

Chapter 2: Preparation Methods of nanomaterials

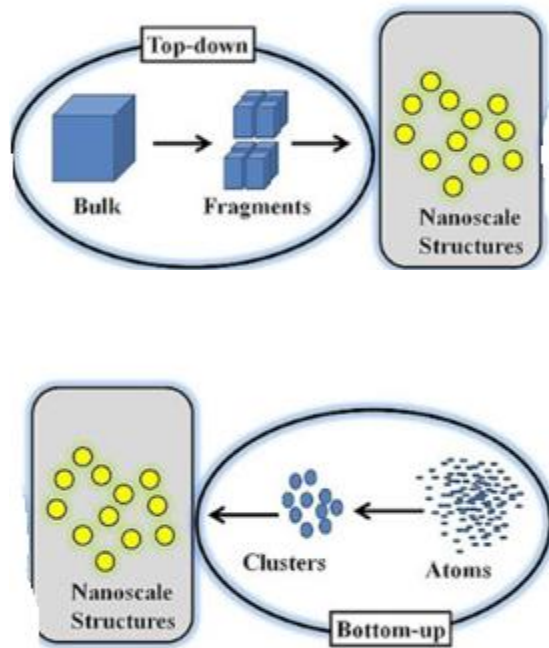
By

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Synthesis of Nanocrystalline Materials

- Two ways can be chosen to prepare nanomaterials
- Methods for creating nanostructures



Physical method

Chemical method

Synthesis of Nanocrystalline Materials

1. Physical method

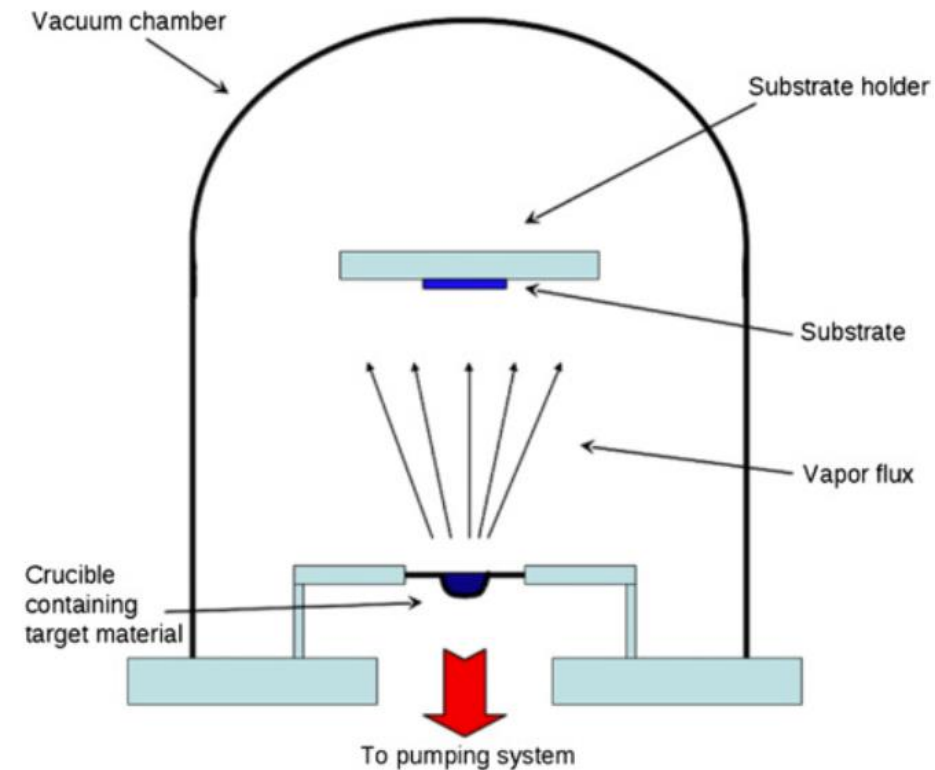
A-Mechanical grinding

- Top-down method where by the structural decomposition of coarser-grained structures.
- It's an advantage method to make nanocrystalline materials:
 - ✓ its simplicity
 - ✓ the relatively inexpensive equipment needed
 - ✓ the applicability to essentially the synthesis of all classes of materials.
- It's a disadvantage method to make nanocrystalline materials:
 - contamination from milling media and/or atmosphere.
 - to consolidate the powder product without coarsening the nanocrystalline microstructure.

