

Principle of Synthesis of Nanocrystalline Thin Films

J K Dongre*, Yuvraj Patil**

*(Department of Physics, Prime Minister College of Excellence, Govt. Autonomous Post Graduate College Chhindwara, India
Email: jdongre6@gmail.com)

** (Raja Shankar Shah University Chhindwara (M.P.) India
Email: yuvrajchhindwara@gmail.com)

Abstract:

Thin film technology plays a vital role in the development of optoelectronic, photonic, and magnetic devices. Thin films are classified into two types based on their application. The first type is the wet chemical method, which does not require high temperatures and expensive equipment. The second is the dry method, which requires high temperatures and expensive equipment, as well as a vacuum environment. In this article, we discuss the basic principles of operation of various thin film fabrication methods.

Keywords — Thin film, CBD, SILAR, Sol-Gel method, Laser Ablation

I. INTRODUCTION

Grove was the first to explain his observation that metal films are formed by the sputtering of positive ions and the cathode. This observation can perhaps be called the beginning of "thin film science." However, thin film research has seen unprecedented growth in industries across diverse fields, including electronics, optics, space science, aircraft, and defence [1]. But currently, advancements can be seen in the use of thin films in new types of devices such as monolithic and hybrid circuits, field effect transistors (FET), metal oxide semiconductor transistors (MOST), sensors, switching devices, cryogenic applications, high density memory systems for computers, etc. Therefore, the present article focusses on the synthesis of thin films with different methods along with their basic principle.

II. PRINCIPLE OF SYNTHESIS PROCESS

Thin films are a materials coating on substrates having thicknesses in the order of few nanometers to a few micrometers [2]. The elemental composition of a thin film depends on the structure, and mechanisms of thin films. The thin films can be mainly categorised in two ways.

1. Wet chemical based

2. Dry process based

Here we discuss on the preparation method which is based on wet chemical synthesis route and dry process route.

A. Chemical Bath Deposition

CBD commonly known as solution growth technology. The method is used to deposit various kinds of materials on to substrate at low temperature and low cost. it doesn't require sophisticated instruments only magnetic stirrer, thermometer, beaker and regulated power supply are required to fabricate thin film onto substrates [3]. numerous materials including chalcogenides and metal oxides and other different kinds of materials fabricated with this technique. The operation principal of CBD is based on precipitation. When the solution is saturated, the ionic product is equal to the solubility product. But when ionic product exceeds the solubility product the solution is supersaturated, precipitation occurs and ions combine on the substrate and in the solution to form nuclei.

The preparation steps are summarized as-

(1) Using necessary reagent and their solution in solvent

- (2) Substrate immersion and rotation in the solution
- (3) chemical reaction and nucleation adhere on the substrate surface and initiate thin film growth
- (4) After a necessary required periods of time the substrate is removed and washed to remove excess reactants from the substrate surface
- (5) Finally, the substrate coated with thin films are annealed at required temperature at given atmosphere

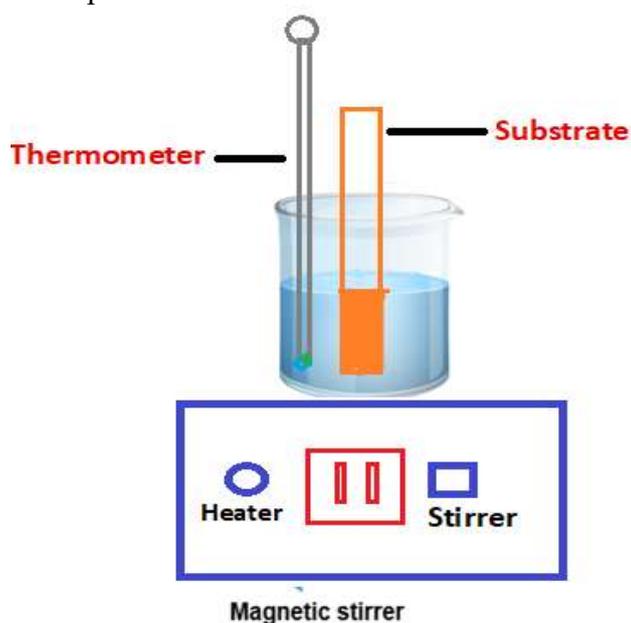


Fig. 1 A typical diagram for CBD

The thickness and microstructures of thin films can be controlled using parameters viz temperature, Ph of solution, and concentration of reagents compound.

The growth mechanism of thin film deposition process is of two types.

