Nanotechnology Course/ Ph-457 Lecture 5

Chapter 2: Preparation Methods of nanomaterials

By

Dr. Marwah Jawad Kadhim

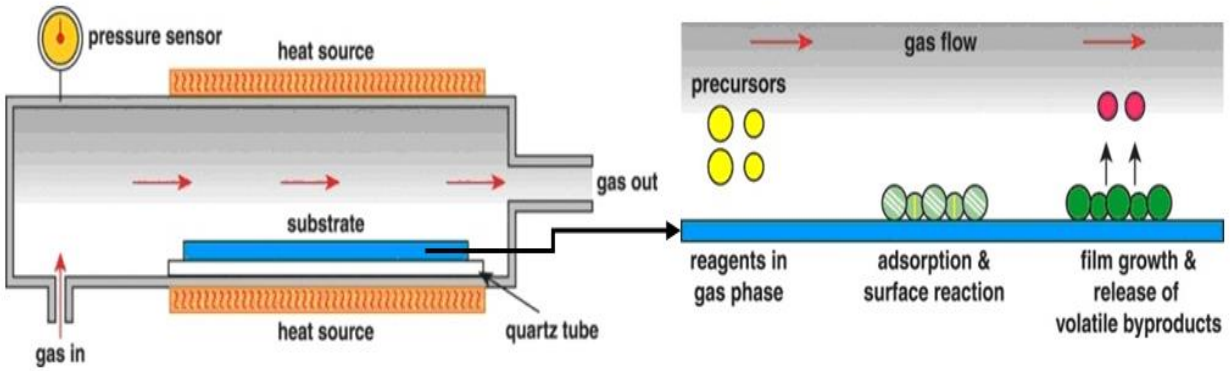
Synthesis of Nanocrystalline Materials

2- chemical method

A- Chemical Vapor Deposition (CVD)

This method is widely used to prepare nanocrystalline materials, especially one-dimensional nanomaterials such as nanowires, nanorods, and nanotubes. CVD involves flowing a precursor gas or gases into a chamber containing one or more heated objects to be coated. Chemical reactions occur on and near the hot surfaces (substrates), producing thin film deposition on the surface. The Figure below shows the substrate's CVD system and thin film growth steps.

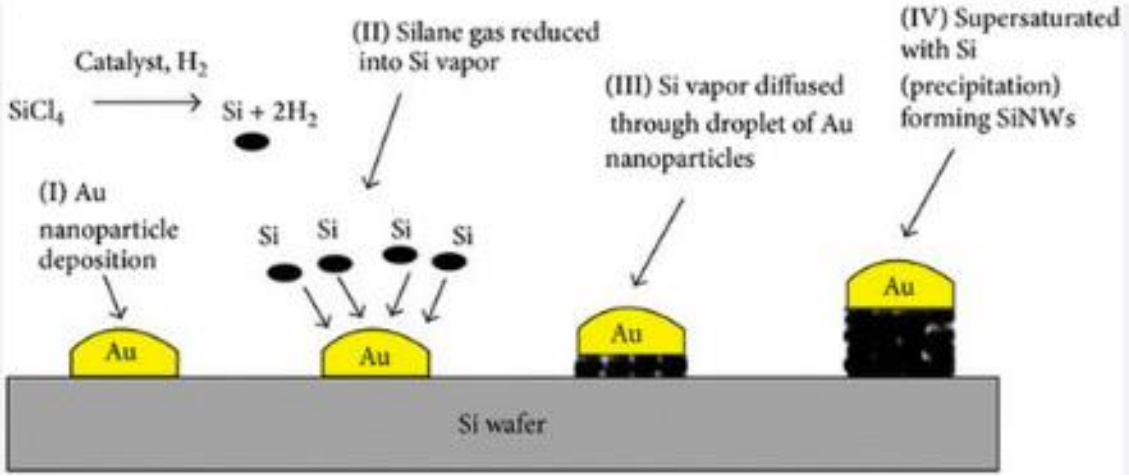
CVD is not cheap and requires gas to transfer the molecules or atoms from the source to the substrates. This method does not allow for controlling the thickness of the prepared thin films, but the product allows for high quality and crystallinity.



Synthesis of Nanocrystalline Materials

- One-dimensional crystalline nanowires are synthesised via a liquid metal-alloy droplet-catalyzed chemical or physical vapour deposition technique conducted within a vacuum deposition apparatus. Au-Si droplets on the substrate surface reduce the activation energy required for conventional vapor-solid development.

- The growth mechanisms referred to as vapor-liquid-solid (VLS). This process employs catalysts such as Au, Zn, Ag, Al, Sn, Ti, and other metals to facilitate the synthesis of nanowires.