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B-Thermal evaporation

- The material designated for evaporation is heated to an appropriate temperature in a specialized container (ceramic crucible, tantalum boat, tungsten spiral wire, etc.) by applying energy (electrical current, electron beam, laser, arc discharge, etc.).
- The thermally emitted atoms or molecules move from the surface of the evaporated substance, creating a coating on the substrate or adjacent walls.
- High Vacuum is 1.3×10^{-4} Pa



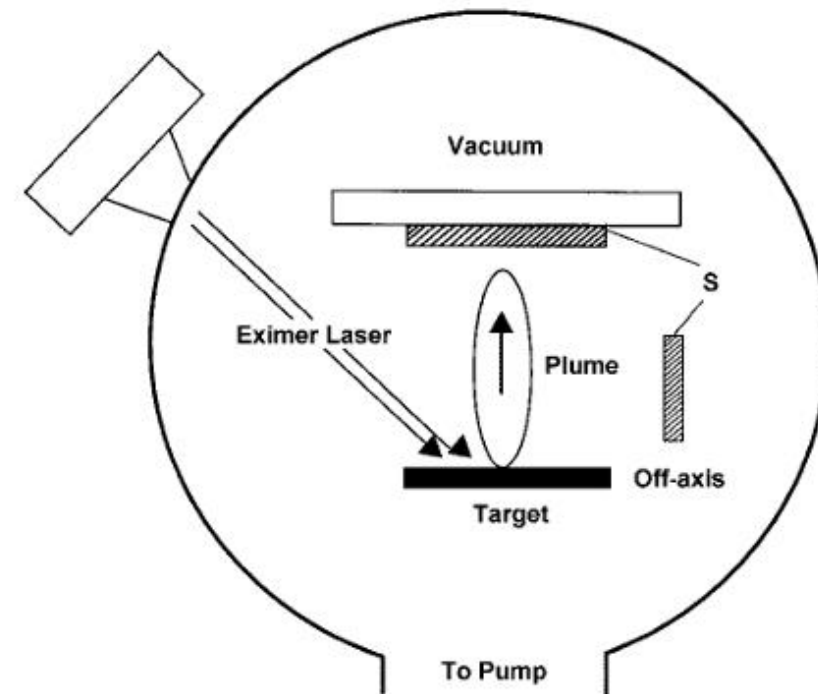
Synthesis of Nanocrystalline Materials

- The procedure typically occurs under High Vacuum, allowing coating particles to travel straight from the source to the substrates without collisions with residual gas.
- Disadvantages
 1. Evaporation sources can be categorized by the method of energy supply.
 2. Cost
 3. Chemical reactions between crucible and evaporation material are possible which can lead to impurities in the film and/or to the destruction of the evaporation source.

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C-Pulsed laser deposition (PLD)

- is an improved thermal process for depositing alloys and/or compounds with a controlled chemical composition. In laser deposition, a high-power pulsed laser such as the KrF excimer laser is irradiated onto the target of source materials through a quartz window. A quartz lens increases the energy density of the laser power on the target source. Atoms ablated or evaporated from the surface are collected on nearby sample surfaces to form thin films.



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- The target material is locally heated to the melting point, melted, and vaporized in a vacuum. The laser pulse may also provide photoemitted electrons from the target to make a plasma plume and the evaporation mechanism may be complex since the process includes the thermal process and the plasma process.
- The PLD has the advantage of being simple in design, and the target has many forms such as a powder, sintered pellet, and single crystal.
- However, at present, the process has a limited area of uniform deposition, and microsized globules or particles are ejected from the target.

