

# An Innovative Method for the Synthesis of Monodisperse ZnO Nanoparticles for Biomedical Applications

Zaur Berishvili, Marina Kipiani, Irakli Kordzakhia, Guram Dekanozishvili

**Abstract**— The growing demand for monodisperse nanostructured materials both for industry and for biomedical applications has led to the development of new technologies for the synthesis of nanoparticles. This concerns, for example, zinc oxide nanoparticles (ZnO NPs), the most widely used in biomedicine. However, the existing methods for the synthesis of ZnO NPs do not always meet the growing application requirements. The article discusses the technology and layout of innovative vacuum equipment for magnetron ion-plasma electrodispersion for the synthesis of ZnO NPs. The proposed method of magnetron ion-plasma electrodispersion of the bulk target material is based on the creation of a compact reactor for the formation of nanoparticles. The method consists in the formation of a flow of charged liquid macrodroplets of the target material over a sputtered target, and a cascade fission zone in the volume of a toroidal magnetron plasma, generally continuously moving above the target surface. The principle of operation of the reactor described in the article shows the possibility of creating ideal conditions for the formation of monodisperse ZnO NPs, uniform in size and chemical composition, as well as the conditions for preventing the enlargement of ZnO NPs as a result of Oswald ripening or agglomeration.

**Index Terms**— Magnetron, synthesis, nanoparticles, reactor, agglomeration, plasma, target.

## I. INTRODUCTION

Zinc oxide nanoparticles (ZnO NPs) are used in industry. In biomedicine, their widespread use is due to their excellent biocompatibility, economy and low toxicity, especially in anti-cancer and antibacterial fields. They are able to trigger the production of excess reactive oxygen species (ROS), release zinc ions, and induce cell apoptosis. It is important that zinc preserves the structural integrity of insulin well, which has allowed the development of effective antidiabetic drugs.

It is interesting for its wide biomedical use as an anticancer agent and for drug delivery [1]. It is generally known that zinc, as an important trace mineral, is widely present in all tissues of the body, including the brain, muscles, bones, skin, and so on. As the main component of various enzyme systems, zinc is involved in metabolism and plays a decisive role in proteins and nucleic acid synthesis, hematopoiesis,

and neurogenesis [2,3,4,5].

ZnO NPs are widely used as an antibacterial and in the treatment of diabetes, as an anti-inflammatory, in the treatment of wounds and in obtaining bioimages [6, 7, 8].

The biological activity of nanoparticles depends on many factors, including surface chemistry, size distribution, particle morphology, and their reactivity. Consequently, the development of nanoparticles with controlled parameters, uniform in size, morphology, and functionality, is required for various biomedical applications.

The most commonly used methods for the preparation of stable ZnO NPs are chemical precipitation, sol-gel, solid-phase pyrolytic, non-dissolution mechanochemical, and biosynthesis.

The most popular method for preparing ZnO NPs is chemical deposition. Controlled parameters in this method mainly include the concentration of the zinc precursor and the precipitant, the molar ratio of the two reactants, and the reaction and calcination temperatures. Bisht et al. synthesized ZnO NPs by chemical deposition. The intermediate products were calcined at 200 °C for 2 h in a muffle furnace to obtain a white fine ZnO powder with a size of  $18.67 \pm 2.2$  nm [9]. Bettini et al. the precipitate (ZnO) is dried in an oven at 100 °C for 6 hours. The obtained ZnO nanoparticles with a flaky structure had a size distribution of about 100 nm. The production of ZnO NPs by chemical deposition is not only simple and easy to control but also a simple industrial method. However, due to the surface effect of nanoparticles, the precursor of nano-oxides obtained by chemical deposition can easily agglomerate.

Sol-gel method Includes three main stages: 1) Preparation of zinc precursor; 2) Preparation of ZnO clusters; 3) Crystal growth. Crystal growth is a self-induced procedure that takes place at room temperature. But the growth rate, shape, and size of the crystal must be well controlled, which presents significant difficulties.

The solid-state pyrolytic method was pioneered by Wang et al. and is characterized by low cost and ease of use when growing high-quality ZnO NPs [10]. The particle size can be adjusted by choosing different pyrolytic temperatures. Using this method, Wang et al. obtained ZnO NPs of different sizes in the range of 8–35 nm.

With the solution lessmechanochemical method, the preparation of ZnO NPs is a two-stage synthesis method. The first step is grinding the material, the second step is the thermal decomposition of the nanoparticles at a very high temperature to obtain ZnO NPs, but the morphology of ZnO NPs is highly dependent on the grinding time of the reagent mixture, longer grinding time results in smaller particle size.

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The resulting ZnO NPs have an average size in the range of 24 to 40 nm.

