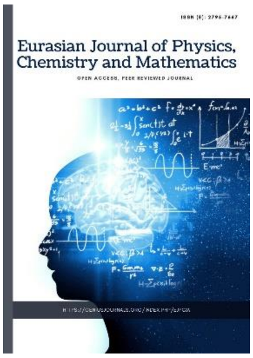


# Technology of Obtaining Carbon Nanotubes and Their Use in Practice History of Nanotechnology Development

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**ABSTRACT**

Article on the history of development of nanotechnologies, author Uktam Dekhkonovich Sherkulov, Republic of Uzbekistan, Navoi State Pedagogical Institute. The article describes what nanotechnology is, the emergence of nanotube research, the creation of electron microscopes, Feynman's lecture, the results of research by world scientists, and a lot of historical information. The article is intended for scientific researchers and graduate students.

**Keywords:** Electron microscope, diffusion length of input atoms, effective mass, crystal lattice, nanoparticles, piecewise "atom" assembly, National Bureau of Standards, scanning, lots of room below.

## Introduction

In the last decade, we have increasingly used words such as “nanotechnology”, “nanomaterials”, “nanosystems”.

These words no longer surprise anyone, since they are constantly associated with the development of new technologies that create materials at the atomic level (usually from 1 to 100 nm). Nanoproducts and nanotechnologies have become a part of our lives. They can be found in building materials.

For all developed and developing countries, nanotechnology is one of the priority areas of science and technology, which is designed to bring revolutionary changes to various sectors of industry, energy, medicine, construction and agriculture. This applies to such areas of production as new materials with unique mechanical and thermophysical properties, nanoelectronic elements with record speed and low energy consumption, sensors with selective sensitivity at the level of individual atoms and molecules, lasers in quantum-sized structures. Photodetectors with high radiation efficiency and characteristics several times higher than modern analogues, etc.

Historically, issues of nanotechnology have been actively studied since the early 60s of the last century. This interest was largely purely scientific without significant financial support from the government. The situation changed in 2000 with the adoption by the US government of the National Nanotechnology Initiative, a government investment program aimed at developing nanotechnology.

The impetus for this work was a similar program adopted two years ago in Japan, which has the highest government priority, and the program was financially supported not only by the government, but also by large private companies. Later, government programs for the development of nanotechnology were adopted in the European Union and China. Since 2007, Russia has been included in the list of these countries. All this quickly yielded practical results.

If in 2009 the global market for goods created using nanotechnology was $254 billion, then by 2017 this figure reached almost $1 trillion, and by 2020 it exceeded $3 trillion.

After the prestigious scientific journal Science named nanotechnology the “breakthrough of the year,” public interest in it increased sharply, and the phrase “new industrial revolution” began to be often applied to nanotechnology. Hundreds of conferences are held almost every year on various aspects of nanotechnology. Hundreds of thousands of articles and monographs have been published, special websites have been created on the Internet, and reports regularly appear about new ideas for the use of nanotechnology or new types of products obtained using nanotechnology.

This article has been prepared for use as an interesting source for undergraduates in the master’s specialty “Methods of teaching exact and natural sciences (physics and astronomy)” when studying the subject “Modern physics and modern problems of its teaching.” The article is divided into 3 parts: 1) History of the development of nanotechnology; 2) Technologies for the production of carbon nanotubes; 3) Use of nanomaterials in practical activities.

The materials presented in the article for graduate students and scientific researchers will shed light on the problems that have arisen in this area, ways to solve them and ways to solve them, the development of science and the introduction of new technologies in the near future, and we hope that these materials will help our researchers to develop skills and abilities in scientific activity.

## History Of Nanotechnology Development

### 1. Review of nanotechnology.

Nanotechnology is a set of methods and techniques for manipulating matter at the atomic and molecular level to produce final products with a predetermined atomic structure. Nanotechnologies make it possible to controllably create and modify objects, including those smaller than 100 nm, with radically new properties and integrate them into larger, fully functional systems.

Radically new properties of nanomaterials are associated, first of all, with the quantization of the energy spectrum of quasiparticles in nanoobjects and structures of reduced size, which is especially noticeable with the radical manifestation of the transformation of semiconductors, magnetic materials, organic and carbon-containing materials, as well as molecular assemblies.

The main reason for the change in properties is that at these scale levels the influence of the laws of quantum mechanics begins to manifest itself, that is, the nanoscale level is the level of transition from classical mechanics to quantum mechanics.

To determine the threshold for the appearance of new properties of the measured object, the concept of critical size d* is introduced.

This value depends on what property of the object is measured in the experiment and what geometric value determines this property - the mean free path of electrons in metals or semiconductors, the diffusion length of input atoms in semiconductors, the de Broglie wavelength of electrons, or the depth of penetration of the electromagnetic field into the surface layer object, etc.

