3 Synthesis of Nanoparticles by Physical Route

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Abstract

Development of nanoscience has evoked new technologies both in sample preparation and device fabrication. Synthesis and development of nanoparticles that are synonyms to quantum-confined atom is an important milestone in this pursuit. In recent years, a significant development with advanced improvements has been been made in the synthesis methods of nanomaterials. The nanoparticles can be prepared by using two well-known approaches, i.e., top-down approach and bottom-up approach. This chapter gives an overview of the various physical routes of synthesis of nanoparticles. Various methods of preparing nanomaterials including mechanical milling, sputtering, laser pyrolysis, laser ablation, electron beam evaporation, and nanolithography are discussed in this chapter. As a summary, this chapter describes the main physical routes for nanoparticle synthesis.

Keywords

Physical synthesis route · Laser ablation · Mechanical milling · Sputtering · Lithography

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3.1 Introduction

There are numerous techniques available for synthesis of nanomaterials which can be in the form of powders, tubes, wires, rods, spheres, or thin films. Nanomaterials can be synthesized by either physical, chemical, biological, or hybrid routes. These different routes yield a different variety of nanomaterials which may be dependent on the route, material used, catalyst, enzyme, starting chemicals, and many more. In this chapter, we have discussed some of the physical routes which are available to synthesize nanoparticles for commercial scale production and have been optimized parametrically for synthesis of nanomaterials.

3.2 Approaches for Synthesis of Nanomaterials

Basically, the synthesis of nanomaterials can be done using two main approaches: top-down and bottom-up (Fig. 3.1).

Top-down approaches: The bulk materials are processed to produce nanomaterials of desired parameters in top-down approach. The physical methods which include top-down approach are physical vapor deposition, mechanical milling, electrospinning, sputtering, laser ablation, electro-explosion, lithography (e-beam), arc discharge, and thermal evaporation.

Bottom-up approaches: The building up of a material from the bottom, i.e., atom by atom, molecule by molecule, or cluster by cluster. The physical methods

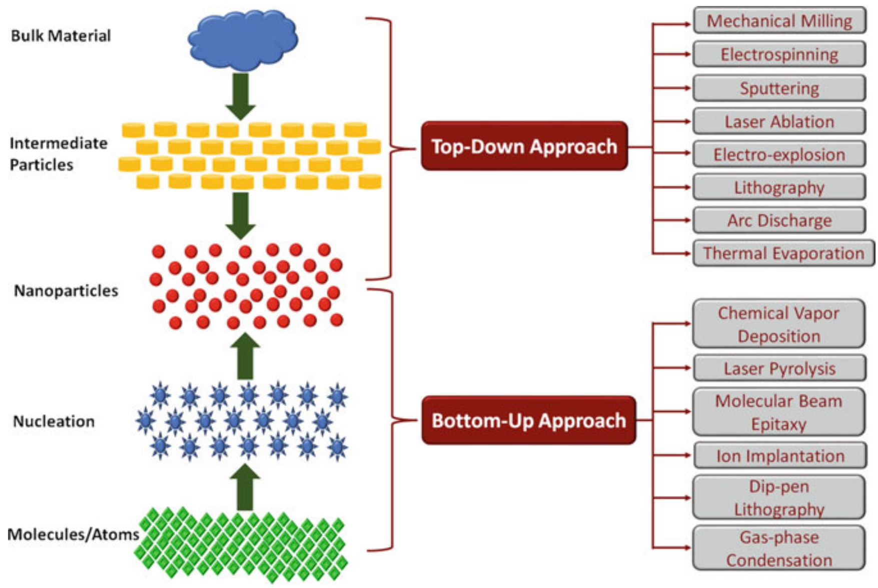


Fig. 3.1 The two approaches, i.e., top-down and bottom-up approaches, for synthesis of nanoparticles

which include bottom-up approach are chemical vapor deposition, laser pyrolysis, molecular beam epitaxy, ion implantation, dip-pen lithography, and gas phase condensation.

