AN OVERVIEW OF NANOPARTICLES: METHODS OF OBTAINING, APPLICATIONS AND CHARACTERISTICS

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ABSTRACT: Lately, the term of “Nanotechnology” becomes very popular. This combines different representations and interpretations of action methods on materials. It means, before proceeding to characterization, methods of obtaining and applications, it is necessary to clarify about which dimensions will be further discussed. Is clear, that the term of “Nanotechnology” becomes obtained from two words: “nano” that means looking into a scale of billionth part (10^{-9}) and “technology” – the application of knowledge in practice. So hard to imagine these dimensions and to clarify a little, it’s important to mention that the thinnest atom of Hydrogen has a diameter of a 10^{-10} m and especially the DNA spiral has the diameter of 1 nm. So, nanotechnology can be defined as a set of technics and methods, based on manipulation with atoms and molecules in a scale from 1 to 100 nm. The usage of special characteristics of materials in a scale of some couples of nanometers discovers new possibilities of development of process technologies in a strong link with electronics, material study, chemistry, medicine, mechanics and a lot of science branches. Obtaining new materials and development of new methods conduits to a technic-scientific revolution in information technology, obtaining new construction materials, producing medical preparations, construction of super exact devices etc. The overview in the following paper is designed to facilitate informed people and a reference for researchers and professionals regarding the know-how transfer, multidisciplinary collaboration and further development of methods of obtaining, applications and characterization on nanomaterials.

KEYWORDS: nanotechnology, nanoparticle, nanomaterial, synthesis, discharge, explosion

1. INTRODUCTION TO NANOTECHNOLOGY

Nanoparticles are materials with dimensions in a range between 1 and 100 nanometers (nm). Their small size impacts completely new approaches regarding unique properties, including physical, chemical, mechanical, technological and application of these in a very different branches of industry and furthermore in our lives.

The first pioneers in nanotechnology are rightfully Richard Feynman, an American physicist who considered in his lesson “Plenty of Room at the Bottom” in early 1959 the possibility of manipulation with separated atoms in scope of creation of new matters [1]. Norio Taniguchi, a Japanese professor who introduced first time the term on nanotechnology in 1974 to describe semiconductor processes regarding pellicle deposition and ion beam processing [2]. Eric Dexler, in his popular book “Engines of creation” (1986) proposed the creation of new devices, so called “molecular machines” and discovered amazing possibilities related to the development of nanotechnology [3].

For a better understanding about which dimensions will be related, it is necessary to mention that the smallest atom of Hydrogen has the diameter of a 1Å (Ångström) that is equal to 10^{-10} m and specially the DNA spiral has the diameter of 1 nm, that is about of 50-100 thousand times thinner than human hair. The upper limit of the field of nano corresponds to the minimal elements of so-called large-scale integration circuits widespread in semiconductors and computer technics. But from another side, many viruses have dimensions of 10 nm, and one nanometer corresponds almost exactly to protein molecule [1].

From previously related, it becomes clear that nanotechnology bring together all technical processes connected directly to atoms and molecules [1].

Exactly for this reason, nanotechnology represents a perspective in the field of obtaining new construction materials, semiconductor elements, information recording devices, precious medical preparations, etc. [1].

Nanotechnology is progressing quickly and could revolutionize numerous industries, significantly benefiting our lives.

Nanoparticles come in various types and classifications, where are characterized by their size, form, and chemical properties [4].

2. METHODS OF OBTAINING OF NANOPARTICLES

There are given the many different principles, this section outlines one of the main concepts that have a crucial part in the evolution of nanotechnology in general. This concept refers to two principles, but fundamentally different, approaches to material processing and the creation of desired products.

These approaches are conventionally referred to as "top-down" and "bottom-up" technologies [1, 4, 8].

Methods for creating nanomaterials can be classified according to various criteria, such as physical, chemical, and green technologies. They can also be divided into electrical and mechanical methods, which are further categorized into electric discharge, electric arc, electric explosion, laser ablation technologies, also should be mentioned that exists technologies as well as powder metallurgy, controlled crystallization from an amorphous state, intense plastic deformation, thin film technologies, electrolytic deposition, Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD), sol-gel method, and so on [4, 6, 8].

In the Figure 1 are presented some classifications of nanomaterials synthesis methods divided by criteria of "top-down" and "bottom-up" approaches which are subdivided into categories of physical, chemical and green methods [4, 8].

2.1 "Top-Down" approach

From the previously related information, results that the "Top-down" approach is based on the different methods of acting on a macro level to materials, by decreasing their size down for obtaining of new matters in a microscopic or nanostructural scale [1, 4, 6, 8, 10, 11].

2.1.1 Sonication

This method of synthesis by sonication of nanoparticles is based on applying of ultrasound (mechanical waves with a frequency more than 20 kHz) on the prepared materials. The sound energy is dispersed for agitation of particles or fibers in liquids. The probes are processed by wave generators in two conditions: as direct action, where sonicator's horn is applied direct to the dry powders or liquid suspensions and indirect sonication, where a container with prepared suspension is inserted in a bath with a liquid through which is propagated the ultrasound [4, 7].

Sonication is a very specific process what involves different physicochemical interactions that influence further disintegration or agglomeration of matters and additional effects of chemical reactions [7].