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D- Sputtering

Sputter deposition is a physical vapor deposition process for depositing thin films. Sputtering means ejecting material from a target and depositing it on a substrate, such as a silicon wafer. Substrates are placed in a vacuum chamber and pumped down to a prescribed process pressure. Sputtering starts when a negative charge is applied to the target material, causing a plasma or glow discharge.

Positive charged gas ions generated in the plasma region are attracted to the negatively biased target plate at a very high speed. This collision creates a momentum transfer and ejects atomic-size particles from the target. These particles are deposited as a thin film into the surface of the substrates.

□ The **two-type** sputtering method:

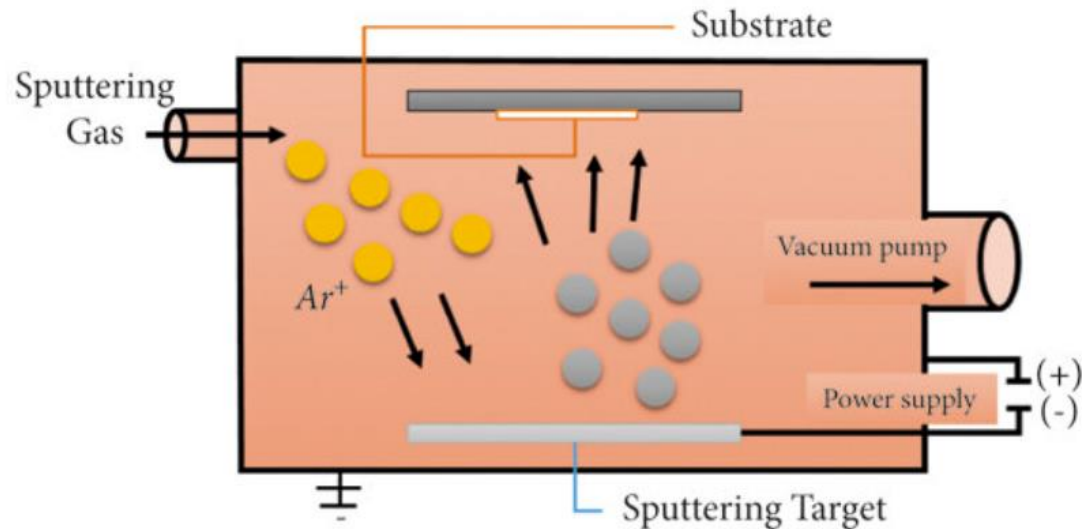
- 1- RF sputtering method
- 2-DC sputtering method

