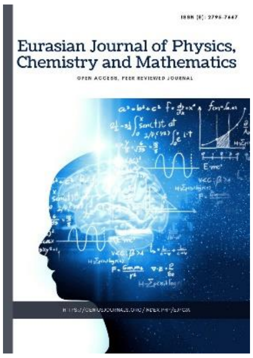

Technology of Obtaining Carbon Nanotubes and Their Use in Practice. Technologies of Obtaining Carbon Nanotubes

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ABSTRACT

The article on carbon nanotube production technology and their practical use (carbon nanotube production technology), author Uktam Dekhkonovich Sherkulov, Navoi State Pedagogical Institute. The article provides information on the technologies for obtaining nanotubes. The main 8 technologies are described in detail. The article is intended for scientific researchers and graduate students. The article is intended for graduate students and scientific researchers.

Keywords: nanotubes, nanotechnologies, nanosystems, thermophysical properties, powder metallurgy, particle appearance centers.

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.

Technologies For Producing Carbon Nanotubes

The main methods for the production of nanomaterials can be divided into a number of technological groups (Fig. 1): methods based on powder metallurgy, methods based on the production of amorphous precursors, surface technologies (creation of nanostructured coatings and modified layers), methods based on the use of severe plastic deformation and complex methods using sequentially or in parallel several different technologies.

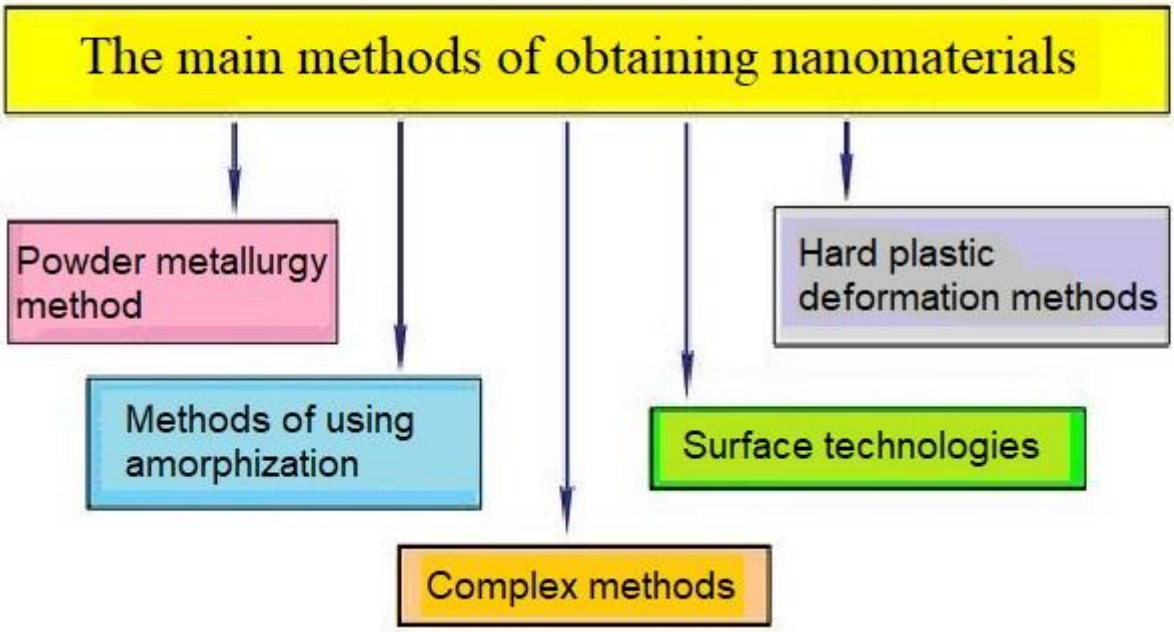

Figure 1. Basic methods for obtaining nanomaterials.

1. Powder metallurgy methods.

These methods can be divided into two groups: methods for producing nanopowders and methods for producing compact products from them. Depending on their options, several methods can be used to obtain nanopowders and form bulk products.

1.1 Methods for obtaining nanopowders.

There are a number of general approaches and features that are common to all methods of producing nanopowders and distinguish them from traditional methods of producing powders [1, 2]:

- high rate of formation of particle centers;
- low particle growth rate,
- the largest size of the resulting particles does not exceed 100 nm,
- small range of particle size distribution;
- stability of obtaining particles of a certain size;
- repetition of the chemical and phase composition of particles;
- high requirements for control and management of production process parameters.