Physicochemical methods for the preparation of ZnO NPs are widespread. Currently, the development of green chemistry is attracting more and more attention, since it is the most environmentally friendly [11]. A wide range of plant extracts is used for the biosynthesis of ZnO NPs. The biosynthetic and environmentally-friendly ZnO NPs synthesis technology is considered to be more environmentally friendly, economical (at low cost), non-toxic, and biocompatible than chemical and physical methods. ZnO NPs obtained by this method have shown great potential for biomedical applications such as its excellent anti-cancer and antibacterial activity. However, all this is associated with the collection of a large number of environmentally friendly plants, the production of extracts from them, and so on. Even so, it is difficult to obtain uniform nano-sized grains.

Physical methods mainly use thermal plasma, for example, powerful plasma jets, electric arc discharges, laser, and electron-beam plasmas, high-frequency induction discharge plasma, etc. These methods can be used to obtain high-quality monodisperse nanomaterials with a close-packed structure, but their widespread use is essential. limited due to the coagulation of particles during their synthesis [12,13].

A known method for producing nanoparticles, which includes dispersing a liquid material through a jet cathode made of a conductive material with a radius of curvature of the tip of not more than 10 microns. An electric field is applied to the cathode, the intensity of which at the top of the cathode tip is at least 107 V / cm. The resulting material droplets at the cathode tip fall into the plasma of an electric discharge, the pulse duration of which is at least 10 microns, and it is created in an inert gas atmosphere at a pressure of 10<sup>-3</sup> -10<sup>-1</sup> Pa. Nanoparticles are formed as a result of cascade fission of macrodroplets in a plasma with certain parameters under conditions of the development of Ray or capillary instability. The liquid nanoparticles formed as a result of such fission solidify after leaving the plasma and are deposited on the substrate. Another example of a method for producing nanoparticles involves dispersing a molten material and feeding the resulting macrodroplets of this material into an electron flow in order to recharge the macrodroplets to a state in which their cascade division begins and nanoparticles are obtained. The device for implementing the above method contains a unit for forming a stream of macrodroplets, a unit for recharging macrodroplets and a unit for deposition of nanoparticles [14, 15, 16].

However, these devices do not provide effective recharging of dispersed macrodroplets of molten material due to their short residence time in the recharging unit, which leads to a significant spread in the sizes of the resulting nanoparticles.

## II. NANOMATERIALS SYNTHESIS TECHNOLOGY

Methods for making metal's nanopowders largely determine their activity. The physical methods of producing nanopowders are mainly based on evaporation in a vacuum, rapid cooling and condensing of metals. In this case, high rates of cooling speed are one of the prerequisites for

receiving active nanopowders. The method of electrodispensing of liquid-phase droplets is widely used in modern nanostructure technologies; it is based on the Rayleigh (capillary) instability. Due to that, if the droplet charge of R radius reaches the critical value

$$Q_{cr} = 8\pi(\epsilon_0\alpha R^3)^{1/2}, (1)$$

the process of droplet distribution begins; here  $\alpha$  is a surface tension coefficient,  $\epsilon_0$  - dielectric constant [17]. The droplets formed during emission process are also unsustainable, and we obtain the cascade decaying of droplets to the sizes

$$d_{min} \approx 8 \cdot 10^{-7} \epsilon_0 \alpha^{-3}, (2)$$

At this size the particles lose charge as a result of the electronic emission [18]. For the majority of materials, these sizes comprise several nanometers and are distributed in a narrow range. Therefore, the methods of electrodispensing are very promising to obtain high-dispersed nanomaterials. In the plasma, metal droplets are charged to a critical value and begin to divide. In this case, macroparticles and, in particular, droplets acquire a negative charge due to collisions with electrons. The magnitude of this charge is determined by the magnitude of the floating potential and depends on the parameters of the plasma.

## III. VACUUM APPARATUS FOR MAGNETRON ION-PLASMA ELECTRODISPERSION

Recently, there has been a process of developing new designs of magnetrons, studying the sputtering processes taking place in them, and their relationship with the characteristics of the materials obtained. The main line of development of magnetron sputtering from the 70s of the last century to the present has been associated with the transition to planar magnetron sputtering systems. In 1973, J. F. Corbani [19] and in 1974 J. S. Chapin [20, 21, 22] proposed the design of stationary magnetron sputtering devices.

Due to the use of crossed electric and magnetic fields in these systems, the efficiency of gas ionization increased, and the density of the magnetron plasma became several orders of magnitude higher than in non-magnetic cathode sputtering devices. At the same time, the ion current density and the target sputtering rate increased significantly, the working gas pressure decreased, and many technical and technological parameters of the magnetron improved. An important feature of stationary magnetron sputtering is the absence of a droplet phase and macroparticles in the flow of the deposited substance, in contrast to thermal evaporation in a vacuum.

