

**RESEARCH**

# A Review on Synthesis Methods of Materials Science and Nanotechnology

Avadhesh Kumar Yadav<sup>1,\*</sup> 

<sup>1</sup>Dept. of Physics,  
F.A.A. Govt. PG College  
Mahmudabad, Sitapur 261203  
India  
(An Associated College of  
University of Lucknow, Lucknow)

**\*Corresponding author:**  
Dr. Avadhesh Kumar Yadav  
E-mail: [yadav.av11@gmail.com](mailto:yadav.av11@gmail.com)

**Web of Science Researcher ID:**  
AAI-2004-2019

## ABSTRACT

Materials science is a fast developing field of research in which a lot of advancements have been reported in few decades. The progress in materials science is essential due to its wide range of applications in solid state fuel cells, display materials, solar cells, energy storage devices, automotive sectors, electronics and environment, mechanical, medical & aerospace industries. Doping and substitutions in host materials are excellent ways of changing the properties of any materials. There are two main forms of materials, thin film and bulk, which are used in research. These two forms of materials are synthesized by two basic approaches, named as top-down and bottom-up. Bottom-up and top-down approaches are comprised of several synthesis methods, e.g., solid state reaction method, hydrothermal method, co-precipitation method, sol gel method, auto-combustion method, melt quench method, evaporation method, sputtering method, pulse laser deposition method, spin coating method and spray pyrolysis method. Each synthesis method has its unique procedure as well as merits and demerits. The solid state reaction route is one of the simplest synthesis method, which requires heating of the materials which were grounded for homogeneous mixing of the various oxide ingredients. The bulk and thin films at low reaction temperatures were prepared by sol-gel synthesis route which provides the high purity products. Co-precipitation method provides homogenous particle size which is a very energy efficient method. Thus, the synthesis method is an essential factor for materials science and nanotechnology research. The present article is an attempt to review the synthesis methods and their merits or demerits.

## KEYWORDS

Materials science, nanotechnology, synthesis methods, merits and demerits of synthesis methods.

## INTRODUCTION

Materials science is an interdisciplinary field of research which involves the design and discovery of materials. The researchers of chemistry, physics and engineering sciences are engaged in the analytical study of materials from ancient times and at the same time their phenomenological studies are carried out in metallurgy and mineralogy. In recent years, after the origin of nanotechnology, the research on materials opens a new scope for researchers and scientists who are working in this field [1-3]. Nanotechnology is an emerging field of research and innovation which is mainly focused on materials and devices for enrichment of properties of the host materials. With the use of this technology, materials may become useful for industrial purposes. Materials engineered via nanotechnology are known as nanomaterials and exhibit

unique electrical, optical, and magnetic properties. These properties also have great importance in electronics and medicine. Researchers are focused on nanomaterials due to controlled synthesis and its property optimization in recent years. The large surface area of nanomaterials form their bulk state makes them potential materials for advanced applications according to current demands. Nanostructured material's discovery involves synthesis methods and characterization technological developments [4-10].

Several exercises have been made by experimenters to optimize the materials for new properties and scopes. During the journey of research in materials science, numerous methods were designed for achieving the required results and goals. Each synthesis method has its unique way of working and procedure. In addition to the procedure, each method has different classes of merits and

demerits. The synthesis methods are mainly classified in top-down and bottom-up approaches. The top-down approach is an attempt to find specific results starting with general matter or bulk. The top-down approach involves the breaking down of large pieces of material to generate the required nanostructures. On the other hand, the bottom-up approach is used to find the results by accumulating a large number of particles into larger nanostructures [11-26]. Furthermore, the synthesis methods are classified in bulk and thin film fabrication. The bulk synthesis methods are mainly solid state reaction route [27-29], hydrothermal method [30,31], co-precipitation method [32,33], melt quench method, sol-gel method and auto combustion method [34,35] whereas thin film preparation methods are divided in two major classes, physical vapor and chemical vapor deposition methods [36]. Thermal evaporation, sputtering and pulse laser deposition methods are main synthesis routes of physical vapor deposition method. On the other hand, sol-gel synthesis route, spray pyrolysis and spin coating methods are main synthesis routes of chemical vapor deposition [38]. The homogeneous mixture of oxides ingredients of raw materials was made by grinding in acetone media and the resultant mixture was heated in a furnace for calcinations. This process was repeated several times to achieve a good homogeneous mixture which is required for solid state reaction methods [39,40]. Reverse drop co-precipitation method needs higher amount of chemicals and consequently requires longer preparation time for preparation of composite nano particles whereas high calcination temperature and a long soaking time are main requirements of synthesis nanoparticles by hydrothermal and sol-gel method requires [41]. The auto combustion route produces a homogenous microstructure in shorter reaction times at lower temperatures. The films prepared by evaporation method have good purity with high rate of deposition but the film composition is not easily controlled by this method. The thickness of the film and their distribution are well controlled by the sputtering

method. The grain structure is found to be better in the sputtering method compared to the evaporation method. The deposition rate of the film is slower by the sputtering method. The pulse laser deposition method finds the excellent importance for controlled crystallization and good stereometry. Moreover, the multilayer growth in pulse laser deposition method is also possible. The spin coating method is one of the chemical vapor deposition methods which provide thin films of uniform thickness and precise control over the thickness of film, whereas the packing density is found to be better in the spray pyrolysis method. The low yield of the film produced by this technology is its main disadvantage [42-105]. Gautam *et. al.* have synthesised numerous glassy and dental materials by using melt – quench method, microwave synthesis method and heat cure method. Particularly, the heat cure method is extensively used for synthesis of dental materials [106-108].

## DISCUSSION OF SYNTHESIS METHODS

### Solid State Reaction Method

One of simplest synthesis techniques in bulk materials research is the solid state reaction route. This method involves non-volatile solids for decomposition by heating. The ingredients are initially ground by mortar and pestle for uniform mixing. The well-mixed ingredients are transferred in either alumina boat or crucible and then placed inside the muffle furnace to provide heat treatment for chemical reactions at high temperatures. During heat treatment, the volatile products like CO<sub>2</sub>, NH<sub>3</sub>, NO<sub>2</sub>, and O<sub>2</sub> are removed and only the oxide forms of the product remain. The final heat treated product is further grounded in agate mortar and pestle for homogeneous mixing. This process also further minimizes the grain size of crystals. The complete process of synthesis by solid state reaction method is shown in the schematic diagram of Fig. 1.



Fig. 1. Schematic diagram of solid state reaction method.

This method has several demerits includes that it requires high power to produce high temperature. The decomposition rate is also very slow and sometimes crystallization does not take place at one step, then repetition is required for crystallization. In actuality, the reaction takes place simultaneously over a period of days and hours at high temperatures (500–2000°C) [42-48].

### Hydrothermal method

Hydrothermal synthesis is a derived geology in the twelfth century by George W. Morey. It consists of two terms, hydro and thermal. Thus, in this method, the crystallization of aqueous solutions occurs at high temperature and high pressure. The single crystals are mainly synthesized by this method using an autoclave (an apparatus consisting of a steel pressure vessel). It is basically a solution reaction-based approach. Between the two ends of the growing chamber, a temperature gradient is kept constant. At the hotter end, the nutrient solute disintegrates, while at the cooler end, it is deposited on a seed crystal, causing it to grow into the desired crystal. The hydrothermal synthesis method is shown in the schematic diagram of Fig. 2 [109]. The nanomaterials were synthesized in a wide range of temperatures by this method. This method plays a significant role in the synthesis of crystals which are not stable at the melting point. Moreover, the crystals can be prepared which have high vapor pressure near the melting points. Hydrothermal method is particularly suitable for the development of high-quality crystals while preserving control over their makeup. Vapor pressure has the ability to influence the shape of materials as well. This method requires the minimum amount of raw ingredients and consequently a minimum loss of material is observed. The pricey autoclave device, safety concerns while the reaction is occurring, and the impossibility of watching the reaction process are the main drawbacks of this approach. It has necessity of high calcination temperature and prolonged soaking time [30,31].