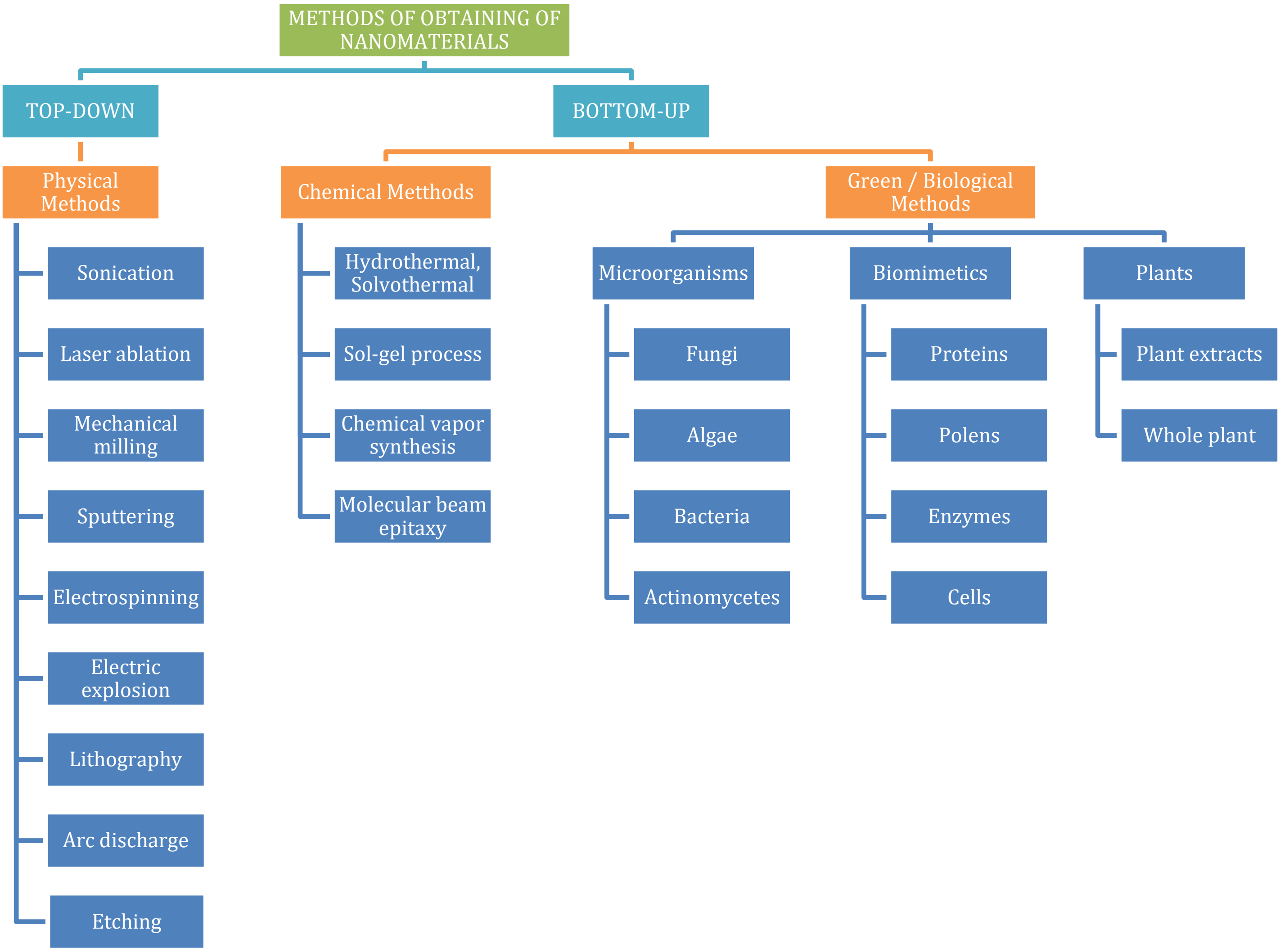

Figure 1. Classification of synthesis methods of nanomaterials [4, 10, 11]

The energy of sonication *(E)* depends of the applied power *(P)* and total amount of processing time *(t)* where ultrasound is propagated on suspension [7].

The equation (1) describes the energy of sonication:

$$E = P \times t \quad (1)\ [7]$$

Ultrasonic devices can function continuously or in the pulsed mode. In the last one, waves are alternated with pauses, where the time of vibrations and breaks can be adjusted. Pulsed mode is recommended because the operating temperature can be better controlled, and this facilitates minimization of parasite effects during the operation [7]

The effect of suspension volume is calculated as density of energy (W·s/mL). This measure indicates the quantity of energy supplied on volume of the suspension. When operating with few quantities the temperature of the suspension will increase faster and most effective cooling will be necessary [7].

The calibration of power can be determined by a calorimetric method and is based on temperature rise measurement in a liquid ambiance vice versa on time as a effect of cavitation what appears in a liquid by an introduced ultrasound sample [7].

At a specific power regime, the rise of temperature in liquid is registered on time and the supplied power can be determined by equation (2) [7].

$$P = \frac{dT}{dt} M C_p \quad (2)\ [7]$$

where P is supplied ultrasonic power (W), T is temperature (K), t is time (s), C_p is the specific heat of the liquid (J/g·K) and M is the liquid mass (g) [7].

2.1.2 Laser ablation

This process attributes to a situation where atoms or thin pieces of raw material are ablated in result of applying laser beam. When the beam is focused at target, the rise of temperature causes evaporation of material, hence the formation of plasma conduces to collision of atoms and predisposition for agglomerates formation. This method is frequently used for obtaining nanomaterials comprising quantum dots of semiconductors, oxides of metals and pure metals, carbon nanowires and nanoparticles [4, 8].

In different ambient conditions, the presence or absence of air or other gases can influence synthesis of nanoparticles, but advantages and disadvantages are provided as high cost for vacuum generating or reducing of pure state of particles by presence of foreign matters or affecting environmental toxicity in result of formation in air. As measures to reduce environmental impact – liquid is more safe option for laser synthesis [4, 8].

There are 3 methods of laser-based obtaining of nanoparticles in fluids: laser ablation in liquids (LAL), laser melting in liquids (LML), and laser fragmentation in liquids (LFL) [4, 8, 9].

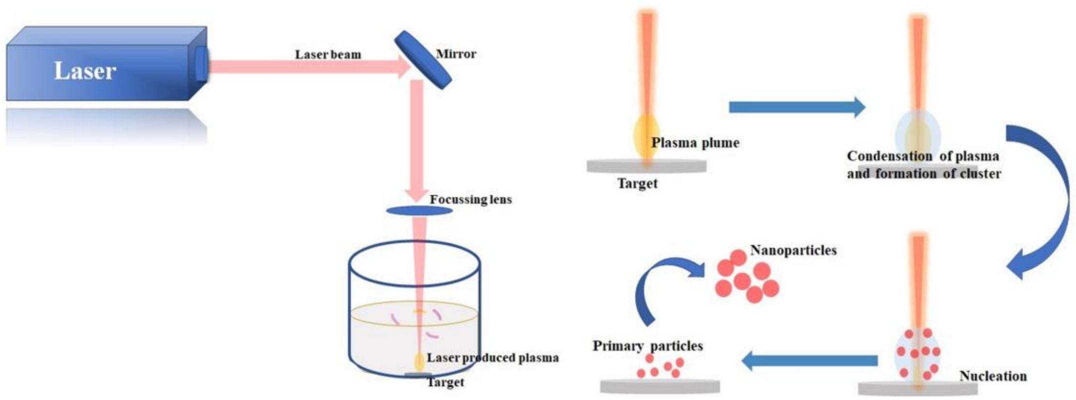

Figure 2. Illustration of experimental scheme and procedure of obtaining particles using laser ablation in liquid [8]

2.1.3 Mechanical milling

This method of synthesis of nanoparticles is also called Ball milling or High Energy Ball Milling (HEBM) and refers to powder metallurgy [4, 10, 11, 12].

The principle of obtaining nanoparticles is based on reducing particle size. The energy is transferred from balls to raw material by frequent collision between them. Into a container that is set into rotational motion are introduced together balls and materials, what determines producing of elastic, plastic and shearing deformations in result of which nanoparticles are obtained. The conditions which affect the process are method: wet or dry, ball-to-powder weight ratio, milling speed and duration, chamber atmosphere [4, 6, 10, 11, 13].

Mechanical milling is a good corelated to cost and performance method and permits to produce blends of various phases and helps the obtaining of composites at nanoscale. The model of production by the described method is shown in Figure 3. Mechanical milling is basically used to produce metallic and alloys nanoparticles and their oxides and carbides, graphite nanoparticles, nitrogen-dopped carbon nanoparticles and polymer nanoparticles as well as poly methyl methacrylate (PMMA) [4, 10, 11].

Principal advantages of mechanical milling are process simplicity, eco-friendly, low-cost, different materials and medium can be selected, a variety of types of nanomaterials synthesis is possible and sizes of nanoparticles, nanostructures and crystallites can be obtained in a range of 1 – 500 nm. Collecting synthesized particles and structures remains the one of the problems that should be resolved when method is applied. The materials may be used for capturing energy and conversion, protection of environment and is appropriate for fine adjustment of composites based on metallic matrix, magnetic nanoparticles etc. [4, 10, 11, 12].

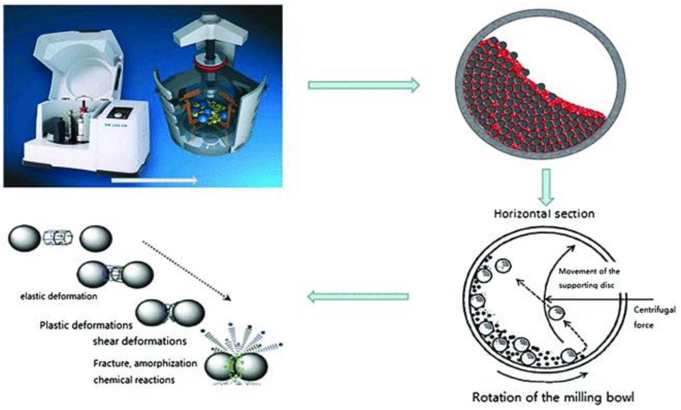

Figure 3. The principle of the ball milling method [10, 13]

2.1.4 *Sputtering*

Obtaining nanoparticles through this method is characterized by removing matter by applying high energy as plasma from bulk material (often this has a shape of a needle) by applying bombardment to target solid surfaces [4, 10, 14].