3.2.1 Physical Vapor Deposition

Physical vapor deposition (PVD) is a commonly used technique for the fabrication of thin films and surface coatings. Physical vapor deposition is characterized by a process in which the material to be deposited is converted into vapor by high-temperature vacuum or gaseous plasma, and then transported to a region of low pressure from its source to the substrate where the vapor undergoes condensation on the substrate to form a thin film. PVD technique results in the formation of coatings with improved properties as compared to the substrate material. All types of inorganic materials and some types of organic materials can be used in PVD technique.

3.2.2 Mechanical Milling

Mechanical milling is one of the simplest and the most cost-effective method of producing nanoparticles of some metals and alloys from their bulk materials. The material development by high-energy ball milling of powders was first established by John Benjamin (1970) and his co-workers at the International Nickel Company in the late 1960s wherein complex oxide dispersion-strengthened (ODS) alloys for high-temperature structural applications were produced (Benjamin 1970). This mechanical alloying method produced fine, uniform dispersions of oxides of Al_2O_3, Y_2O_3, and ThO_2 in nickel-based superalloys. Mechanical milling technique involves placing a suitable powder charge (typically, a blend of elemental) in a high-energy mill, along with a suitable milling medium. The mechanical milling helps to reduce the particle size and also to achieve new phases. This method produces nanomaterials of different phases which makes it suitable for production of nanocomposites. There are different types of mills being used in this technique such as vibratory, planetary, rod, tumbler, etc. (Kulkarni 2015). The size of the container used for milling depends upon the quantity of sample to be prepared. The kinetics of mechanical milling depends on the energy transferred to the powder from the balls during milling which is governed by many parameters such as the type of milling (dry or wet), the powder supplied to drive the milling chamber, milling speed, temperature, and duration of milling. Either tungsten carbide or hardened steel balls are put in the container with the desired bulk material. The initial/bulk material being used can be of arbitrary size and shape. After putting the balls and the bulk material in the container, it is closed with the use of tight lids. The mass ratio of balls to material which is most suitable to get nanomaterials is 2:1, i.e., if the container is more than half filled, then the efficiency of the milling is reduced. Use of heavy milling balls increases the impact energy of collision, but at the same time the defect density also increases. The temperature during milling depends on

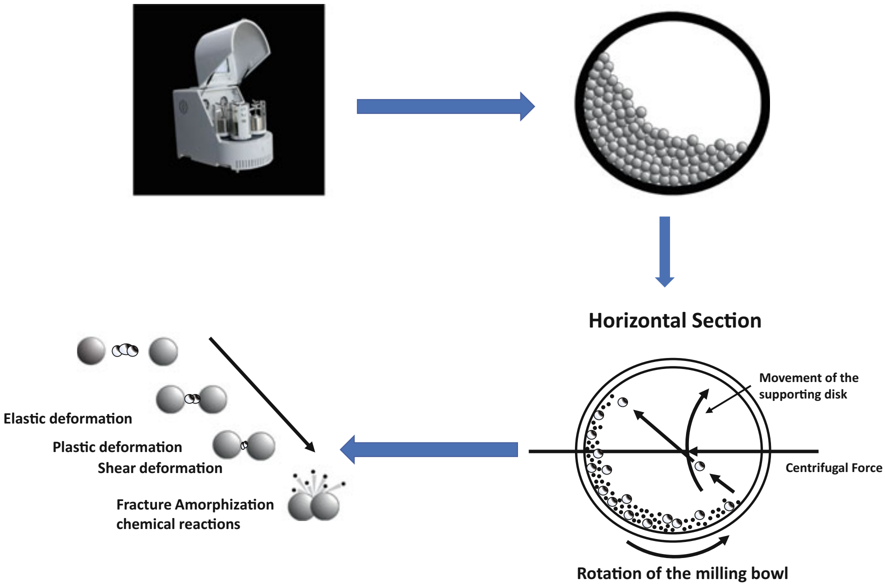


Fig. 3.2 The principle of the ball milling method. Reprinted with permission from ref. Zhuang et al. (2016). Copyright: ©2016, John Wiley & Sons, Ltd.