Calculations show that different phenomena occur in organic and inorganic materials and the critical size d* lies between approximately 1 and 100 nm. For example, let's calculate the de Broglie wavelength λDB of electrons in a semiconductor:

$$\lambda_{DB} = \frac{h}{p} = \frac{h}{m^* \cdot v},$$

Here h - is Planck’s constant, p - is the quasi-momentum of the electron in the crystal, m* - is the effective mass of the electron, v - is the velocity of the free electron. Considering that typical values of the effective mass of electrons in semiconductors range from 0.1 me to me (me is the mass of a free electron), de Broglie wavelengths range from 3 to 30 nm.

Thus, quantum-size effects for electrons in semiconductors appear at object sizes from 1 to 100 nm. The sharp change in the properties of nanomaterials compared to bulk materials of the same chemical composition is explained by the effect of a sharp increase in the surface area of the nanoparticles that make up the nanomaterial. As the size of nanoparticles decreases, the percentage ratio between surface and bulk atoms changes. As a result, the influence of surface atoms on the properties of the object becomes decisive.

1 (a) - in the figure, the dependence of the total surface of an object consisting of many particles on the size of these particles; In Fig. Figure 1 (b) shows a similar distribution depending on the number of particles and atoms located on the surface.

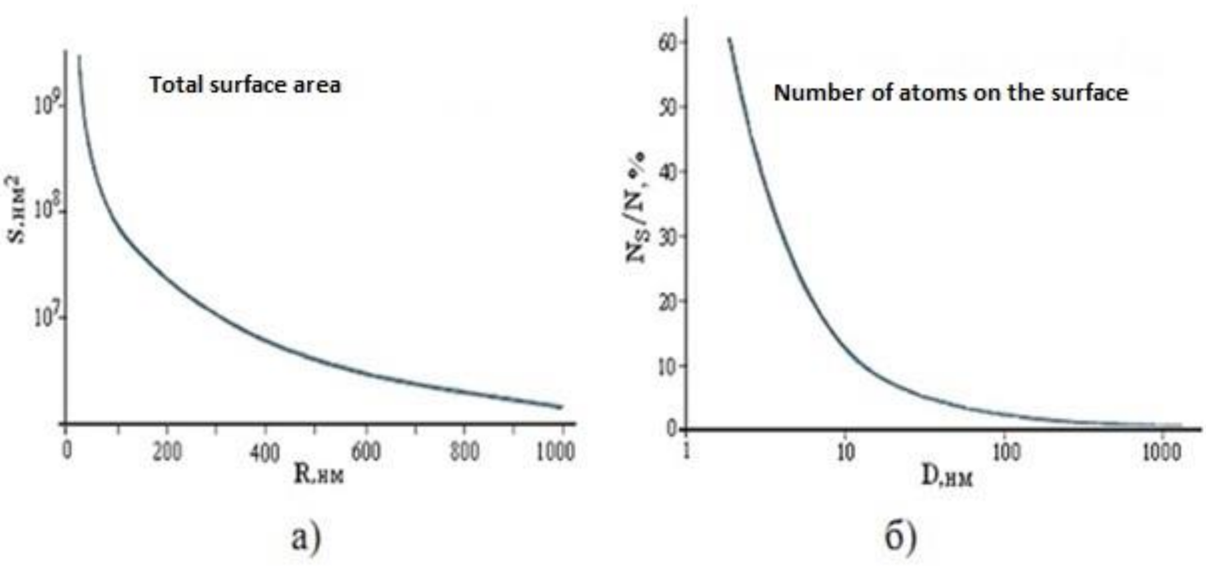



**Figure 1.** Dependence of the total surface area of an object (a) and the number of atoms on its surface (b) on the size of the particles that make up this object.

It is known that surface phenomena include surface tension, surface activity, capillary phenomena, wetting, adsorption, etc. The main physical reason explaining the nature of these phenomena is that the interactions between surface and bulk atoms are different. Atoms located on the surface of a body or on the surface of the particles that make up the body are in special conditions. The forces acting on surface atoms located at the nodes of the crystal lattice are “unilateral”, that is, only neighboring atoms act on surface atoms, and forces from all sides act on volumetric atoms. Therefore, the properties of surface atoms differ from the properties of similar bulk atoms. This leads to the manifestation of new properties such as melting point, electrical conductivity, transparency area and magnetism.

For objects of macroscopic dimensions, the basic physical and chemical properties do not depend on size. For nanoobjects, the same properties can vary greatly. This applies to structural and phase changes, magnetization processes, thermal and electrical conductivity phenomena, optical phenomena and others. In this case, almost all the main characteristics of the substance change, for example, the parameters of the crystal lattice, the mobility of charge carriers, the optical absorption spectrum, the melting point, etc. For example, reducing the size of nanoparticles to a value of the order of 10 nm leads to a decrease in the melting temperature $T_{melt}$ by several tens of percent. compared to volumetric objects consisting of crystallites with dimensions several times larger. When the size is reduced to 1-2 nm, the melting temperature decreases several times. This effect was observed experimentally on many metals, in particular Al, Ag, Au, Cu, Ga, In, Sn, etc. As an example, in Figure 2 shows the dependence of $T_{melt}$ of gold nanoparticles Au and cadmium sulfide CdS on their size (the dotted line indicates $T_{melt}$ for bulk objects).