This method is suitable for obtaining metallic nanoparticles and thin films of nanomaterials. Depositing by sputtering is accompanied by excited gaseous ions bombardment to surface and generating physical detachment of atom clusters that depends also by incident gaseous-ion energy [10, 16, 17].

The sputtering procedure can be prepared in several conditions, using radio-frequency diode, magnetron, rectifier diode etc. Generally, sputtering happens in a cavity which is prepared with gas inside. High voltage application between electrodes ionizing gas and are producing ions what are accelerated in an electric field in direction of target electrode. Magnetron sputtering method is utilized to produce nanofilms and is interesting because sputtered material stays the same as target materials with few impurities in comparison with lithography has advantage of a cost-effective method. The factors that impact nanoparticles formation are the distance to target-substrate, ion quantity, time of deposition and the sort of targets used stuff [10, 18, 19, 20].

Formation of nanoparticles is a dependent function to the distance between electrodes and can be described by Equation (3), where k_B – is the constant o Boltzmann, T – is temperature, p – the pressure, d – the diameter of the atoms which are sputtered [20].

$$\ell = \frac{k_B \cdot T}{\sqrt{2}\pi d^2 p} \quad (3)\ [20]$$

According to Equation (3), the freeway of the atoms can be decreased by temperature drop T, or by increasing the pressure p [20].

The configuration of one method for obtaining of nanostructures by sputtering deposition is shown on Figure 4.

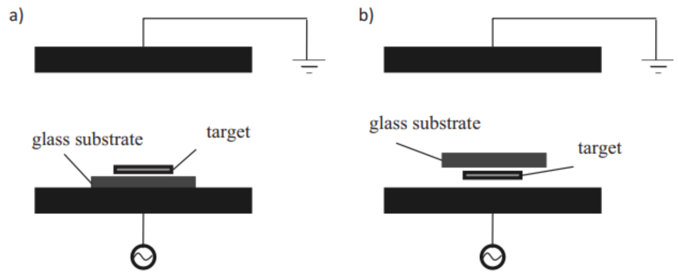

Figure 4. Design of sputtering deposition principle, (a) target is situated on the top, (b) target is situated on the bottom [20].

2.1.5 *Electrospinning*

Electrospinning represents a most simple approach of obtaining nanostructures, predominantly nanofibers, the most of which are polymers and permit them to obtain in a diapason of multiple of 10 of nanometers and increase to some micrometers [4, 10, 21, 23].

The electrospinning system for obtaining nanofibers consists of source for applying high voltage, spinneret and a plate designed for collection of fibers which is connected to ground [23].

The principle of work is pulling the charged threads from the melted material [4., 10, 21, 22, 23].

The working principle is shown in Figure 5, where is shown the obtaining in a random form aligned fibres, or reeled on a drum and obtaining core-shell wires and hollow fibres [23].

A connected method is also developed by Moldovan researchers who formed micro and nanowires by pulling capillary melted metal insulated in a glass shell and levitate in a high-frequency electromagnetic induction field [22].

Other researchers reported the creation of organic, inorganic and hybrid nanomaterials [4, 10].

The influential parameters for assuring of electrospinning process are viscosity, applied electric field, spinneret collector revolutions and feed rate [23].

The advantage of the method is economic cost-effective and feasible to produce in laboratories for different nanofibers applications as well as dye-sensitized solar cells, dye degradation, water-splitting, fuel cells etc. [23].

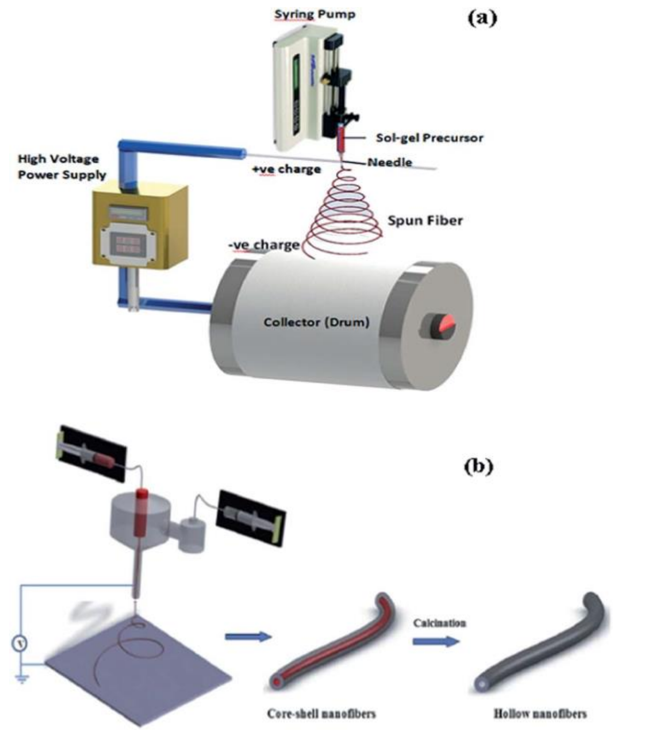

Figure 5. Electrospinning working principle (a) for production of random aligned or reeled on a drum nanofiber, (b) core-shell and hollow nanofibers [23].

2.1.6 Electric explosion

An interesting and fast-growing method became the principle of electric explosion that is based on the effect that occurs when an electrical impulse with a duration of 10^{-5} – 10^{-7} s of a high-density current ~ 10^4 – 10^7 A/mm2 flows through a wire with thickness of 0,1 – 1 mm. Dispersed powders of particles with nano-sizes are obtained by this method [4, 24, 25, 26].

The electric explosion of wire consists in a fast modification of metal physical condition as a outgrowth of intensive energy release in time of current flow in impulse with high density [25].

Electric explosion is accompanied by generation of shock waves and offers the possibility of fast heating of metals with a velocity of about 10^{10} K/s and grown-up temperatures, about T > 10^4 K [24, 26].

At initial state, Joule-Lentz wire heating effect aim a linear dilatation with a speed of 1 – 3 m/s. When an explosion occurs, in result of impulse current flow, the wire is overheated at temperature that exceeds melting point and metallic wire material explodes with a speed of about 5 · 103 m/s and disperses in explosion direction. Pressure and temperature at margin of shock wave became values of hundreds of MPa and respectively 10^4 K. In result of fast condense of vapors flow – small particles are obtained [24].

Figure 6 is presented the electrical circuit of installation.

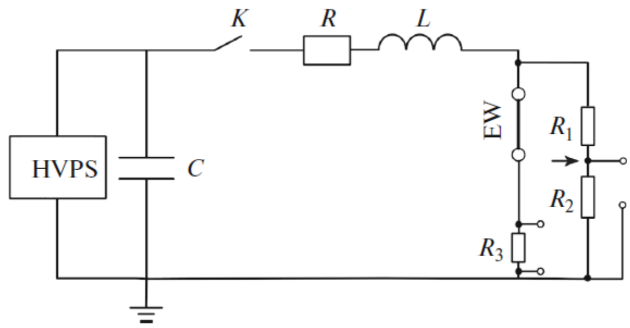

Figure 6. Electrical circuit installation for obtaining nanoparticles by electric explosion of wire [27].

Electrical circuit of installation consists of the following components: *EW* – electric wire, *C* – capacitor what is loaded using a power source for high-voltage *HVPS* with voltage U_0. In time of spark switch *K* is actioned, a high current impulse traverse in the circuit. *R* – is the resistance and *L* – is the inductance of circuit components. Then current impulse flows wire – it is exploded. The data about explosions are functions of impulse time, current and voltage what are registered with aim of current shunt R3 and voltage dividers R1 and R2 [27].