powder used, milling media, and kinetic energy of the ball. The temperature of the powder during milling influences the diffusivity and defect concentration in the powder inducing the phase transformations. Higher temperature is likely to result in intermetallic phases, while lower temperature results in the formation of amorphous phases. Sometimes, there are chances of addition of some kind of impurities from balls. The gases being used to make the inert atmosphere may also serve as impurities if they are not of high purity. The basic principle of ball milling is displayed in Fig. 3.2 (Zhuang et al. 2016). This technique is mainly used to produce oxide-like ferrites and carbide-strengthened aluminum alloys, wear-resistant spray coatings, aluminum-/nickel-/magnesium-/copper-based nano-alloys, and many other nanocomposite materials (Yadav et al. 2012). Carbon nanomaterials prepared through ball milling are considered to be a novel class of nanomaterials as they have varied applications in the fields of energy storage, energy conversion, and environmental remediation (Lyu et al. 2017; Kammakakam and Falath 2021). Different types of nanoparticles can be synthesized using ball milling like ZnO nanoparticles of size 5–110 nm which were prepared depending on different milling speed and milling time (Salah et al. 2011; Giri et al. 2007; Damonte et al. 2004). CuO nanoparticles of size 11–20 nm were synthesized from metallic powder of size 60 micrometer (Khayati et al. 2013; Yang and Chen 2017). TiO_2 nanoparticles in the range of 10–37 nm were prepared by ball milling (Khayati et al. 2013; Yang and Chen 2017; Carneiro et al. 2014; Salari et al. 2008; Yadav et al. 2015), and Ag_2O powder (5–40 μm) was ball-milled to nanoparticles of size 14 nm in 95 h (Khayati and Janghorban 2013).

3.2.3 Electrospinning

Electrospinning is another one of the simplest top-down methods for synthesis of nanomaterials which are usually fibers of a variety of materials which are mainly polymers (Ostermann et al. 2011). Development of coaxial spinning method was one of the breakthroughs in the electrospinning technique. Coaxial electrospinning is an effective technique for synthesis of core-shell nanofibers at a large scale. The length of the ultrathin nanofibers synthesized using this technique may go up to several centimeters. The spinneret in this technique has two coaxial capillaries, i.e., one of the capillaries with viscous liquid is used to generate shell, and the other capillary with nonviscous liquid is used to generate shell. A schematic diagram of coaxial electrospinning is shown in Fig. 3.3 (Du et al. 2012). This technique is highly helpful in development of core-shell as well as hollow organic, inorganic, polymeric, and hybrid nanomaterials (Kumar et al. 2014). Nanocomposite fibers by electrospinning can be divided into blending, post-modification, and posttreatment methods. In the blending method, metal nanoparticles are mixed with the polymer solution to form a uniform precursor solution. This mixed solution is directly electrospun and various materials interfaces are formed. The process is beneficial in terms of ease of preparation and high yield and therefore is widely used in preparation of fluorescent and electrochemical sensing interfaces. In post-modification, metal nanoparticles are adsorbed or modified onto the nanofibers to obtain the metal nanoparticle-based interface. Posttreatment method is conducted on the obtained metal nanoparticle-based interfaces, such as calcining to obtain the interface of the new structure.

3.2.4 Sputtering

Sputtering is a technique which is being widely used for producing thin films of nanomaterials (Kulkarni 2015; Ayyub et al. 2001). The main advantage of using this

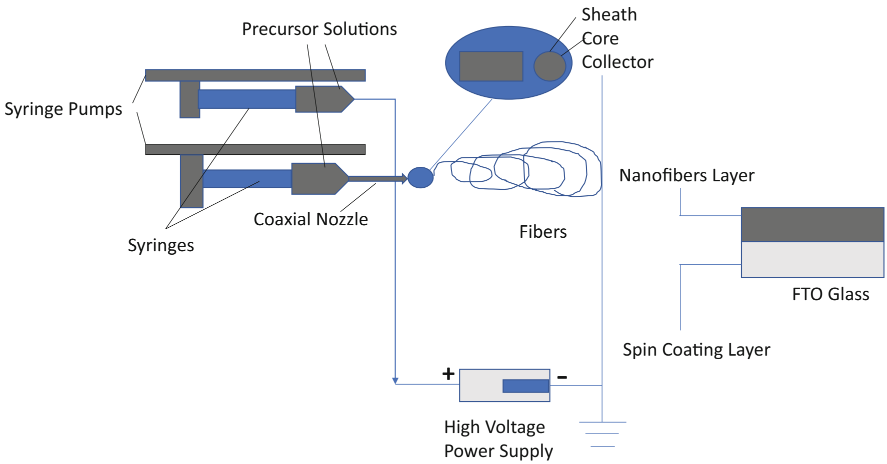


Fig. 3.3 A schematic diagram of coaxial electrospinning. Reprinted with permission from ref. Du et al. (2012). Copyright: ©2012, Elsevier Ltd. All rights reserved