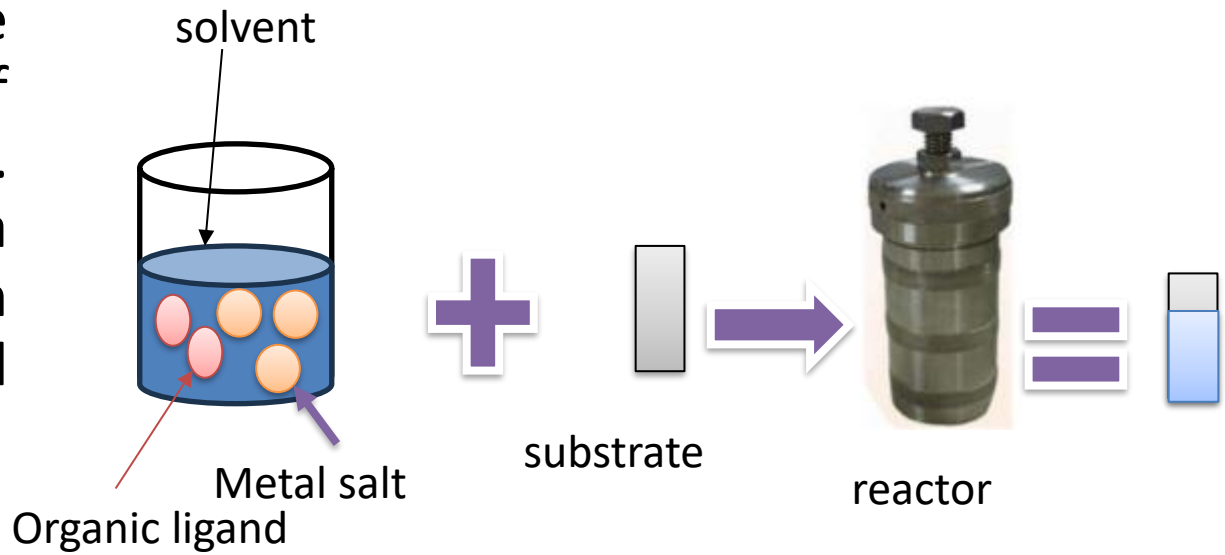


Nanoscale Research Letters,11(1),2016

Synthesis of Nanocrystalline Materials

B- Hydrothermal and Solvothermal

- Hydrothermal synthesis refers to the synthesis by chemical reactions of substances in a sealed, heated solution above the ambient temperature and pressure.
- The hydrothermal synthesis of single crystals depends on the solubility of minerals in high-pressure hot water. The crystal development occurs in a steel pressure chamber known as an autoclave, where a nutrient is provided alongside water.
- A temperature gradient is maintained at the opposite ends of the growth chamber so that the hotter end dissolves the nutrient and the cooler end causes seeds to take additional growth.



Synthesis of Nanocrystalline Materials

Advantages:

- Most materials can be soluble in a proper solvent by heating and pressuring the system close to its critical point.
- Adjusting parameters such as reaction temperature, reaction time, solvent type, surfactant type, and precursor type allows for easy and precise control of the size, shape distribution, and crystallinity of the final product.
- Significant improvement in the chemical activity of the reactant, the possibility to replace the solid-state synthesis and materials that may not be obtained via solid-state reaction may be prepared through hydrothermal/solvothermal synthesis.

Disadvantages:

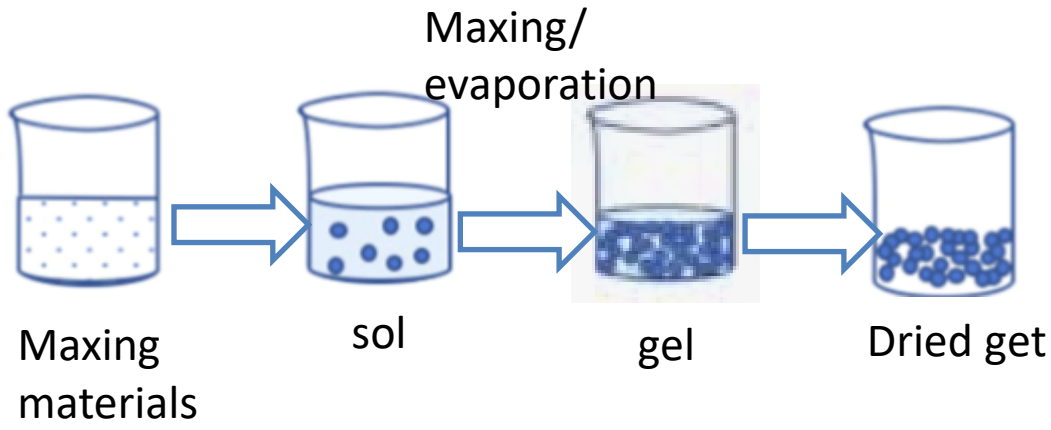
- The need for expensive autoclaves.
- Safety issues during the reaction process.
- Impossibility of observing the reaction process

Synthesis of Nanocrystalline Materials

C- Sol-Gel

The sol-gel process is a technique for synthesizing solid materials from molecular precursors. This chemical process involves the gradual transformation of the sol (or solution) into a gel-like diphasic system comprising both a liquid and a solid phase, with morphologies varying from discrete particles to continuous networks of polymers or nanomaterials.