The parameters of wire such as thickness and installation values or RLC circuit influences size and distribution of particles as result of electric explosion [26, 27].

At high frequencies and with larger wire diameters, the skin effect causes most of the energy from a current pulse to be concentrated in the wire's surface layer. This results in uneven heating across the wire's cross section. In order of reducing of irregularity – the wire's radius needs to be thinner than shell depth *δ*, what is described by Equation 4,

$$\delta = \sqrt{\frac{2\rho}{\mu\omega}} \qquad (4)\ [27]$$

where *ρ* – is electrical resistivity, *μ* – material permeability, and *ω* – frequency of oscillation, conditioned by value of inductance *L* and capacity *C*, shown on Equation 5 [27].

$$\omega = \frac{1}{\sqrt{LC}} \qquad (5)\ [27]$$

The accumulated energy *W* is determined as described in Equation 6 [26, 28].

$$W = 1/2\, CU^2 \qquad (6)\ [26, 28]$$

Energy of discharge through the Joule-Lentz heating effect and in the same time of explosion can be expressed by Equation 7 [28],

$$W = \int_{t_i}^{t_j} i u_R dt \quad (7)\ [28]$$

where t_i and t_j are the start and end of the operation, and i – is registered current and u_R – resistive voltage when explosion is happening [28].

2.1.7 Lithography

This technique uses focused beams of electrons or light in order to produce nanoscale patterns such as electronic integrated circuits, micro-electro-mechanical system devices, digital micromirrors for display applications, pressure sensors etc. [4, 10, 29]

Lithography can be categorized in two methods: masked and unmasked lithography [10, 29].

Masked lithography uses masks or molds in order of patterns assignment onto a large area at the same time and permits fabrication with increased efficacy up to several tens of wafer shells on hour [4, 29].

To masked lithography refers to some types of methods: soft lithography, photolithography, nanoimprint lithography [29].

- Photolithography – moreover used technique for integrated circuits and electronics production. This uses an exposition of a polymer which is sensible to light, called photoresist to ultraviolet light (UV) to obtain necessary pattern. At first, UV light with wavelength in diapasons from 193 to 436 nm is lighted using a photomask what is a feature of transparent substrate with opaque zones for exposing on the substrate coated photoresist [29, 30, 31].

In the field, which is exposed, they are dissolved the polymeric chains of photoresist, then obtaining a chemical solution with increased characteristics of solubility so-called developer.

Afterwards, the exposed photoresist is deleted in a developer to generate necessary pattern of photoresist [29].

Nanopillars as well as nanowires and nanorods with diameter of 150 – 500 nm was obtained by Z. Szabo´ et al., Figure 7, [32] through the following method: it has been selected two substrates – one of single crystal ZnO, Zn-terminated c-polar (0001) and a direct current reactive magnetron sputtered thin film of ZnO (150 nm) on Si wafer [32,33].

The wafers after cleaning and dehydration were spin-coated with a photo-resist whit thickness of 200 nm and pre-heated on a plate with 110°C for 60s. The hexagonal close-packed monolayer of Stöber silica and polystyrene spheres with different diameters (180 – 700 nm) were submitted on the resist using Langmuir–Blodgett method in a chamber to exclude UV exposition. After exposure at 0,4 – 0,6 s with 405 nm UV light in a mask aligner, in a several time the with aim of 1:20 HF solution, nanospheres have been removed. In a one part of developer reported to 2 parts of water, the development has been followed by a 10–15 min long after-baking procedure at 110°C in a heater. The nanorods of Zinc oxide have been obtained hydrothermally from the window pattern on the single crystal of Zinc oxide or on ZnO thin pellicle covered Si substrates. [33, 34].

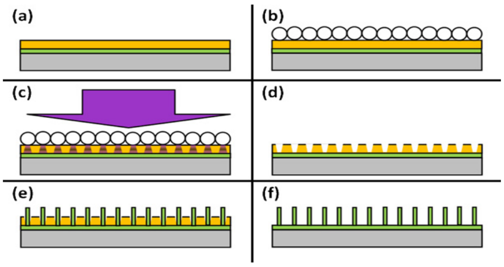

Figure 7. Design of nanosphere photolithography aimed with chemical process, (a) – photoresist spin coating on ZnO surface, (b) – Langmuir-Blodgett deposition of nanospheres, (c) – exposure, (d) – nanospheres removal and photoresist development, (e) – hydrothermal process of ZnO nanopillars, (f) – deleting in acetone of the photoresist [33].

There are 3 types of photolithography: projection printing, proximity printing and contact printing, presented in Figure 8. The contact printing and proximity printings place the mask in contact or in a nearfield to photoresist. The projection, or so-called “stepper” applies a system with optical lens for projection of a deep ultraviolet pattern from a laser with a wavelength in the range of 193 or 248 nm on photoresist that permit to obtain reduction of pattern-size by 2 – 10 times [29].

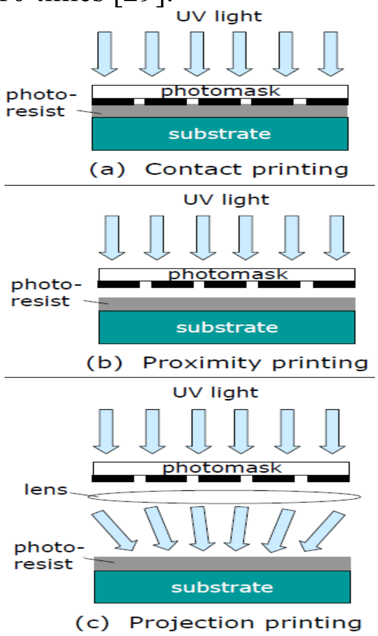

Figure 8. Schematic representation of 3 types of photolithography: (a) – contact printing, (b) – proximity printing, (c) – projection printing [29].

The *maskless lithography* is producing random patterns by a series writing without using masks. They are several types of maskless lithography: electron beam lithography, focused ion beam lithography, scanning probe lithography [29].

- Electron beam lithography (EBL) uses an accelerated electron beam what in order to make an exposure is focused on an electron-sensitive resist [29].

Further, the electron beam spot with a diameter of several nanometers is scanned on the area of resist in a point-to-point manner to obtain patterns [29].

The resolution of electron or ion beam lithography methods is situated in the range of 5 – 20 nm related to ultra-short-wave lengths of beams up to several nanometers. These methods are suitable for obtaining prototypes and devices at nanoscale [29].

Previously, the approach of electron beam lithography was so expensive, but recent research make possible to equip scanning electron microscopes (SEM) with modules for patterns generation, that makes possible electron beam spot scanning within cautious zones to create patterns at nanoscales as systems of electron beam lithography [29].

On the Figure 9 is presented the model of electron beam lithography.

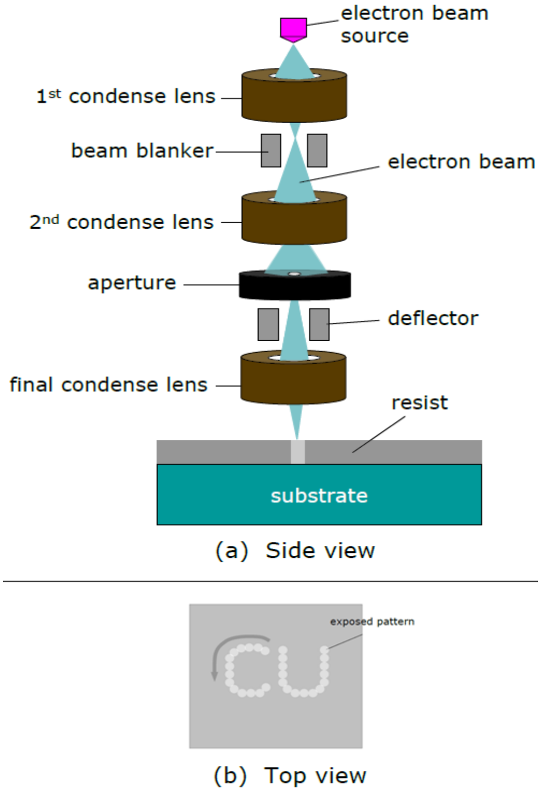

Figure 9. Electron beam is focused on a pellicle of resist for creation of pattern by exposing point-by-point: (a) – side view of the installation, (b) – top view of the exposed pattern by a serial writing [29].

