm) or the distance along which the coherence of the wave function is preserved. The critical smallest size is considered to be the size at which the crystal lattice retains its properties, for example, 0.5 nm for iron and 0.6 nm for nickel. The other important characteristic of the wave is the coherence length or the distance over which the wave is preserved in phase (usually < 10 nm), high specific surface area 400-500m^2/g, surface~ 220 m^2/g, SSA and Density ~ 2.1 g/ml at 25 degrees (dust).

The object of the present work is the nanoelement carbon nanotube or nanofiber, belonging to the field of nanostructures. This is a single-atom layer of carbon, formed into a cylinder with a diameter and height of several nanometers [1,2]. Carbon compounds, called fullerenes by their discoverer B. Fuller, are their new allotropic form of existence - clusters C60 and C70 - along with graphite and diamond. Today, all types of molecules with a closed spatial structure bear the general name fullerenes. Fullerenes and nanotubes have been the subject of study since their discovery (N. Croto, R. Kerlu, R. Smalley, 1985, Nobel Prize 1996 [3]). They attracted greater attention when they found application in the products of electric arc evaporation of graphite (Prof. S. Ishima, 1991). The most stable form contains 60 carbon atoms arranged in 20 hexagons and 12 pentagons, which form a closed network (in C70, 25 hexagons and 12 pentagons, respectively, similar to a rugby ball). Each pentagon is bordered only by hexagons; each hexagon has three neighboring pentagons and three neighboring hexagons. Fullerenes have been in mass production since 1990, produced using the technology presented in [4]. A graphite electrode is used, evaporated in a carbon laser arc with a protective helium gas, which produces a large variety of fullerenes S60 and S70 of the C12 isotope. The C60 molecule can form fullerites or crystals doped with various elements. The strength of such pipes exceeds tens of times that of steel; they withstand heating to a temperature of 2500 ^{0}C and a pressure of a thousand atmospheres. Carbon can also produce molecules with a gigantic number of atoms, for example, C1000000, located at the vertices of hexagons (Figures 1,2).

Like spherical fullerene structures, cylindrical structures, or nanotubes, are also single-layer (single-walled) or multilayer (multi-walled). Usually, they are closed on both sides and resemble cocoons. The inside of the tube can be filled with different materials. Thus, the shell (tube) and the filling in it make it possible to create nanomaterials with new properties - a combination of the properties of both components. The material in some nanotubes is even crystallized, and its atoms are strictly ordered.

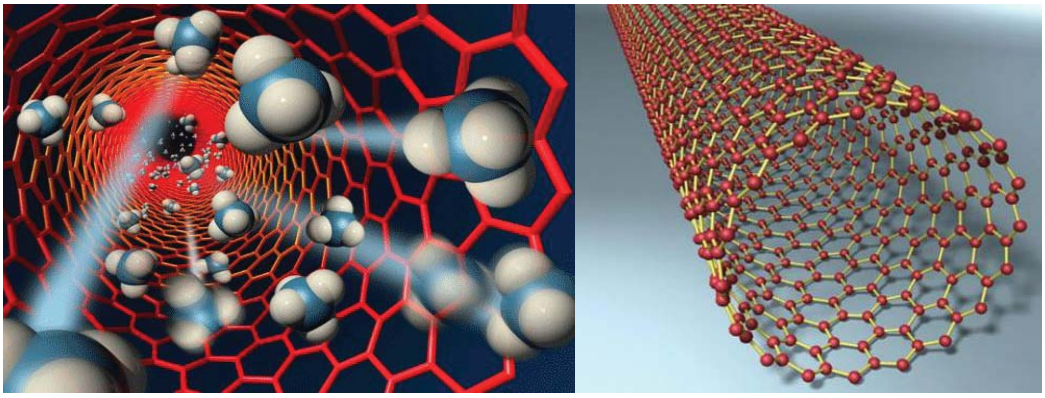

Figure 1: Surface effect in a nanotube and a high porosity carbon tube.