A common feature of powder nanoparticles obtained by any method is their tendency to combine into aggregates and agglomerates [3]. As a result, it is necessary to take into account not only the sizes of individual nanoparticles, but also the sizes of their aggregates. It is impossible to make a clear terminological distinction between aggregates and agglomerates, but it is known that in aggregates the connection between crystallites is stronger and intercrystalline porosity is smaller [3]. Aggregated powders require higher temperature and/or pressure than non-aggregated powders to achieve a certain material porosity during the subsequent compaction process.

All methods for producing nanopowders can be divided into two groups. The first group includes technologies based on chemical processes, and the second - on physical processes.

1.1.1. Chemical vapor deposition technology.

This group of technologies is based on the use of chemical reactions of metal compounds in the gas phase. In this case, in a certain zone of the reaction chamber, these compounds thermally decompose with the formation of nanodust and solid deposits and enter into chemical reactions with the formation of gaseous substances or dust and gaseous substances [2,4]. Metal halides (mainly chlorides), alkyl compounds, carbonyls and oxychlorides can be used as raw materials. The size of the resulting particles can be controlled by temperature and deposition rate. Using this technology, nanopowders of silicon, boron, titanium oxides, zirconium oxide, aluminum oxide, nitrides, silicon carbonitride, titanium diborides with particle sizes from 20 to 600 nm are obtained [2].

In the group of technologies under consideration, two main methods can be distinguished: transfer through the gas phase and subsequent regeneration with decomposition [4].

Another developed method can be attributed to chemical vapor deposition technology, which is a high-temperature hydrolysis method [5]. It is based on the interaction of compounds, mainly chlorides, in a hydrogen-oxygen flame. With their help it is possible to obtain multicomponent compounds. In particular, nanopowders of SiO_2, TiO_2, Al_2O_3 and ZrO_2 were obtained.

1.1.2. High-energy thermonuclear technologies.

This group of technologies is based on the use of reactions occurring at high speed under conditions far from equilibrium under high-energy influence. Two methods have been used to obtain nanopowders: detonation and plasma-chemical [2].

Detonation synthesis is based on the impact of a shock wave with a pressure of up to several tens of GPa on a mixture of initial reagents. Using this method, for example, diamond nanopowder with an average particle size of 4 nm is obtained from a mixture of graphite and metal powder under the influence of an explosion of organic substances with a high carbon content and low oxygen content [6]. Nanopowders of various morphological forms of carbon and oxides of Al, Mg, Zr, and Zn were also obtained.

Plasma-chemical synthesis is carried out using low-temperature plasma from arc or glow discharges (conventional, high-frequency or microwave discharges. Metals, halides or other compounds are used as feedstock.

Due to the fairly high plasma temperature (up to 10,000 K) and high interaction rates, the transition of almost all initial substances into a gaseous state and their subsequent interaction and condensation of products in the form of nanopowder with particles of regular shape, having sizes from 10 to 200 nm, is ensured. The highest temperatures and power are achieved when using installations with arc plasmatrons, and the purest and most uniform nanopowders are obtained when using microwave plasmatrons [2]. When using active media containing carbon, nitrogen, boron or oxygen, nanopowders of carbides, nitrides, borides and oxides of various elements, as well as multicomponent compounds, are obtained by plasma-chemical synthesis. [2,7,8]. When using reducing media, it is possible to obtain powders of refractory metals from oxides [2,8]. Laser heating can also be used as a source for creating and maintaining plasma by heating [9]. Fullerene nanopowders are obtained in this way.

1.1.3. Technologies of precipitation from solutions.

This group of technologies is one of the most studied methods for the production of nanopowders [2]. A common feature of this group is the conduct of chemical reactions in solutions of salts in water. Several different methods are used. In the case of the chemical precipitation method, after preparing solutions of metal salts, suitable conditions for precipitation are created and a precipitating agent is added and the metal oxide powder is precipitated while separating the hydroxide precipitate. Deposition conditions are adjusted by changing pH, temperature, and adding buffer solutions. The most commonly used precipitants are ammonia solutions, ammonium carbonate, oxalic acid, and ammonium oxalate, and soluble nitrate salts are preferably used as precipitated substances. As a result, oxide nanopowders are obtained. If necessary, metal nanopowders can be obtained by heat treatment in a reducing environment. The method has found quite wide application for the production of multicomponent powders, when several compounds are precipitated from multicomponent solutions at once [2, 10]. The main disadvantage of the method is the use of large volumes, a significant content of impurities in the powders and a large scatter of particle sizes [2].