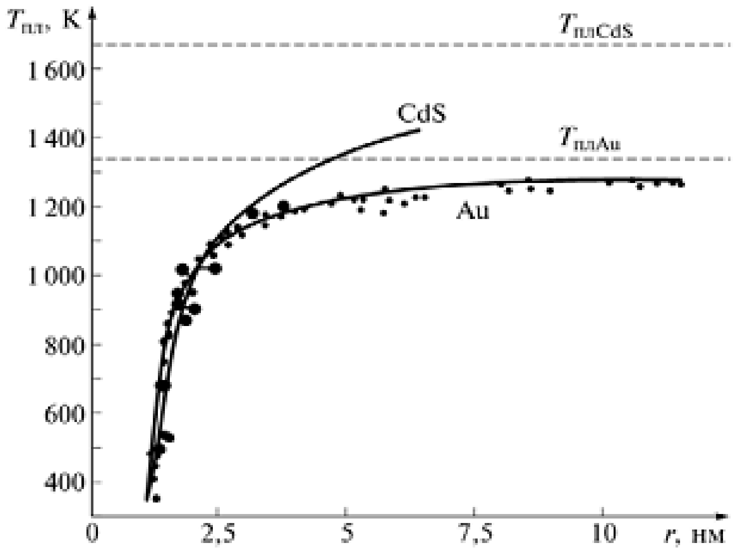



**Figure 2.** Dependence of the melting temperatures of gold (Au) and cadmium sulfides (CdS) on the sizes of nanoparticles $T_{melt}$.

## 2. Origins and first steps of nanotechnology.

Conducting research on the creation of nanoobjects and the use of their properties for practical purposes requires appropriate tools. The creation of such instruments began in the first half of the 20th century.

In 1928, a design for a close-up optical microscope was proposed. In 1932, E.Ruska and M.Knoll (Germany) created a transmission electron microscope (TEM) designed to study nanometer-sized objects. A few years later, Siemens (Germany) launched the first such microscope with a viewing resolution of 10 nm. At the end of the 50s of the last century, fundamentally new technologies appeared that made it possible to obtain nanostructured materials.

Let's take a look at some of the important discoveries that led to the development of modern nanotechnology. In 1959, the famous American physicist R.Feynman (who later received the Nobel Prize) gave a famous lecture at the American Physical Society Christmas party entitled “Plenty of Room Below: An Invitation to the New World of Physics.” In it, he disagreed with the assertion of some physicists that all important discoveries have already been made and there is no point in studying physics. R. Feynman expressed his opinion on new directions of research in physics. In particular, he proposed the idea of synthesizing new substances by manipulating individual atoms. In it, he was the first to consider the possibility of creating nanoscale objects in a completely new way - by assembling “atoms” piece by piece. He drew the attention of those gathered to the fact that the currently known laws of physics in no way prohibit the assembly of objects “atom-on-atom”, which in principle allows the synthesis of any substance according to its chemical formula. This, in his opinion, shows unprecedented prospects. R.Feynman gave simple calculations, from which it follows that if each letter of the alphabet is represented by 6-7 bits of information and 100 atoms are used to record each bit, then they can be placed not only on the surface, but also in volume, then the entire collection of the British encyclopedias can be “packed” into a volume approximately equal to a child’s head. This shows that there is indeed a lot of space in the subatomic world. As R.Feynman noted, the laws of physics do not prohibit the assembly of nanoobjects by placing “atom on top of atom.” The manipulation of atoms is very realistic and does not contradict the laws of nature. At this stage, this is due to the fact that the available tools for the practical implementation of such an atomic assembly are too large to solve such a problem. However, scientists need tools that will allow them to manipulate individual atoms. Biologists, for example, have been studying objects such as

DNA molecules for decades. They know that DNA contains the code for an organism's structure, but they need the right tool to decipher the code. Therefore, biologists and biophysicists have long been waiting for the creation of a new microscope that allows them to study nanometer-sized objects. R.Feynman with his lecture aroused the imagination of his colleagues, and also initiated a scientific race in the study of the molecular world. The tools for such research were not created until twenty years later.

In 1966, Russell Young, an American physicist working for the National Bureau of Standards, invented the piezo motor, which is today used in scanning microscopes and positioning nanoinstruments with an accuracy of 0.01 angstroms (1 nm = 10 Å).

In 1968, Alfred Cho and John Arthur, employees of the scientific department of the American company Bell, developed the theoretical basis for nanoprocessing of surfaces.