technique is development of stoichiometric thin films which makes the technique more cost-effective. Thin films/nanoparticles are obtained by bombarding the solid surfaces by high-energy particles, i.e., gas and plasma. It is the phenomena of deposition of nanoparticles when highly energetic gaseous ions are bombarded on the target surface resulting in physical ejection of small atomic clusters (Son et al. 2017; Wender et al. 2013; Shah and Gavrin 2006; Bharti et al. 2020). It is generally carried out in an evacuated chamber, in which sputtering gas is introduced. The cathode (target) is supplied with a high voltage, and free electrons collide with the gas to produce gas ions. The positively charged ions are strongly accelerated in the electric field toward the target resulting in the ejection of atoms from the surface (Munoz-Garcıa et al. 2009; Nam et al. 2020). Some important factors which are responsible for determining the size and shape of the nanoparticles are substrate temperature, energy of particles, and annealing duration (Bharti et al. 2020). There can be many ways through which the high-energy particles can be bombarded on the substrate and depending on change of source material according to which it can be classified into magnetron, radio-frequency diode, and DC diode sputtering (Wender et al. 2013). Development of multilayer thin films/magnetic films for spintronic applications is also possible using this technique (Kulkarni 2015). A schematic representation of DC magnetron sputtering is shown in Fig. 3.4 (Son et al. 2017). Different nanoparticles of silver, gold, iron, ferrites, copper, copper oxide, and zinc oxide have been synthesized using sputtering (Asanithi et al. 2012; Hu et al. 2013; Xing et al. 2016; Peng et al. 2003; Gunnarsson et al. 2015; Jaiswal et al. 2015; Das et al. 2016; Rashid et al. 2015).

3.2.5 Laser Ablation

Laser ablation technique involves synthesis of nanoparticles by usage of a powerful laser beam which hits the target material and ablates the surface. The target/source/precursor material is vaporized due to the high energy of the laser irradiation which results in nanoparticle formation. The laser beam condenses a plasma which produces nanoparticles by irradiating the different metal substrates (Amendola and Meneghetti 2009). This technique is very useful for reduction of metal to nanoparticles. The stable nanoparticles are synthesized using laser ablation techniques and do not need any stabilizing agent or chemicals. Different types of nanoparticles of silver, gold, copper, copper oxide, tin oxide, etc. can be manufactured by using laser ablation (Maciulevičius et al. 2013; Wender et al. 2011; Al-Azawi and Bidin 2015; Khumaeni et al. 2017; Valverde-Alva et al. 2015; Tajdidzadeh et al. 2014; Pyatenko et al. 2004; Hajiesmaeilbaigi et al. 2005; Gondal et al. 2009; Al-Dahash et al. 2018; Mintcheva et al. 2018; Boutinguiza et al. 2013; Singh et al. 2016; Abdulateef et al. 2016; Gondal et al. 2013).

