VELAMMAL ENGINEERING COLLEGE, CHENNAI 66

DEPARTMENT OF PHYSICS

FUNDAMENTALS OF NANOSCIENCE

UNIT 3 – SYNTHESIS OF NANOMATERIALS

PART B QUESTION AND ANSWER

1. What are two routes through which nanoparticles can be synthesized?

- **Top down approach**: Involving breaking down bulk materials to nano sizes.
  Example: Mechanical alloying
- **Bottom Up approach**: where the nano particles are made by building atom by atom.
  Example: Chemical vapour deposition

2. Mention few techniques for synthesis of nano phase materials.

i. Mechanical alloying
ii. Inert gas condensation
iii. Sol-gel technique
iv. Electro-deposition
v. Laser synthesis
vi. Spraying

3. Write down the advantages of pulsed laser deposition.

1. Single walled carbon nanotubes of 10-20 nm diameter and 100 micrometer long can be produced by this technique.
2. In this technique more than 85% of graphite is converted into CNT.
3. The presence of catalyst prevents the growth of fullerenes instead a selective growth of nanotube is achieved.
4. The nanotube diameter can be controlled by the reaction temperature.

4. Explain the principle of Sol-Gel method.

Sol gel method involves formation of 'sols' in a liquid and then connecting the sol particles (or some sub-units capable of forming a porous network) to form a network. By drying the liquid, it is possible to obtain powders, thin films or even monolithic solid.

5. Explain vapor phase deposition methods?

Vapour phase deposition technique is used to fabricate thin films, multilayers, nanotubes, nanofilaments and nanosized particles of different materials. These

materials can be organic or inorganic. There are generally two types of vapour phase deposition techniques. They are: i. Physical Vapour Deposition (PVD) and ii. Chemical Vapour Deposition (CVD).

6. Briefly describe Physical vapour deposition technique.

Physical vapour deposition (PVD) is a technique by which a metal, ceramic or a compound can be converted into a gaseous form and then deposited on the surface of the substrate.

PVD methods are subdivided into,

1. Evaporation
2. Sputtering

7. List out few applications of Physical vapour technique.

1. PVD is used to produce the deposit of various metals, alloys or compounds in the form of coatings or films for :
   - Optics (Ex: Antireflection coatings)
   - Electronics (Ex: Metal contacts)
   - Mechanics (Ex: Hard coatings on tools)
2. PVD coatings are generally used to improve hardness, wear resistance and oxidation resistance.
3. PVD coatings used in wide range of applications such as aerospace, automotive, surgical or medical, cutting tools, dies and moulds for material processing.

8. What are the advantages and disadvantages of physical vapour deposition technique?

Advantages:

1. Ultra-pure films or particles can be produced by PVD technique.
2. Almostany type of inorganic material can be used as well as some kinds of organic materials.
3. The process is more environmentally friendly.

Disadvantages:

1. High capital cost.
2. Some processes operate at high vacuum and temperatures requiring skilled operators.
3. The rate of coatingdeposition is usually quite slow.

9. Briefly describe chemical vapour deposition technique.

Chemical vapor deposition (CVD) is a process whereby a solid material is deposited from a vapor by a chemical reaction occurring on or in the vicinity of a normally heated substrate surface. The solid material is obtained as a coating, a powder, or as single crystals. Chemical vapour deposition is a bottom up approach.

10. What are the advanatages of chemical vapour deposition techniques?

- Increased yield of nanoparticles.
- This method is used to produce *defect free nanoparticles*.
- More complex oxides such as $BaTiO_3$ and composite structures can be formed.
- Due to the simplicity of the experiment, the scaling up of the unit for mass production in industry is achieved without any major difficulties.
- The rate of deposition is of the order of few hundred microns per hour.
- It can be used for producing metallic and ceramic compounds.

11. Mention few applications of chemical vapour deposition technique.

- It is used to produce the produce the semiconductors and related devices like Integrated circuits, sensors and optoelectronic devices.
- It is used to produce the nanomaterials like Metals and alloys, Carbides, Nitrides, Oxides etc.
- It is used to produce coatings such as wear resistance, corrosion resistance etc.

12. What is the principle of Ball milling approach?

In ball milling, small hard balls are allowed to rotate inside a container (drum) and then it is made to fall on a solid with high force to crush the solid into nanoparticles. It is a top-down approach.

13. List out the advantage and disadvantage of hydrothermal synthesis process?

Advantages:

a) Many geometries including thin film, bulk powder, single crystals can be prepared.

b) Thermodynamically stable novel materials can also be prepared easily.

c) Easy and precise control of size, shape distribution and crystallinity can be obtained.

Disadvantages:

a) The need of expensive autoclaves.

b) Safety issues during the reaction process.

c) Impossibility of observing the reaction process.

14. Explain the principles of pulsed laser deposition?

The laser pulse of high intensity and energy is used to evaporate carbon from graphite.

These evaporated carbon atoms are condensed to form nanotubes. This is Bottom-up approach.