In 1972, the first close-up optical microscope with a resolution of 0.05 wavelength λ was created at IBM in Zurich, Switzerland. In the early 90s, scientists who developed this idea managed to create a near-field optical microscope with much higher resolution.

In 1974, physicist N. Taniguchi (Japan) introduced the term “nanotechnology,” by which he proposed to understand methods for creating objects less than one micrometer in size. A few years later, the physics of quantum dots and quantum wires became the basis for nanoscale objects. In 1981, G. Gleiter (USA) proposed a classification of nanomaterials according to their chemical composition and phase distribution, where the concept of “nanocrystallinity” was first introduced. Later, the terms nanostructured, nanophase, nanocomposite and others began to be used.

In 1981, employees of the IBM branch in Zurich (Switzerland) G. Binnig and G. Rohrer created a scanning tunneling microscope, which made it possible to “see” a three-dimensional picture of the arrangement of atoms on the surface of the body of electrically conductive materials. Five years later, both scientists received the Nobel Prize in Physics for their work in scanning tunneling microscopy. In the same year, a group of scientists under the leadership of G. Binnig created and manufactured an atomic force microscope, the scanning function of which turned out to be more powerful than that of a tunnel microscope.

In 1985, American physicists Robert Curl, Harold Krotoy and Richard Smalley created technology that could accurately measure objects with a diameter of one nanometer.

In 1986, an atomic force microscope was created, which, unlike a tunnel microscope, is not conductive, but allows it to interact with any materials.

The discovery of new carbon nanomaterials gave a powerful impetus to the development of nanotechnology. Previously, science knew that there are two main crystalline allotropic modifications of carbon - graphite and diamond. However, it turned out that there are several more modifications of carbon with unique properties. We are talking about fullerenes and carbon nanotubes, as well as graphene.

Carbon nanotubes were discovered in 1991 by Japanese scientist S.Iijima. Carbon nanotubes, like fullerenes, have unique physical properties. They are very durable and have record values of electrical and thermal conductivity. They have high chemical activity, that is, they are able to interact with other substances. In addition, other atoms can be introduced into fullerenes and carbon nanotubes, which makes it possible to obtain materials with new properties. Later it turned out that nanotubes can be synthesized not only from carbon atoms, but also from other chemical elements. In particular, in 1992, nanotubes based on $MoS_2$ and $WS_2$ were synthesized.

Fullerenes were first synthesized in 1985 as a result of joint research by future Nobel laureates G.Croto (Great Britain), R.Curl (USA) and R.Smalley (USA). During experiments studying the properties of graphite vapors obtained as a result of laser irradiation of a graphite target, large aggregates consisting of a large number of carbon atoms were identified (aggregates with 60 atoms were often encountered in experiments). Scientists have suggested that such aggregates are molecules of carbon atoms, and are shaped like a soccer ball. In it, all carbon atoms form a closed surface with 20 hexagonal and 12 pentagonal sides. The size of such molecules, called fullerenes, was about 1 nm.

In 1998, S.Dekker (Netherlands) created a transistor based on a carbon nanotube. Almost immediately after this, publications began to appear on the creation of field-effect nanotransistors of a similar design. All this contributed to the emergence of the technological foundations of nanoelectronics.

## 2.1. Creation of a scanning tunneling microscope.

In 1981, G.Binnig and G.Rohrer created a scanning tunneling microscope (STM), which for the first time allowed scientists to “see” individual atoms. The operating principle of STM is shown in Figure 3. First, an electrically conductive probe (needle) approaches the sample surface at a short distance using a piezoelectric motor (z-piezomanipulator). The radius of curvature of the probe and the distance between it and the sample are tenths of a nanometer. In the presence of voltage, a tunnel current flows between the probe and the sample, the magnitude of which depends on the distance between the probe and the sample.

The probe is moved parallel to the sample surface using x- and y-piezoelectric motors. In this case, the magnitude of the tunnel current is maintained constant using a feedback system (control system). Based on the analysis of feedback signals, the computer creates an image of the sample surface on the monitor.

STM uses a sharp metal needle as a probe. The maximum spatial resolution of STM is mainly determined by the radius of curvature of the needle tip and its mechanical rigidity. If the mechanical stiffness in the longitudinal and transverse directions is sufficiently small, mechanical, thermal and quantum fluctuations of the needle tip can significantly distort the STM resolution. Metals with high hardness and chemical resistance - tungsten or platinum - are usually used as probe materials. By studying the surface of some crystalline samples using STM, Binnig