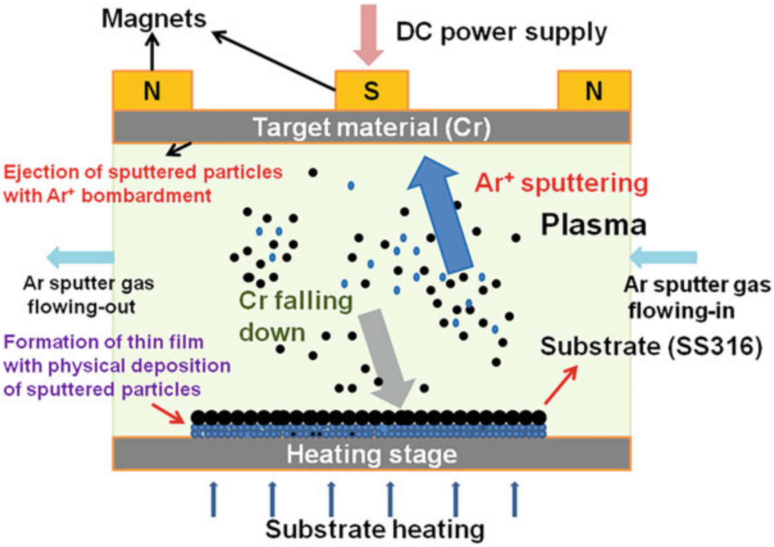


Fig. 3.4 A schematic diagram of the DC magnetron sputtering process. Reprinted with permission from ref. Son et al. (2017). Copyright: ©2017, Elsevier Ltd.

3.2.6 Electrical Explosion

Electrical explosion of metal wires is another upcoming technology for the synthesis of nanoparticles with increased activity. In this method, a high-density current pulse is passed through a metal wire resulting in heating up of the wire quickly. The wire explodes with the formation of explosion products, which form nanoparticles while passing a gas atmosphere. This technique results in the possibility of obtaining nanopowders of metals, alloys, and oxides and nitrides of metals (Lerner et al. 2016; Llyin et al. 2012). Also, this method shows stability in properties of nanopowders obtained with high activity in chemical processes. Ease of changes in process parameters, small-sized nanoparticles, and high energy efficiency of the process characterized by low energy losses for heating the environment are some of the other advantages of this method.

3.2.7 Lithography

This term lithography can be defined in two distinct ways. First is the general definition which is by Google "lithography," seven of the first ten hits will show the art world and a process invented by Aloys Senefelder in 1788 (Britannica Concise Encyclopedia 2005). And second is "the process of printing from a plane surface (as a smooth stone or metal plate) on which the image to be printed is ink-receptive and the blank area ink-repellent" given by the Merriam-Webster

Online Dictionary (Mollenstedt and Speidel 1960). The second definition gives a much clearer and boarder view of lithography. Lithography can be of many types depending on the source being used for imprinting, i.e., ion beam lithography, e-beam lithography, and X-ray lithography.

3.2.7.1 Ion Beam Lithography

In this process a beam of heavily charged and high momentum ions is used to fabricate a high-resolution pattern on the surface of substrate. It is therefore useful in developing integrated circuits of nanometer scale. The ion beam penetration power can be adjusted by fluctuation of the ion energy (Fig. 3.5).

3.2.7.2 Electron Beam Lithography

Electron beam lithography is used to generate patterns over a substrate using focused electron beam of small wavelength. This lithographic process comprises of three steps: exposure of the sensitive material, development of the resist, and pattern transfer. An electron-sensitive film or resist is placed on the sample; thereafter it is exposed by spin coating spun at 1000–6000 rpm to form a coating. The energy deposited during the exposure creates a latent image that materializes in the course of chemical development**.** When exposed to the electron beam, the resistance solubility changes to the point where it becomes selectively more soluble in the exposed part or in unexposed parts depending upon the type of resist. The resist can be removed by