- Ion beam lithography (IBL), as well as electron beam method uses an accelerated ion beam in place of electron beam to reach a metallic pellicle on a substrate [29].

The technique is very popular for construction of semiconduction devices, ultrathin films and superconducting Josephson devices, superconducting quantum interference devices etc. [37, 38, 39].

Focused ion beam (FIB) conducts immediate etching and lay down of necessary forms without a process based on resist. The etching rate is a dependent function of the next factors: the quantity of gallium or helium ions are adjusted by condenser lens, aperture size and time for dosing. If the amount of the focused ion beam meets a specific threshold for inducing superconductivity in silicon, it becomes possible to create the wanted superconducting patterns directly, without relying on a process based on resist [37, 38, 39].

- Scanning probe lithography employs a pointed sample in an atomic-force microscope (AFM) to heating, scratching, oxidizing, or transfer materials onto a substrate's surface for creating nanoscale patterns. Among these methods, dip-pen nanolithography (DPN) is the most used for specific deposition of molecules or nanoparticles on a substrate. This technique can be conducted in ambient conditions without the need for large electromagnetic fields or shear forces [29, 40, 41].

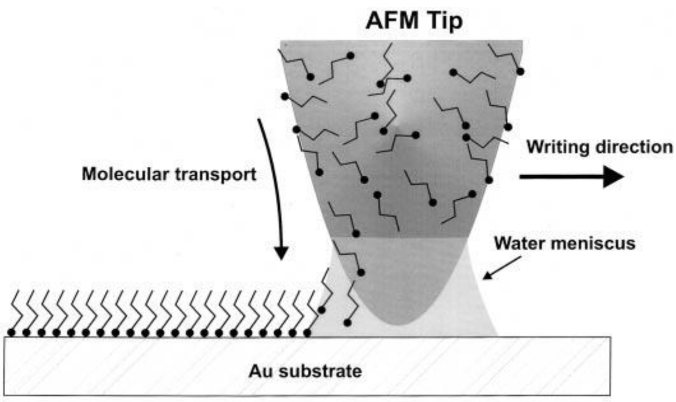

Figure 10. Schematic illustration of "dip-pen" nanolithography. A water meniscus obtained between the tip of atomic force microscope, coated with 1-octadecanethiol (ODT) and the gold substrate. The dimension of the meniscus, controlled by relative humidity is affecting the transport rate of ODT, the effective tip-substrate contact area, and resolution of "dip-pen" nanolithography [40].

2.1.8 Arc discharge

This kind of technology is based on the vaporization of material in an arc discharge between electrodes before condensation, nucleation and further development of nanomaterials [4, 10].

The advantage of technology consists in the simplicity of method. The electric arc discharge can happen in air or in protective atmosphere. Electric arc pulverization is an over technique for obtaining nanoparticles. The method consists of pulverizing electrodes of carbon composite with additive components [42].

The electric arc discharge conducts temperature increases and vaporization of electrode, obtaining in result the gas-plasma, what is formed in a reactor chamber and is deposited on its walls. The anode vaporization is deposited on cathode. The collection of material is realized by water cooling system [43].

Another experimental installation is presented in Figure 11 and the principle of work is described as following: a tube from quartz glass is fulfilled with argon. This tube is submerged fully into a cryogenic medium. With the aim of precise positioning, the arc is maintained between graphite cathode and aluminum anode. The positioning of electrode is ensured by vertical positioner in a range of 2 – 10 mm, powered by a stepper motor. The 10 mm thick aluminum with purity of 99,7% is used as source of aluminum, and with the same thickness and purity graphite rod is used as cathode in order of a carbon source. The electrodes are powered by DC supply. The work parameters such as voltage and waveform were recorded by oscilloscope. The flow of argon gas is an important parameter what is monitored by a flowmeter. In process of DC arc discharge the surfaces of electrodes are emanating aluminum and carbon particles, which are converged within the near of arc plasma and carbon-aluminum nanoparticles are obtained. The flow of argon gas transports particles into liquid nitrogen [44].

The method of arc discharge is frequently used for obtaining carbon nanoparticles, as well as fullerenes, carbon nanotubes, metal oxides etc. used for different applications e.g. energy storage, sensors etc. [4, 10, 42, 43, 44].

The energy released in the process of arc discharge is described by the same relationship as the Equation 6 [27, 28].

At fixed electrode interstice and a constant capacity, the frequency is described by the constant current charging the capacitor *IC* and voltage at which discharge happens, Equation 7:

$$I_C = C\frac{dV_d}{dt} \qquad (7)\ [45]$$

where *C* is capacity, V_d – is voltage of discharge [45].

The mass loss is a dependence of frequency, Equation 8:

$$f = \frac{I}{CV_d} \qquad (8)\ [45]$$

where *f* – is electrical frequency of every discharge, and *I* – is applied current on condenser [45].

The concentration of particles *n* obtained in a electric discharge can be determined as shown in Equation 9:

$$n = N_0\frac{f}{V} \qquad (9)\ [45]$$

where N_0 is the number of primary particles obtained one each electric discharge, and *V* – is the volumetric flow rate [45].

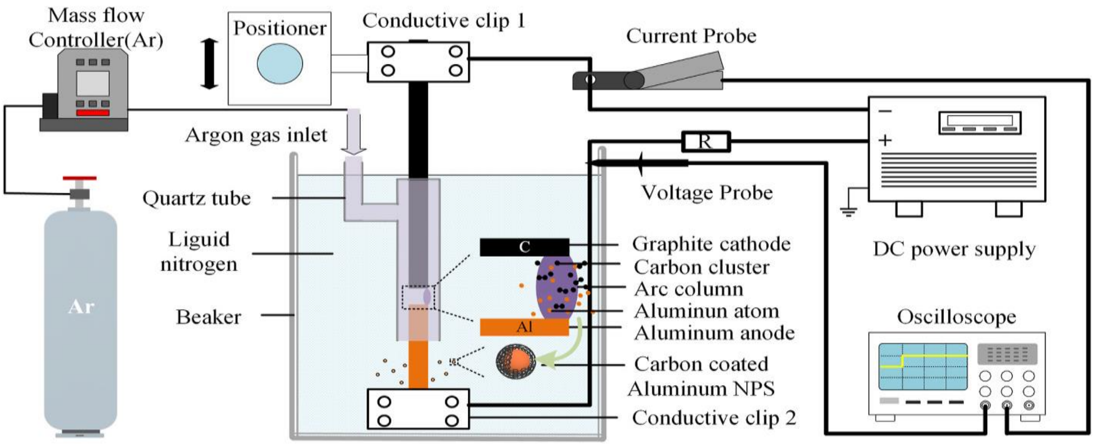

Figure 11. Schematic of the installation for obtaining aluminum-carbon nanoparticles [44].

2.1.9 *Etching*

Etching is a process of obtaining nanoparticles, nanolayers are removed by using chemicals. It is possible to do the etching in two methods: liquid and plasma etching. The technologies are used for increasing morphological properties of nanoparticles [4].