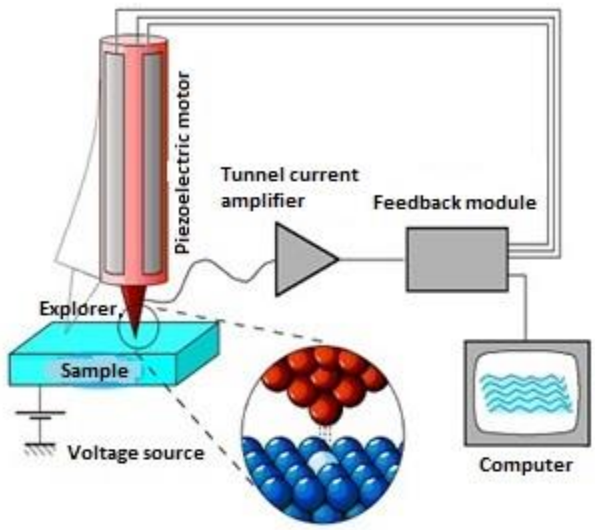



**Figure 3.** Simplified diagram of a scanning tunneling microscope.

and Rohrer were able to “see” relief irregularities one atom high. Modern scanning microscopes have a vertical resolution of 0.01 nm and a horizontal resolution of about 0.2 nm. An important factor limiting the functionality of the microscope was supposed to be the electrical conductivity of the sample. However, by the end of 1986, there were more than 40 scanning microscopes operating in laboratories around the world.

## 2.2. Creation of an atomic force microscope.

In 1986, Binnig and his colleagues developed an atomic force microscope (AFM), which made it possible to "see" not only electrically conductive materials, but also any samples. The operating principle of the ECM is shown in Figure 4. In it, a needle mounted on an elastic cantilever scans the surface of the sample. The forces of interaction between atoms on the surface of the sample and atoms located at the tip of the probe cause a deflection of the cantilever, which can be recorded in one way or another, for example optically. This makes it possible to restore the topography of the sample surface.

In 2009, IBM scientists in Zurich, Switzerland, succeeded in creating a microscope with a resolution of ~100 pm and using it to obtain a three-dimensional image of the pentacene molecule, which has a structure of five benzene rings.

In Fig. Figure 5 shows an ACM image of such a molecule. Above is a diagram of an ACM image of a pentacene molecule. A pentacene molecule is schematically depicted under the needle. Below is an actual AFM image of the molecule.

The pictures were taken at a temperature of -268ºC. The resulting images

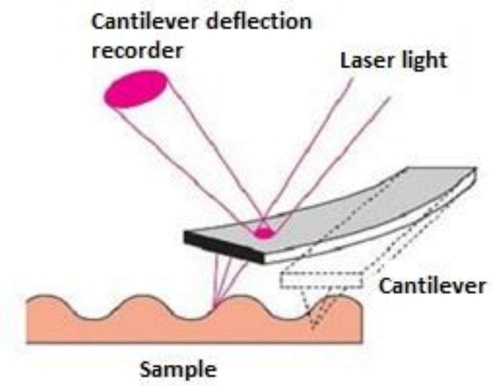



**Figure 4.** Simplified diagram of an atomic force microscope.

Clearly show the arrangement of previously only benzene rings, hexagonal structures of carbon atoms and even light hydrogen atoms. The side length of the benzene ring, clearly represented by individual atoms, is only 0.14 nm.

Tools have emerged that allow individual atoms to be manipulated. Individual atoms can be moved using a special probe with an electric field and a very small radius of curvature. To demonstrate this capability, in 1989, IBM employees created their company logo on a nickel base and placed it using atoms xenon Figure 6 shows a sequence of images of the nickel base during the formation of the abbreviation "IBM" from 35 xenon atoms. The height of the letters is 5 nm. The whole procedure took 22 hours.

The experiment, carried out in deep vacuum conditions at extremely low temperatures, was purely demonstrative in nature - all 35 atoms that were not chemically bonded to the base then "escaped" from their places. However, subsequent studies reliably confirmed the possibility of determining the valence of atoms on the surface of various materials without the use of ultra-low temperature technology.

In Figure 7a shows a "dancing child" consisting of carbon atoms assembled on a metal substrate. It took ten thousand atoms to obtain such a structure. Figure 7b shows the process of "assembling" a circle of 48 copper atoms on the surface of iron. These photographs confirm the steps involved in constructing a circle of 48 copper atoms on the surface of iron.

## 2.3. Discovery of fullerenes.

For decades, carbon was thought to have two allotropes: graphite and diamond. Sometimes amorphous carbon is added to them in powder form. In the mid-1960s, hexagonal diamondlonsdaleite was discovered, named after