A generally recognized disadvantage of planar magnetron sputtering systems is the narrow and non-uniform zone of erosion of the sputtered target, which limits its economic performance. Despite this, stationary planar magnetron sputtering systems are widely used both in industry and in research work to obtain promising materials [23, 24, 25, 26, 27, 28, 29, 30].

There have been attempts to create magnetron sputtering devices with a rotating magnetic unit, but due to design flaws, they have not found wide application [31, 32].

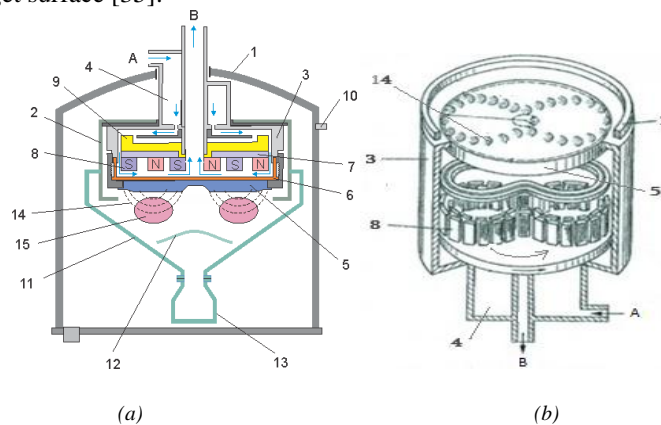
A real breakthrough in magnetron deposition methods is

the emergence of a new generation of magnetron sources - a planar magnetron sputtering device with a rotating magnetic unit, which significantly expands the functional and technological capabilities of the industrial production of promising materials [33, 34].

The proposed method of magnetron ion-plasma electrodispersion of a bulk target material is based on the creation of an innovative, compact reactor for the formation of nanoparticles, which consists in the formation of a stream of liquid charged macrodroplets of the target material over the sputtered target and a zone of cascade fission in the volume of a magnetron plasma of a toroidal shape, generally continuously moving over target surface [35].

This method can be successfully used to get ZnO NPs.

A model of vacuum equipment for magnetron ion-plasma electrodispersion for obtaining ZnO NPs (Fig. 1) contains a vacuum chamber 1 and an innovative planar magnetron sputtering device mounted in it, containing anode 2, cathode unit 3, the liquid cooling system of the cathode unit 4, disk target 5 from ZnO, disc target holder 6, disc 7 made of soft magnetic material, the matrix of permanent magnets 8, disc holder in the form of a hollow cylinder with blades 9, working gas supply system 10, funnel-shaped hopper 11, separation device 12 and receiving container produced by ZnO NPs 13.



**Fig.1** a) Vacuum apparatus for magnetron ion-plasma electrodispersion;  
b) Innovative planar magnetron sputtering device.

The vacuum apparatus for magnetron ion-plasma electrodispersion works as follows: after reaching the ultimate vacuum ( $10^{-4}$  Pa) in the vacuum chamber 1, inert gas argon (Ar) is fed into it through the working gas supply system 11. As a result, working pressure is created in the vacuum chamber about ( $10^{-1}$ - $10^{-2}$ ) Pa. The coolant supplied to the cooling system of the cathode assembly 4 through the branch pipe A passes through the cathode assembly 3 and flows out at the end from the outlet pipe B. The coolant acts on the blades and drives the disk holder 9, disk 7 with a matrix of permanent magnets 8 fixed to it. The turbulent flow of coolant moves along a spiral path in the channel of the liquid cooling system 4 of the cathode assembly. Due to its turbulence, the coolant acquires an increased ability to absorb heat (approximately 3-4 times), which ensures effective cooling of the cathode assembly 3 as a whole. Located above the stationary target 5, the matrix of permanent magnets 8 rotates in a plane parallel to the surface of the target 5, so that its axis of rotation passes through the center of the disk target 5 [30]. This causes the formation of a rotating magnetic field 14 in the form of a closed-loop and contributes to the localization of the generated magnetron plasma 15 in the immediate vicinity of the target 5. The separation device 12 serves for selection and is located above the receiving container of the produced ZnO NPs 13.

