American Journal of Nano Research and Applications
2017; 5(3-1): 56-59
http://www.sciencepublishinggroup.com/j/nano
doi: 10.11648/j.nano.s.2017050301.22

The Induction Method of Production of Nanocrystalline Particles

Zaur Gamishidze

Department of Physics, Batumi Shota Rustaveli State University, Batumi, Georgia

Email address:
zaurgamishidze@yahoo.com

To cite this article:
Zaur Gamishidze. The Induction Method of Production of Nanocrystalline Particles. *American Journal of Nano Research and Applications*. Special Issue: Nanotechnologies. Vol. 5, No. 3-1, 2017, pp. 56-59. doi: 10.11648/j.nano.s.2017050301.22

Received: February 3, 2017; **Accepted**: February 4, 2017; **Published**: February 28, 2017

Abstract: Currently a lot of methods of production of nanoparticles, allowing the quite precisely control size, shape and structure of the nanoparticles have been developed. In particular, in the condensation method of production of nanocrystalline particles (powders and films), the initial macrobodies are first evaporated, and then the resultant vapor is condensed until the nanoparticles of the desired size are formed. All methods of production of nanoparticles require a powerful flow of energy from the external source. For this very reason we could use the method of induction heating for production of nanoparticles, a version of this method is considered in this paper. The induction method has a number of advantages such as rapid heating, high concentration and exact localization of energy with heating, high and uniform quality, etc., which allows exact automatic controlling the process and avoiding the complex maintenance.

Keywords: Induction Method, Nanoparticles, Heating Effect, Nanocrystalline Films, Condensed State

1. Introduction

Isolated nanocrystalline particles (nanoparticles) are usually produced by metal (alloy or semiconductor) evaporation at controlled temperature in the low pressure inert atmosphere or vacuum with subsequent vapor condensation near the cold surface or behind it. The atoms of the substance evaporated in the rarefied inert atmosphere lose their kinetic energy owing to the collisions with gas atoms and form segregations (clusters) more readily as distinct from the vacuum evaporation. Depending on the conditions of metal evaporation (gas pressure, temperature of the substrate and its state etc.), the condensation of the metal could proceed both in the volume and on the surface of the reaction chamber. For volume condensates, spherical particles are characteristic, whereas the particles of surface condensates have cuts. This is the simplest method of production of crystal nanopowders.

The basis for the method of synthesis of nanoparticles from supersaturated metal vapors is the classical nucleation theory. This theory is based on the assumption that incipient clusters of a new phase (nanoparticles) are described by the model of a spherical liquid drop.

There are a number of versions of devices for obtaining the nanoparticles (powders or clusters), which differ in the evaporation procedure: laser-induced, thermal, arc-discharge, plasma or solar-energy evaporation [1 – 4]. These devices also differ in the design of one or another unit [5 – 7]. All these methods allow us to study both the physical-chemical characteristics of nanoparticles in the gas phase (before the vapor deposition on the substrate) and the properties of powders and films obtained in the result of deposition. The devices based on the principle of evaporation–condensation differ in the means of supply of the evaporation material and power for evaporation, in working media, organization of condensation and the system of collection of the produced powder (or films). The metal evaporation may occur from a crucible, or the metal / alloy gets into the heat and evaporation zone in the form of wire, injected metallic powder or in the liquid spray. The energy can be supplied by direct heating, through the wire, using an arc discharge in plasma, by induction heating with the current of high and medium frequency, using the laser radiation or e-beam heating. The evaporation and condensation may proceed in vacuum, in an immobile inert gas or in a gas stream including a plasma spray.