According to study of R.G. Ayvazyan and K. Jacoby – the kinetic regularities of silicon nanoparticles obtaining in the result of quartz etching in a hydrofluoric acid, the strong stability of quartz etching nanoparticles in acid explain structural characteristic dependence of having a fluorinated layer on surface of Si-F boundaries surrounding the grains of silica. The pieces obtained by this method have a big importance in construction of semiconductors. Fused quartz is frequently used for CVD chambers, etching and epitaxial processes [46].

Another approach for the etching process consists in the development of electroless technology of plating so-called etching to obtain nanoparticles of metals or nanostructures of silicon. By metal assisted chemical etching (MACE) makes it possible to obtain nano depositions for different applications, as well as micro-electrical mechanisms, biosensor, energy caption, mass-spectroscopy etc. The method of deposition of droplets is a simple technique that uses a pipette to place a little bit of deposition or etchant solution on a substrate of silicon, facilitating either deposition of droplets or etching [47].

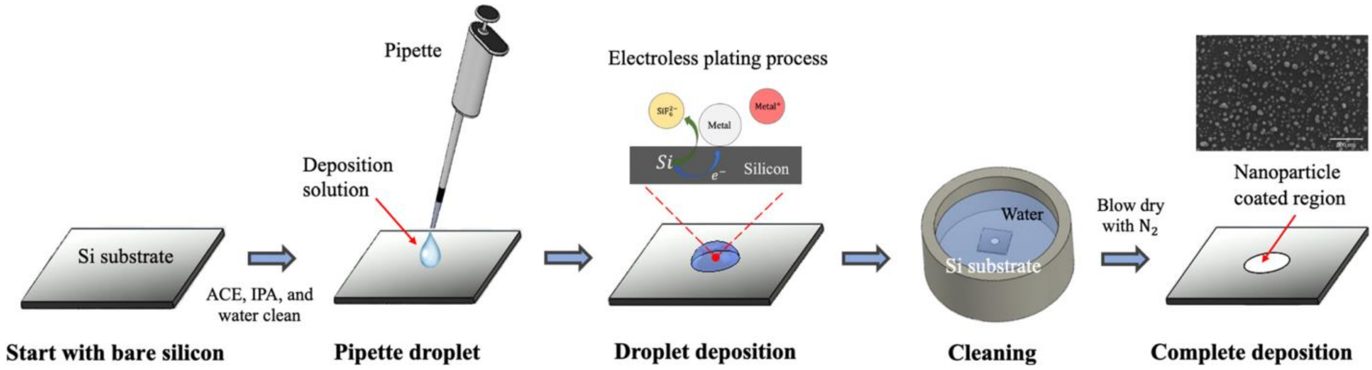

Figure 12. Schematic representation of droplet deposition process [47].

2.2 "Bottom-Up" approach

These approaches are based on miniaturization of material components, forwarded by self-assembly process as a result of nano formation of structures. In these methods, the nanostructures are obtained by assembly of atoms or molecules in particular [1, 4, 6, 8, 10, 11].

2.2.1 Hydrothermal, Solvothermal

In hydrothermal synthesis the nanoparticles are obtained by chemically reacting nanomaterials at specific pressure and temperature in organic solvents or in enclosed aqueous solution [4]. It is possible to be realized in two ways: continuous hydrothermal systems and batch hydrothermal systems. [11].

Nanomaterials are accomplished through heterogenous reaction produced in liquid medium aimed at increased pressure and temperature appropriate to critical point in hermetic recipients. The solvothermal principle is almost the same as hydrothermal with the specificity, that it happens in a non-liquid medium [10].

According to recent studies, with aim of this method it is possible to obtain nano structural materials as well as nanowires, nanorods, nanosheets, nanospheres, metallic, polymer particles, magnetic particles at dimensions in a range of about 10 nm etc. for applications in various fields such as medicine, electronics, super-capacitors, photocalysts, electro-chemical devices etc. This method is recommended by its simplicity and relatively low-cost technology [4, 10, 11].

The solvothermal method, which requires high reaction temperatures, leads to significant changes in the precursors before the formation of nano metal oxide framework (NMOF) particles. Key factors influencing the nucleation and growth of NMOF particles include temperature, time, and heating rates. To facilitate surface adsorption, pre-synthesized MOFs are suspended in a solution with functional molecules, as illustrated in Figure 13. Importantly, this approach imposes no limitations on the pore size or types of functional groups that the MOFs can incorporate [48].

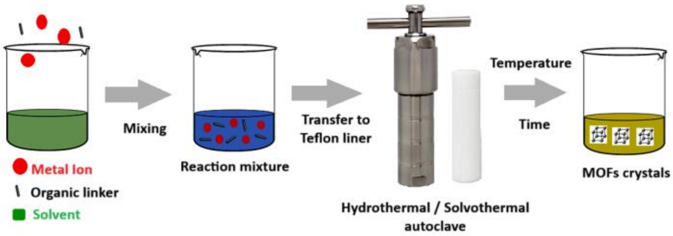

Figure 13. Schematic synthesis on MOF nanoparticles by hydrothermal / solvothermal methods [48].

2.2.2 Sol-gel process

This technique is used for the development of different applications of high-quality metal-oxide-based nanomaterials. It involves condensing, hydrolyzing, thermal decomposition of metal alkoxides or precursors in order to produce stable solutions. The liquid precursor is transformed in sol, and the sol is transformed in a structure like a network, as-called gel, from which is the name of technology – sol-gel [4, 10, 11, 49, 51, 52].

There is a so complicated phenomenon of transition from sol–gel and is not fully understood, in special speaking about models that can completely explain the process. At the early researching, the sol–gel process has been treated as a supposable alternative way to obtain glasses at lower temperatures. The process has proven to be expensive for mass production of glass, but it has an advantage regarding obtaining of films, hybrid organic and inorganic materials, nanofibers and nanoparticles [49, 50].

Although hard, but in simplified steps, the process can be described as follows: in first instance, the hydrolysis of oxides of metals happens in water or in alcohol to form a sol. In the second place, the condensation is happening and viscosity of solvent increases to form a porous structure that should be aged. In time of condensation or polycondensation, hydroxo- (M-OH-M) or oxo (M-O-M) bridges form, becoming into metal-hydroxo-polymer or metal-oxo-polymer composition in solution. In the time of maturation, polycondensation keep on and the structure continues changing as same as characteristics and porosity. The last one decreases and the space between colloidal particles rises. After aging, follows the drying, where water and organic solvents are deleted from gel. In the end, calcination happens to obtain nanoparticles [10, 49, 50, 51, 52, 53, 54].

2.2.3 Chemical vapor synthesis

A variety of sub technologies are coalesced in chemical vapor synthesis principle, such as chemical vapors precipitation (CVP), chemical vapors deposition (CVD), chemical vapors condensation (CVC), chemical vapors reaction (CVR), atomic layer deposition (ALD) [11, 57].

In CVD, the nanostructures are obtained in a result of deposition on a surface through chemical reaction of precursors in a vapor-phase at high temperatures. A precursor should have appropriate properties to satisfy the requirements of technology: chemical purity, high volatility, stability in time of vaporization, economic efficient, non-dangerous, long shelf-life and last – during decomposition, do not create residual impurities [10, 11, 57].

