IOSR Journal Of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 15, Issue 2 Ser. I (Mar. – Apr. 2023), PP 69-72
www.Iosrjournals.Org

Nanomaterials: Synthesis and Applications

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Abstract
Nanotechnology is a promising science with wide applications from cosmetics, food products, clothing, and household appliances to fuel catalyst, disease treatment, and renewable energies. Nanotechnology is also being applied to a variety of industrial and purification processes including construction materials, nanomachining of nanowires, nanorods, graphene, water filtration, and wastewater treatment.
Their applications are becoming wider in "nanomedicine" by interfacing the nanomaterials with biological molecules or structures, "green technology" to enhance the environmental sustainability and "renewable energy" to develop the new ways to capture, store, and transfer energy. For instance, carbon nanotube productions have been used for applications in energy storage, automotive parts,thin-film electronics, coatings, and so forth.
Nanomaterials are found as important and keep growing in the field of Nanoscience and Nanotechnology and in recent years researchers are investing much effort on the synthesis and applications of various nanomaterials, due to their potential applications in science and industry. For example, biocompatible nanomaterials are applied directly or they are used to replace natural materials tofunction or to be in contact with the living systems.
This special issue provides some new research and developments in nanomaterials. It presents the principles of the synthesis and fabrication of self- assembly nanostructures and their applications and also some important tools andchallenges associated with these techniques for engineers and scientists.

I. Introduction

There are a large number of techniques available to synthesize different types of nanomaterials in the form of colloids, clusters, powders, tubes, rods, wires, thin films etc. There are various physical, chemical, biological and hybrid techniques available to synthesize nanomaterials. The technique to be used depends upon the material of interest, type of nanostructure viz., zero dimensional, one dimensional, or two dimensional material size, quantity etc.

Physical methods

Ball milling: It is used in making of nanoparticles of some metals and alloys in the form of powder. Usually the mill contains one or more containers are used at a time to make fine particles. Size of container depends upon the quantity of interest. Hardened steel or tungsten carbide balls are put in containers along with powder or flakes (<50 um) of a material of interest. Initial material can be of arbitrary size and shape. Container is closed with tight lids. The containers are rotated at high speed(a few hundreds of rpm) around their own axis. Additionally they may rotate around some central axis and are therefore called as 'planetary ball mill'. When the containers are rotating around the central axis, the materialis forced to the walls and is pressed against the walls. But due to the motion of the containers around their own axis, the material is forced to other region of the container. By controlling the speed of rotation of the central axis and container as well as duration of milling, it is possible to ground the material to fine powder whose size can be quite uniform. Some of the materials like Co, Cr, W, Ni-Ti, AlFe, Ag-Fe etc. are made nanocrystalline using ball mill. Large balls, used for milling, produce smaller grain size and larger defects in the particles. The process may add some impurities from balls. The container may be filled with air or inert gas. However, this can be an additional source of impurity. A temperature rise in the range of100 to 1100 C is expected to take place during the collisions. Cryo-coolingis used to dissipate the generated heat.

Melt Mixing: It is possible to form or arrest the nanoparticles in glass. Structurally, glass is an amorphous solid, lacking long range periodic arrangement as well as symmetry arrangement of atoms/molecules. When a liquid is cooled below certain temperature, it forms either a crystalline or amorphous solid (glass). Nuclei are formed spontaneously with homogenous (in the melt) or inhomogeneous (on the surface of other materials) nucleation, which can grow to form ordered, crystalline solid. Usually, metals form crystalline solids but, if cooled at very high cooling rate, they can form amorphous solids. Such solids are known as metallic glasses. Even in such cases the atoms try to reorganize themselves into crystalline solids.

Addition of elements like B, P, Si etc. helps to keep the metallic glasses in amorphous state. It is possible to form nanocrystals within metallic glasses. It is also possible to form some nanoparticles by mixing the molten streams of metals at high velocity with turbulence. On mixing thoroughly, nanoparticles are formed.