The sol-gel process was developed specifically for the production of oxide ceramics [11]. The process includes the following stages: preparation of alkoxide solutions, their catalytic interaction with subsequent hydrolysis, condensation polymerization, further hydrolysis. An oxide polymer (gel) is obtained as a product of the process. It is subjected to aging, washing, drying and heat treatment. The disadvantage of the method is the complexity of the hardware design, but the advantage is the high purity and homogeneity of the synthesized compounds, as well as the possibility of obtaining a variety of nanopowders.

The method of liquid-phase reduction from solutions is used to obtain only metal nanopowders with low values reduction potential (copper, silver, nickel) [12]. It consists of preparing a solution of an organic metal salt, followed by adding a strong reducing agent and separating the precipitated metal nanopowder. The particle size of the resulting powder is 20-40 nm and the spread of particle sizes is very low. An example of the use of this method is the production of copper nanopowder using an aqueous solution of hydrazine hydrate with lithium sulfate and a solution of copper nitrate in 4-methylpentanol [2]. These solutions are mixed and an emulsion is obtained, after separation of which the copper nanopowder is in the organic phase. To obtain the powder itself, it is separated, filtered and dried.

Hydrothermal synthesis method uses chemical reactions hydrothermal decomposition and oxidation that occur in water environments at elevated temperatures (100-370^{0}C) and pressures (up to 100 MPa) [11]. The method makes it possible to obtain oxide nanopowders with a narrow particle size distribution. The disadvantage of the method is the high cost and complexity of the equipment, as well as the frequency of the synthesis process [2].

The microemulsion method includes the following steps: preparation of an emulsion from two immiscible liquids – aqueous solution and oil, precipitation of metal hydroxide within drops of aqueous phase by adding an organic precipitant, separating the components, drying the precipitation product. There is data on the production of Y_2O_3 powder with spherical particles up to 800-1000 nm in size and silver powder with a size of 2-2.5 nm using this method [13].

The cryochemical method for producing metal oxide nanopowders involves dissolving salts, rapid freezing of the resulting solutions, sublimation of the solvent, and thermal decomposition of the residue. Using this method, copper and yttrium oxide powders and powders of the Al_2O_3 - 10 wt.% ZrO_2 - 2 wt.% MgO system were obtained [14-16]. The advantages of this method include the possibility of obtaining homogeneous nanopowders of complex composition [2].

1.1.4. Technology of decomposition of unstable compounds.

Currently, this technology is considered as a promising method for producing nanopowders with a particle size of 20-300 nm [2].

The most studied is ***the thermal decomposition*** of azides, oxalates, perchlorates, styphnates, permanganates, carbonates, hydrates, citrates, acetates, hydroxides, alcoholates [17,18]. The process involves three reactions: thermolysis, oxidation and hydrolysis. The advantages of this method include low process temperature, small reaction volumes, the absence of labor-intensive and ineffective operations of washing and filtering the final products, controlled dispersion, good sintering ability and high purity of the resulting powders. The disadvantage of the method under consideration is the difficulty of controlling and regulating particle sizes with the simultaneous competitive occurrence of two processes - decomposition of the initial compound and sintering of the particles of the final product under the influence of temperature. Moreover, the powders obtained by this method are characterized by high chemical activity [2]. To obtain nanopowders of metal oxides, it is promising to use alcoholates (alcohol derivatives of metals) as starting products. At the same time, it is possible to deeply purify alcoholates from compounds of other metals due to their volatility and solubility in organic solvents. Other examples of using the method include the production of magnesium oxide nanopowder by thermal decomposition of magnesium carbonate trihydrate and the production of nanopowders of iron, cobalt, nickel and copper with particle sizes of 100-300 nm by pyrolysis of their formates at a temperature of 470-530 K [2].

Another method belonging to this group is ***the radiation decomposition of compounds***. Using this method, by decomposing silver azide, silver nanopowder was obtained, in which there were mainly two groups of particles - with a size of 5-30 nm and 170-220 nm [19]. In this case, particles up to 100 nm in size had a spherical shape, and large particles had a faceted shape. The same method can also produce Pd and Cd nanopowders, which have very high chemical resistance.

