

# Review on Synthesis Techniques of Multilayer Nanostructures using Bottom-up Approach

Karamjit Kaur, Anil Kumar\*

Amity University Haryana, Gurugram, India

Corresponding Author: Anil Kumar

**Abstract-** The process of synthesis of nanostructures and nanomaterials plays a crucial role in physical properties and associated phenomena acquired by them and hence is a deciding factor in various potential applications of the materials. The parameters like particle size, porosity, yield stress, ductility strain hardening, etc are strongly dependent on the method used for preparation of nanostructures. Thus, the know-how of skill of fabrication and processing nanomaterials and nanostructures is the first corner stone in field. This paper summarizes different synthesis techniques for nanostructures and nanomaterials. The parameters crucial for selection of technique are also presented.

**Keywords**—multilayer nanostructures, bottom up approach, top down approach, thin film deposition.

## I. INTRODUCTION

This paper focus on the different existing techniques for synthesis of nanomaterials, the selection of potential process for preparation of multilayered nanostructures such that material with optimum characteristics may be obtained. The synthesis techniques may be broadly classified as top down methods and bottom up methods. [1] [2] [3] [4]. The basic principle behind both approaches is summarized in Figure 1.

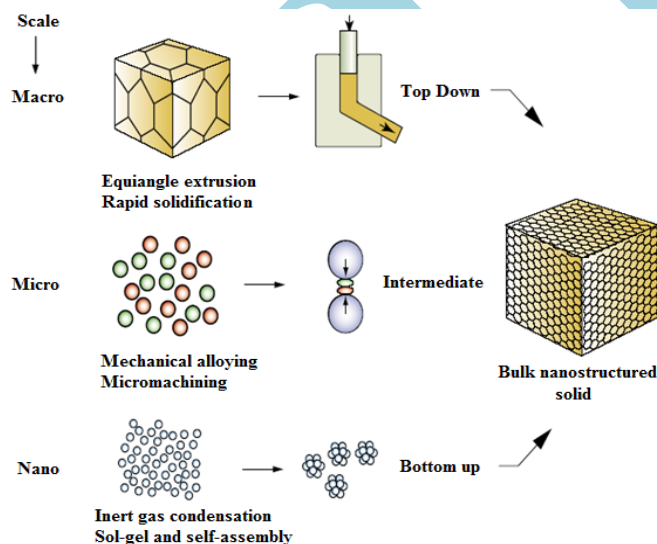


Figure 1: Top down and Bottom Up approach for synthesis of nanomaterials

## II. TOP-DOWN APPROACH

In the top-down approach, the nanostructure is obtained by breaking down the microstructure of a bulk material. It often uses the traditional workshop or micro-fabrication methods

where externally controlled tools are used to cut, mill and shape materials into the desired shape and order. The approach implements physical techniques like milling, abrasion, quenching, lithography etc. Although materials of particle size ranging from tens to hundred nanometres can be easily formed, but have relatively broad size distribution and varied particle shape. In addition, they may contain impurities from milling medium, defects in the surface structure of material so synthesized since the techniques such as lithography and etching can cause major damage to the crystallographic patterns of surface. For example, nanowires made by lithography are not smooth and may contain a lot of impurities and structural defects on surface. Such imperfections may significantly influence the physical properties and surface chemistry of nanostructures and nanomaterials, since the surface over volume ratio in nanostructures and nanomaterials is very large. The technique is generally used for production of bulk materials, which requires much lower sintering temperatures. Although very fine particles can be produced, this process is difficult to design and control so as to produce the desired particle size and shape. It is also limited to materials with very poor thermal conductivity but a large volume change.

The major top-down techniques include:

- Solid phase transformation and re-crystallization
- Plastic deformation processes like Equal channel angular pressing (ECAP), Accumulative roll bonding (ARB), High pressure torsion (HPT)
- Mechanical milling (MM) and mechanical alloying (MA)
- Micro-patterning techniques like lithography, inkjet printing

## III. BOTTOM-UP APPROACH

Bottom-up approach refers to the approach to build a material up from the bottom: atom-by-atom, molecule-by-molecule, or cluster-by-cluster through chemical process. Nanostructures

and nanoparticles are synthesized by homogeneous nucleation from liquid or vapour, or by heterogeneous nucleation on substrates. Nanoparticles may be prepared by phase segregation through annealing appropriately designed solid materials at elevated temperatures, by confining chemical reactions, nucleation and growth processes in a small space. It implements the know-how of chemical properties of molecule that self-organize into some useful conformation, or based on positional assembly.