15. What are the various stages involved in laser ablation technique?

- Laser radiation interaction with the target.
- Dynamic of the ablation materials.
- Deposition of the ablation materials with the substrate.
- Nucleation and growth of a thin film on the substrate surface.

16. What are the advantages of pulsed laser deposition?

- It provides a good *control over impurities*.
- High quality samples can be grown in 10-15 minutes.
- It can be used for growing wide range of materials including Oxides, metal, semiconductors and even polymers.
- Single walled carbon nanotubes of 10 – 20 nm diameter and 100 micrometer long can be produced by this technique.
- In this technique more than 85% of graphite is converted into carbon nanotube.
- The presence of catalyst prevents the growth of fullerenes and a selective growth of nanotube is achieved.
- The nanotube diameter can be controlled by the reaction temperature.
- Other advantages are clean, flexibility, low cost and fast response.

17. What does sonication mean?

Sonication is a process in which sound waves are used to agitate particles in solution. Such disruptions can be used to mix solutions, speed the dissolution of a solid into a liquid (like sugar into water), and remove dissolved gas from liquids.

18. Are Sonicators dangerous?

Sonicators are high-frequency sound generators used to disrupt cells or shear nucleic acids. Laboratory personnel must be concerned about two of the major hazards associated with sonicators. The first hazard is hearing damage caused by high frequency sound.

19. What are the various stages involved in sol-gel synthesis?

Hydrolysis – Formation of Solution

Condensation or Ageing – Formation of gelatin

Drying – Removing the solvent from the gel

20. Explain the advantages of sol-gel synthesis method.

- The use of synthetic chemicals rather than minerals enables high purity materials to be synthesized.
- It involves the use of liquid solutions as mixtures of raw materials.
- Since the mixing is with low viscosity liquids, homogenization can be achieved at a molecular level in a short time.
- As the precursors are well mixed in the solutions, they are likely to be equally well-mixed at the molecular level when the gel is formed; thus on heating the gel, chemical reaction will be easy and at a low temperature.
- Changing physical characteristics such as pore size distribution and pore volume can be achieved.
- Incorporating multiple components in a single step can be achieved. Producing different physical forms of samples is manageable

VELAMMAL ENGINEERING COLLEGE, CHENNAI 66

DEPARTMENT OF PHYSICS

FUNDAMENTALS OF NANOSCIENCE

UNIT 4 – Characterisation of Nanomaterials

PART B QUESTION AND ANSWER

1. **What are the significances of XRD?**
    - Measure the average spacing's between layers or rows of atoms
    - Determine the orientation of a single crystal
    - Find the crystal structure of an unknown material
    - Measure the size, shape and internalstress of small crystalline regions
2. **State the principle of X-ray diffraction**

    X-ray diffraction is based on constructive interference of monochromatic **X-rays** and a crystalline sample. These **X-rays** are generated by a cathode **ray** tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample.
3. **What is the basic principle of electron microscopy?**
    - Particles such as electrons possess wave like properties and have shorter wavelength
    - Like light being focussed by lenses, electrons can be focussed by suitable electric and magnetic field
4. **What is the principle of SEM?**

    When the accelerated primary electrons strike the sample, it produces secondary electrons. These secondary electrons are collected by a detector to produce a three dimensional image of the sample.
5. **In what principle does the TEM works?**

    Electron are made to pass through the specimen and the image is formed in the fluorescent screen, either by using transmitted beam (bright field image), or by using diffracted beam (dark field image).
6. **What are the advantages and disadvantages of TEM?**

    Advantages:

    1. It has high resolution

2. The magnification is 1,000,000 times greater than the size of the object
3. It can be used to examine the specimen of size upto 0.2 nm

Disadvantages:

1. The specimen should be very thin
2. It is not suitable for thick sample
3. 3– dimensional image cannot be obtained

7. Mention few applications of TEM.

1. The main application of TEM is in nano-sciences (nano-tubes, micro machine etc), used to find the internal structure of nano materials.
2. It is used to find the 2 – dimensional image of very small biological cells, virus, bacteria etc.
3. It is used in thin – film technology, metallurgy, biochemistry, micro- biology etc.
4. It is used to study the compositions of paints, papers,fibers, composite materials, alloys etc.

8. Differentiate between SEM and TEM.

S.No	SEM	TEM
1	Scattered electrons or secondary electrons are used to form the image	Transmitted elecrons through the specimen is used to form the image
2	Scanning of electron beam on the specimen is required	Scanning is not reqiured
3	Even a thick specimens can be analyzed	Very thin sample has to be used
4	Very good contrast	Contrast is poor

9. Explain the sample preparation process involved in TEM.

For TEM, samples must be cut into very thin cross-sections. This is to allow electrons to pass right through the sample. After being fixed and dehydrated, samples are embedded in hard resin to make them easier to cut. Then, an instrument called an ultramicrotome cuts the samples into ultra-thin slices (100 nm or thinner). TEM samples are also treated

with heavy metals to increase the level of contrast in the final image. The parts of the sample that interact strongly with the metals show up as darker areas.