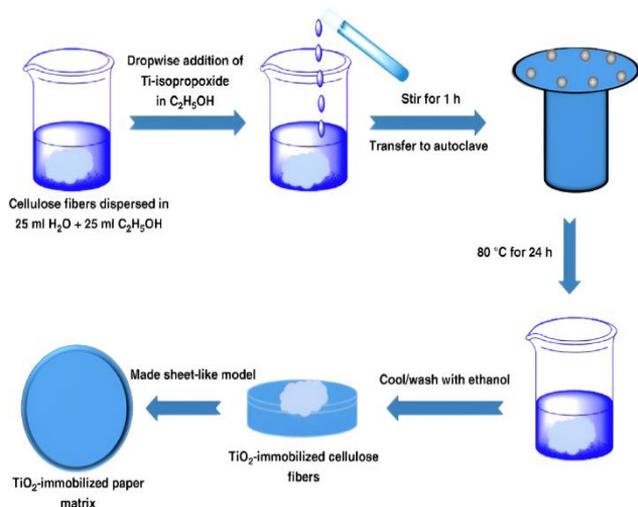
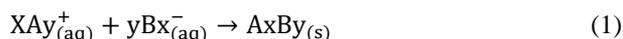


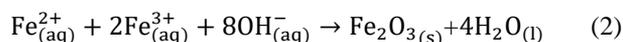
Fig. 2. Schematic diagram of hydrothermal synthesis method [109].

### Co-precipitation method

It entails the co-occurrence of the processes of nucleation, growth, coarsening, and agglomeration. In this procedure, soluble metal salts were used to create the stoichiometric solution, which was then predicated as hydroxide, oxalate, etc. The end product is produced by filtering, drying, and heating the resulting combination. The precursor is then heated to cause it to break down into the desired product. The homogenous products in this process are produced at relatively low temperatures, although occasionally a suitable precursor cannot be found. The super saturation conditions necessary to induce precipitation are usually the result of a chemical reaction as given in Equation 1.



For example, the synthesis of magnetite by using the aqueous mixture of ferrous and ferric solution in the form of hydrated chloride salts in alkaline medium, in 1/2 ratio. Initially, 10 mL of the HCl 2 mol·L<sup>-1</sup> was added to prevent Fe<sup>2+</sup> oxidation and precipitation Fe<sup>3+</sup> in the form of hydroxide. Their chemical reaction for the synthesis of magnetite can be understood by equation 2 [110].



The synthesis procedure of co-precipitation method is shown in Fig. 3 [111]. This preparation method is relatively quick and easy to use, allowing for simple modification of the particle surface state and overall homogeneity by controlling particle size and composition. This technique of synthesis uses no organic solvents and is exceedingly energy-efficient. It has a number of significant limitations, including the inability to utilise on uncharged species and the precipitation of trace contaminants in the final product. If the reactants precipitate at highly different rates, the process takes a long time and does not operate properly. More chemicals are needed, and preparation takes longer time to complete the synthesis process [62-66].

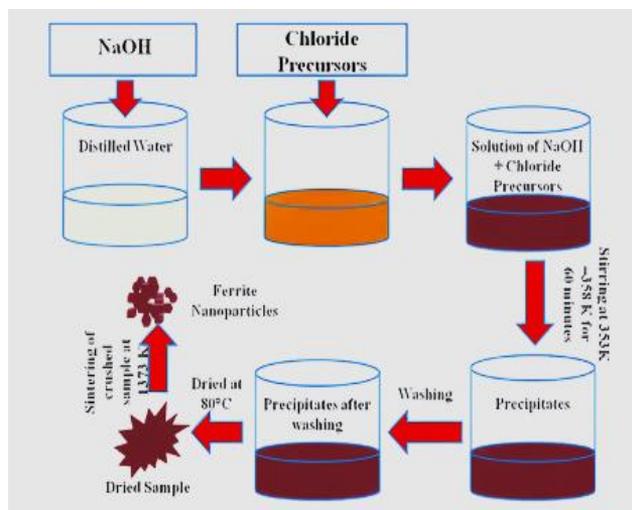
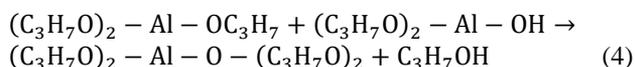
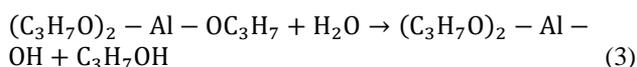


Fig. 3. Schematic diagram of co-precipitation method [111].

### Sol-Gel method

The sol-gel process is a wet chemical method which is used for the preparation of nanostructures of various metal oxides in both bulk and thin film research. The molecular precursor of especially metal alkoxide is dissolved in water or alcohol in this method. The precursor is converted into a gel by suitable heating and stirring by using a magnetic stirrer on a hot plate. During the conversion of the precursor to gel, hydrolysis/alcoholysis occurs. After the hydrolysis process, the wet gel is converted into drying either by burning of alcohol or other methods depending on the desired properties and application of the gel. The dried gel is powdered and finally calcined for the decomposition and removal of the organic species. The sol-gel method is described in the schematic diagram of Fig. 4 [112].

Alkoxide and water react to create hydroxy metal alkoxide, which then reacts with metal alkoxide to cause gelation through a condensation process. The reactions are shown in equations 3 and 4.



Due to the low reaction temperature, the sol-gel method is a practical way to synthesise substances and has considerable control over the material composition of the end products. This method is used to prepare the high purity products. Ceramics and thin films of metal oxides, which have important uses in sensors and solid state fuel cells, are also processed using this technique [48-52].

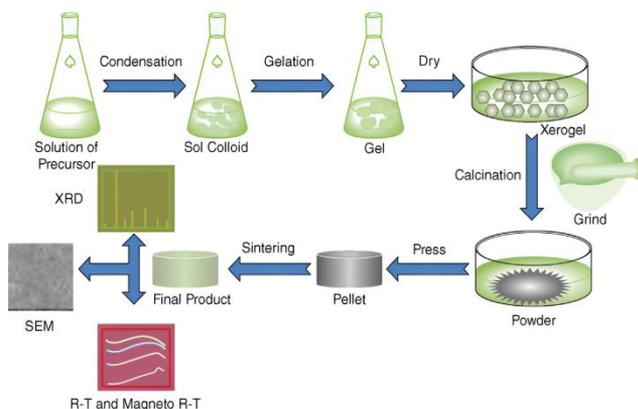


Fig. 4. Schematic diagram of Sol-Gel method [109].

### Melt-Quench method

Glasses are specifically made using the melt quench process. According to the composition of glasses, the right proportions of reagent and chemical raw materials were weighed out appropriately, combined with acetone medium in an agate mortar, and dried in this technique. An alumina or platinum crucible is filled with thoroughly combined and dried powder, and the crucible is then placed within a high

temperature programmable furnace. The furnace is set to a temperature that will cause components to melt. For homogenization and refinement, the combined powder of materials is melted and kept at melting temperature in the furnace for 10 to 30 minutes.

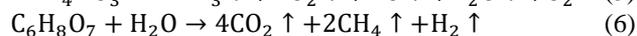
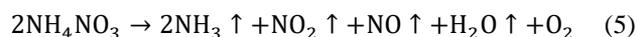


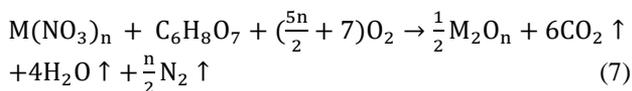
Fig. 5. Schematic diagram of melt quench method.