1.1.5. Use of restorative processes.

The most famous of this group is ***the method of hydrogen reduction of metal compounds*** [2,4]. Metal compounds (hydroxides, chlorides, nitrates, carbonates) enter into a reduction reaction in a current with hydrogen at a temperature of about 500 K. The chemical reduction reaction using the example of metal chloride can be written as: $MeCl_2 + H_2 \leftrightarrow Me + 2HCl$. This method can usually produce powders of iron, tungsten, nickel, rhenium, molybdenum, copper, calcium; There is also the possibility of obtaining powders of alloyed alloys and steels [4]. The resulting metal nanopowders are characterized by a low impurity content and a narrow particle size distribution [2].

This group also includes the chemical-metallurgical method. In accordance with this method, the synthesis reaction of low-water hydroxides is first carried out by gas-phase interaction, and then the resulting hydroxides are heat treated in a reducing environment, for example, in hydrogen [17,18]. As a result, nanopowders of iron, nickel, cobalt, molybdenum, tungsten, and copper are obtained. If heat treatment is carried out in air, then nanopowders of oxides, for example Al_2O_3,

TiO_2, ZrO_2 or their compositions, are obtained. The advantages of the method are the small spread of nanopowder particles in size, low impurity content, relatively inexpensive technological equipment, and easy transition from the production of one powder to the production of another.

1.1.6. Physical vapor deposition methods.

These methods for producing nanopowders are currently most widely used. This is due to the fact that the technologies for evaporating a substance using various high-intensity energy sources and its subsequent deposition from the vapor phase are sufficiently mature, are easily controlled and provide high requirements for the purity of the resulting nanopowder, especially when using chambers with a controlled atmosphere. In the latter case, vacuum chambers or chambers filled with inert gases - helium or argon, xenon - are most often used. When metals evaporate in a vacuum or an inert gas, the metal atoms that have passed into the gas phase (vapor) tend to unite into particles of the order of several nanometers, which are then deposited on a cooled substrate [19]. This group of methods makes it possible to obtain complexly alloyed powders, and alloys of a given composition can be obtained both by evaporation of a pre-alloyed material and by the simultaneous evaporation of individual components. The particle size of the resulting powders, depending on the type of method and technological parameters, can range from 5 to 100 nm [2].

Depending on the type of evaporation process, the following types of methods can be distinguished.

Thermal evaporation. With this method, the evaporated substance is heated in a crucible. Currently, different heating methods are used, usually using high-intensity energy sources: high-frequency induction, electron beam, electric arc, plasma, laser.

Explosive evaporation. This method is currently being developed rapidly. It is based on the release of a very large amount of energy in a short period of time. In this case, the material evaporates, and then, due to a rapid increase in volume, it cools with the condensation of vapors into small particles [2,19]. In some cases, part of the material may not have time to evaporate, melts and explodes into liquid droplets. An additional factor promoting atomization may be the release of gases dissolved in the source material [19].

To supply the required amount of energy, a powerful pulse of electric current, an arc discharge or a laser pulse are used [19]. The most widespread version of this technology is the explosion of a wire with a diameter of 0.1-1 mm under the influence of a current pulse with a duration of 10^{-5}-10^{-6} s, a voltage of 10-15 kV and a current density of 104-106 A/mm2 [2,19 ,21,22]. In this case, the current discharge is created by a capacitor. The size and structure of particles are controlled mainly by changing the density and speed of the supplied energy [2]. The method makes it possible to produce high-purity spherical powders with particle sizes up to 5-10 nm, including from metals with a high melting point and high chemical activity [2,19]. There is also data on the production of Al_2O_3 and TiO_2 nanopowders from ordinary ceramic powders using a similar method [23]. The disadvantages of the method are: significant energy consumption and, as a consequence, the relative high cost of the resulting nanopowders and the difficulty of removing micron-sized particles that arise from melt droplets [2].

Evaporation in a flow of inert gas (levitation-jet method). With this method, metal evaporation is carried out in a flow of inert gas, for example from a drop of melt at the end of a wire, heated by a high-frequency magnetic field [24,25]. The size of the resulting particles depends on the gas flow rate - with an increase in speed it can decrease from 500 to 10 nm with a simultaneous decrease in the scatter of particle sizes [24,25]. The method under consideration produces, in particular, Mn and Sb nanopowders. [24-26]. The latter powder was amorphous due to the high rate of quenching in a gas stream. There is a variant of the method under consideration, called the cryogenic melting method [27]. It consists in the fact that the wire is melted in a liquid with a very low temperature, for example in liquid nitrogen.

