

## Synthesis of Nanostructure Materials

There are a large number of techniques used to synthesize different types of nanomaterial in the form of colloids, powders, tubes, clusters, wires, rods, thin films etc. As nanotechnology is an interdisciplinary subject there are therefore various physical, chemical, biological, and hybrid techniques available to synthesize nanomaterials.

### Top-Down and Bottom-Up Technique:

There are two different approaches of nanomaterial synthesis and fabrication. One is top-down and the other is bottom-up approach.

In top-down approach a bulk material is taken and machined it to modify into the product of desired shape and size.

Examples: Lithography, Etching and Ball milling is an important top-down approach. In ball milling technique macrocrystalline structures are broken down to nanocrystalline structures.

Bottom-up approach is used to build something from basic materials. In this technique atoms and/or molecules come together to form nanomaterials of required size and shape by controlled deposition or reaction parameters.

Examples: Sol-Gel technology, Chemical Precipitations, Electrodepositions, Physical and Chemical vapour deposition (PVD, CVD), Epitaxial growth, Laser ablation etc.

### Lithography:

Lithography is a top-down approach for nanomaterials synthesis.

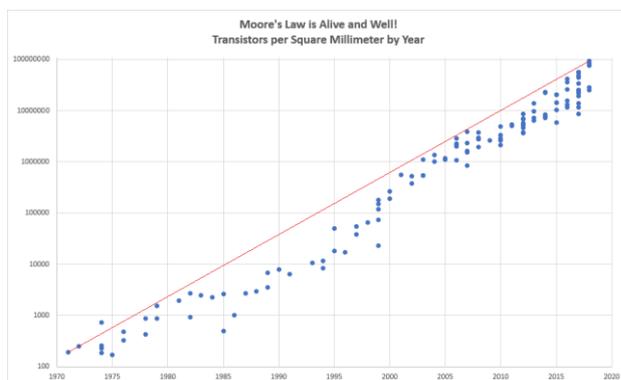
The word 'Litho' is a greek word which means stone. Hence lithography means carving a stone or writing on a stone.

It is used to mean a process in which sample is patterned by removing some part of it or sometimes even organizing some materials on a suitable substrate.

It is used in IC technology. British Engineer G.W.A Dummer first proposed that an entire circuit should be directly made on a silicon substrate instead of wiring together the different components.

### Moore's law:

In 1959 James Moor predicted that in every 18 months the number of a transistor on a chip doubles or the processing power of a computer doubles.



The lithography technique involves transfer of some predesigned geometrical pattern (called master or mask) on a 'resist' cover semiconductor (silicon wafer) or directly patterning using suitable radiation.

'Resist' is a radiation sensitive material.

Generally there are three types lithography process based on incident radiation.

1. Photolithography
2. X-ray lithography
3. Electron beam lithography

### **Photolithography:**

Photolithographic process uses light to transfer a geometric pattern from a photomask to a light sensitive chemical (photoresist) on the substrate.

There are two types of photoresist i.e positive photoresist and negative photoresist.

**Positive photoresist:** The photoresist which *degrades* under exposure of light is known as positive photoresist.

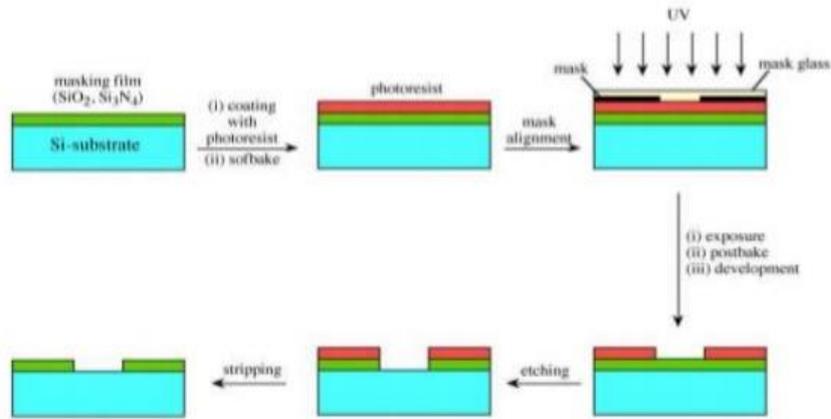
**Negative photoresist:** The photoresist which *hardens* under exposure of light is known as negative photoresist.

**Photomask:** A photomask is generally quartz plate or silica glass coated with chromium with different required design. The quartz is transparent to UV light whereas the chromium metal is opaque to UV light.

Various structures can be generated on the substrate on the basis of different pattern of photomask and then different silicon based electronic and photonic devices can be developed.