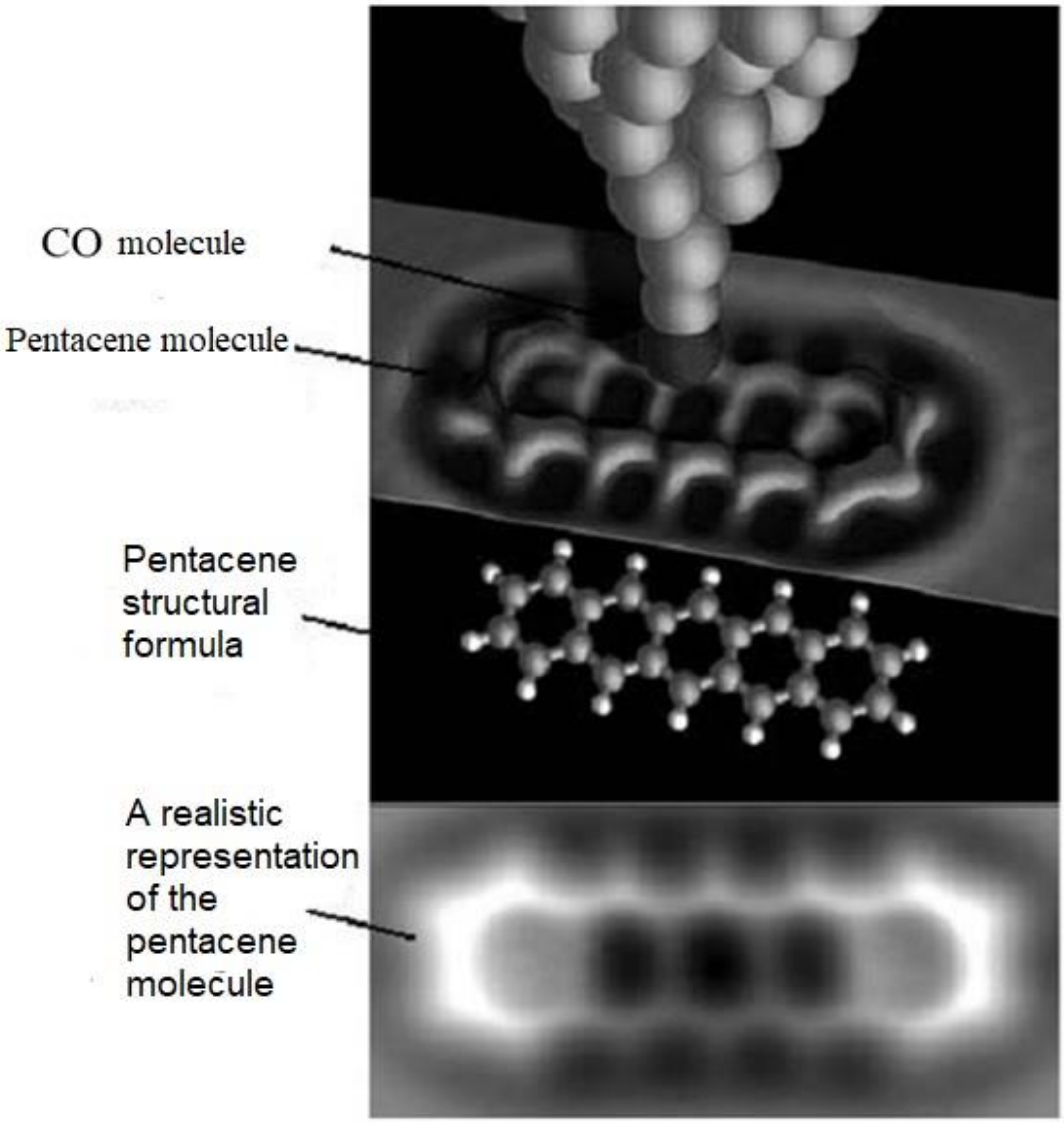



**Figure 5.** ACM image of a pentacene molecule.

british crystallographer K.Lonsdale. At the same time, two forms of carbon were discovered in the USSR: α-carbyne, the bond between carbon atoms of which is described by the formula (–C≡C-)n, and β-carbyne, the bond by the formula (=C=C=)n. And although the possibility of the presence of carbon compounds in a closed framework structure was recognized by chemists, the discovery of a new stable carbon framework structure by R.Curl, G.Croteau and R.Smalley in 1985 became a scientific sensation.