2. Models

This paper deals with the possibility of application of one of the versions of induction heating to obtaining of nanoparticles (powders or films). The induction heating has the following advantages: rapidness of heating, high concentration and precise localization of energy under heating, high and uniform quality, the possibility of accurate automated control of the process, elimination of complex maintenance procedures, no smoke or other harmful emissions with heating, etc. The main drawback of the induction heating is that it is impossible to heat up dielectrics by this method. According to the principle of induction heating, the induction device can be presented as a freely connected transformer in which the turns of primary winding are connected magnetically to the turn of secondary winding formed by melting. Thus, we can assume that the ratio between the current induced in the melt and the current in the winding is approximately equal to the number of turns in the winding. The maximum current density is induced around the perimeter of the melt, decreasing exponentially in the melt depth towards the center. The depth of current penetration is

$$h = \sqrt{2\rho/\mu_o\mu f} = 503\sqrt{\rho/f}$$

where, ρ is the resistivity of melted metal, μ_o and μ are the absolute and relative permittivity, respectively; f is the current frequency in the winding.

The most amount of current flows in the distant layers of the melt, within the thickness equal to the penetration depth. The power generated in these layers can be estimated by the formula

$$P \approx I^2 R$$

where, I is the current in the melt, $R = 2\pi\rho/rh$ is the resistance of the melt.

The evaporation rate υ is determined by vapor pressure p, evaporation temperature T and molecular mass M,

$$\upsilon = 5.834 \cdot 10^{-2} p (M/T)^{1/2} \approx$$

$$\approx 6 \cdot 10^{-4} (M/T)^{1/2}$$

where, υ is the evaporation rate (g / cm^2 $\cdot$ s) and p is the saturated vapor pressure (10^{-2} Hg mm).

The temperature dependence of vapor pressure in a general form is described by the formula

$$\log p = AT^{-1} + B$$

where, A and B are the constants characteristic of the given substance. Coefficient A is related to the average evaporation heat value (J) over the given temperature range

$$A = \Delta Q/19.14.$$

In accordance with the sign of the process heat effect and the temperature dependence equation, the saturated vapor pressure increases with the increasing temperature. At certain temperature, the saturated vapor pressure equal to the ambient atmosphere pressure is reached; at this temperature the liquid boils. The boiling point and boiling temperature usually correspond to the 0.1 MPa pressure. The metal boiling temperature decreases when the external pressure decreases. In vacuum the boiling process transfers into evaporation from the open surface. The evaporation temperature of the substance is the temperature at which the pressure of its vapor reaches 10^{-2} Hg mm.

If the pressure of the boiling substance (metal) vapor does not exceed 1.33 Pa at temperature Tthen, under the working pressure in the vacuum chamber of the order of 10^{-2} Pa or lower, the molecules and atoms of the boiling substance reach the surface of the substrate without collisions with each other and with the molecules of residual gases. In this case there is realized a molecular mode of evaporation and condensation for which spatial distribution of the substance evaporated from a plane surface is proportional to cosφ (φ is the angle between the direction of vapor propagation and the normal to the surface). The amount of the substance that deposits on the opposite surface also depends on the location of this surface in respect to the evaporator. The number of particles reaching the substrate surface is inversely proportional to the squared distance between the evaporator and the substrate. These laws are basic when analyzing the regularities of film formation on the surfaces of various shapes.

Upon metal evaporation and interaction of the melt surface with residual gases, collisions play a primary part. This process is characterized by the mean free path λ. When it is equal to the melt surface radius, there exists an area bounded by a certain surface where the metal atoms undergo a large number of collisions. This area firstly plays a role of the source of vapor, instead of the melt surface, and secondly it is a shielding screen of the melt surface preventing its direct interaction with the residual atmosphere in the vacuum chamber.

The mean free path of metal atoms at the melting point depends on many factors and can be calculated by the formula [8]

$$\lambda = \frac{kT}{4\sqrt{2\pi}r^2 p} = 5.8 \cdot 10^{-3} \frac{T}{r^2 p}$$

where T is the temperature, p is the metal vapor pressure, r is the molecular radius, $r = 1.33 \cdot 10^{-8} (M/\rho)^{1/3}$, where M is the atomic weight and ρ is the metal density.