- (i) 'Ion-by-ion growth'
- (ii) cluster-by-cluster growth

B. Successive Ionic Layer Adsorption and Reaction (SILAR) Method

The thin film grown by this technique have controlled thickness and composition. The method is modified version of CBD. SILAR is based on the successive adsorption of ions present in solution onto the surface of a substrate, followed by a chemical reaction to form a thin film layer.

The SILAR method consists of four steps [4]:

First step: In the first step substrate is dipped into a cation solution, ions get adsorbed on substrate surface, and formed double layer along with inner layer (cations) and charge-balancing outer layer (anions).

Second step: The substrate is rinsed with double distilled water to remove the loosely bonded ions from the double layer.

Third step: The immersion of substrate in the anion-based precursor solution the ions diffuse from solution to inner layer reacted with cations and form the first solid thin-film layer.

Fourth step: The immersion of substrate in double distilled water to remove the loosely bonded ions from the surface.

The basic operation principle of SILAR method is shown in the figure 2.

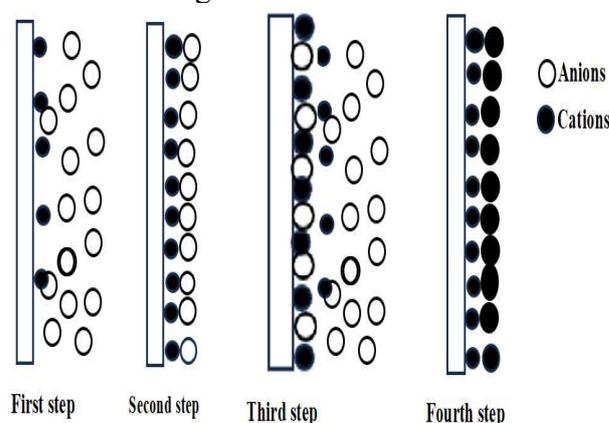
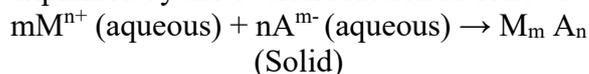


Fig. 2: Schematic view of thin-film growth during one SILAR cycle (Re-draw the image Ref. [5])

These four sequential steps complete one cycle of SILAR. The thickness of thin film produced by single cycle of SILAR is very small. The desired film thickness depends on the number of repeated cycles of SILAR.

In a SILAR process the formation of thin film can be explained by the overall reaction as follows:



The three major factor concentrations, pH and counter-ions of the precursor solutions influence the deposition of thin films in the SILAR method.

C. Sol-Gel method

Abelmann discovered that orthosilicates hydrolysed in air to form gels, thus beginning a new era of sol-gel chemistry in 1846.

Sol-gel synthesis methods typically consist of two separate stages: solution and gelation. "Sol" describes a stable mixture of polymer particles in a solvent, whereas "Gel" describes a three-dimensional structure with a liquid phase inside a continuous network,

To prepare nanomaterials by sol-gel method there are three steps [4]

- (1) sol preparation,
- (2) gel formation, and
- (3) subsequent heat treatment.

In first step the metal inorganic salt (or metal organic alkoxide) dissolve in a liquid solvent (viz water/organic solvent) to form a uniform solution. To this precursor salt solution an additive is added to hydrolyze, alcoholize, or polymerize and form sol system.

In second step gel can be formed either **aging the sol** since it is concentrated or partial evaporation of the sol and dried at low temperatures in a vacuum to obtain a colloidal powder (or dry gel film).

In third step applying Subsequent heat treatment (such as sintering, carbonization, or high-temperature reaction) uniform functional nanomaterials from powders (or aerogels). The formation of nanomaterial through sol-gel method is depicted in figure 3.

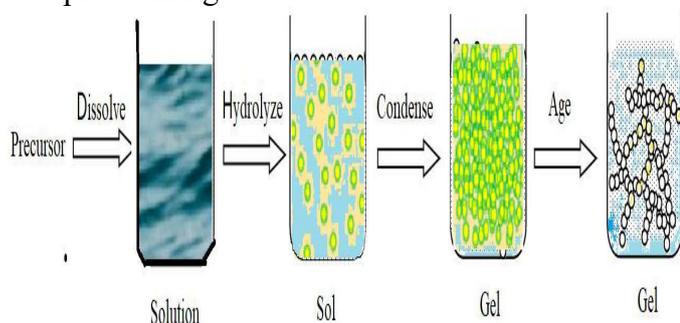
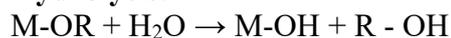


Figure 3: A typical diagram shows basic process of sol-gel method

In the hydrolysis process, metal alkoxide precursors react with water, to form a homogeneous solution of precursors