1.1.7. Melt spraying.

This group of methods is based on rapid spraying and cooling melt of the starting material. This technology makes it possible to obtain powders size not less than 100 nm. At the same time, the resulting powders with a particle size of 0.5-10 microns have a nanocrystalline (and in some cases amorphous) structure [19] and, therefore, can also be classified as nanomaterials, and the technology for their production can be classified as nanotechnology. Processes for obtaining powder can be carried out in a protective atmosphere. Currently, the following three variants of this technology are mainly used to obtain nano- and/or nanocrystalline powders.

Contact cooling using a water-cooled disk or drum. This method is based on feeding molten material onto a rapidly rotating water-cooled disk or drum, which is made of materials with high thermal conductivity [19]. Typically, copper is used as such a material. This ensures a cooling rate of up to 108 K/s. The surface of the drum or disk is made rough (toothed), since in the case of a smooth surface it will be possible to obtain foil, strip or wire with a thickness of about 10-50 microns with an amorphous or nanocrystalline structure.

The resulting powder has a flaky particle shape [19]. This particle shape can lead to a non-uniform structure and anisotropy of properties in products formed from such powders. In this regard, the powders obtained by the method under consideration are usually additionally subjected to mechanical grinding. This is the main disadvantage of the method.

Impact spraying of the melt. With this method, a stream or drops the melt is mechanically broken into small particles upon impact with intensely cooled, rapidly rotating metal blades [19,30]. A cooling rate of up to 107 K/s is provided. As in the previous method, the powder particles have an irregular shape and in order to obtain high-quality products with a uniform structure during subsequent molding, it is necessary to additionally subject the powder to mechanical grinding.

Electrohydrodynamic melt spraying. This method uses electrostatic forces to atomize the melt. The melt jet is fed into a nozzle with a hole diameter of about 80 mkm, before which the ring electrode is located. A constant voltage of 3-20 kV is applied to it. As a result, positively charged small drops of the melt fly out of the nozzle, forming powder particles after cooling. The particle size, depending on the material and technological parameters, can be 100 nm – 10 mkm [19]. The disadvantage of this method is the very low productivity (2 g/h from one nozzle).

1.1.8. Mechanical grinding.

Mechanical grinding of material particles (pre-formed powders, granules, ground ingots) is one of the most common methods for producing powders. It is especially easy to obtain powders based on brittle materials. Powders of plastic, high-strength and amorphous materials are more difficult to obtain. In this case, the danger of excessive heating of the material and its contamination with wear products of the working parts of technological equipment increases [19].

During mechanical grinding using mills, a decrease in the particle size of the material occurs as a result of intense crushing between the working parts of the mill. Depending on the type of material and the required properties of the nanopowder, planetary, ball and vibration mills are mainly used [2]. The average particle size of the resulting powders can range from 5 to 200 nm. Another option for the method could be the use of attritors and simoloyers - high-energy grinding devices with a stationary drum body and stirrers that transmit movement to the balls in the drum [3]. Attritors have a vertical drum arrangement, and simoloyers have a horizontal drum arrangement. In this case, the grinding of the ground material occurs mainly due to abrasion rather than impact. The main disadvantage of the method is contamination of the powder due to wear of the working parts of the equipment.

In ***the countercurrent fluidized bed grinding method***, powder particles are crushed by colliding with each other [19]. In this case, the processes of mutual collision of particles accelerated to high speeds in a gas stream occur in the middle of the fluidized layer formed by these particles. Only a very small fraction of particles comes into contact with the walls of the chamber in which the grinding process is carried out [31,32]. At the bottom of the working chamber there is a system of nozzles from which gas comes out under high pressure. The resulting gas jets meet each other in the center of the lower part of the chamber, loosen the substance being ground and form a fluidized layer. In this layer, the particles being ground move at high speeds from the edges to the center of the chamber. From the grinding zone, the flow of particles is carried away by gas jets to the upper part of the installation, which has a separator for separating particles by size. Particles smaller than a certain size are carried away with the gas flow into the filter system, where they are separated from the gas flow and end up in a storage hopper. The separator directs large particles back to the grinding zone.

The powders obtained by this method meet high requirements for purity, are highly homogeneous and contain particles of approximately the same size. The intense gas flow significantly reduces the heating of particles during grinding. This allows the processing of amorphous and nanocrystalline powders. The main disadvantage is the complexity and high cost of technological equipment in the case of obtaining powders with nano-sized particles.

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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