3. Results

Varying the evaporation rate, the substrate temperature, and the pressure and composition of the gas, it is possible to obtain crystal nanoparticles over the range of $(3 - 100)$ nm.

One of the possible designs of the device for production of nanoparticles by the abovementioned method is shown in the Figure 1. The device consists of a working chamber, a vacuum system and a control panel. The working chamber (1) is made in the form of a cylindrical cap from quartz (or stainless steel with viewing windows). Inside the working chamber, there is a crucible (2), a stencil (mask) (3), providing the desired shape of the nanocrystalline layer, and a substrate (4), which the evaporated substance is condenses on. The vacuum system represents diffusion and rotary vacuum pumps connected in parallel (12). Diffusion vacuum pumps perform pumping of the working volume through a hole. The initial removal of the bulk of air from the working volume of the device is performed by the rotary pump via the pipeline connected with the pumped volume via a valve (13).

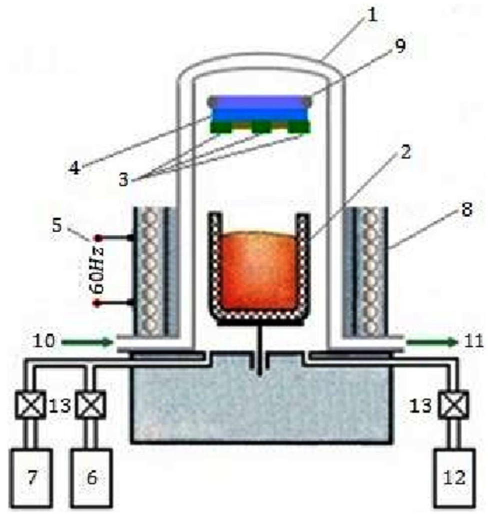

Figure 1. *1 – quartz housing of the reaction chamber; 2 – crucible with the sample; 3 – plate with holes (stencil); 4 – substrate; 5 – power network; 6 – cylinder with an inert gas; 7 – cylinder with a mixture of the inert gas and oxygen; 8 – inductor for heating the sample; 9 – substrate heater; 10 – water cooling (inlet); 11 – water cooling (outlet); 12 –vacuum pumps; and 13 – valves.*

The control panel consists of a power source heating the substance to be evaporated. A simple design of the induction melting system involves direct feeding of current to the induction coil from the power grounded network ($220\,\text{V}$) providing load power of $2kW$ (Figure 2). The capacitor cell serves to compensate a low power factor (cos φ) of the inductive coil.

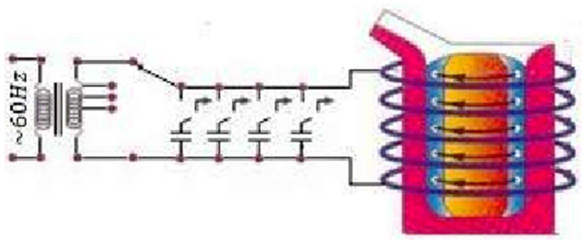

Figure 2. *Schematic diagram of the control panel of the induction melting system.*

The control panel includes the control units of ionization and thermocouple gauges, for determination of the melting rate and the thickness of obtained films, the substrate temperature, etc. The microprocessor temperature controller that automatically forms the power value and controls the converter power can be used to control the temperature. The microprocessor temperature controller is a single-channel controller with a universal input for connecting the sensors, an additional input for remote control, a data processing unit forming the signals controlling the output devices, and a bidirectional interface.

The inert gas flow rate can be controlled by the readings of the reducer unit mounted on the cylinder with the inert gas (6). The flow rate of the inert gas should not be high, as the inert gas will cool the crucible. Before opening the device and taking out the sample, they passivate the sample by passing the mixture of the inert gas and oxygen (7) [9].

