علم النانو في الفيزياء الطبية

Nanoscience in Medical Physics

CHAPTER FOUR

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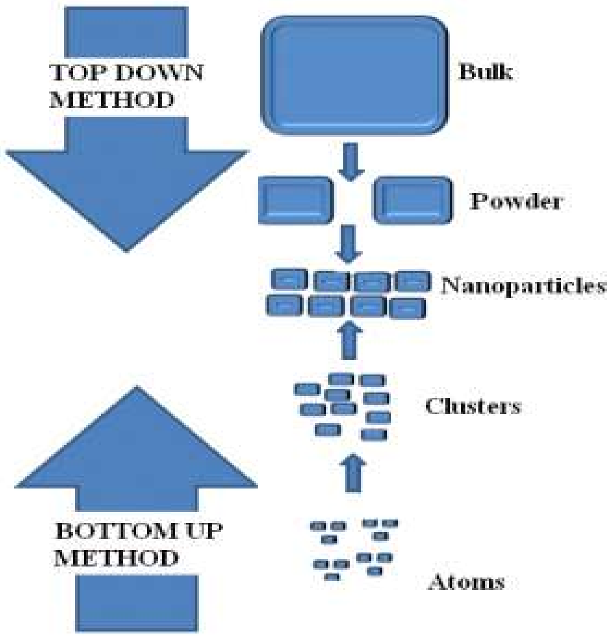
Nano-Material Fabrication Methods

Nano-materials deal with very fine structures: a nanometer is a billionth of a meter (1nm = 10^{-9} m). This indeed allows us to think in both the 'bottom up' or the 'top down' approaches (Fig. 3.1) to synthesize nanomaterials, i.e. either to assemble atoms together or to dis-assemble (break, or dissociate) bulk solids into finer pieces until they are constituted of only a few atoms. This domain is a pure example of interdisciplinary work encompassing physics, chemistry, and engineering up to medicine.



Schematic illustration of the preparative methods of nanoparticles.

Several physical and chemical methods under specific growth conditions at low and high temperatures are used for growth of nanostructures.

There are two <u>main ways</u> to fabrication nano-materials;

1- **Top-down Method: Top-down method is reduced in size of material from large size to smallest size or nano-scale. These require to create smaller material by using larger ones to their assembly. These techniques include;**

   i) Mechanical Grinding

   ii) Pulls Laser Deposition

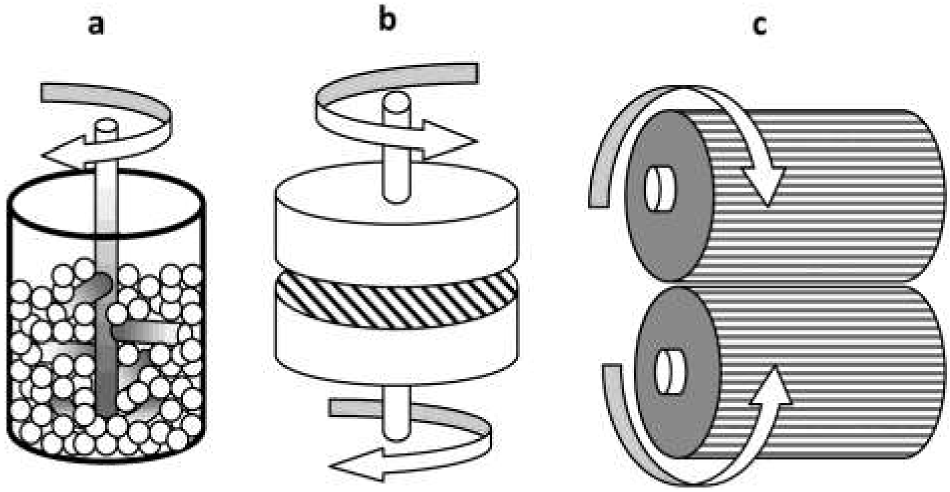
2- **Bottom-up Method: The bottom-up method is build larger structures of nano-materials by atom or molecule. These techniques include;**

   i) Thermal Evaporation Using Tube Furnace Tube Furnace

<u>Types of fabrication methods of nano-materials</u>

Mechanical Grinding (Milling)

Mechanical grinding is a typical example of 'top down' method of fabrication of nano-materials, where the material is prepared not by cluster assembly but by the structural decomposition of coarser-grained structures. Mechanical milling is typically achieved using high energy shaker, planetary ball, or tumbler mills (mills vibrators, non-vibrators, and electric mixers), as shown in Fig. 3.2.



Schematic representation of the principle of mechanical grinding.

- **What is mechanical grinding (milling)?**

Mechanical grinding is a typical example of 'top down' method of fabrication of nano-materials by decomposition of structures or materials, where achieved using high energy mills vibrators, non-vibrators, and electric mixers.