10. Differentiate Optical microscope and electron microscope

Optical Microscope	Electron Microscope
Lenses used are made of glass	Lenses used are electromagnets
Focal length of the lenses are fixed	Focal lengths of the lenses can be varied by changing the current through the coil
The objective lens can be varied for different magnification	The objective lens is fixed and the magnification is altered by changing the focal length of projector lens
Depth of field is small	Depth of field is large
The source is normally placed at the bottom	The source is placed at the top

11. What is AFM?

Atomic force microscopy (AFM) is a type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit.

12. Mention the advantages and disadvantages of AFM?

Advantages

1. Easy sample preparation
2. Accurate height information
3. Living systems can be studied
4. 3D image facilities

Disadvantages

1. Data is dependent on the tip

2. Damage of tip and sample is possible
3. Limited magnification range
4. Limited scanning speed

13. Mention few applications of AFM.

1. Unfolding of proteins can be studied
2. Biomolecules can be imaged
3. Studies of antibody & antigen binding can be studied
4. Study surface frictional forces

14. What is scanning probe microscopy?

Scanning probe microscopes (SPM) are a family of tools used to make images of nanoscale surfaces and structures, including atoms. They use a physical probe to scan back and forth over the surface of the sample. During the scanning process, a computer gathers data that are used to generate an image of the surface. SPM are different are different from optical microscopes because the user doesn't " see " thesurface directly. Instead the tool "feels" the surface and creates an image to represent it.

15. Explain the principle of dynamic light scattering.

Dynamic light scattering (DLS) is based on the Brownian motion of dispersed particles. When particles are dispersed in a liquid they move randomly in all directions. The principle of Brownian motion is that particle is constantly colliding with solvent molecules. These collisions cause a certain amount of energy to be transferred, which induces particle movement. The energy transfer is more or less constant and therefore has a greater effect on smaller particles.

16. Write down the Stokes - Einstein relation for calculating particle size in DLS method?

$$D = \frac{k_B T}{6\pi\eta R_H}$$

Where,

D Translational diffusion coefficient [m²/s] – "speed of the particles"

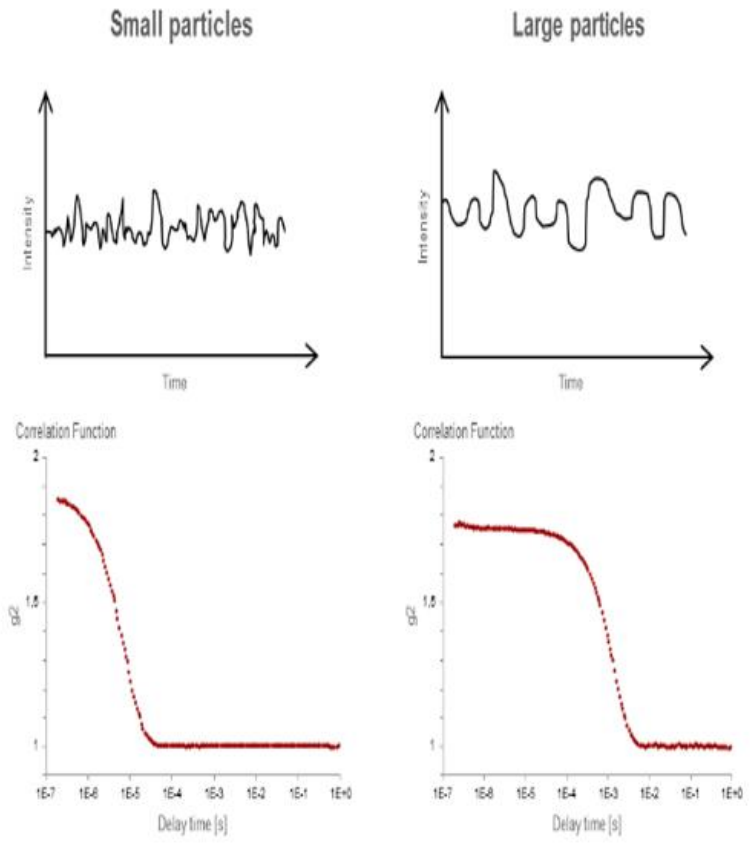
k_B Boltzmann constant [m²kg/Ks²]

T Temperature [K]

η Viscosity [Pa.s]

R_H Hydrodynamic radius [m]

17. Draw the intensity and correlation function of large and small particles in DLS method?



18. Write down the limitations of dynamic light scattering?

1. It measure the hydrodynamic radius of the particle, not able to measure the actual size of the particle
2. The particle having size greater than 1000 nm are not measured by this method
3. Size of solid particles are not measured by DLS

19. How to perform nanoindentation?

Nanoindentation is a powerful technique where the indentor tip of known geometry is projected in to the specific site in the material to be tested, increasing load is applied and when it reaches the designated maximum value, partial unloading is performed until desired depth is attained. The holding segment is introduced which allows the material to relax before unloading. The process is repeated many times and the position of the indentortip and the surface is monitored with differential transformer.

20. Mention some of the limitations in nanoindentation methods?

1. Limited depth of indentation
2. Complex calibration
3. Difficult to detect the surface of the sample in soft tissues
4. Sensitivity and accuracy of measurements