The principal bottom-up techniques includes:

1. Vapour to Solid synthesis through Inert Gas Condensation, Sputtering, Laser Ablation, Plasma Assisted Physical and Chemical Vapour Deposition (PVD & CVD)
2. Solid to solid synthesis through Fast Solidification, Electrodeposition, Aerosol Conversion, Sol Gel Methods, Spark Erosion, High Pressure Solidification.

Top-down and bottom-up approaches play very important role in modern industry and most likely in nano technology as well. There are advantages and disadvantages in both approaches. The top down approach is used for bulk production but the main issue involved is the imperfection of surface structure, significant crystallographic damage to the processed patterns and introduction of internal stress. These imperfections in turn lead to extra challenges in the device design and fabrication. Also, when structures fall into a nanometre scale, there is a little chance for top down approach and all the tools we have possessed are too big to deal with such tiny subjects. Bottom up approach promises a better chance to obtain nano structures with less defects, more homogeneous chemical composition and better short and long range ordering. This is because the bottom-up approach is driven mainly by the reduction of Gibbs free energy, so that nanostructures and nanomaterials such produced are in a state closer to a thermodynamic equilibrium state. They are assumed to be capable of producing devices in parallel and much cheaper than top-down methods, but could potentially be overwhelmed as the size and complexity of the desired assembly increases. The different synthesis techniques implementing bottom-up approach includes Gas Phase Methods, Physical and Chemical Vapor Deposition Method, Microwave Synthesis, Physical Method (E- Beam), Sol Gel Method, Hydrothermal Method etc. [5]

### Gas Phase methods

The gas phase methods used for synthesis of nanostructures usually involves homogeneous nucleation in the gas phase and subsequent condensation and coagulation. In order to generate nanostructure layer, an environment of super-saturation is to be created where the vapour phase mixture is thermodynamically unstable relative to formation of the solid material to be prepared in nanoparticulate form. The gas molecules react chemically to form a condensed phase. If the degree of super-saturation is sufficient, and the reaction and hence the subsequent condensation kinetics permit, particles will nucleate homogeneously. Once nucleation occurs, remaining super-saturation can be relieved by condensation or reaction of the vapour-phase molecules on the resulting

particles, and particle growth will occur rather than further nucleation. The required situation of super-saturation may be achieved by different physical or chemical methods. The physical methods involve some form of cooling of the monomers, by expansion, by mixing with a cooling gas or by heat transfer to the surrounding whereas chemical methods involves set of chemical reactions which produce a non-volatile condensable product. These reactions are usually decomposition reactions initiated by a rise in temperature and used extensively in laser and flame reactors. [6] [7] [8] [9]. The chemical vapour deposition technique is usually used where large surface area coatings is to be done in short span of time, while physical deposition is done for experimental purposes. The different type of physical and chemical vapour deposition techniques are summarized in Figure 2.2. The different physical processes available may be broadly classified under two heads namely evaporation and sputtering. In evaporation, the growth species are removed from the source by thermal means. In sputtering, atoms or molecules are freed from solid target through impact of plasma of gaseous ions. [10].