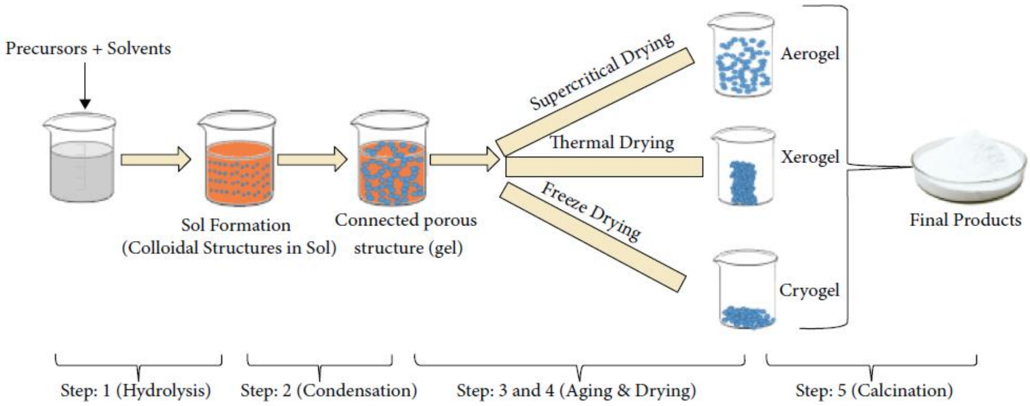

Figure 14. Simplified schematic of several steps in sol-gel process [52].

Growth of nanostructures through these methods permits the creation of 0D, 1D, 2D and 3D types of nanomaterials, where 0D – are nanoparticles and quantum dots; 1D – nanorods, nanowires, nanobelts; 2D – atomically thin layered materials; 3D nanostructures as polycrystal, dendrimers bulk materials etc. [55, 56, 57].

The molecules of precursors in reaction vessel deposits on substrate's surface. The technique permits to prepare almost all types of inorganic materials at a relatively low cost. The CVD process can pass in different layouts, but basically the system requires: chamber, substrate on which the growth is happening at suitable pressure and temperature [10, 11, 57].

An example of the CVD method is shown in Figure 15.

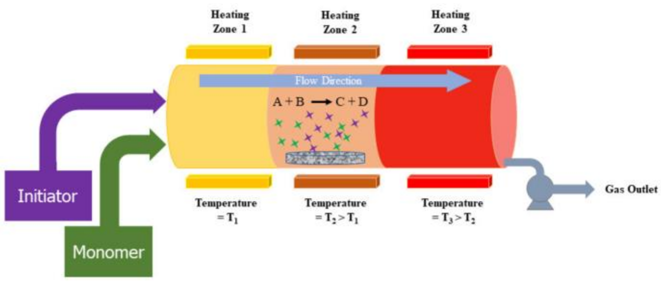

Figure 15. A model of three-zone chemical vapor deposition system [57].

2.2.4 Molecular beam epitaxy

Molecular beam epitaxy (MBE) is a technique that permits to create a high-accurate single crystal layers with atomic dimensional control. It makes it possible to obtain specific nanostructures for different applications such as optoelectronic devices etc. The real-time progress of growth control is the main advantage of technology for manufacturing [4, 58, 59].

MBE technology can be described as follows: they are a source of material and a plasma medium for generating active particles. The particles are grown on a substrate of material with preventive etching in a high-vacuum to ultra-high-vacuum chamber at temperatures above 700°C for a specified time to remove residual impurities and further exposition at plasma for a specified time [57, 60, 61, 62, 63].

2.2.5 Green synthesis: Fungi

The green synthesis uses natural extracts of plants, microorganisms or proteins, enzymes, cells etc. instead of industrial obtained chemicals. Modern science is actively researching available plants to find necessary applications. Research shows that green synthesized nanoparticles like gold, silver, copper, zinc, platinum, nickel, iron, cobalt etc. can be obtained with anticancer, antibacterial, antioxidant etc. properties [4, 11, 64].

Fungi are appealing agents for the biogenic production of iron nanoparticles because of their high metal tolerance and ease of handling. Additionally, they produce substantial amounts of extracellular proteins that enhance the stable state of the nanoparticles. Obtaining a significant quantity of synthesized nanoparticles with the help of fungi is relatively simple. This is due to their exceptional ability to secrete extracellular enzymes, the practical and efficient use of biomass, and their ease of cultivation, which leads to rapid growth rates. [65, 66, 67].

Metal oxide (MeO) nanoparticles are produced using fungi via intracellular or extracellular enzyme reduction and biomimetic mineralization. The enzymes within and outside the fungal cells act as reducing agents, facilitating nanoparticle formation [66, 67].

2.2.6 Green synthesis: Algae

An exact and quantitative approach to synthesizing nanoparticles can be achieved through either extracellular or intracellular methods, referring on the specific properties of the algae. Algal cultures or extracts contain various reducing agents that are believed to influence the creation of metallic nanoparticles extracellularly [65].

Figure 16. Schematic of nanoparticle production by algae green synthesis [66].

Algae are eukaryotic marine organisms capable of oxygenic photosynthesis, ranging from unicellular forms like Chlorella to multicellular types such as brown algae, which lack traditional plant structures like leaves and roots. These organisms are increasingly recognized for their sustainable applications in nanoparticle synthesis, contributing to economic development and metal reduction, thanks to their low toxicity and biological value. Their extracts contain a variety of bioactive compounds, including carotenoids, enzymes, minerals, chlorophylls, phycobilins, lipids, carbohydrates, polyphenols, and polyunsaturated fatty acids. Microalgae can bio-remediate toxic metals, transforming them into more manageable forms, while macroalgae possess active metabolites like cyclic peptides, alkaloids, proteins, polyketides, polysaccharides, lipids, and glycerol, which aid in nanoparticle production. Nanoparticles can be synthesized using two primary methods: intracellular and extracellular. The intracellular method utilizes nitrogen fixation and photosynthesis during metabolic processes, whereas the extracellular method occurs outside the cell, where metal ions attach to the algal surface and are reduced through metabolic exudates like pigments, enzymes, and non-protein molecules such as lipids and antioxidants [66, 68].

2.2.7 Green synthesis: Bacteria

Bacteria are unicellular organisms that grow rapidly in warm, moist environments. Many can produce nanoparticles, including silver, zinc oxide, gold, magnesium, arsenic, and iron oxide. To reduce metal ions, bacteria utilize various anionic functional groups, reducing sugars, proteins, enzymes, and other biomass components. Both gram-positive and gram-negative bacteria are used for nanoparticle synthesis. Gram-positive bacteria have cell walls rich in anionic functional groups, composed of peptidoglycan, lipoteichoic acids, teichoic acids, polysaccharides, and proteins, which facilitate cation biosorption and reduction. Additionally, some studies have explored metallic nanoparticle synthesis using yeast [11, 65, 66, 67].

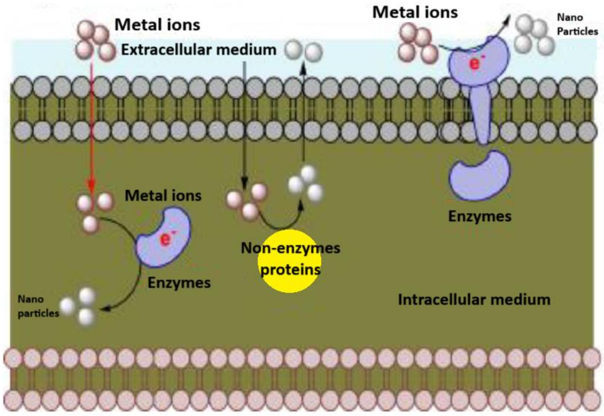

Figure 17. Schematic of intracellular and extracellular synthesis of metal oxyde nanoparticles in bacteria-cells [66].

2.2.8 Green synthesis: Actinomycetes

Prokaryotic bacteria and actinomycetes are frequently used in the production of nanoparticles of metal oxides. Actinomycetes, which share traits with both fungi and bacteria, have shown promise in this area. Studies have demonstrated that Au-nanoparticles can be synthesized intracellularly using alkali-tolerant Rhodococcus species, with a greater concentration of nanoparticles present in the cytoplasmic membrane than in the cell wall. Due to their underutilization, actinomycetes are becoming increasingly recognized as potential candidates for metal nanoparticle synthesis [11, 66, 69].