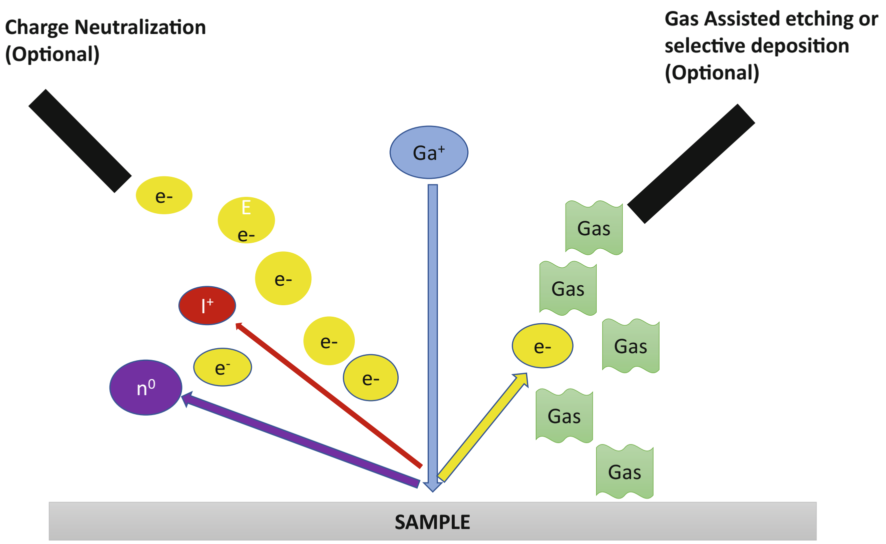


Fig. 3.5 Focused ion beam lithography. Reprinted with permission from ref. Sutter et al. (2008). Copyright: ©2017, Elsevier Ltd. License Number 5134251485537

immersing it in a solvent which is often termed as developing. The intensity and energy of the electron beam, thickness of the resist, and irradiation time are some of the important parameters to be taken into account. Etching (chemical removal of the layer from the surface of substrate) and liftoff (creating a photoresist profile that ensures separation between the thin film coating in desired and undesired areas of the pattern) are the main pattern transfer methods that can be applied. Polymethyl methacrylate (PMMA) is the commonly used resist material due to its advantages of high resolution and easy processing (Fig. 3.6).

3.2.7.3 X-Ray Lithography

X-ray lithography is a masked lithographic process used to transfer patterns from a mask to a resist on the surface of substrate by X-rays. X-ray lithography is a masked lithographic technique that uses X-rays to transfer patterns from a mask to a resist on a substrate's surface. The procedure begins with the application of a mask and resist to the wafer surface, followed by exposure. The photoresist is penetrated by X-rays that emanate from a synchrotron and are selectively passed or inhibited by the patterned mask absorber, depositing energy to expose the resist. In this procedure, gold is commonly utilized as an absorber. To generate nano-patterns or nanostructures on the substrate, the same etching and developing technique as photolithography is used (Betancourt et al. 2021; Sebastian et al. 2020) (Fig. 3.7).

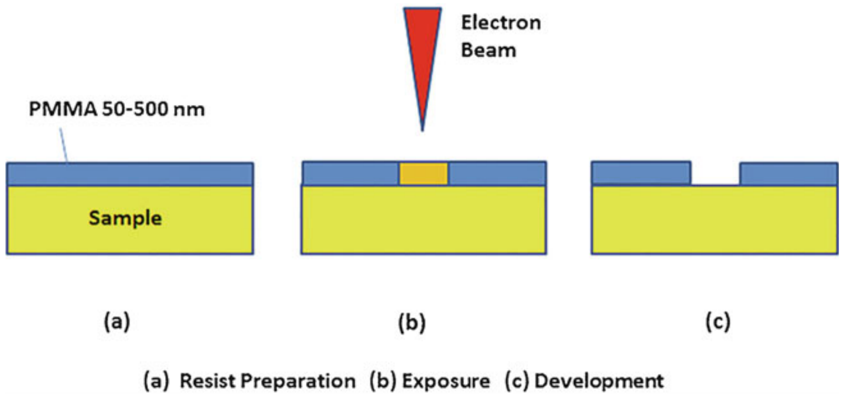


Fig. 3.6 Electron beam lithography. Reprinted with permission from ref. Llyin et al. (2012). Copyright: ©2017, Elsevier Ltd. License Number 5134251485537