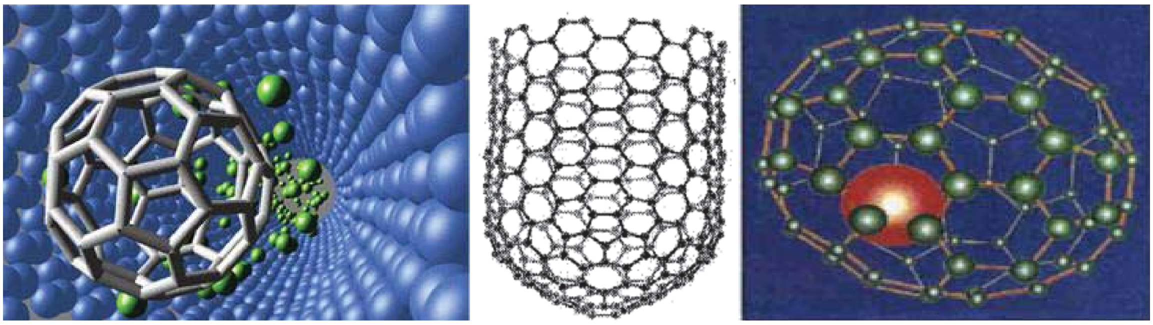

Figure 2: Closed structures of nanotubes and fullerenes.

It is convenient to denote nanotubes by a pair of numbers (coordinates). They are obtained by imposing the graphite plane that closes each tube on a coordinate system so that one end lies at the origin of the coordinate system, and the other is rotated until its coordinates become integers. According to the direction of the six-atom rings relative to the axis of the tube or by morphology, they are divided into armchair, zigzag, and chiral. The most interesting properties are possessed by spiral tubes (chiral), whose properties (e.g., the width of the closed zone) depend very strongly on their diameter and the pair of numbers that characterize them. The number of possible diameters is unlimited, from which it follows that the number of tubes with different electronic properties is also unlimited. The geometry of the structural pentagons, hexagons, and heptagons, the type of symmetry, and their deformation determine their unique chemical and physical properties. Nanotubes are good conductors and semiconductors (depending on the material) and allow the size of microcircuits to be reduced according to Moore's law. They are used for displays and nanodiodes, as well as for blades in atomic microscopes, probes, or very thin pens. Changing the size and shape of carbon nanotubes (1% carbon content and 99% air) leads to an extremely high degree of porosity (Figure 1) on the right. Modification of polymer nanotubes is also carried out by plasma by 2 methods: hydrophilization and hydrophobization. For the production of nanotubes, the catalysis operation with special catalysts is used (Figure 3): hydrodesulfurization of thiophene nanocomposite.

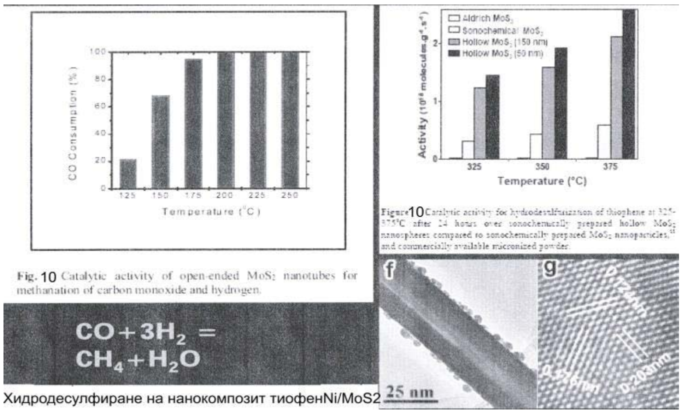

Figure 3: Catalysis and graphs of its course with different catalysts.

$$CO + 3H_2 = CH_4 + H_2O \quad (1)$$

Fullerenes are synthesized by the method of graphite evaporation at reduced pressure of helium in an electric arc discharge with a power of about 30kW and an arc current of about 1000 A (5). The content of fullerenes in the obtained carbon material is 15%. Laser irradiation of thermal black carbon (a product of thermal decomposition without access to air) also produces fullerenes C60 and C70. C60 layers with a thickness of 2 - 10 nm have also been obtained by thermal sputtering in vacuum at a speed of 0.002-0.05 nm/sec on mica, glass, NaCl with anisotropy, and APP substrates at a temperature of T = 20 ºC - 250 ºC. Nanolayers can grow island-like (on amorphous substrates) or layer-wise, with layers on mica being best obtained at a temperature of 215 ºC. The roughness class of the layer decreases with increasing thickness. By adding impurities, the properties of the layer can be adjusted, and a semiconductor behavior similar to that of silicon can be obtained.