Hydrolysis:



In the condensation process when the metal alcohol group undergoes to heating forms a metal oxide

bond (M-O-M). The second step, in which M-OH is added, forms a polymer network with M-O-M linkages. Subsequently, the removal of solvents leads to gel formation. The overall reaction is

Condensation:



D. Laser Ablation Method

Rapidly heating a solid target with a high-energy laser causes it to melt and vaporize, forming a plasma plume that cools and condenses into nanoparticles in a liquid or gas. This is the basic idea behind laser ablation for nanomaterials [6]. In the method particle size is controlled by the parameters like pulse energy, wavelength, repetition rate, and spot size. Solvent type and their pH, and temperature influence stability influence the morphology, and agglomeration. Using this method high-purity nanomaterials including metals, oxides, quantum dots, and carbon nanotube with minimal contamination can be synthesized. The operation principle for formation of nanomaterials is depicted in the figure 4.

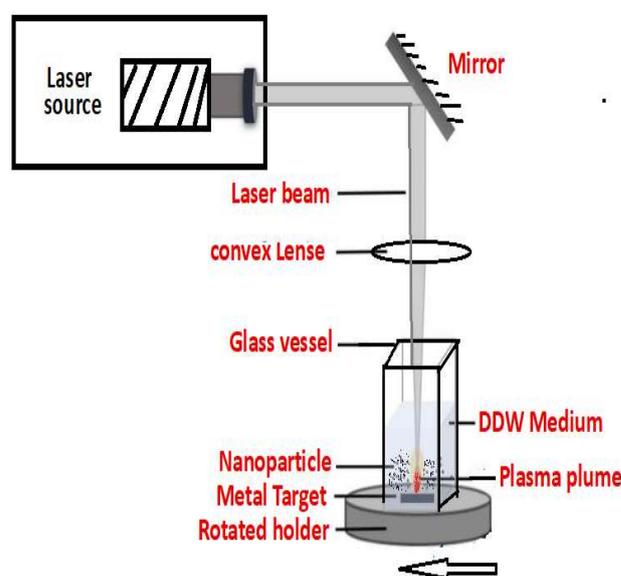


Fig. 4 schematic diagram for Laser Ablation Method (Re-draw the image Ref. [5])

E. Thermal Evaporation Techniques

The fabrication process is based on principle of evaporation of metal or any desired materials at elevate temperature under vacuum environment.

Finally, the vapor is allowed to condense on a substrate to form a thin film [7].

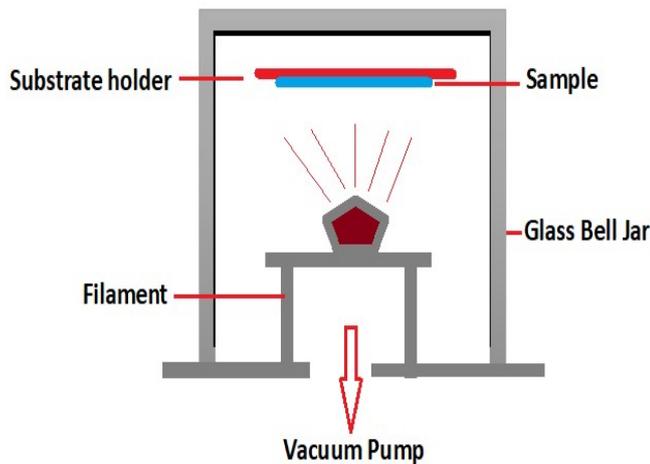


Fig. 5: show a schematic diagram for Evaporation Techniques (Re-draw the image Ref. [8])

Typically, in thermal evaporation, a vacuum is highly needed. The necessary temperature in chamber is produced by using a resistive heater or an electron beam. Therefore, under vacuum atmosphere the material gets evaporate and condenses to forms a solid film on substrate, The film fabrication parameters including the substrate temperature, deposition rate influenced the growth.

F. Spin coating

This technique produces thin films by pouring a solution onto a substrate at high spin. This creates a uniform film thickness [9].

The prepared thin films are utilized in microelectron optoelectronics, and materials research since it is a simple and reproducible method. By using Spin coating method numerous kinds of films including polymer films, oxides materials, and organic/inorganic hybrid materials can be fabricated. The major advantage of this method is that the control over the thickness of coated materials controlled by different coating parameters such as spin speed, solution viscosity and spinning time.