4. Discussion

For evaporation the substance is heated. The metal to be deposited on the substrate (4) is placed in the crucible (2) made of a refractory metal, for example, tungsten. When the crucible with the weighed metal sample is placed in the inductor (8) with the magnetic field varying with the current varying on its surface, there will generate vortex currents causing the heating of the surface owing to resistance [6]. The heating effect increases as increases the field intensity, and it depends on the properties of the material and on the distance between the coil and the surface.

Being heated, the substance melts and then transforms into a vapor state. Propagating from the melt, the vapor of the melted metal in the form of an atom beam falls on the substrate and deposits on its surface. When the vapor deposits on the substrate, the metal atoms transform from the gaseous phase into the condensed one forming a layer in the form of a thin film. The quality and strength of films depend to a great extent on the substrate surface finish.

Hence, the substrate surface is to be polished and thoroughly cleaned beforehand. If the substrate is placed on the plate with, e.g. round, holes (3), during condensation there are formed films shaped like round spots, i.e. they will correspond to the holes in the mask. Thus, we can impart desired shapes and sizes to the films with the help of the mask. A number of factors influence the film formation, the substrate temperature being the main one. The substrate can be heated by a special heater (9) up to $100 - 3000$ °C. With the heated substrate, internal strain in the film is partially removed, and the film adhesion to the substrate improves. Depending on the substrate temperature, there are realized different mechanisms of condensation determining largely the structural state and properties of nanoparticles.

5. Conclusions

The proposed method allows obtaining the films of different thickness. As mentioned above, the film thickness is controlled by varying the rate or the duration of condensation. The structure and properties of the thin films obtained by induction heating condensation to a great extent are determined by the condensation conditions and depend on the nature of the evaporated substance and matching of its structure with the substrate structure, the evaporation rate, the angle of incidence of the atom (molecular) beam onto the substrate, and the film thickness.

Depending on the certain conditions of induction heating and evaporation, the films from the same substance can have the following structures: single-crystalline structure, if the film represents a solid crystal lattice of the atoms of this material, colloid (fine-grain) structure consisting of the crystals 100 Å and more in size, and amorphous structure characterized by the absence of the crystalline lattice.

References

[1] D. M. Cox, D. J. Tevor, R. L. Whetten, E. A. Rohlfing, and A. Kaldor, "Magnetic behavior of free-iron and iron oxide clusters," Phys. Rev. B, vol. 32, p. 7290, 1985.

[2] W. A. de Heer, P. Milani, and A. Chatelain, "Spin relaxation in small free iron clusters," Phys. Rev. Lett., vol. 65, p. 488, 1990.

[3] F. Fendrych, L. Kraus, O. Chayka, P. Lobotka, I. Vavra, J. Tous, V. Studnicka, and Z. Frait, "Preparation of nanostructured magnetic films by the plasma jet technique," Chem. Monthly, vol. 133, p. 773, 2002.

[4] B. Martínez, A. Roig, X. Obradors, and E. Molins. "Magnetic properties of γ-Fe_2O_3 nanoparticles obtained by vaporization condensation in a solar furnace," J. Appl. Phys., vol. 79, p. 2580, 1996.

[5] Eds. L. Maissell and R. Glang, "Thin Film Technology. Handbook, vols. 1 & 2," Moscow, Soviet Radio, 1977.

[6] V. N. Chernyaev, "Physicochemical Processes and Radio Electronic Technologies," Moscow: Higher School, 1987.

[7] Z. M. Gamishidze, "Determination of parameters of the induction melting method for synthesis of nanocrystalline particles," In: "Abs. 3rd Int. Conf. Nanotechnol.," Tbilisi: Georg. Tech. Univ., p. 40, 2014.

[8] E. Fromm and E. Gebhardt, "Gases and Carbon in Metals," Moscow, Metallurgy, 1980.

[9] A. S. Zolkin, "Metal vapor source for thin film research and technology: Review," J. Vac. Sci. Technol. A, vol. 15, p. 1026, 2016.