The melt was poured into an aluminum/copper mould and compressed into a thick aluminium plate before being immediately transferred to a preheated programmable muffle furnace for annealing at a temperature 1/3 of the melting temperature for 3-6 hours to eliminate any remaining stresses caused by the temperature gradient during rapid cooling. After annealing, the glasses were cooled within the furnace. The schematic diagram of the melt quench process for creating glass is shown in Figure 5. This technique's key advantage is that it is relatively straightforward and inexpensive. Although this approach is widely available commercially, its homogeneity and reproducibility are quite poor. Power usage is really high. Another significant drawback of this technology is the requirement for two furnaces [67-70, 108].

### Auto Combustion Method

Combustion synthesis, commonly known as self-propagating high temperature synthesis (SHS), is a successful, affordable method for creating very pure and uniform nanocrystalline powders. It is a wet chemical technique that is comparable to the Pechini and sol-gel techniques. By using a sol-gel process, an aqueous solution of the necessary metal salts and organic fuel is created in the form of a gel. The gel is then burned to cause it to burn, producing a fluffy product with a lot of volume and surface area. Citric acid is utilised in this procedure as a complexant to create a homogenous precursor called xerogel. Figure 6 shows the schematic diagram of the processing of the sample by auto combustion method [113]. The chemical reactions occurs in the auto combustion method are shown in the given equations [114].





Because the salts used are economical and there is no expensive machinery utilised in the processing of materials, it is a quick and easy procedure. The main drawback is that determining the ideal fuel to oxidizer (F/O) ratio is not always simple. A product with an undesirable composition is frequently produced when the amount of fuel for combustion is estimated strictly according to stoichiometric F/O [53-60].

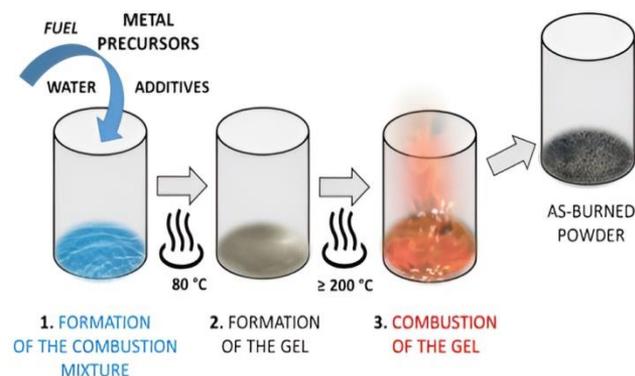


Fig. 6. Schematic diagram of auto combustion method [113].

## PHYSICAL VAPOUR DEPOSITION METHOD

Physical vapour deposition (PVD) is a method of fabricating new materials that involves depositing a thin layer of material on a substrate. There are various processes in the preparation of thin films. The first material is prepared in accordance with the thin film's preferred method of preparation. The substance to be deposited is then physically transformed into vapour and transferred from its source via a low-pressure area to the substrate. To create the thin film, the vapour is finally condensed on the substrate. The films made by PVD method are sometimes harder and more corrosion resistant than other coating methods. This method can be employed with both inorganic and organic coating materials on diverse groups of substrates. The line-of sight transfer is typical for PVD methods. This method requires high temperatures and vacuum for its operation, thus, it is not easy for operating personnel. Moreover, a water cooling system dissipates large heats. There are three techniques used for PVD. First technique is thermal evaporation, the second one is sputtering technique, and the last one is pulse laser deposition.

### Thermal Evaporation Method

The thermal evaporation method is a reasonably simple and popular technique for forming thin films of materials. This technique involves using a resistive heat source to

evaporate a solid substance in a dry atmosphere. The ingredients for which a film needs to be made are heated in a high vacuum chamber till vapour is produced. The evaporated material or vapor steam traverses the vacuum chamber from  $10^{-6}$  to  $10^{-7}$  torr with thermal energy and coats the substrate. Because of the high vacuum, there is no chance of colliding with or being interfered with by other gas molecules or atoms as they are being carried to the substrate.

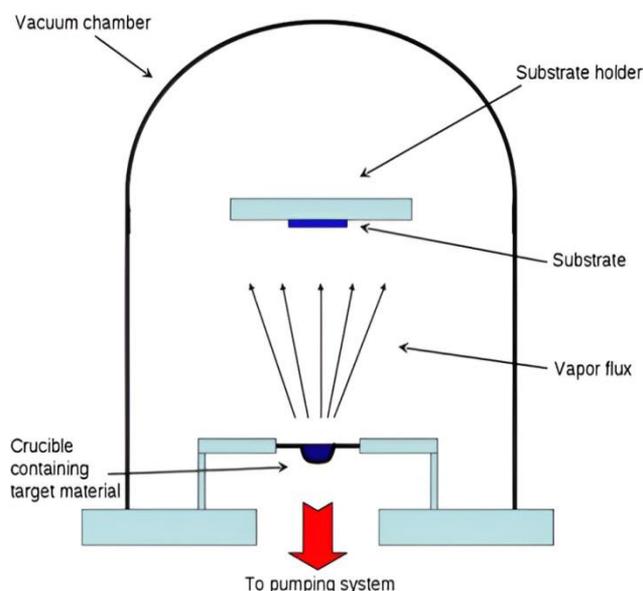


Fig. 7. Schematic diagram of thermal evaporation method [115].

A tungsten filament (resistive evaporation) or an electron beam is then used to heat the crucible (e- beam evaporation). In order to evaporate a substance, radio frequency (RF) energy can be used to warm the source material. A crucible containing the source is encircled by an RF induction heating coil in this method. It is called inductive heating evaporation to refer to this process.

The schematic diagram in Fig. 7 describes the thin film preparation by thermal evaporation method [115-116]. High film deposition rates, less substrate surface damage from impinging atoms during film formation, good film purity due to the high vacuum conditions utilised during evaporation, and a reduced tendency for inadvertent substrate heating are all benefits of the evaporation process. In contrast to these benefits, a fundamental disadvantage of this technology is that it is more difficult to manage the film composition and cannot perform in situ substrate surface cleaning [38,71,72].

### Sputtering Method

Sputtering is a frequently used process for creating thin films. In this procedure, high-energy particles collide with a material's surface to eject atoms or molecules, which then condense as a thin film on the substrate. Surface atoms are physically ejected off a solid surface through momentum transfer caused by an energetic bombardment particle with

an atomic size, which is a nonthermal vaporisation process. Generally speaking, compared to vacuum deposition, the source-to-substrate distance is small. Using an ion cannon or low pressure plasma (5 mTorr), high-energy ions are shot at a solid surface (the sputtering target) in a vacuum, causing the sputtered particles to collide with the substrate very infrequently or not at all. One of the most popular methods for depositing different metallic films, such as those made of aluminium, aluminium alloys, platinum, gold, and tungsten, as well as insulating and semiconductor films like CuO, ZnO, and SnO<sub>2</sub>, on wafers is RF or reactive sputtering. The schematic diagram for the sputtering process is shown in Fig. 8 [116-117]. This method has several benefits, including the ability to sputter from large-size targets, the simplification of the deposition of thin films with uniform thickness over large wafers, and the ease with which the film thickness can be adjusted by simply adjusting the deposition time and operating parameters. Evaporative deposition makes it easier to control the composition of the material as well as other film characteristics including step coverage and grain structure. The rates of oxide deposition with this method are not very high, and it is not cost-effective. Ionic bombardment can easily destroy some substances, such as organic solids [38, 73-88].