Nanotechnology is not intended (or at least its capabilities are not limited) only to synthesizing small and large nano- or micro-objects on a nanoscale. Moreover, assemblers (nanorobots-assemblers) are able to build mega-objects the size of continents and planets. Theoretically, there is no limit to the number of atoms that can build a fullerene structure. The only rule that determines whether a structure can exist or not is its energy stability. If carbon atoms continue to fall on the surface of a closed fullerene structure during growth, then multilayer structures usually arise. They resemble Russian matryoshka dolls, can reach very large sizes, and consist of hundreds of layers. At their core, sometimes accidentally falling atoms are captured - both individually and in large quantities. Foreign atoms can be enclosed even in the smallest single-layer fullerene structures. For example, up to 3 atoms with a smaller atomic radius can be incorporated into C60.

As a summary from a technological point of view, it can be said that nanoobjects are divided into 4 main classes:

- Three-dimensional particles and polycrystals obtained by plasma synthesis, thin-layer regeneration, wire explosion, sol-gel methods, etc.;
- Two-dimensional objects – thin and inverse layers and heterostructures obtained by molecular layering methods, chemical methods for micro- and nanostructuring (CVD) [5], ion layering, etc.;
- One-dimensional objects – nanowires and fibers (whiskers), which are obtained by the method of molecular layering, introduction of substances into cylindrical micropores, self-cleaning surfaces, nanotubes, or by protonation (4);
- Zero-dimensional clusters (b.a. in some sources the type is absent or called colonies), quantum dots, individual molecules, atoms, and nanoparticles (Figure 4).

Methods of manufacturing nanotubes

Nanotechnologies for production are usually divided into three directions:

- Preparation of electronic elements, nanomaterials, and circuits from several atoms using nanoscale technologies, new methods for reading and storing information;
- Creation of nanomechanisms, robots, and machines with the size of a molecule using the approach of E. Drexler; nanomechanics;
- Direct manipulation of atoms, molecules, and nanotubes, formation of quantum dots and molecules, and assembly of active nanostructures from them. DNA nanotechnology, which uses self-assembly of complex nanostructures [6].

The subject of this publication is the latter direction. Methods for growing carbon nanotube arrays on substrates are known. Usually, chemical vapor deposition (CVD) is applied in the presence of a catalyst or between inert microparticles with sizes from 50 nm to 30 μm. However, the point here is to obtain arrays of nanotubes or so-called mats - a forest of tubes that are oriented vertically to the substrates. Mats with a height of 20 μm to 1 mm were obtained, synthesized using two types of iron catalysts. In the first type, a 5 nm high Fe layer was deposited on a silicon substrate, and in the second type, a 1 nm Fe layer on a 10 nm thick ceramic buffer layer (Al_2O_3). In both cases, there is a 1 μm oxidized SiO_2 layer on the substrate, and the diameter of the tubes is 10 - 15 nm [7]. The synthesized carbon nanotube arrays were first placed in an ethanol solution by scientists from the United States, and then dried at room temperature. During drying under the action of capillary forces, a new structure was formed. In the array synthesized with the first catalyst, compactness and reorganization of the tubes into cells are obtained, shown in