- **What are the properties of mechanical grinding (milling)?**

(i) Simple method.

(ii) Cheap equipment.

(iii) Possibility of making of all types of materials.

(iv) Possibility for increasing the quantities of material.

- **What are the properties of mechanical grinding (milling)?**

(i) Impurity from milling media and/or atmosphere.

(ii) Product is not uniform in size of nano-crystalline or micro-structure.

- **What are the factors affecting mechanical grinding (milling) speed?**

(i) Rotational or vibration speed

(ii) Size and number of the balls

(iii) Ratio of the ball to powder mass

(iv) Time of milling

(v) Milling atmosphere.

(vi) Milling in cold liquids can reduce the dimensions of powder particles.

(vii) Stop oxidation is necessary to get a good size and uniform of particles.

Nano-particles are produced by the cut process during grinding. Milling in cold liquids can greatly reduce the dimensions of powder particles. As with any process that creates fine particles, an suitable step to stop oxidation is necessary. Hence, this process is very restrictive for the production of non-oxide materials since then it requires that the milling take place in an inert atmosphere and that the powder particles be handled in an appropriate vacuum system or glove box. **This method of synthesis is suitable for producing amorphous or nano-crystalline alloy particles, elemental or compound powders.** If the mechanical milling imparts sufficient energy to the constituent powders a homogeneous alloy can be formed. Based on the energy of the milling process and thermodynamic properties of the constituents the alloy can be rendered amorphous by this processing.

Thermal Evaporation Using Tube Furnace

Thermal evaporation method using tube furnace is considered to be one of the important methods employed to synthesize nanostructures. In addition, this represent the simplest method to produce nanoparticles is by heating the desired material in a horizontal or vertical tube furnace as shown in Figure 3.3 (a and b), which containing the desired material. This method is appropriate only for materials that have a high vapour pressure at the heated temperatures that can be as high as 2000°C.

Thermal evaporation using tube furnace uses to growth of nano-materials through heating of powder in the tube furnace. During the heating process, the atoms of powder will evaporated into an atmosphere inside the tube furnace. The vapor of the powder will condense into the substrate with or without the inert carrier gas (e.g. He, Ar) to produce nano-particles.

To carry out reactive synthesis, materials with very low vapour pressure have to be fed into the furnace in the form of a suitable precursor such as organometallics, which decompose in the furnace to produce a condensable material. The hot atoms of the evaporated matter lose energy by collision with the atoms of the cold gas and undergo condensation into small clusters via homogeneous nucleation. In case a compound is being synthesized, these precursors react in the gas phase and form a compound with the material that is separately injected in the reaction chamber. The clusters would continue to grow if they remain in the supersaturated region. To control their size, they need to be rapidly removed from the supersaturated environment by a carrier gas. **The size of nano−materials or micro−materials and its distribution are controlled by only three parameters:**

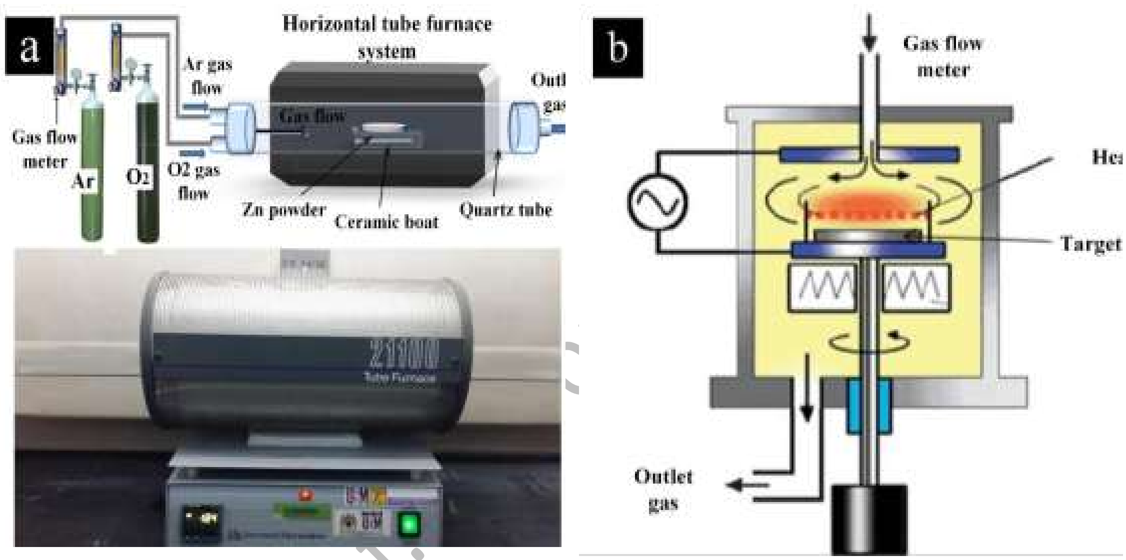
- **What are the factors affecting thermal evaporation using tube furnace?**

i) Rate of evaporation

ii) Rate of condensation

iii) Rate of gas flow

iv) Type of substrate



Schematic of the set-up used for the growth of nanostructures by thermal evaporation method using tube furnace, (a) horizontal tube furnace (b) vertical tube furnace