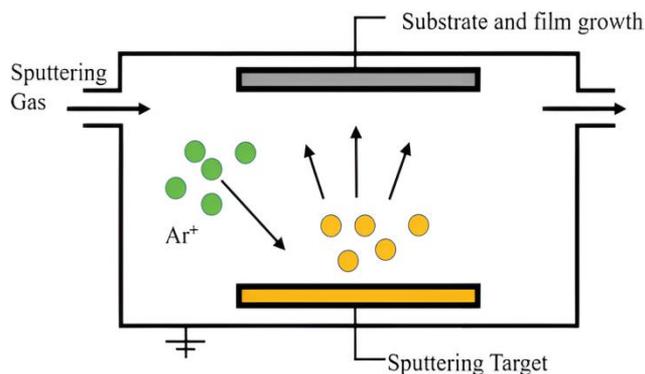


Fig. 8. Schematic diagram of sputtering method [113].

### Pulsed Laser Deposition Method

Turner and Smith were the first to use a high energy pulsed laser as an external power source to ablate the source or target material in order to demonstrate pulsed laser deposition (PLD), a form of PVD technology. The chosen material is vaporised using the laser in this instance. A brief but intense interaction between the laser pulse and the material leads to ablation through a series of intricate events. In the PLD setup, an external vacuum chamber is used to house a high energy pulsed laser, either a Nd:YAG or UV excimer laser with an intensity of 10<sup>8</sup>–10<sup>9</sup> W/cm<sup>2</sup>. The target surface of the laser beam is its focal point, and a small volume of air is where it is absorbed. A high pressure gas is created in the surface layer because the energy density of the absorbed radiation is sufficient to dissolve any chemical bond between the molecules. A supersonic jet is ejected normally toward the target surface as a result of

the pressure gradient. The laser beam's energy is heavily absorbed by the particle cloud, causing a hot plasma expansion in the deposition chamber. After a few hundred or thousands of laser pulses, the ablated species condense on the substrate and create a thin film.

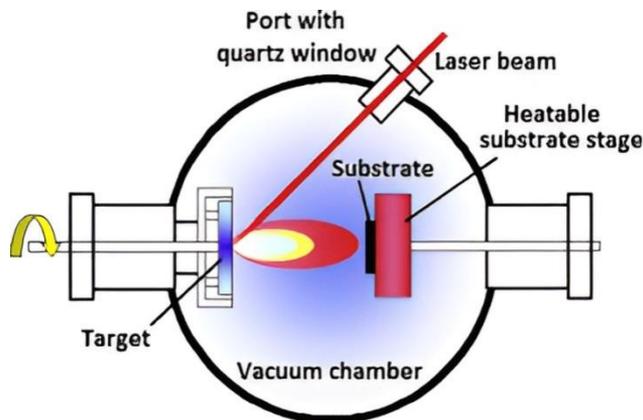


Fig. 9. Schematic diagram of pulse laser deposition method [117].

When the technique is done correctly, the film develops epitaxially and has a stoichiometry that is identical to the target. By rotating the target, rastering the beam, or using a combination of the two, deep surface pitting brought on by repeated ablation is prevented. In order for the substrate to barely touch the ablation plume's tip, the substrate and target are positioned just a few millimeters apart. The pulse laser deposition process is illustrated schematically in Fig. 9 [117]. Stoichiometry maintenance, multilayer development, and crystallinity control are all benefits of this method [38,116].

### CHEMICAL VAPOUR DEPOSITION

A substance or compound from the gas phase condenses on the surface of a substrate during a chemical reaction that creates a solid thin film deposit, which is known as a chemical vapour deposition (CVD) process. The production of thin films and coatings using chemical vapour deposition techniques are finding significant use in a variety of fields, including the construction of solid-state electronic devices, micro-electromechanical system devices, ball bearings, cutting tools, and nuclear reactor components. Chemical deposition techniques include sol-gel, dip-and-spin coating, spray pyrolysis, etc. [97-100].

Chemical vapour deposition (CVD) technique is defined as a technique in which a compound or compound from the gas phase condenses on a substrate surface where a chemical reaction occurs, which leads to the production of a solid thin film deposit. Chemical vapour deposition processes for producing thin films and coatings have found increasing applications in such diverse technologies as the fabrication of solid-state electronic devices, micro-electromechanical system devices, the manufacture of ball bearings, cutting tools and nuclear reactor components.

Sol-gel, dip and spin coating, spray pyrolysis, etc. are chemical deposition techniques [97-100].

### Spin Coating Method

Spin coating is a method to fabricate thin films of uniform thickness around 1-10  $\mu\text{m}$  by using centrifugal force which requires a liquid-vapor interface. A liquid is often positioned in the centre of a circular surface and quickly spun to create homogeneous films. To create thin films using this approach, there are four phases. The substrate is wetted consistently in the first stage by pouring a regulated amount of the precursor over it. If necessary, the precursor can additionally be cleaned of bigger particles using a sub-micron filter. The substrate is spun at the proper rotational speed in the second stage to remove the extra fluid. While the substrate rotates more quickly, the top of the fluid layer exerts inertia. Spiral vortices may arise as a result of the twisting motion produced by these two forces. However, in typical circumstances, the precursor is sufficiently thin to continue spinning in conjunction with the substrate and no indication of a thickness differential is present. In the end, the substrate moves at the correct speed and the rotational acceleration precisely cancels out the viscous shear drag. The thinning behaviour of the fluid is dominated by viscous forces in the third stage. The development of homogenous films is influenced by the fluid thinning. However, edge effects are occasionally noticed as well. The whirling fluid moves uniformly outside. If there is too much fluid present, drops must form at the borders so that they can fly off. As a result, there may be an edge effect since the thickness at the edges of the substrate may be slightly greater than at its centre. The fluid begins to evaporate in the fourth and final stage, controlling its own thinning tendency. In this process, the solvent phase is eliminated, and the sol is transformed into a dense ceramic. The fluid evaporates as a result of its temperature rising as a result of the fast rotation. As a result, the leftover solution becomes more viscous, which causes the coating to "gel." Typically, the final movie has an amorphous form. Despite the fact that the third and fourth stages, i.e., viscous flow and evaporation, happen simultaneously, the viscous flow effect predominates sooner rather than later [116]. The spin coating method's processing is shown in Fig. 10 [118].

To preserve its viscosity, the sol needs to be stored in an airtight flask; otherwise, it turns into gel and cannot be utilised for film deposition.

The viscosity of the coating solution, rotating speed, annealing temperature, and time length are the elements that impact film thickness. When heated, gel-coated films become porous and sintered. The main benefits of this technology are the uniformity of the thin film and the exact control over film thickness. The spin coating process for creating thin films has a low operational cost. This approach allows for the very quick fabrication of the films.

The drawback is that big area samples are challenging. The high-speed spinning becomes challenging as the substrate or film gets larger, which makes it challenging to

thin the film. The spin coating process has a relatively low material efficiency. Only 2%–5% of the material is often dispensed onto the substrate for the manufacture of thin films, whereas 95%–98% of the material is typically thrown off and disposed of during the process [38, 89–96].