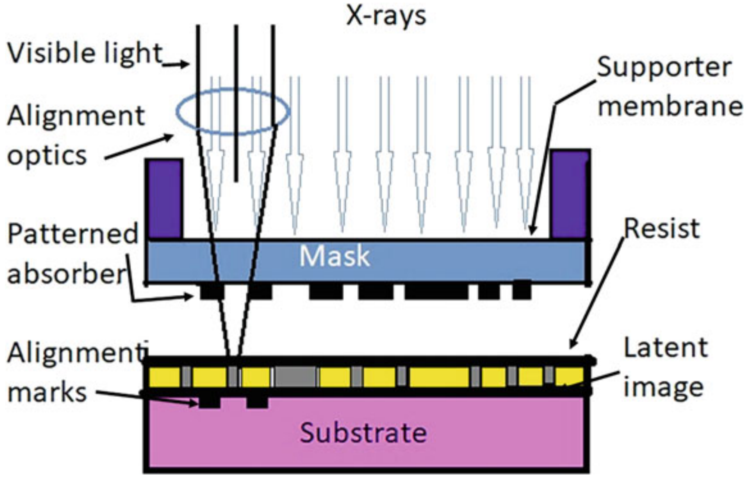


Fig. 3.7 Process of X-ray lithography. Reprinted with permission from ref. Sutter et al. (2008). Copyright: ©2017, Elsevier Ltd. License Number 5134251485537

Bottom-up approach: The basic molecular and atomic units are being used for synthesis of nanoparticles in bottom-up approach. The physical methods which include bottom-up approach are chemical vapor deposition, laser pyrolysis, molecular-beam epitaxy (MBE), ion implantation, dip pen lithography, and gas-phase condensation.

3.2.8 Laser Pyrolysis

Laser pyrolysis is one of the important, easy, and effective vapor phase nanopowder synthesis methods. In this technique, a laser beam is used selectively to heat a gas stream of nanoparticle precursors. The reaction between the laser beam and gaseous precursors will enhance the temperature, and hence the precursor gets decomposed, thereby inducing nucleation of nanoparticles. The powders are deposited where they will be obtained. One of the most important advantages of laser pyrolysis is its flexibility to synthesize nanoparticles of diverse materials using appropriate precursors of 15–20 nanometers at 100 g/h. Some other advantages of this technique are flow of reaction, high level of purity, and excellent chemical and physical properties. A study reported the laser pyrolysis synthesis of zinc sulfide and zinc oxide nanoparticles with an average diameter below 10 nm using a low-power CO_2 laser and controlling the operating parameters (Malekzadeh et al. 2020).

3.2.9 Chemical Vapor Deposition

Chemical vapor deposition (CVD) is a vacuum-based deposition process for producing high-quality, high-performance solid materials. CVD is a method for depositing nonvolatile solid thin films on substrates that involves chemical reactions between an organometallic or halide compound and other gases. CVD differs from PVD in that it uses a multidirectional deposition method to deposit material onto the substrate, whereas PVD uses a line-of-site impingement method. CVD is frequently used in microfabrication techniques to deposit materials in a variety of morphologies, such as monocrystalline, polycrystalline, amorphous, and epitaxial.

In contrast to PVD, in CVD, a mixture of gases interacts chemically with the bulk surface of the material, causing chemical breakdown of some of the specific gas elements and the formation of a solid coating on the base material's surface (Jones and Hitchman 2008; Shah and Tali 2016).

CVD is an important material preparation technology that is used to make precious metal thin films and coatings. A variety of CVD processes exist, including atmospheric pressure chemical vapor deposition (APCVD), low-pressure chemical vapor deposition (LPCVD), plasma-enhanced chemical vapor deposition (PECVD) or plasma-assisted chemical vapor deposition (PACVD), and laser-enhanced chemical vapor deposition (LECVD). Hybrid techniques, which combine physical and CVD properties, have also arisen (Fig. 3.8).

3.2.10 Molecular-Beam Epitaxy (MBE)

In thermal evaporation, the molecular-beam epitaxy (MBE) technique is the most reliable deposition process. A typical MBE system is depicted in Fig. 3.9. The system is a controlled MBE process in which a computerized process control unit controls the evaporation rate of the source materials in situ. Esaki has successfully deposited a man-made superlattice structure formed of thin alternating layers of GaAs and GaAlAs, as illustrated in Fig. 3.9 (Adachi and Wasa 2012).