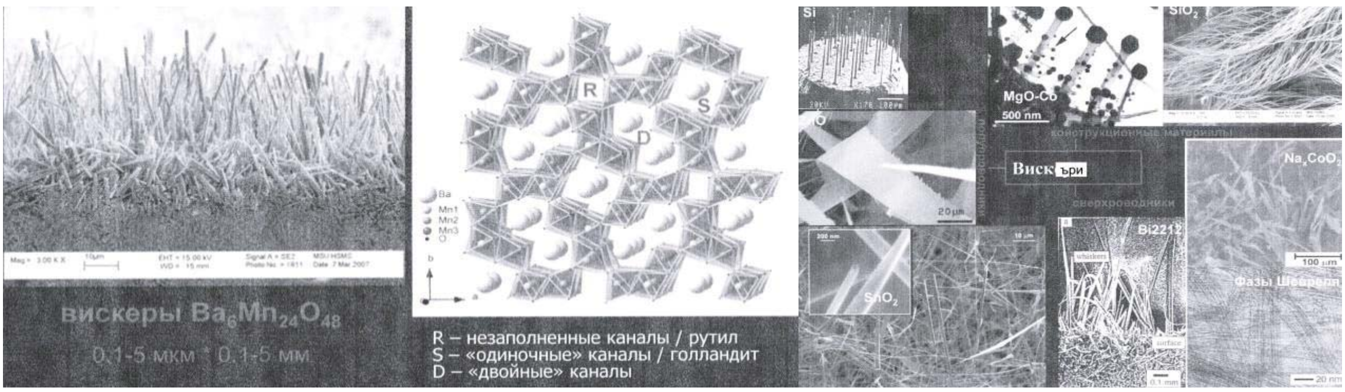

Figure 4: Types of whiskers: a combination of unique structure and shape in tunneling manganese oxide, chemical sensor SnO_2, quantum rivets, and others (www.nanometer.ru, goodilin@iniorg.chem.msu.ru).

Figure 5a. The height of the cell walls is comparable to that of the initial array, 50 μm. The light areas in the center of the cells are areas of the substrate without tubes (Figure 5b).

For the 1 mm high array synthesized with a buffer layer catalyst, the picture changes abruptly. Upon drying, the nanotubes form separate dense islands with an average distance between them of about 700 μm (Figure 6a), and there are no lying tubes on the substrate. These results show that the formation of new structures is influenced by two factors: the magnitude of the capillary forces and the strength of adhesion of the tube roots to the substrate, which manifest themselves oppositely in the two variants, respectively.

A method for obtaining the material «SWNT solid» from compacted and oriented carbon single-walled tubes is also proposed by Japanese scientists [7]. The array preserves the properties of the individual tubes - flexibility, electrical conductivity, and large surface area, giving a different shape. The universal method is based on compacting the "forest" under the action of a liquid (water, acetone, alcohol, dimethylformamide, liquid nitrogen, oleic acid, various oils) and the use of capillary attraction. The difference is the use of long (on the order of mm) vertical tubes with a diameter of 2.8 nm. The resulting array has a density of 0.03 g/cm3 and 97% empty space. Adhesion occurs in two stages – upon wetting and upon evaporation. Figure 7 shows a dense material obtained after introducing water drops into the center of a 1 x 1 cm sample. If the process is not controlled, the material in Figure 7a is obtained. After wetting, under the action of capillary attraction, the "forest" is reduced horizontally by 20%, and after drying, by 4.5 times in both directions. In this way, the density increases 20 times, and hence the hardness 70 times, at a constant height. Control over the process parameters allows the production of unique structures of different shapes – needles, plates, microvolcanoes, etc. (Figure 7c).

Another approach to compaction of HT arrays is used by scientists from Singapore and the USA [8], which consists of pre-introducing into the array of compacted HTs of different configurations and synthesized with different catalysts. These round or oblong (oval) holes act as centers of drying. One of the samples is made of multi-walled carbon nanotubes with a diameter of 30 - 40 nm and a height of 9 to 200 μm, depending on the growth time. They were obtained by the CVD [9,10] method with Fe catalyst on a Si wafer with an intermediate layer of aluminum and have a diameter of the cylindrical hole of approximately 3 μm (Figure 8a). When liquid water (10 μml) is dropped, a thin film is formed, which subsequently dries and self-organization of HT occurs, initially forming dry zones around the centers of drying. Under the action of surface tension, the tubes contract and form ridges. If the holes have a circular cross-section, a cellular or nested structure is formed; if the holes are oval, a structure of ditches is formed.

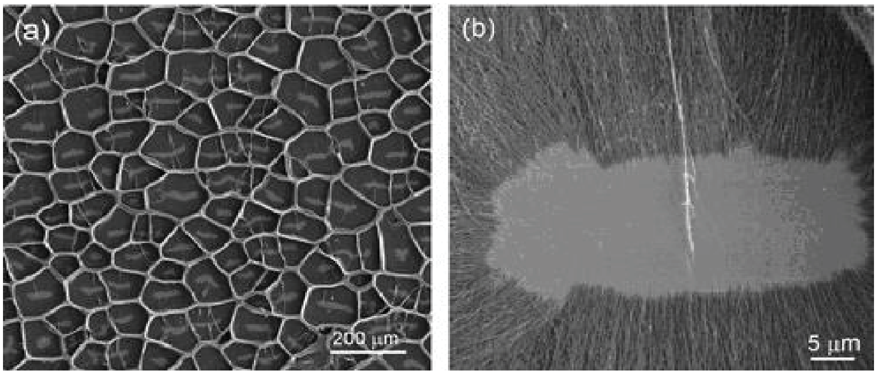

Figure 5: (a) SEM image of a nanotube cell structure synthesized with 5 nm Fe/SiO_2. (b) Enlarged image of the bottom of the cell.