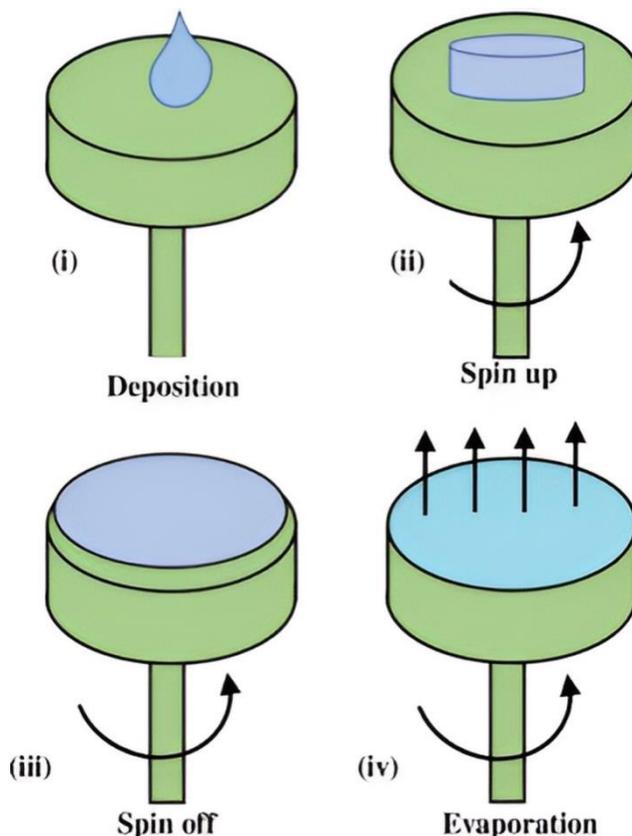


Fig. 10. Schematic diagram of spin coating method [118].

### Spray Pyrolysis Method

A cluster of liquid or vapour atoms from various chemical species engage in a thermally sparked reaction in the process known as spray pyrolysis. The spray method entails misting a solution (often aqueous) containing salts of the atoms that make up the desired molecule onto a substrate that is kept at a high temperature. As soon as the sprayed droplets touch the hot substrate, they undergo pyrolytic breakdown, which results in the formation of the product as single crystals or crystallites. In the vapour phase, excess solvent, other volatile byproducts, and other byproducts escape. The hot substrates provide the thermal energy necessary for deposition, species recombination that follows, and crystallisation of the crystallites again. The spray nozzle uses a carrier gas that may or may not be involved in the pyrolytic reaction to optimise the chemical solution into a spray of tiny droplets. By scanning either or both the spray head and the substrate using electromechanical systems, as indicated in the schematic figure of Fig. 11 [119], a large area homogeneous coverage of the substrate is accomplished. The chemicals used for

spray pyrolysis must meet the following requirements: the desired thin film material must be produced by a thermally activated reaction between the different species/complexes dissolved in spray solutions, and the remaining chemical components, including the carrier liquid, must be volatile at the pyrolysis temperature. The aforementioned conditions can be satisfied for a given thin film material by a variety of chemical mixtures, and depending on the deposition parameters, the structure and morphology of the films can change significantly [120].

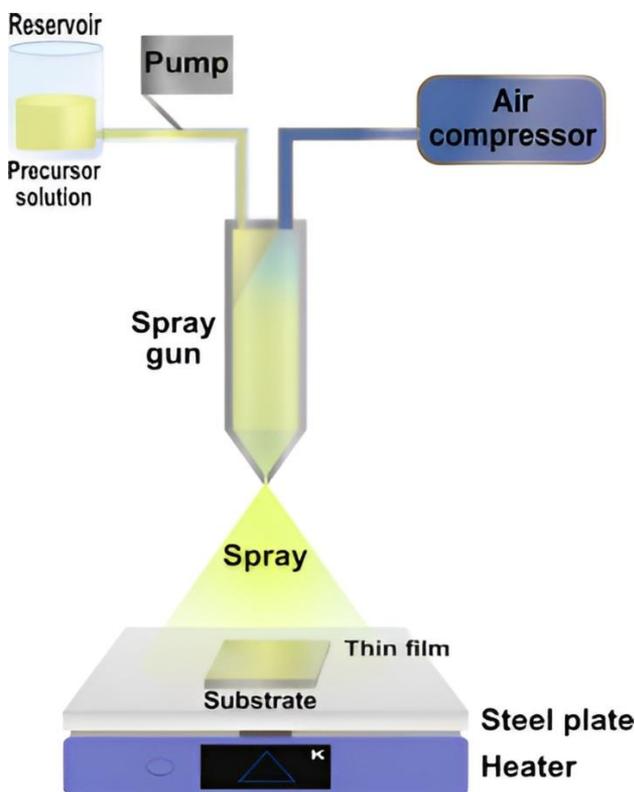


Fig. 11. Schematic diagram of Spray Pyrolysis Method [119].

Both chlorides and nitrates can be used to create oxide films. Thus, deposits of  $\text{In}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{SnO}_2$ , and  $\text{ZnO}$  are possible. Oxide films have also been produced using organometallic compounds. Their low deposition temperature is a benefit. The substrate's characteristics, the chemical make-up and concentration of the spray solution, as well as its additives and spray parameters, all affect how quickly the films grow when subjected to spray pyrolysis. In general, the spray-deposited films are robust and adherent, mechanically tough, pinhole-free, and temperature- and time-dependently stable. The films' topography is typically rough and is determined by the spray circumstances. Deposits range from amorphous to polycrystalline depending on the ingredients' chemical reactivities and droplet mobility. By using the spray pyrolysis process, high density packed porosity and uniform thickness films are created. The film produced with

this technology has a low yield. This procedure is found to improve the thin film's shape, it has been seen [38,101–103].

## CONCLUSION

Materials science is a diverse field of experimental research. The materials are mainly classified in four categories: metals, polymers, semiconductors, and ceramics. Modern society is heavily dependent on advanced materials, for example, lightweight composites for faster vehicles, optical fibres for telecommunications, and silicon microchips for the information revolution. Moreover, materials science is vital for developments in nanotechnology, quantum computing, batteries, and nuclear fusion, as well as medical technologies such as bone replacement materials. The sources, processing, and fabrication of these materials are different according to the use, requirements, and applications. Thus, the fabrication of materials plays a significant role in the development of any device. A detailed study of synthesis methods in experimental materials science and nanotechnology is reviewed in the present article. Top down and bottom-up approaches are used for the synthesis of nanomaterials. Top approach starts from the big structure of the material to attain the smaller structure of nano the range whereas the bottom up approach deals with starting from the smallest structure to attain the required material. These two approaches use several experimental methods for the synthesis of bulk as well as thin film materials. The simplest method of synthesis of bulk materials is the solid state reaction route. This method requires heating and grinding for homogeneous mixing of the various oxide materials. In addition to this, high temperature is necessary in the solid state reaction route. Hydrothermal method is one of the significant methods for the synthesis of nanomaterials from room temperature to very high temperatures. The crystals, which are not stable at the melting point, are prepared by hydrothermal method easily. This method requires expensive autoclave instruments. Co-precipitation method provides homogenous particle size, it is very energy efficient because it does not use organic solvent. This method is not useful for uncharged species. It is time consuming and does not work well when the reactants have very different precipitation rates. The sol-gel method is a method which is used to prepare bulk and thin films at low reaction temperatures. The samples prepared by sol-gel method are high purity products. The melt quench method is particularly used in the synthesis of glasses. The auto-combustion method is a fast and simple processing method, but it is not always easy to define the optimal F/O ratio in an auto combustion method.

The thin film preparation methods have mainly physical vapor deposition and chemical vapor deposition. These two methods use several synthesis routes and each method has certain merits and demerits. The evaporation method has a rate of high film deposition and more difficult

to control of film composition as well as the absence of the capability to do in situ cleaning of substrate surfaces. Sputtering technique provides the deposition of thin films with uniform thickness over large wafers. It has step coverage and well grain structure compared to evaporation. The last PVD method, PLD, has the advantages of good crystalline control, multilayer growth, and maintenance of stoichiometry. The spin coating method gives the control over the thickness of the film by maintaining the viscosity. Sometimes the sol gets converted into gel and cannot be used for the deposition of films. The spray pyrolysis methods were used to develop strong and adherent, mechanically hard, pinhole free, and stable films with time and temperature.