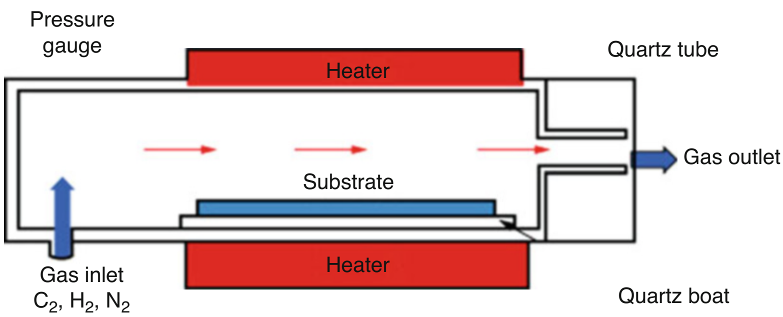


Fig. 3.8 CVD setup. Reprinted with permission from ref. Jones and Hitchman (2008). Copyright: ©2017

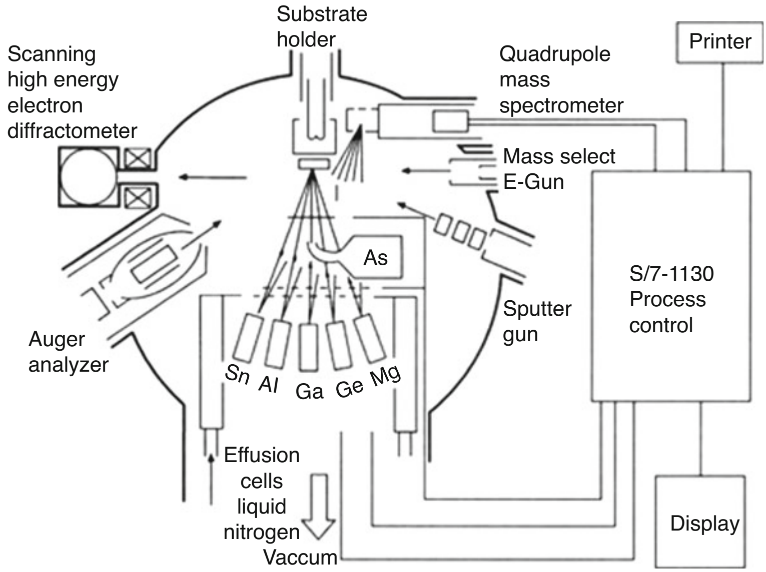
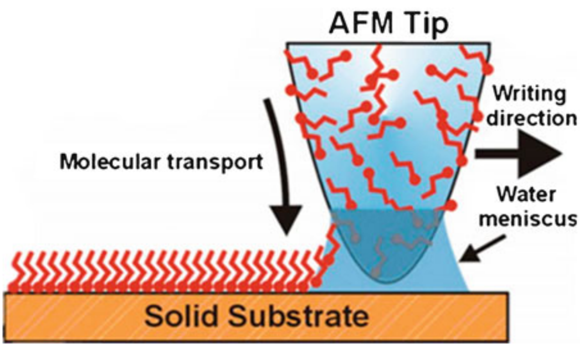


Fig. 3.9 A typical MBE system. Reprinted with permission from ref. Adachi and Wasa (2012). Copyright: ©2017

Fig. 3.10 Schematic representation of DPN. Reproduced with permission from ref. Piner et al. (1999). Copyright 1999 American Association for the Advancement of Science



3.2.11 Dip Pen Lithography

Figure 3.10 (Piner et al. 1999) depicts the invention of DPN, which was published in a Science article in 1999. The Mirkin group employed AFM tips to transport molecules (particularly, alkanethiols, ODT, and MHA) directly onto a gold thin film in a manner similar to a dip pen with 30 nm line width resolution. The "nib" in this case was an AFM tip, the "paper" was a solid-state substrate, and the molecules with a chemical affinity for the substrate were employed as "ink" to be directly written into the "paper" by the "nib." The feature size was dependent on the tip scanning speed and the ink transport rate. The meniscus and humidity were employed to aid molecule transport and build chemisorbed nanostructures on a surface. DPN uses a positive printing mode to easily deposit different types of molecules at particular spots without the use of a stamp, resist, or specialized tool.

3.3 Conclusion

A large number of nanoparticles and nanocomposites can be fabricated by using the methods and techniques described in this chapter. Different physical methods have been developed to obtain nanoparticles of various shapes and sizes, including mechanical milling, laser ablation, e-beam evaporation, electric deposition, and lithographic techniques. But to know the optimum conditions to improve the reproducibility and the quantity is an important parameter in nanoparticle synthesis.

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