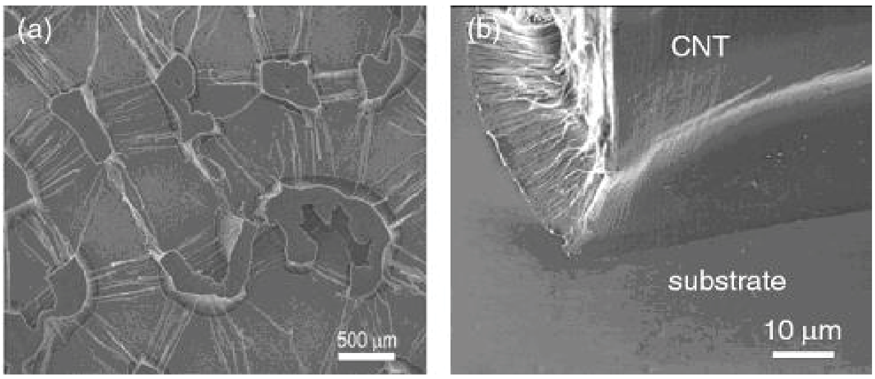

Figure 6: (a) Structure formed during drying of the array with a height of 1 mm, synthesized with a catalyst of 1 nm $Fe/Al_2O_3/SiO_2$. (b) Enlarged image of the end of the individual islands.

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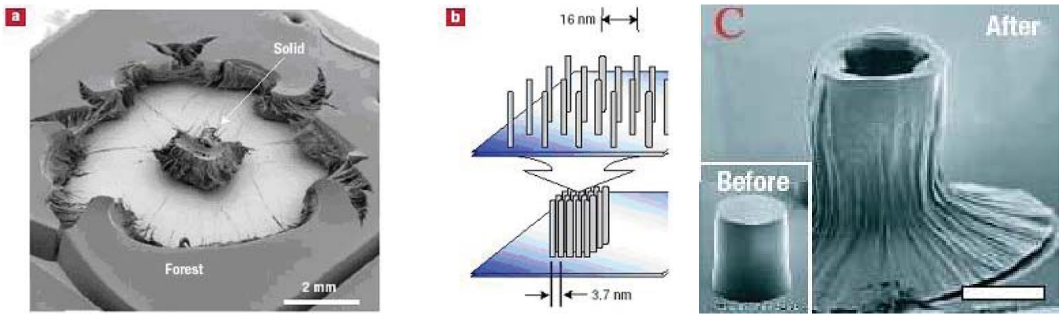

Figure 7: SEM image (a) and schematic (b) of the compaction of oriented HTs under the action of a single drop of liquid, (c) – "microvolcano" before and after the collapse (the scale shown is 250 μm).

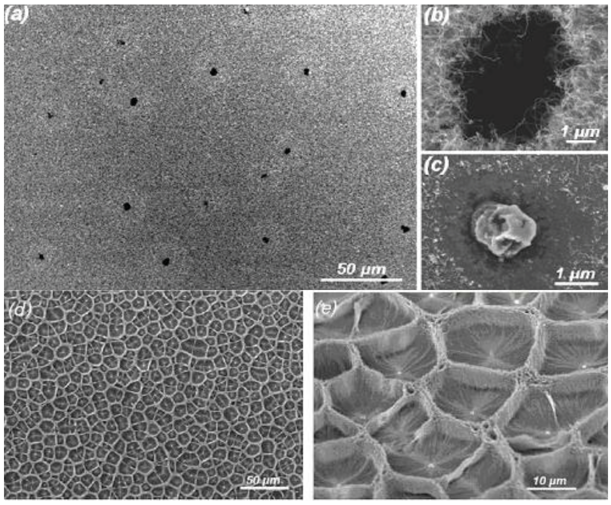

Figure 8: (a) 12 μm BNT array with internal microholes; (b) enlarged image of the microholes; (c) aluminum microparticle obtained by sputtering the sublayer; (d) formed cellular structure; (e) in the center of the nest are visible bright spots – Al microparticles.