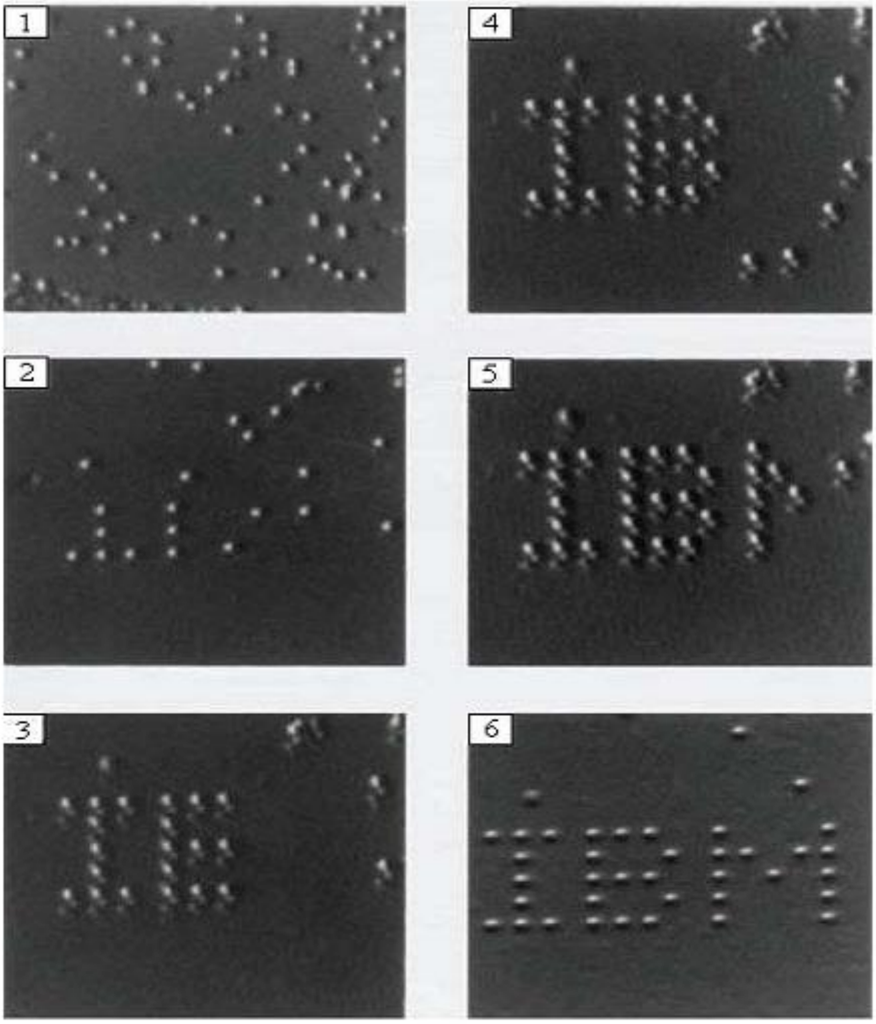
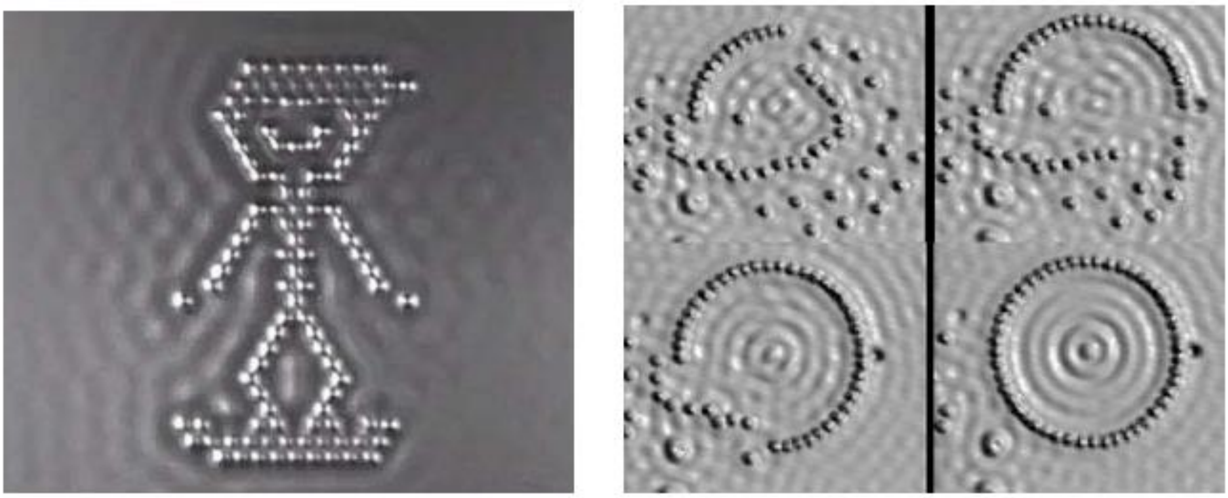


**Figure 6.** The process of forming the acronym "IBM" using nickel based xenon atoms.



а) б)

**Figure 7.** Examples of atomic structures obtained using AFM: a “dancing child” of carbon atoms on the surface of a metal (a) and a circle of copper atoms on the surface of iron (b).

The new type of carbon is called fullerene. The structure of fullerene is very similar to the frame of a regular soccer ball, made from pieces of leather (Fig. 8a). Fullerene consists of 60 carbon atoms. Its structure is fundamentally different from the structure of other types of carbon - graphite and diamond. Fullerene is named after the architect and inventor B. Fuller, who designed and built the first geodesic dome - a hollow spatial steel spherical structure made of straight rods (Fig. 8b).

Fullerene, which has a closed molecular structure, is called an atomic microcluster. Microclusters of various materials, including iron and lead, have already been synthesized. Microclusters - are a new phase of solids with unusual chemical and physical properties.