## ACKNOWLEDGEMENTS

The author gratefully acknowledges the Government of Uttar Pradesh for providing financial support under 'Research and Development Scheme' wide letter no. 46/2021/603/Seventy-4-2021-4(56)/2020. The author is also thankful to the publishers Elsevier, Springer Nature, IOP Science for permitting the reproduction of cited contributions for academic purposes. The author acknowledges the authors of the cited articles.

## CONFLICTS OF INTEREST

The authors declared no potential conflicts of interest.

## REFERENCES

- Asim, N.; Sopian, K.; Ahmadi, S.; Saeedfar, K.; Alghoul, M.A.; Saadatian, O.; Zaid, S. H., *Renewable and Sustainable Energy Reviews*, **2012**, 16, 5834–5847.
- Bhadeshia, H. K. D. H., *ISIJ International*, **1999**, 39 966-979.
- Weller, H., *Advanced Materials*, **1993**, 5, 88-95.
- Hulla, J. E.; Sahu S. C.; Hayes, A. W., *Human and Experimental Toxicology*, **2015**, 34, 1318–1321.
- Sanchez, F.; Sobolev, K., *Construction and Building Materials*, **2010**, 24, 2060–2071.
- Khan, I.; Saeed, K.; Khan, I., *Arabian Journal of Chemistry*, **2019**, 12, 908-931.
- Roco, M. C., *Journal of Nanoparticle Research*, **1999**, 1, 1–6.
- Altug, H.; Oh, S. H.; Maier, S. A.; et al., *Nat. Nanotechnol.* **2022**, 17, 5–16.
- Stater, E.P., Sonay, A.Y., Hart, C.; et al., *Nat. Nanotechnol.* **2021**, 16, 1180–1194.
- Heinrich, A.J., Oliver, W.D., Vandersypen, L.M.K.; et al., *Nat. Nanotechnol.* **2021**, 16, 1318–1329.
- Isaacoff B. P.; Brown, K. A., *Nano Lett.*, **2017**, 17, 6508–6510.
- Liu, Y.; Mai, S.; Li, N.; Yiu, C. K.; Mao, J.; Pashley, D. H.; Tay, F. R., *Acta biomaterialia*, **2011**, 7, 1742–1751.
- Kumar, S.; Bhushan, P.; Bhattacharya, S., Fabrication of Nanostructures with Bottom-up Approach and Their Utility in Diagnostics, Therapeutics, and Others. In: Bhattacharya S., Agarwal A., Chanda N., Pandey A., Sen A. (eds) Environmental, Chemical and Medical Sensors. Energy, Environment, and Sustainability. Springer, Singapore, **2018**.
- Escudero, A.; Carrillo-Carrión, C.; Romero-Ben, E.; Franco, A.; Rosales-Barrios, C.; Carmen Castillejos M.; Khiar, N., *Inorganics*, **2021**, 9, 58.
- Natsuki, J.; Natsuki, T.; Hashimoto, Y., *International Journal of Materials Science and Applications*, **2015**, 4, 325-332.
- Abbasi, E.; Milani, M.; Aval, S. F.; Kouhi, M.; Akbarzadeh, A.; Nasrabadi, H. T.; Nikasa, P.; Joo, S. W.; Hanifepour, Y.; Koshki K. N.; Samiei, M., *Crit Rev Microbiol.* **2016**, 42, 173-80.
- Cele, T., Preparation of Nanoparticles". Engineered Nanomaterials - Health and Safety, edited by Sorin Avramescu et al, IntechOpen, 2020. 10.5772/intechopen.90771.
- Iravani, S.; Korbekandi, H.; Mirmohammadi, S.V.; Zolfaghari, B., *Res Pharm Sci.*, **2014**, 9, 385-406.
- Patil, N.; Bhaskar, R.; Vyavhare, V.; Dhadge, R.; Khaire, V.; Patil, Y., *Int J Curr Pharm Res*, **2021**, 13, 11-16.
- Ealias A. M., Saravana kumar, M. P., *Materials Science and Engineering*, **2017**, 263, 032019.
- Ijaz, I.; Gilani, E.; Nazir A.; Bukhari, A., *Green Chemistry Letters and Reviews*, **2020**, 13, 223-245.
- Rafique, M.; Sadaf, I.; Rafique M. S.; Tahir, M. B., *Nanomedicine, and Biotechnology*, **2017**, 45, 1272-1291.
- Zahoor, M.; Nazir, N.; Iftikhar, M.; Naz, S.; Zekker, I.; Burlakovs, J.; Uddin, F.; Kamran, A. W.; Kallistova, A.; Pimenov, N., et al., *Water*, **2021**, 13, 2216.
- Niculescu, A. G.; Chircov, C.; Grumezescu, A. M., *Methods*, **2021**, <https://doi.org/10.1016/j.ymeth.2021.04.018>,
- Amina, S. J.; Guo, B., *Int J Nanomedicine*, **2020**, 15, 9823-9857.
- Kumar, P.; Mahajan, P.; Kaur, R.; Gautam, S., *Materials Today Chemistry*, **2020**, 17, 100332.
- Noah, N. M., *Journal of Nanomaterials*, **2020**, 2020, 1-20.
- Mai, A.; Haanappel, V.A.C.; Uhlenbruck, S.; Tietz, F.; Stöver, D., *Solid State Ionics*, **2005**, 176, 1341–1350.
- Selvaduray G.; Sheet, L., *Materials Science and Technology*, **1993**, 9, 463-473.
- Ghorbani, H. R., *Orient J Chem*, **2014**, 30, 1941-1949.
- Komarneni, S.; Fregeau, E.; Breval, E.; Roy, R.; *Journal of the American Ceramic Society*, **1988**, 71, 26-28.
- Tavakoli, A.; Sohrabi M.; Kargari, A., *Chem. Pap.*, **2007**, 61, 151–170.
- Pandya, N.; Kulkarni, R. G.; Parsania, P. H.; *Materials Research Bulletin*, **1990**, 25, 1073-1077.
- Li, X.; Zhang H. B.; Zhao, M. Y., *Materials Chemistry and Physics*, **1994**, 37, 132-135.
- Deganello, F.; Marci, G.; Deganello, G., *Journal of the European Ceramic Society*, **2009**, 29, 439–450.
- Wang, N.; Fuh, J.Y.H.; Dheen, S.T.; et al. *Bio-des. Manuf.*, **2021**, 4, 379–404.
- Shoukat, R.; Khan, M.I., *Microsyst Technol*, **2021**, 27, 4183–4192.
- Swihart, M. T., *Current Opinion in Colloid & Interface Science*, **2003**, 8, 127-133.
- Ravindranathan, P.; Komarneni, S.; Roy, R., *Journal of Materials Science Letters*, **1993**, 12, 369-371.
- Agnoli, S.; Favaro, M.; *J. Mater. Chem. A*, **2016**, 4, 5002-5025.
- Sivakumar, M.; Gedanken, A.; Zhong, W.; Jiang, Y.H.; Du, Y.W.; Brukental, I.; Bhattacharya, D.; Yeshurunc, Y.; Nowik, I., *Journal of Materials Chemistry*, **2004**, 14, 764–769.
- Ikesue, A.; Furusato, I.; Kamata, K., Editor, Greskovich, C., *Journal of American ceramic society*, **1995**, 78, 225-228.
- Zhu Y.; Zhou, Y., *Applied Physics A*, **2008**, 92, 275–278.
- Elilarassi, R.; Chandrasekaran, G., *J Mater Sci: Mater Electron*, **2010**, 21, 1168–1173.
- Dao-hua, L.; Shao-fen, H.; Jie, C.; Cheng-yan, J.; Cheng, Y., *Materials Science and Engineering*, **2017**, 242, 012023.
- Quiroz, A.; Chavira, E.; Espinosa, J.; Palomino-Merino, R.; Marinero, E.; Nishioka M.; Garcia-Vázquez, V., *Materials Sciences and Applications*, **2015**, 6, 16-22.
- Basyrova, L. R.; Maksimov, R. N.; Shitov, V. A.; Aleksandrov, E. O., *AIP Conference Proceedings*, **2017**, 1886, 020003.
- Ahmed, H.; Khan, S.; Khan, W.; Naseem S.; Nongjai, R., *Philosophical Magazine Letters*, **2018**, 98, 365-374.
- Hayat, K.; Gondal, M.A.; Khaled, M. M.; Ahmed, S.; Shemi, A. M., *Applied Catalysis A: General*, **2011**, 393, 122–129.
- Abdullah, K. A.; Awad, S.; Zaraket, J.; Salame, C., *Energy Procedia*, **2017**, 119, 565-570.
- Aziz, M.; Abbas, S. S.; Rosemaria, W.; Baharom, W., *Materials Letters*, **2013**, 91, 31-34.
- Liu, T.; Xu, Y., *Materials Chemistry and Physics*, **2011**, 129, 1047-1050.
- Theingi, M.; Tun, K. T.; Aung, N. N., *SciMedicine Journal*, **2019**, 1, 151-157.
- Toksha, B.G.; Shirsath, S.E.; Patange, S.M.; Jadhav, K. M., *Solid State Communications*, **2008**, 147, 479-483.