### **Basic steps of Photolithography Process:**

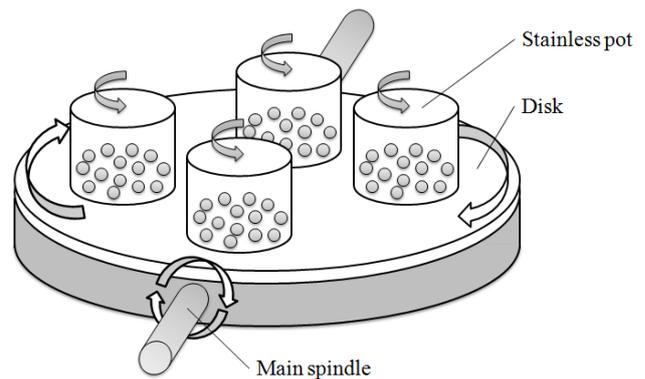
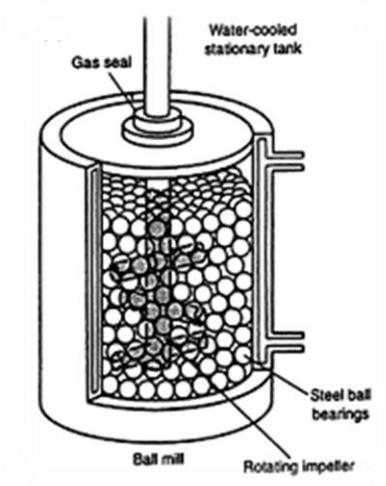
1. Surface preparation
2. Deposition of barrier layer ( $\text{SiO}_2$ )
3. Photoresist coating
4. Soft baking
5. Photo Mask aligning
6. Expose to UV
7. Developing
8. Hard baking
9. Etching
10. Resist stripping



### Ball Milling:

High energy ball milling is one of the simplest top down approach of making nanopowder of some metals and alloys.

Hardened steel or tungsten carbide balls are used in different size of containers depending on required quantity. Powder or flakes ( $<50\ \mu\text{m}$ ) of different materials are put into the container. Generally the mass ratio of balls to material is taken as 2:1 and the container is less than half filled to enhance efficiency of milling. After closing the container or the containers are allowed to rotate at high speed (a few hundreds of rpm) around their own axis or some central axis and are therefore called as 'planetary ball mill'. Heavy milling balls increase the impact energy on collision. The container may be filled with air or inert gas. Larger balls are used to produce smaller grain size but larger defects in the particles.



Planetary ball mill

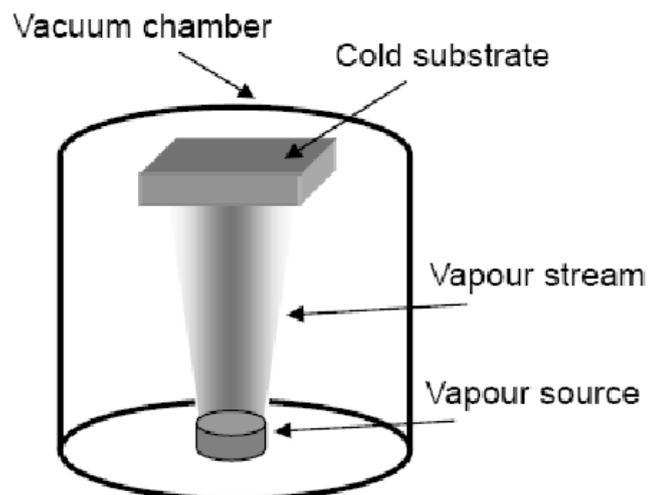
### Uses and Advantages:

Some metals and alloys like Co, Cr, W, Al-Fe, Ni-Ti, Ag-Fe etc and different types of metal oxide nanoparticles such as  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{BaTiO}_3$ , FeO,  $\text{Fe}_2\text{O}_3$ , etc are synthesized using ball mill. Few milligrams to several kilograms of nanopowder can be synthesized in a short time. This method can also be used to make a variety of carbon-based nanomaterials such as CNT (carbon nano tube).

### Limitation:

Although this is a very simple method for synthesizing nanoparticles, there are some impurities arise from rotating balls and the inert gas also.

### Gas phase condensation:



One of the most widely used methods for the synthesis of nanomaterials is the gas condensation process. By this method, a metallic or inorganic material is vaporised using thermal evaporation sources, electron beam evaporation devices or sputtering sources in an atmospheric pressure of 1-5 Torr He (or another inert gas). In high vacuum conditions, the atoms leave the source in straight line paths and continue until they impinge upon a solid surface which is called a substrate. The substrate is at temperatures much lower than the source temperature and the impinging atoms give up their excess energy to the surface where they must adhere and grow a film. In the presence of the inert gas molecules, the evaporated atoms lose kinetic energy by collisions, so that clusters form in the vicinity of the source by homogeneous nucleation in the gas phase and grow by coalescence and incorporation of other atoms.

## **Physical Vapour Deposition (PVD)**

Physical vapour deposition (PVD) describes a variety of vacuum deposition technique which can be used to produce thin films and coatings on a substrate. PVD is a process in which the material goes from a condensed phase to a vapour phase and then back to a thin film condensed phase. The most common PVD processes are sputtering and evaporation.

Applications:

PVD is used in the manufacture of items which require thin films for optical, mechanical, chemical or electronic functions.

PVD is used to make

1. Semiconductor devices such as thin film solar cells.
2. Aluminized PET (Polyethylene terephthalate) film for food packaging and balloons.
3. Titanium nitride coated cutting tools for metalworking.

Advantages:

There are various advantages of PVD technique over any other technique.

1. This process is versatile and deposits almost any material amenable to vaporization.
2. No or little scope of chemical reaction in flight or in deposit.
3. Little or minimal damage to substrate.

Limitation:

1. Non-uniformity of thickness.
2. Line of sight process-shadowing.
3. Difficult to evaporate materials with low vapour pressures.