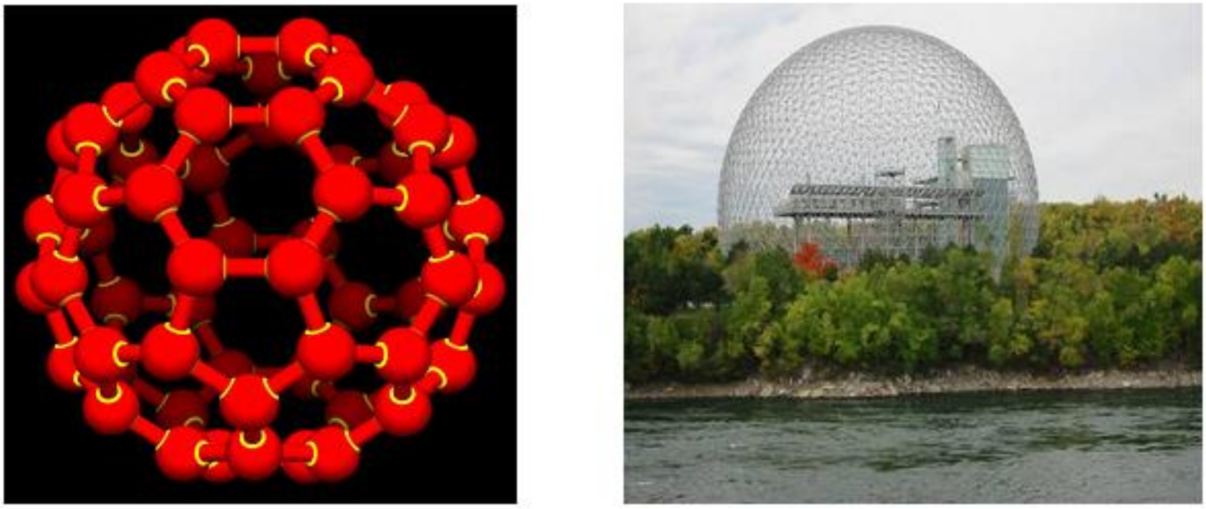


**Figure 8.** Model of the C60 fullerene molecule (a) and the Montreal Biosphere Building by architect B. Fuller (b).

The main one is to increase the activity of the reaction. They easily attach atoms of other substances to themselves and form materials with fundamentally new properties. On their basis, a new stereochemistry of carbon has emerged, which makes it possible to purposefully create new organic molecules and, consequently, substances with a given shape and properties.

The activity of fullerenes allows them to be used in technological processes associated with growing crystals, carrying out selective catalytic changes and, above all, obtaining materials with completely new electronic, magnetic and optical properties. These are, for example, polymer materials with high electrical conductivity and magnetic properties, new catalysts, highly selective sorbents, new classes of superconductors, semiconductors, magnets, ferroelectrics and nonlinear optical materials. It is possible to create a new material that is 200 times stronger than steel.

Fullerenes with a typical cluster size of about 1 nm can be used as “nanoparticles” to create new materials, including for ultra-dense recording of information. Films based on fullerenes solve the problem of cleaning contaminated surfaces. The increased reflectivity of the fullerene-coated surface when irradiated with a laser allows the aircraft to become virtually invisible to laser radars. New technologies for the synthesis of diamond and diamond-like compounds of extremely high hardness are being created.

## Conclusion

To summarize, it should be noted that the study of the nanostate of substances and their application in technology is one of the promising areas of modern materials science. Along with the obvious advantages and prospects of using a new class of materials, the main problems in this area have already clearly emerged today. First of all, they are associated with the difficulty of obtaining pure substances, without foreign impurities, with the same size of structural components. Therefore, the task of regulating the dispersity and other properties of nanomaterials during their production using technical and methodological techniques is relevant. Solving this problem would significantly expand the range of these materials and increase the efficiency of using the resulting product.

Another problem is the high cost of nanomaterials. The cost of the final product greatly depends on the production method, but it significantly exceeds that of traditional materials. In this regard, the problem of finding cheap raw materials and reducing the cost of technology arises. In addition, nanopowders are subject to requirements to prevent their pyrophoricity and ensure explosion safety.

The main ways to solve these problems are outlined. Control of technological parameters of synthesis processes allows you to regulate the dispersion, shape and other properties of the resulting products, expanding the scope of their application.

Reducing the cost of nanomaterials, in turn, could be facilitated by the use of cheaper types of raw materials, for example, waste from various industries, such as scrap, sludge, spent etching solutions, and a reduction in pyrophoricity should be ensured by the development of special technologies and passivation methods.

All of the above leads to the fact that active developments are currently underway to solve a number of problems to increase the efficiency of methods for obtaining, studying the properties and using nanomaterials in modern technology and the industries of the future.

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