Physical Vapor Deposition: It involves material for evaporation, an inert gas or reactive gas for collosion of material vapor, a cold finger on which clusters or nanoparticles can condense, a scraper to scrape the nanoparticles and piston- anvil (an arrangement in which nanoparticle powder can be compacted). All the processes are carried out in a vacuum chamber so that the desired purity of the end product can be obtained. Metals or high vapor pressure metal oxides are evaporated or sublimated from filaments or boats of refractory metals like W, Ta, Mo in which materials to be evaporated are held. Size, shape and even the phase of evaporated material can depend upon the gas pressure in deposition chamber. Clusters or nanoparticles condensed on the cold finger (water orliquid nitrogen cooled) can be scraped off inside the vacuum system. The process of evaporation and condensation can be repeated several times until enough quantity of material falls through a funnel in which a piston- anvil arrangement has been provided.

Ionized Cluster Beam Deposition: It is useful to obtain adherent and high quality single crystalline thin films. The set up consists of a source of evaporation, a nozzle through which material can expand into the chamber, an electron beam to ionize the clusters, an arrangement to accelerate the clusters and a substrate on which nanoparticle film can be deposited, all housed in a suitable vacuum chamber. Small clusters from molten materialare expanded through the fine nozzle. The vapor pressure, ~10 torr to 10-2torr needs to be created in the source and the nozzle needs to have a diameter larger than the mean free path of atoms or molecules in vapor form in the source to form the clusters. On collision with electron beam clusters get ionized. Due to applied accelerating voltage, the clusters are directed towards the substrate. By controlling the accelerating voltage, it is possible to control the energy with which the clusters hit the substrate. Thus it is possible to obtain the films of nanocrystalline material using ionized cluster beam.

Laser Vaporization: In this method, vaporization of the material is effectedusing pulses of laser beam of high power. The set up is a ultra high vacuum or high vacuum system equipped with inert or reactive gas introduction facility, laser beam, solid target and cooled substrate. Clusters of any material of which solid target can be made are possible to synthesize. Usually laser giving UV wavelength such as excimer laser is necessary because other wavelengths like IR or visible are often reflected by some ofthe metal surface. A powerful beam of laser evaporates the atoms from a solid source, atoms collide with inert gas atoms (or reactive gases) and coolon them forming clusters. They condense on the cooled substrate. The method is often known as laser ablation. Gas pressure is very critical in determining the particle size and distribution. Simultaneous evaporation of another material and mixing the two evaporated materials in inert gas leadsto the formation of alloys or compounds

Laser Pyrolysis or Laser Assisted Depositon: Here a mixture of reactant gases is decomposed using a powerful laser beam in presence of some inert gas like helium or argon. Atoms or molecules of decomposed reactant gases collide with inert gas atoms and interact with each other, grow and are then get deposited on cooled substrate. Many materials like Al2O3, WC, Si3Ni4 etc. are synthesized in nanocrystalline form by this method. Here too, gas pressure plays an important role in deciding the particle sizeand their distribution.

Sputter Deposition: In sputter deposition, some inert gas ions like Ar are incident on a target at a high energy. The ions become neutral at the surface but due to their energy, incident ions may get implanted, get bounded back, create collision cascades in target atoms, displace some of the atoms in the target creating vacancies, interstitials and other defects, desorb some adsorbents, create photons while loosing energy to target atoms or even sputter out some target atoms/molecules, clusters, ions and secondary electrons. Sputter deposition is a widely used thin film deposition technique, specially to obtain stoichiometric thin films from target material. Target material may be some alloy, ceramic or compound. It is avery good technique to deposit multilayer films for mirrors or magnetic films for spintronic applications. Sputter deposition can be carried out using Direct Current (DC) sputtering, Radio Frequency (RF) sputtering or magnetron sputtering. In all these methods, one uses discharge or plasma of some inert gas atoms or reactive gases. The deposition is carried out in a required gas pressurized high vacuum or ultra high vacuum system equipped with electrodes, one of which is a sputter target and the other is asubstrate, gas introduction facility etc