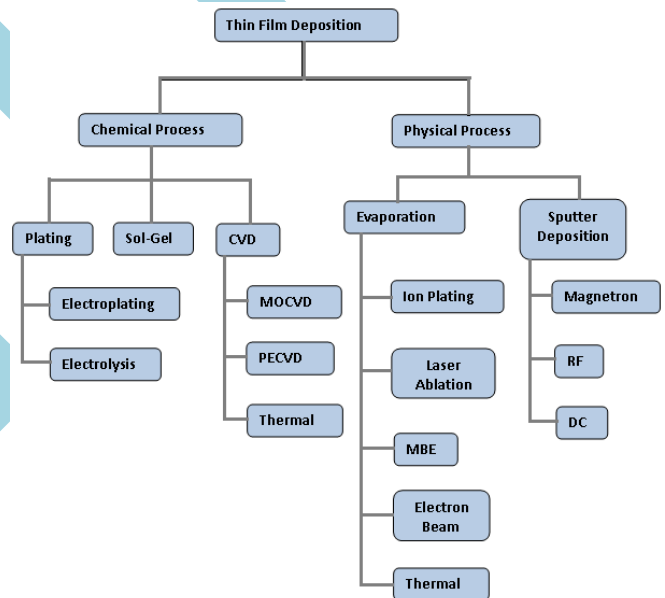


Figure 2: Various Physical and Chemical Vapour Deposition techniques

### Microwave Synthesis

This technique is similar to the chemical vapour deposition employing plasma instead of high temperature for decomposition of the metal organic precursors. The method uses microwave plasma in a 50 mm diameter reaction vessel made of quartz placed in a cavity connected to a microwave generator. A precursor such as a chloride compound is introduced into the front end of the reactor. The major advantage of the plasma assisted pyrolysis in contrast to the thermal activation is the low temperature reaction which reduces the tendency for agglomeration of the primary particles. Additionally, it has been shown that by introducing another precursor into a second reaction zone of the tubular reactor, e.g. by splitting the microwave guide tubes, the

primary particles can be coated with a second phase. For example, it has been demonstrated that ZrO<sub>2</sub> nanoparticles can be coated by Al<sub>2</sub>O<sub>3</sub>. In this case the inner ZrO<sub>2</sub> core is crystalline, while the Al<sub>2</sub>O<sub>3</sub> coating is amorphous. While the formation of the primary particles occurs by homogeneous nucleation, it can be easily estimated using gas reaction kinetics that the coating on the primary particles grows heterogeneously and that homogeneous nucleation of nanoparticles originating from the second compound has a very low probability. [11] [12]

#### Sol-Gel Method

Sol-gel technology is a colloidal chemistry technology, which offers possibility to produce various materials with novel, predefined properties in a simple process and at relatively low process cost. The sol is a name of a colloidal solution made of solid particles few hundred nm in diameter, suspended in a liquid phase while gel can be considered as a solid macromolecule immersed in a solvent. Sol-gel process consists of chemical transformation of a liquid (the sol) into a gel state and with subsequent post-treatment and transition into solid oxide material. It can be effectively implemented for preparing ceramic and glass materials in different shapes like spherical, thin films, fibers, membranes etc. The sol is usually prepared by hydrolysis and condensation reaction of inorganic metal salts or different organic compounds. The processes like spin coating, dip coating, precipitation, spray pyrolysis, emulsion techniques etc can be used to make different shaped materials.

#### Pulsed Laser Method

Pulsed Laser methods is used for synthesis of nanoparticles and films implementing the laser beam as the primary source of ablation for generating materials directly from a solid sections. Since the nanoparticulate layers of very minute size may be easily manufactured, the technique find potential applications in manufacturing analytical fields like coupled plasma emission spectrometry, ceramic particles and coatings. The process of synthesis of nanoparticles involves generation of aerosol and fog through liquification and then solidification of droplets. The general dynamics of both the aerosol and the fog favours the aggregation process and micrometer-sized fractal-like particles are formed.

#### Thermolysis

The techniques involves generation of nanoparticulate by heating the sample at higher temperatures (650oC and above). Owing to high temperature, the compounds of solid material decompose itself and combine to form colloidal metal particles. The technique is used to produce nanomaterials with crystalline structures and larger surface areas. [13]

#### Hydrothermal Process

For generation of metal oxide nanoparticles, the hydrothermal process is commonly used. In the process, the reaction takes place with aqueous solution placed in highly pressurized chamber called autoclave. The methodology used can easily

help to control grain size, reaction temperature, pressure, solvent properties, additives and aging time. [14]

Each of these methods has their own advantages and disadvantages depending on the composition and properties of nanomaterials. Among these methods, vapor deposition method is most widely used owing to its advantages such as low cost, fast synthesis, capability of mass or continuous production and control over certain aspects of nanomaterials synthesized. Considering the various advantages of technique and the availability of resources, the present work implemented Sputtering and High Vacuum Thermal Deposition for manufacturing of the required nanostructures. In this chapter, the basic principles involved with these techniques are discussed followed with the method of preparation and experimental set ups used.

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