55. Hankare, P. P.; Sanadi, K. R.; Garadkar, K. M.; Patil, D. R.; Mulla, I. S.; *Journal of Alloys and Compounds*, **2013**, 553, 383-388.
56. Kadam, R. H.; Biradar, A. R.; Mane M. L.; Shirsath, S.E., *Journal of Applied Physics*, **2012**, 112, 043902.
57. Zhang, J.; Gao, L., *Materials Chemistry and Physics*, **2004**, 87, 10-13.
58. Mehnane, H. F.; Wang, C.; Kondamareddy, K. K.; Yu, W.; Sun, W.; Liu, H.; Bai, S.; Liu, W.; Guo S.; Zhao, X. Z., *RSC Adv.*, **2017**, 7, 2358-2364.
59. Iwasaki, T.; Mizutani, N.; Watano, S.; Yanagida T.; Kawai, T., *Journal of Experimental Nanoscience*, **2012**, 7, 355-365.
60. Verma, S.; Joy, P.A.; Khollam, Y.B.; Potdar, H.S.; Deshpande, S.B., *Materials Letters*, **2004**, 58, 1092- 1095.
61. Machmudah, S.; Widiyastuti, W.; Prastuti, O. P.; Nurtono, T.; Winardi, S.; Wahyudiono; Kanda H.; Goto, M., *AIP Conf. Proc.*, **2014**, 1586, 166-172.
62. LaGrow, A. P.; Besenhard, M. O.; Hodzic, A.; Sergides, A.; Bogart, L. K.; Gavriilidis, A.; Thanh, N. T. K.; *Nanoscale*, **2019**, 11, 6620-6628.
63. Ahn, T.; Kim, J.H.; Yang, H.M.; Lee J.W.; Kim, J.D., *J. Phys. Chem. C*, **2012**, 116, 6069.
64. Sagadevan, S.; Chowdhury, Z.Z.; Rafique, R.F. *Method, Mat. Res.*, **2018**, 21, 1-5.
65. Tinwala, H.; Shah, D.V.; Menghani, J.; Pati, R., *J. Nanosci. Nanotechnol.*, **2014**, 14, 6072-6
66. Saravanan, T.; Raj, S. G.; Chandar, N. R.; Jayavel, R., *J Nanosci Nanotechnol.*, **2015**, 15, 4353-7.
67. Yadav, A.K.; Singh, P., *J Mater Sci: Mater Electron*, **2015**, 26, 9443-9450.
68. Yadav, A.K.; Gautam, C. R.; Singh, P., *RSC Adv.*, **2015**, 5, 2819-2826.
69. Sindhu, S.; Sanghi, S.; Agarwal, A.; Sonam; Seth, V.P.; Kishore, N., *Physica B: Condensed Matter*, **2005**, 365, 65-75.
70. Alderman, O. L. G.; Hannon, A. C.; Feller, S.; Beanland R.; Holland, D., *J. Phys. Chem. C*, **2017**, 121, 9462-9479.
71. Pan, C.A.; Ma, T.P., *J. Electron. Mater.*, **1981**, 10, 43.
72. Jayaraj, M. K.; Draeseke, A.D., Tate J., Sleight, A.W., *Thin Solid Films*, **2001**, 397, 244.
73. De A.; Ray, S., *J. Phys. D: Appl. Phys.*, **1991**, 24, 719.
74. Schiller, S.; Beister, G.; Buedke, E.; Becker H.J.; Schicht, H.; *Thin Solid Films*, **1982**, 96, 113-120.
75. Miyata, N.; Miyake, K.; Koga K.; Fukushima, T., *J. Electrochem. Soc.*, **1980**, 127, 918.
76. Budzyńska, K.; Leja, E.; Skrzypek, S., *Sol. Energy Mater.*, **1985**, 12, 57.
77. Minami, T.; Kakumu, T.; Shimokawa K.; Takata, S., *Thin Solid Films*, **1998**, 317, 318.
78. Phillips, J.M.; Kwo, J.; Thomas, G.A.; Carter, S.A.; Cava, R.J.; Hou, S.Y.; Krajewski, J.J.; Marshall, J.H.; Peck, W.F.; Rapkine D.H.; van Dover, R.D., *Appl. Phys. Lett.*, **1994**, 65, 115.
79. Sreenivas, K.; Sudersena Rao T.; Mansingh, A., *J. Appl. Phys.*, **1985**, 57, 384.
80. Geoffroy, C.; Campet, G.; Portier, J.; Salar-denne, J.; Couturier, G.; Bourrel, M.; Chabagno, J.M.; Ferry D.; Quet, C., *Thin Solid Films*, **1991**, 202, 77.
81. Miao, W.-N.; Li, X.-f.; Zhang, Q.; Huang, L.; Zhang, Z.-j.; Zhang L.; Yan, X.-j., *Thin Solid Films*, **2006**, 500, 70.
82. Webb, J.B.; Williams D.F.; Buchanan, M.; *Appl. Phys. Lett.*, **1981**, 39, 640.
83. Ghosh, S.; Sarkar, A.; Bhattacharya, S.; Chaudhuri S.; Pal, A.K.; *J. Cryst. Growth*, **1991**, 108, 534.
84. Choi, B.H.; Im, H.B.; Song J.S.; Yoon, K.H., *Thin Solid Films*, **1990**, 193-194, 712.
85. Sarkar, A.; Ghosh, S.; Chaudhuri S.; Pal, A.K., *Thin Solid Films*, **1991**, 204, 255.
86. Minami, T.; Sonohara, H.; Takata S.; Sato, H., *Jpn. J. Appl. Phys.*, **1994**, 33, 1693.
87. Tate, J.; Jayaraj, M.K.; Draeseke, A.D.; Ulbrich, T.; Sleight, A.W.; Vanaja, K.A.; Nagarajan, R.; Wager J.F.; Hoffman, R.L., *Thin Solid Films*, **2002**, 411, 119.
88. Kim, K.K.; Kim, H.S.; Hwang, D.K.; Lim J.H.; Park, S.J., *Appl. Phys. Lett*, **2003**, 83, 63.
89. Adurodija, F.O.; Izumi, H.; Ishihara, T.; Yoshioka H.; Motoyama, M., *J. Vac. Sci. Technol. A*, **2000**, 18, 814.
90. Suzuki, A.; Matsushita, T.; Aoki, T.; Mori A.; Okuda, M., *Thin Solid Films*, **2002**, 411, 23.
91. Kim, H.; Pique, U.A.; Horwitz, J.S.; Murata, H.; Kafafi, Z.H.; Gilmore, C.M.; Chrisey, D.B., *Thin Solid Films*, **2000**, 377-378, 798.
92. Naghavi, N.; Rougier, A.; Marcel, C.; Guéry, C.; Leriche J.B.; Tarascon, J.M., *Thin Solid Films*, **2000**, 360, 233.
93. Yanagi, H.; Inoue, S-i; Ueda, K.; Kawazoe, H.; Hosono H.; Hamada, N., *J. Appl. Phys.*, **2000**, 88, 4159.
94. Kudo, A.; Yanagi, H.; Hosono H.; Kawazoe, H., *Appl. Phys. Lett.*, **1998**, 73, 220.
95. Ueda, K.; Hase, T.; Yanagi, H.; Kawazoe, H.; Hosono, H.; Ohta, H.; Orita, M.; Hirano, M., *J. Appl. Phys.*, **2001**, 89, 1790.
96. Guo, X.L.; Tabata, H.; Kawai, T., *Opt. Mater.*, **2002**, 19, 229.
97. Maruyama T.; Fukui, K., *Jpn. J. Appl. Phys.*, **1990**, 29, L1705.
98. Maruyama, T.; Fukui, K., *J. Appl. Phys.*, **1991**, 70, 3848.
99. Hu, J.; Gordon, R.G., *J. Appl. Phys.*, **1991**, 71, 880.
100. Gong, H.; Wang, Y.; Luo, Y., *Appl. Phys. Lett.*, **2000**, 76, 3959.
101. Shanthe, E.; Banerjee, A.; Dutta, V.; Chopra, K.L., *J. Appl. Phys.*, **1982**, 53, 1615.
102. Kaneko, H.; Miyake, K., *J. Appl. Phys.*, **1982**, 53, 3629.
103. Afify, H.H.; Momtaz, R.S.; Badawy, W.A.; Nasser, S.A., *J. Mater. Sci.: Mater. Electron.*, **1991**, 2, 40.
104. Biswas, P.K.; De, A.; Pramanik, N.C.; Chakraborty, P.K.; Ortner, K.; Hock, V.; Korder, S., *Mater. Lett.*, **2003**, 57, 2326.
105. Thakur, P.; Chahar, D.; Taneja, S.; Bhalla, N.; Thakur, A., *Ceramics International*, **2020**, 46, 10, 5740-15763.
106. Kumari, S.; Hussain, A.; Avinashi, S.K.; Mishra, R.K.; Rao, J.; Behera, S.; Gautam, R.K.; Gautam, C.R.; *Ceramics International*, **2023**, 49, 31412-31427.
107. Kumari, S.; Hussain, A.; Rao, J.; Singh, K.; Avinashi, S.K.; Gautam, C.R.; *Materials Chemistry and Physics*, **2023**, 295, 127089.
108. Gautam, C.; Madheshiya, A.; *J Mater Sci: Mater Electron*, **2020**, 31, 12004-12025.
109. Tatarchuk, T.; Peter, A.; Najar, B. A.; Vijaya, J.; Bououdina, M., Photocatalysis: Activity of Nanomaterials, Book Editor(s): Hussain, C. M.; Mishra, A.K., **2018**, 209-292, Wiley-VCH Verlag, Weinheim, Germany.
110. Petermele, W. S.; Fuentes, V. M.; Fascinelli, M. L.; Silva, J. R. d.; Silva, R. C.; Lucci, C. M.; Azevedo, R. B. D., *Journal of Nanomaterials*, **2014**, 2014, 1-10.
111. Thakur, P.; Sharma, R.; Kumar, M.; Katyal, S. C.; Negi, N. S.; Thakur, N.; Sharma V. and Sharma, P., *Mater. Res. Express*, **2016**, 3, 075001.
112. Tatarchuk T. ; Peter, A.; Najar B. A.; Vijaya J.; Bououdina, M. Photocatalysis: Activity of Nanomaterials. Wiley, USA, **2018**, 10.1002/9783527808854.ch8.
113. Deganello, F.; *Mater. Today*, **2017**, 4, 5507-5516.
114. Niu, B.; Zhang, F.; Ping, H. et al., *Sci Rep*, **2017**, 7, 3421.
115. Martín-Palma, R.J.; Lakhtakia, A. Vapor-deposition techniques, in: *Engineered Biomimicry*, Elsevier BV, **2013**, 383-398.
116. Shukla, R.K.; Pandey, M. Ceramic-conducting polymer composites for sensing applications, Elsevier BV, **2022**, 455-524.
117. Shi, F. Introductory Chapter: Basic Theory of Magnetron Sputtering, **2018**, [Online First], IntechOpen, DOI: 10.5772/intechopen.80550. Available from: <https://www.intechopen.com/online-first/63559>.
118. Bellardita, M.; Paola, A. D.; Yurdakal, S.; Palmisano, L. Preparation of Catalysts and Photocatalysts Used for Similar Processes, Heterogeneous Photocatalysis, Relationships with Heterogeneous Catalysis and Perspectives, Elsevier, **2019**, 25-56.
119. Sami, B.; Abdullah, Y.; Sharafi, A.; Ali, H. Self-Cleaning of Surfaces and Water Droplet Mobility, Elsevier, **2019**, 45-98.
120. Gorup, L. F.; Amorin, L. H.; Camargo, E. R.; Fernando, T. S.; Cincotto, H.; Biasotto, G.; Ramesar, N.; Porta, F. D. A. L. Methods for design and fabrication of nanosensors: the case of ZnO-based nanosensor, *Nanosensors for Smart Cities Micro and Nano Technologies*, Elsevier, **2020**, 9-30.