Electric Arc Deposition: This is one of the simplest and useful methods, which leads to mass scale production of fullerenes and carbon nanotubes. It requires water cooled vacuum chamber and electrodes to strike an arc between them. The positive electrode itself acts as the source of material. If some catalyst are to be used, there can be some additional thermal source of evaporation. Inert gas or reactive gas introduction is necessary. Usuallythe gap between the electrodes is ~1mm and high current ~50 to 100 amperes is passed from a low voltage power supply (~12-15 volts). Inert gas pressure is maintained in the vacuum system. When an arc is set up, anode material evaporates. This is possible as along as the discharge can be maintained. By striking the arc between the two graphite electrodes, it is possible to get fullerenes in large quantity. In case of fullerenes, the formation occurs at low helium pressure as compared to that used for nanotube formation. Also, fullerenes are obtained by purification of soot collected from inner walls of vacuum chamber, whereas nanotubes are found to be formed only at high He gas pressure and in the central portion of the cathode. No carbon nanotubes are found on the chamber walls Ion Implantation: In this method high energy (few keV to hundereds of keV) or low energy (<200 eV) ions are used to obtain nanoparticles. Ions of interest are usually formed using an ion gun specially designed to produce metal ions, which are accelerated to high or low energy towards the substrate heated to few hundered of C. Depending upon the energy f the incident ions, various other processes like sputtering and generation of electromagnetic radiation may take place. It is possible to obtain single element nanoparticles or compounds and alloys of more than one element. In some experiments it has been possible to even obtain doped nanoparticles using ion implantation. There is possibility of making nanoparticles using swift heavy ions (few MeV energy) employing ion accelerators like a pelletron.

Molecular beam epitaxy (MBE): This technique of deposition can be used to deposit elemental or compound quantum dots, quantum wells, quantum wires in a very controlled manner. High degree of purity in materials is achievable using ultra high vacuum (better than torr). Special sources of deposition known as Kundsen cell (K-cell) or effusion cell are employed to obtain molecular beams of the constituent elements. The rate of deposition is kept very low and substrate temperature is rather high in orderto achieve sufficient mobility of the elements on the substrate and layer bylayer growth to obtain nanostructures.

Thermolysis: Nanoparticles can be made by decomposing solids at high temperature having metal cations, and molecular anions or metal organic compounds. The process is called thermolysis. For example, small lithium particles can be made by decomposing lithium azide, $LiN3$. The material is placed in an evacuated quartz tube and heated to 400 C. At but 370 C $LiN3$ decomposes, releasing $N2$ gas, which is observed by an increase in the pressure on the vacuum gauge. In a few minutes the pressure drops back to its original low value, indicating that all the $N2$ has been removed. The remaining lithium atoms coalesce to form small colloidal metal particles. Particles less than 5nm can be made by this method. Passivationcan be achieved by introducing an appropriate gas.

Pulsed laser method: Pulsed lasers have been used in the synthesis of nanoparticles of silver. Silver nitrate solution and a reducing agent are flowed through a blenderlike device. In the blender there is a solid disk, which rotates in the solution. The solid disk is subjected to pulses from a laser beam creating hot spots on the surface of the disk. Silver nitrate and the reducing agent react at these hot spots, resulting in the formation of small silver particles, which can be separated from the solution using a centrifuge. The size of particles is controlled by the energy of the laser and rotation speed of the disk. This method is capable of a high rate of production.

Nanoparticles are essentially a bridge between atoms/molecules and bulk form ofthe material. The bulk materials demonstrate constant physical behaviours regardless of the size of the object. However, nanoscaled materials exhibit size dependent properties, and in most cases these properties are remarkably differentfrom the bulk counterparts. Therefore, as: (a) size of a material approaches nanometer scale, and (b) the surface to volume ratio increases, the materials undergo changes in their properties. On the other hand, in bulk materials (usuallylarger than one micrometer in size), the ratio of surface are to the volume of the material is negligible. Thus it may be concluded that the astonishing properties of nanoscaled materials are caused by the larger surface area of the nanoparticles,and the surface dominates the behaviour of such particles.