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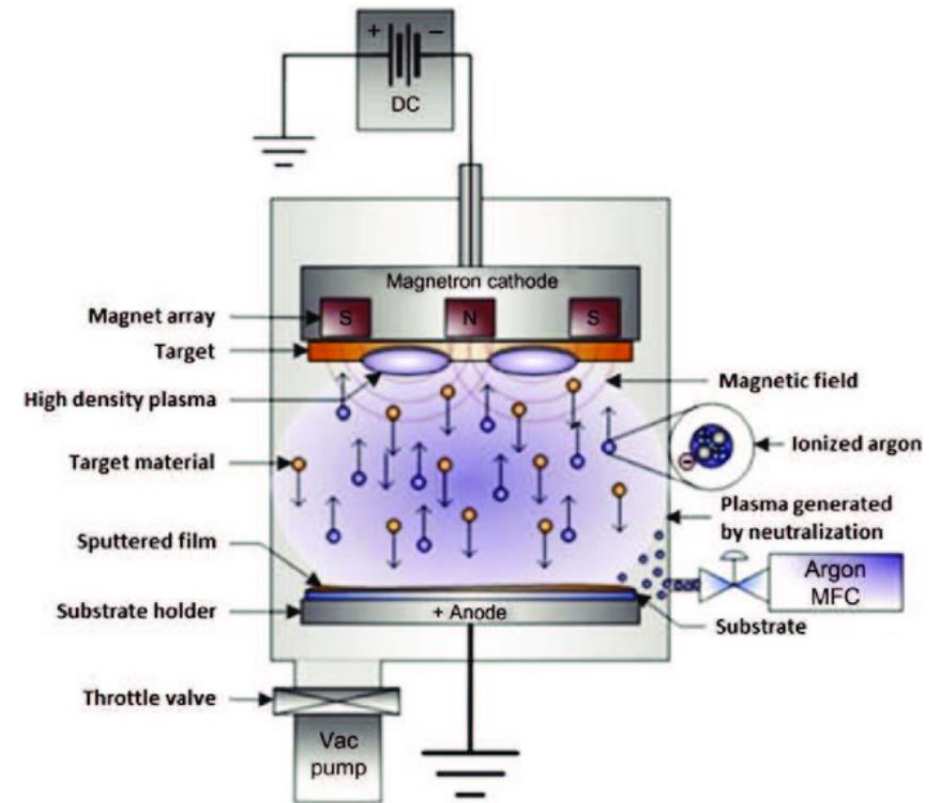
- In sputtering method, particle to particle collisions will involve an elastic transfer of momentum, which can be utilized to apply a thin film to the substrate. In this technique, ions are accelerated toward a target by utilizing electric fields. These ions are usually derived from either an ion gun or from exciting a neutral gas into ion plasma. As the ions are accelerated and bombard the target surface, they dislodge target atoms and other ions. The ejected atoms attach themselves to the substrate, and a thin film of the target material is produced.
- Depending on the target material either RF or DC sputtering may be used. If the target material is a conductor, a constant voltage can be used to accelerate the ions (DC sputtering) As the ions strike the surface, the resulting charges can move freely about the material to prevent any charge buildup. However, if the material is an insulator, the conduction bands will not allow free charge movement.

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- RF sputtering diagram

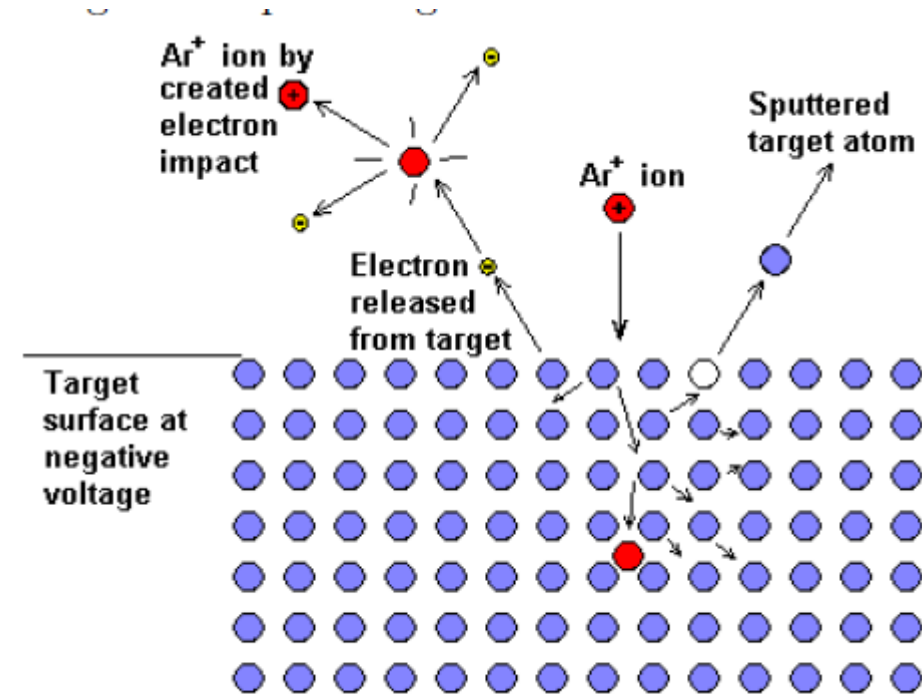


- DC sputtering diagram



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- As the ions strike the surface, their charge will remain localized, and over time, the charge will build up, making it impossible to bombard the surface further. To prevent this, alternating current is used at a frequency above 50 KHz. A high frequency is used so that the heavy ions cannot follow the switching fast enough, and only electrons hit the surface to neutralize the charge (internet).