To ensure a uniform zone of erosion of the sputtered target and the stability of the stationary state of the plasma, its region above the target must be closed and have a constant width. The expression for the axial line determining the optimal configuration of the plasma region:

$$\varphi + C = \frac{\sqrt{r^2 - r_0^2}}{r_0} - \arccos \frac{r_0}{r} \quad (3)$$

Where  $\varphi$  and  $r$  are coordinates of the current points in

polar coordinates, the pole of which coincides with disc target center,  $r_0$  is the radius of the non-diffusion zone of the disc target 5,  $C$  is the constant rate determined from the closure conditions of the involutes. When  $r=R$ ,  $\varphi = \frac{\pi}{n}$ , where  $\pi = 3,14 \dots$ ,  $n = 1,2,3,\dots$ ,  $R$  is the outside radiustarget sputtering zones. The eponymous poles of the permanent magnets of each group are located along the corresponding closed contour composed of the involute line of a circle of radius  $r_0$ , with one of the matrices of magnets located outside the specified contour line, and the second group of magnets inside the contour line. The design of the matrix of the magnetic system is such that for any rotation mechanism a uniform distribution of the ion bombardment intensity is provided, and the corresponding sputtering zone keeps the profile of the bottom of the target erosion zone flat [36, 37]. The width of the erosion zone on the surface of target 5 can be regulated by changing the distance between the opposite poles of permanent magnets and is a new, controlled, independent technological parameter of the magnetron sputtering mode [38].

#### IV. REACTOR FOR THE FORMATION AND SYNTHESIS OF ZnO NPs

The formation of an active sputtering zone between the inputs and outputs of the magnetic field lines on the target surface occurs when an HF (high frequency) electric field and a rotating magnetic field are applied to the target surface. The gas discharge on the target surface has the shape of a closed-loop and coincides with the contour of the "treadmill for electrons", and the region of high-density magnetron plasma of a toroidal shape is located directly above the target erosion zone. Formation of cathode spots i.e. the dispersion of macrodroplets is carried out by ionization of the working

gas atoms in the region of the magnetron plasma, and the acceleration of the formed ions occurs in the immediate vicinity of the target surface in the dark cathode region in the glow discharge plasma.

One of the features of the magnetron sputtering system is the localization of an anomalous glow discharge plasma above and near the erosion zone ("electron treadmill") of the target. This plasma has an almost toroidal shape, and the degree of ionization reaches its maximum value in its central part, on the trajectory of a closed-loop above the sputtering zone. As a result of ion bombardment, electrons emitted from the surface of the disk target are captured between the inputs and outputs of the magnetic field lines, which ensures their movement along complex cycloidal trajectories over the active sputtering zone. As a result, the electrons find themselves in a trap, created on the one hand by the magnetic field lines that return electrons to the surface of the target's active zone, and on the other hand, by the target surface, which repels the electrons. The difference between our approach lies in the choice of more severe regimes of ion bombardment of the target in the erosion zone, in which the formation of cathode spots and dispersion of the target material becomes possible.

The electrons make cyclotron motion in this trap until several ionizing collisions with the working gas molecules or energy transfer to macro droplets of the dispersed target material occur, as a result of which the electrons lose the energy received from the electric field. Macro drops are charged to a floating potential proportional to the plasma temperature, and at an electron energy of 20-30 eV, the charge of macro droplets reaches a critical value at which they become unstable and begin to divide. Thus, most of the energy of an electron, before it reaches the anode, is used to excite macro droplets, which significantly increases the efficiency of the process of transferring the target material into nanoparticle materials.

As a result of the bombardment of the target by ions of the working gas in the erosion zone of the sputtered target, cathode spots are formed and the dispersed flow of macro droplets of the target material is supplied to the region of the magnetron plasma. Cascade fission of macro droplets occurs in the volume of a toroidal plasma under the condition of additional charging of macro droplets and the development of Rayleigh or capillary instability in the plasma. Ideal conditions are created for the preparation of monodisperse nanoparticles, in our case ZnO NPs of the required size with a uniform distribution over the size of NPs, chemical composition, etc.

The process of cooling and solidification of nanoparticles formed as a result of cascade fission occurs outside the plasma region in the vacuum space between the target surface and the receiving container produced by ZnO NPs. This eliminates the danger of nanoparticle enlargement as a result of Oswald ripening or agglomeration. An abrupt stop of fission occurs when the size of daughter nanoparticles reaches several nanometers. The final size of nanoparticles depends only on the surface tension and work function of the molten metal. This size for most metals is a few nanometers. The formed daughter nanoparticles cool extremely quickly (cooling rate ~ 106 K / s) and turn into solid nanoparticles.

## V. CONCLUSION

Currently, ZnO nanoparticles continue to be intensively investigated due to their unique properties. However, their wide practical use is still limited due to the lack of effective synthesis methods. This work reveals an innovative method and technology for the synthesis of ZnO NPs, which make it possible to obtain spherical nanoparticles of specified sizes. In the case of interest from firms and companies conducting research on the use of ZnO NPs in health care and biomedicine, it is realistic to make progress in the development and popularization of an innovative method of magnetron ion-plasma electrodispersion and technology for the synthesis of monodisperse ZnO NPs.

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