Applications:

- Surface coatings in biological applications:

The surface of the nanoparticles should be polar to provide good aqueoussolubility and prevent nanoparticle coagulation. Highly charged surfaces lead to non-specific interactions, while the polyethylene glycol terminated cells avoid
non-specific bindings. Biomolecules can be attached to the nanoparticles to direct them to specific sites in the body, even specific organelles in a cell, or to monitor individual protein or RNA molecules. Most commonly used tags to mark the nanoparticles include monoclonal antibodies, aptamers, streptavidin or peptides. These tags must be attached with the nanoparticles covalently, and also, their quantity per nanoparticles must be controlled for efficient operation. Multivalentnanoparticles have several tags attached to them which may cause their clustering, thereby activating the cell signaling paths, giving stronger anchoring. On the other hand, monovalent nanoparticles bear single binding sites, thus prevent cluster formation. These nanoparticles are suitable to track the behaviourof individual protein molecules.

- **Health and Safety**

There are various speculations both medically as well as environmentally that nanoparticles are hazardous. Owing to their huge surface areas, these particles are highly reactive or catalytic.As they are extremely small, they can pass through the cell membrane and may interact with the cell organelles. Presently, this interaction is not very well understood. Nonetheless, the nanoparticles are unlikely to enter the cell nucleus, Golgi complex, or other cell organelles mainly because of the particle size and intercellular aggregation.

- Nanomedicine

Nanomedicine implies the application of nanotechnology for medical uses. It includes medical uses of nanomaterials and biological devices, nanoscaled biosensors, etc. Future generation applications include biological nanoscaled machines. However, the possibilities of toxicity andenvironment impact of nanomaterials is an important concern.

Nanomaterials can be manipulated to perform various specialised functions. This can be achieved by interfacing the nanomaterials with various biomolecules or structures. Since the nanomaterials are similar in size to various biomolecules and structures, they can be used for in vivo as well as in vitro biomedical research and applications. Till now, various diagnostic devices, contrast agents, analytical tools, physical therapy applications, and drug delivery vehicles, etc., have been developed by integrating nanomaterials with biology.

- Cancer Treatment

Owing to the large surface area to volume ratios, nanoparticles can attach multiple functional groups to it, which can locate and bind to specific tumor cells. Furthermore, the small size of nanoparticles (10- 100 nm) allows them to preferentially accumulate at tumor sites (as tumorslack an effective lymphatic drainage system). The limitations to conventional cancer chemotherapy such as drug resistance, lack of selectivity as well as solubility, can be overcomeby using nanoparticles

- **Imaging**

Nanoparticles have great potential as in vivo imaging tools and devices. Nanoparticle based contrast agents, images (e.g, ultrasound) can have favourable distribution and enhanced contrast. Nanoparticles can aid visualisation of various stages in cardiovascular problems suchas blood pooling, angiogenesis, atherosclerosis, etc. Due to their small size, nanoparticles canbe very useful in oncology, especially in imaging. QDs when used for MRI (magneticresonance imaging), create exceptional images of tumor sites. Cadmium selenide nanoparticles glow on exposure to UV. When injected, they enter the cancer cells, thereby highlighted them.Nanoparticles are much brighter than organic dyes and can be excited only with single light source. Thus, use of fluorescent QDs can create much higher contrast images at lower costs incomparison to organic dyes. The disadvantage is that QDs are made from toxic materials.

- Sensing

Nanotechnology-on-a-chip is analogous to the lab-on-a-chip technology. Sensor test chips containing thousands of nanowires, able to detect proteins and other biomarkers left behind bycancer cells, could enable the detection and diagnosis of cancer in the early stages from a fewdrops of a patient's blood

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