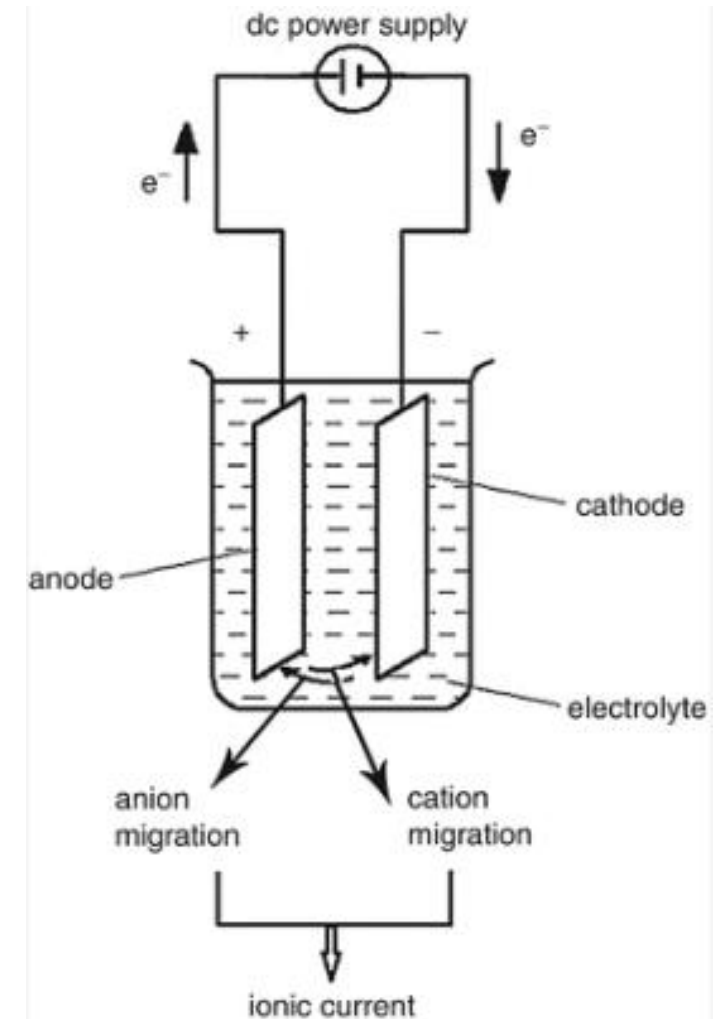


Synthesis of Nanocrystalline Materials

E- Electrodeposition method

the process of coating a thin layer of one metal or semiconductor on top of a different metal or conductive layer to modify its surface properties.

The electrodeposition method is used in electroplating. Generally, two metal electrodes are immersed in the specific electrolyte in the solution. Then, the external electric field is applied to deposit the required metal on the working electrode (cathode).



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- The thickness of the metal films is controlled by varying the electrode potential and current density. Typically, a milliwatt-range DC power supply is given to the cathode and anode electrodes, where the positive ions move toward the cathode, discharge, and chemically react to synthesize the desired material.
- The direct current (DC) power source is the externally applied unidirectional current source across the electrode system.
- The flow of current is the flow of electrons in the circuit. In the electrolyte solution, the electrical current flows in the form of ions. The negative ions (anions) attract toward the anode, whereas the positive ions (cations) attract toward the cathode upon applying the appropriate external power source.

THANK YOU