### AUTHORS BIOGRAPHY



**Dr. Avadhesh Kumar Yadav** serves as Assistant Professor in Dept. of Physics, F.A.A. Govt. PG College, Mahmudabad Sitapur. Dr. Yadav has been actively involved in Materials Science, Nanotechnology and glass/glass ceramics research for high energy storage and sensing devices since 2010. He has published more than 50 research articles in journal of international repute including 4 review articles and 5 books. The citation of his research articles are more than 1970 since November 2023. He is also editor/associate editor of Frontiers of Public Health, Current Chinese Science, Coroviruses, Ceramic science and engineering. He has reviewed research articles for more than 80 international journals of Elsevier, Springer, Wiley, Taylor & Francis, Nature, ACS, RSC etc.



This article is licensed under a Creative Commons Attribution 4.0 International License, which allows for use, sharing, adaptation, distribution, and reproduction in any medium or format, as long as appropriate credit is given to the original author(s) and the source, a link to the Creative Commons license is provided, and changes are indicated. Unless otherwise indicated in a credit line to the materials, the images or other third-party materials in this article are included in the article's Creative Commons license. If the materials are not covered by the Creative Commons license and your intended use is not permitted by statutory regulation or exceeds the permitted use, you must seek permission from the copyright holder directly.

Visit <http://creativecommons.org/licenses/by/4.0/> to view a copy of this license

### GRAPHICAL ABSTRACT

In graphical abstract the synthesis methods used in materials and preapration nanomaterials are given with their simple merit and demerit . .