The same as previously described technologies, by using actinomycetes, the nanoparticle generation may take place either in extracellular medium, or in an intracellular medium [69].

The method's advantage consists in a commercial valuable because of its property of saprophytic behavior [11].

2.2.9 Green synthesis: Biomimetics

Biomimetic synthesis is a method that produces nanoparticles in live organisms or by using biological material. The biomimetics become a popular method for researchers because of their properties exceed traditional methods of physical and chemical obtaining of nanoparticles, especially they are low toxicity, low cost, simplicity, availability, and applicability. Besides all, the characteristics and applications of obtained nanoparticles are biocompatibility, possibility to reuse metal salts, semiconductors, electronic and information technology, medical drug delivery, agricultural and construction materials applications [70].
Some of the advantages of biomimetic synthesis are illustrated on Figure 18.

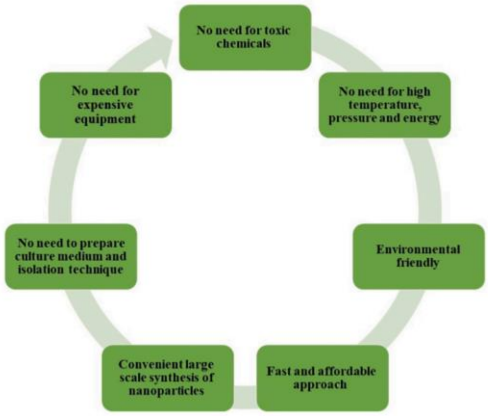

Figure 18. Advantages of biomimetic synthesis of nanoparticles [70].

The synthesis of plant extracts is an environmentally friendly and cost-effective method that eliminates the need for intermediate primary groups. The secondary metabolites found in plants can serve as both reducing and stabilizing agents, helping to control the growth of nanoparticles of metals and promoting the obtaining of small nanoparticles with enhanced characteristics [70].

A key factor in preparing biomimetic production is the size of particles what are interacting with a biological system. Studies have shown that for particles that mimic cells, a size closer to that of the target cell leads to a more pronounced effect on it. A critical element of biomimicry in obtaining nanoparticles is the choice of main material. In this context, yeasts, bacteria, mushrooms and plants serve as important natural resources for biomimetic production of nanoparticles [70, 71].

2.2.10 Green synthesis: Plants

Plants in comparison with other types of bio technologies are very accessible and a big variety of them is known. The cells used are safer than bacteria fungi for example. Plant extracts contain active compounds as well as terpenoids, alkaloids, phenols, tannins, and vitamins which are known for their use in medical and environmental scopes. The construction blocks of this compound represent simple hydrocarbons, monoterpenoids, isoprenoids, carotenoids, sesquiterpenoids, turpentine, and sterols. Metabolites from these small molecular blocks form complex metabolites, such as the phytol tails in the chlorophyll [70, 72, 73].

The obtaining of metallic nanoparticles using plant extracts basically unfolds in three fundamental steps: first phase, activation phase, and last phase. In the initial phase, nucleation and reduction of metallic ions occurs, leading to the formation of reduced metallic atoms. This process is crucial as it lays the groundwork for subsequent particle growth. The concentration of these atoms drives the development of various nanostructures, including nanohexadrons, nanoprisms, nanotubes, and other irregular shapes. During the activation phase, these nascent particles continue to grow and undergo structural transformations, influenced by the plant extracts. The present phytochemicals serve not only as reducing agents but also as stabilizers, preventing uncontrolled aggregation and ensuring the particles maintain a specific morphology. Finally, in the termination phase, the growth of nanoparticles is halted, resulting in stable entities that exhibit desirable characteristics. The plant extracts play a pivotal role in this phase as well, helping to lock the nanoparticles into their optimal conformations and ensuring their stability for various applications [74, 75].

3. CONNECTED APPLICATIONS

According to authors P. Topala, V. Besliu and L. Marin, a connected method to arch discharge, but a little different, called Electrical Discharge in Impulse (EDI) permits to obtain nanostructures, such as nanotubes and fullerenes. As is shown on works [76, 77], the model of formation consists in some phases: ignition of electric arch with installation of conductive channel, dissociation of almost atmospheric components (O, N, H ions) with previous phase electrons, interaction with surface elements and resulting activation of this, transfer of carbon resulting together with intensive oxidation and causing erosion and a substrate film obtaining with crystallization from plasma medium [78].

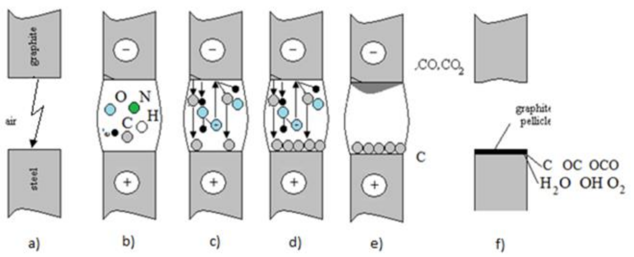

Figure 19. Physical schematic of obtaining the graphite pellicle on the piece surface under the influence of electric discharges in impulse [78].

On Figure 20 is shown the surface morphology obtained with aim of this method.

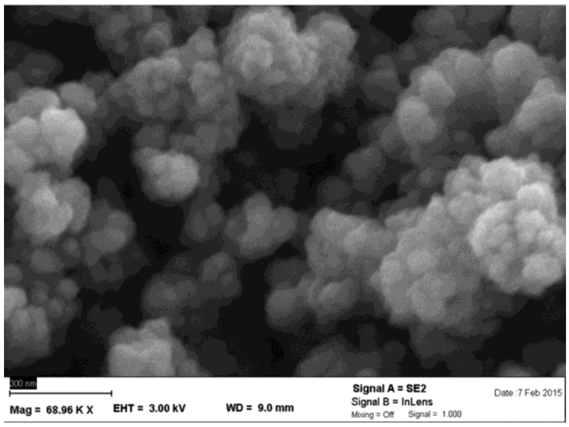

Figure 20. Morphology of surface of cathode pyrolytic graphite deposited on tool-electrode after the influence of plasma EDI (electrical discharge in impulse) [78].

The structures were tested with Thermal Gravimetric Analysis (TGA) of the carbon pellicle in a nitrogen atmosphere and show that some structures as carbon nanotubes and fullerenes the molecules of nitrogen were dopped into fullerenes [78, 79, 80, 81], a simulated model of which is shown on Figure 21.

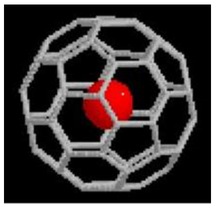

Figure 21. Schematic presenting of the characteristics of obtained space structures of nitrogen atoms [78].

A range of different applications of the structures described in the work [78] was proposed, as well as anti-socket / anti-blocking properties of graphite films, increasing the wear resistance, increasing corrosion resistance and lastly but not the last – the nanosensing properties of obtained structures [78].

4. CONCLUSIONS

Nanomaterials field of study presents a big interests and challenge for research of the domain, because they are a large variety of technologies, and every experimental setup gives opportunity to obtain new structures with new properties that can be useful for different issues in our lives. The nanostructures seem to be a solution for a revolutionizing technology in fields, such as agriculture, biomedicine, construction materials, chemistry, physics, computers, IT technology, semiconductors, sensing devices, nanorobots, solid lubricants and so on. A range of materials can be obtained from metallic, oxides, alloys, polymers, etc. all of them are important in further research, just as a problem-solving equation in which we can achieve a lot of knowledges about how nature works.

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