Pulsed-Laser Deposition

In principle technique of pulsed-laser deposition (PLD) is an simple technique, which uses pulses of laser energy to remove material from the surface of a target, as shown in Figure 3.4. The PLD has been used to deposit high quality films of materials for more than a time. The technique uses high power laser pulses (typically ~108 Wcm^{-2}) to melt, evaporate and ionize material from the surface of a target. This happening produces a transient, highly luminous plasma plume that expands rapidly away from the target surface. The ablated material is collected on an appropriately placed substrate upon which it condenses and the thin film grows. Applications of the technique range from the production of superconducting and insulating circuit components to improved wear and biocompatibility for medical applications. In spite of this widespread usage, the fundamental processes occurring during the transfer of material from target to substrate are not fully understood and are consequently the focus of much research.

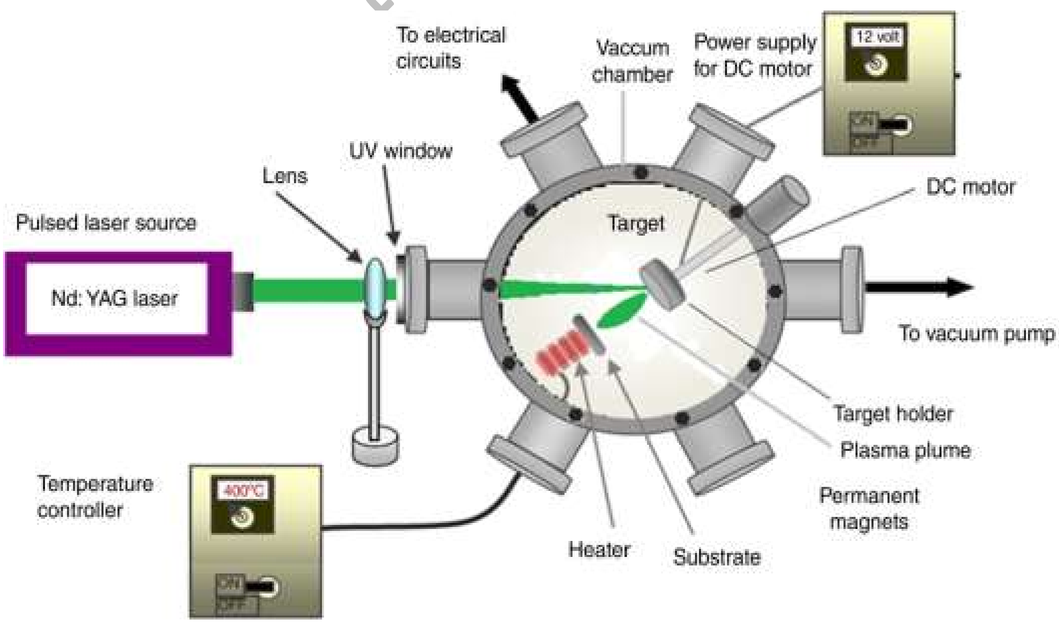


Figure 3.4: Schematic of the PLD system.

The vaporized material, containing neutrals, ions, electrons etc., is known as a laser-produced plasma plume and expands rapidly away from the target surface (velocities typically ~106 cms-1 in vacuum).Film growth occurs on a substrate upon which some of the plume material recondenses. In practice, however, the situation is not so simple, with a large number of variables affecting the properties of the film, such as laser fluence, background gas pressure and substrate temperature. These variables allow the film properties to be manipulated somewhat, to suit individual applications. However, optimization can require a considerable amount of time and effort. Indeed, much of the early research into PLD concentrated on the empirical optimization of deposition conditions for individual materials and applications, without attempting to understand the processes occurring as the material is transported from target to substrate.

The technique of PLD was found to have significant benefits over other film deposition methods, including (<u>properties of PLD technique</u>):

i) The capability for transfer of material from target to substrate

ii) The accurate chemical composition of a complex material can be reproduced in the deposited film.

iii) High deposition rates

iv) Controlled film thickness in real time by simply turning the laser on and off.

v) The fact that a laser is used as an external energy source results in an extremely **clean process**

vi) Getting multi-layer films to be deposited without the need to break vacuum when changing between materials**.**