Methods for synthesis of graphene nanotubes and superlattices

Different methods are used for the synthesis of nanotubes. Single-walled nanotubes and fullerenes, as mentioned above, are obtained in an arc discharge using metal catalysts or accelerators. The process is favored by the addition of S, Y, Bi, and Pb to the graphite anode. By the method of carbon deposition on aluminum membranes in 46% HF, nanotubes with a diameter of about 200 nm and the possibility of incorporating lithium into their structure for creating galvanic cells were obtained. A third option is the pyrolysis of a mixture of polyfurfuryl alcohol and polyethylene glycol (18 parts in vacuum at a temperature of 450 °C) in the presence of Cs as a catalyst and subsequent heating of the mixture at temperatures of 50, 350, and 500 °C for 16 hours, in which the initial amorphous structure is oriented, and short nanotubes are obtained. The same are elongated at room temperature for 6 weeks. The formation of fullerenes in this way is associated with defects in amorphous carbon and is not recommended. A summary of most methods for the production of various objects through nanotechnology is given by the "Angstrom Technology" project from Japan [11].

Nanotube analysis and characterization

Purity analysis: Determination of the purity of carbon nanotubes to ensure compliance with specific standards.

Precise measurements of CNT dimensions using advanced microscopy techniques. Measurement of diameter and length: quantum size, coherence length, internal organization, strong side interactions, and large surface area to volume ratio.

Thermogravimetric Analysis (TGA): Assessment of thermal stability and composition by measuring weight loss under controlled conditions.

Elemental analysis: Quantitative determination of elemental composition to assess quality and potential contaminants [12]. However, challenges related to dispersion, coupling, and cost-effectiveness make CNTs impractical for many manufacturers.

Other Techniques and Developments are: Laser Ablation and Arc Discharge - Historically earlier fabrication methods that are also used. High-pressure splicing: Nanotubes can be joined by exchanging some sp^2 bonds for sp^3 bonds to produce longer strands by disproportionation of carbon monoxide under high pressure. Nanolithography and nanoprinting: Techniques used to precisely position nanostructures in devices [6]. Argon Plasma - uses argon plasma to decompose carbon gas into CNTs. Chemical Liquefaction - uses chemical reactions to convert carbon atoms into CNTs. Mechanical Abrasion - uses mechanical forces to abrade CNTs from graphite surfaces [5]. CVD, electric arc discharge, and laser ablation are the three most common methods for producing CNTs.

Application

Applications for carbon nanotubes:

- Automotive parts;
- Electronics: circuits, batteries, supercapacitors, transistors based on CNTs, sensors;
- Photovoltaic technology - including solar panels, LEDs, sensors, transistors, field emission devices, fuel cells, actuators (devices that power physical motion), optics;
- Absorbents and Catalysts;
- Military industry - body armor;
- Biomedicine: Drug delivery, biosensing, bioimaging, nanorobotics, gene therapy, and tissue regeneration;
- Agriculture: Bioremediation, water purification;
- Carbon nanotubes can be added to a material or substance (nanocomposite) to improve strength. This is useful, for example, in the production of sportswear or materials used in projectile deflection, including bullet-proof vests [13].

Products and technologies are widely used in multiple strategic emerging industries, including but not limited to new energy vehicles, new polymer materials, elastomer materials, aerospace, rail transportation, wind power generation, and hydrogen energy storage.

Shandong Tanfeng New Material Technology Co., Ltd is a high-tech enterprise dedicated to the research and development of carbon nanotubes, the production of new nanomaterials, and application development. The company's products include carbon nanotube powder, carbon nanotube conductive paste, silicon-carbon anode material, single-walled carbon CNTs, and composite materials [13].

Conclusion

Nanoobjects are divided into 4 main classes, and nanotechnologies for their production are usually divided into three directions. The subject of this publication is the direct manipulation of atoms, molecules, and nanotubes and the assembly of active nanostructures from them, analyzing the parameters, development, and application of nanotube production methods. Potential for future research: With the improvement of dispersion techniques and bonding technologies, future applications of CNTs in carbon fiber composites could yield better results.

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