The fabrication principle is divided in four stage-

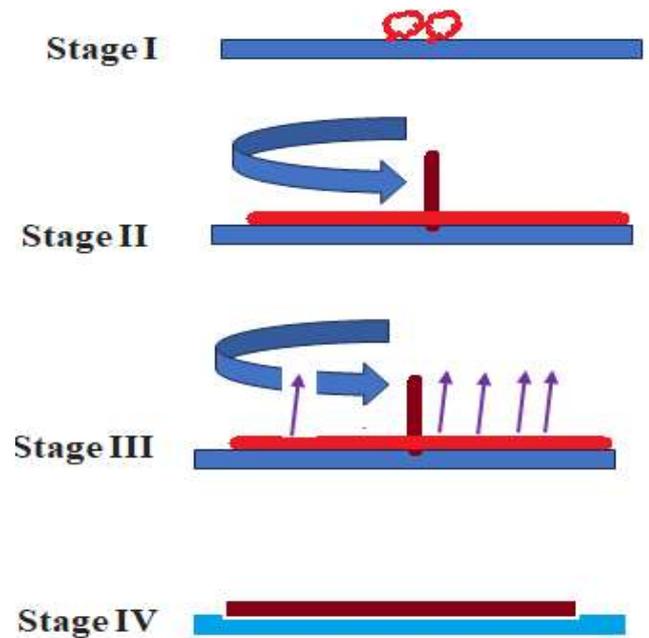


Fig. 6 Schematic of spin coating technique (Re-draw the image Ref. [9]).

In Stage 1, a few drops of the solution containing the material are injected onto the substrate using a syringe.

In Stage 2, the substrate is spun at a high speed, approximately in the range (typically 500- 5000 RPM). Centrifugal force then spreads the droplets onto the substrate, forming a thin film on the substrate. The film thickness is determined by the interaction between centrifugal force and surface tension.

In Stage 3, the rapid spinning of the substrate evaporates the solvent, forming a thin, semi-solid film on the substrate.

In Stage 4, the substrate containing the film is dried. Finally, we obtain a solid thin film deposited on the substrate.

The thickness of film obtained in the spin coating method can be expressed by following equation [10]

$$t = \frac{\eta}{\omega}$$

Where η = is used for the viscosity of a solution,
 ω = denotes angular velocity which is measured in (rad/s)

The above equation clearly predicts that-
 (1) Higher spin speeds lead thinner films.

(2) Lower solution viscosities also lead thinner films.

III. CONCLUSIONS

Thin films materials are utilized in semiconductor devices, integrated circuits, solar cells, light-emitting diodes, micro-electromechanical systems (MEMS), and also used in so many other new cutting technologies. In present article we have discussed in detail the various thin film deposition techniques, including CBD, SILAR, Sol-Gel method, Thermal Evaporation, Laser Ablation and Spin-coating.

REFERENCES

- [1] A. Goswami Thin Film Fundamentals, NewAge International India 1996,
- [2] D. Abou-Ras, T. Kirchartz, U. Rau, U. (Eds.) Advanced Characterization Techniques for Thin Film Solar Cells; Wiley-VCH Verlag GmbH and Co. KGaA:Weinheim, Germany, 2016
- [3] JK Dongre, V Nogrinya, M Ramrakhiani, Structural, optical and photoelectrochemical characterization of CdS nanowire synthesized by chemical bath deposition and wet chemical etching, Applied surface science 255 (12), 6115-6120
- [4] Mohammad Mansoob Khan, Photocatalysts Synthesis and Characterization Methods, ELSEVIER, 2025.
- [5] D. Mitzi, Solution Processing of Inorganic Materials, John Wiley & Sons, Inc., Hoboken, New Jersey, 2008
- [6] A. J., Hadi, U.M. Nayef., F.A.H. Mutlakand M.S. Jabir., Laser-ablated zinc oxide nanoparticles and evaluation of their antibacterial and anticancer activity against an ovarian cancer cell line: in vitro study. 18 (2023) 2091-2101.
- [7] F. T. Z. Toma1, Md. S. Rahman, K. Md. Amjad Hussain, S. Ahmed, Thin Film Deposition Techniques: A Comprehensive Review, J Mod Nanotechnol 2024; 4: 6
- [8] P Tyagi, M.R.Johan, Bin MR Johan. Titanium dioxide/graphene composites for dye-sensitized solar cell applications. Green Sust Proc Chem Environ Eng Sci, 2022; 2022: 313-339

[9] R Srinivasan, T G Ramesh, G Umesh, and C S Sundar, Experimental Techniques in Physics and Materials Science: Principles and Methodologies World Scientific 2024.

[10] A. Chandra and A. Chandra, Synthesis and Characterization of Materials Books clinic Publishing Bilaspur, Chhattisgarh, India 2025