Precipitation can form ultrafine and uniform ceramic powders. These powders, of single and multicomponent composition, can be produced at a nanoscale particle size for dental and biomedical applications.



Synthesis of Nanocrystalline Materials

- The **general mechanism sol-gel** of synthesis of metal nanoparticles is as follows:

✓ Uniformly mix the raw materials in the liquid phase.

✓ Carry out hydrolysis and polycondensation reactions to form a stable, transparent sol system.

✓ After various processes, such as drying and sintering, materials with microstructures and even nanostructures can be prepared.

✓ Colloidal particles slowly aggregate after the aging of sols to form gels with three-dimensional network structures.

Disadvantages:

➢ Relatively longer in reaction time.

➢ Organic solvents may be harmful to the human body.

Advantage

- Starting materials are at the molecular level, and the final products prepared are homogeneous.
- Products are relatively higher in purity.
- Enables synthesis at a low temperature.
- Controllable degree of porosity.
- Easy to prepare in various sizes and convenient to carry out.
- Allows for fine control of the product's chemical composition, especially
- This allows small quantities of dopants to be introduced in the sol and uniformly dispersed in the final product
- suitable for preparing multicomponent materials.

Synthesis of Nanocrystalline Materials

D- Chemical Bath deposition CBD

CBD is an easy technique that requires the placement of a substrate in a tank filled with a highly saturated solution of dilute aqueous precursors, including metal salts, complexing agents, and pH buffers.

- The deposition mechanism is largely the same for all such materials. A soluble salt of the required metal is dissolved in an aqueous solution, to release cations. The non-metallic element is provided by a suitable source compound, which decomposes in the presence of hydroxide ions, releasing the anions. The anions and cations then react to form the compound

Synthesis of Nanocrystalline Materials

A soluble salt of the required metal is dissolved in an aqueous solution to release cations. The non-metallic element is provided by a suitable source compound which decomposes in the presence of hydroxide ions, releasing the anions. The anions and cations then react to form the compound.

Advantage

1- a very simple and stable

2- economically reproducible technique

3- cost-effective

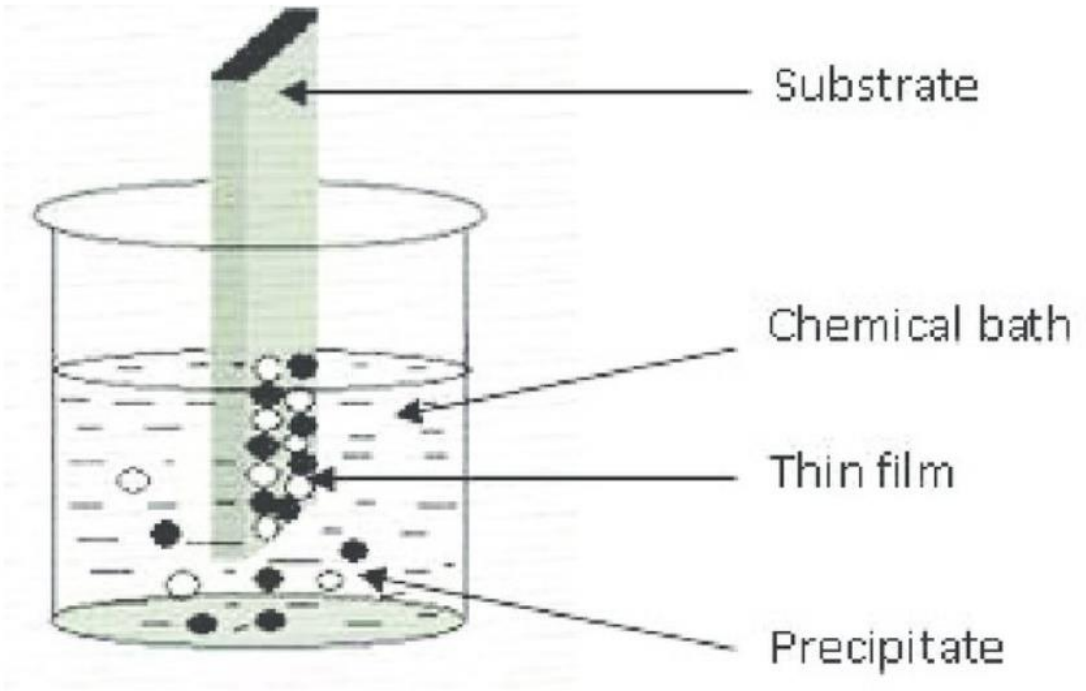
4- can be applied in large area deposition at low temperatures.

5- used to deposit thin films of a wide range of materials

Synthesis of Nanocrystalline Materials

The thin film growth in CBD occurs via the following four possible mechanisms.

1. Simple Mechanism (Ion-by-Ion)
2. Simple Cluster (Hydroxide) Mechanism
3. Complex-Decomposition Ion-by-Ion Mechanism
4. Complex-Decomposition Cluster Mechanism



American Institute of Physics,1728, 020489 (2016)


THANK YOU