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Topic for

Semester – VI, Paper – DSE A2 Nanomaterials and Applications

Understanding Nanostructures

I. Introduction to Nanostructures

The nanostructure is an object or material characterized by dimensions at the scale of nanometers, generally from 1 to 100 nm. Because of this small value, they differ in their behavior from objects of large sizes and display a whole range of new "exotic" properties.

A. Definition of Nanostructures

The word "nanostructure" means a material or system in which at least one of the dimensions is in the nanometer range. One nanometer is a billionth part of a meter. Nanostructures can either be present in nature, like DNA molecules, or can be prepared artificially and used in products, for example, in an electronics nanoparticle.

B. Importance and Applications in Various Fields

Nanostructures, with their highlighted characteristics, find applications of crucial importance in many fields. Some of the critical areas in nanostructures find their application are:

1. **Electronics:** Nanostructures are used in electronic devices to enhance performance, minimize size, and consume less power. In this category, nanowires and nanotubes are indispensable.
2. **Materials Science:** Nanotechnology has revolutionized material design, improving strength and conductivity in these materials, among many other properties. For example, in materials science, some of the nanostructured materials used are thin film and nanocomposite.
3. **Biomedicine:** In this case, nanostructures are prepared for drug delivery, imaging, and diagnostics. A prominent place in the targeted delivery of drugs and medical imaging technologies is occupied by nanoparticles and nanorods.
4. **Energy:** Nanostructures find applications in diverse energy-related technologies, for example, solar cells, batteries, and fuel cells. Nanostructured materials offer higher energy conversion efficiency and better storage capacity.
5. **Environmental Remediation:** Nanostructures are used to treat environmental remediation, such as water and air filtration, using them. Nanomaterials can very well remove pollution and impurities in water.

C. Overview of 1D, 2D, and 3D Nanostructures

1. **1D Nanostructures:** One-dimensional nanostructures are materials that, after synthesis, acquire a dimension in the nanometer scale range; for example, nanowires, nanotubes, or nanorods. They present interesting electrical and mechanical properties and, hence, are valid for applications such as sensors and nanoelectronics.
2. **2D Nanostructures:** 2D nanostructures refer to materials with dimensions within the nanometer scale, which exist as nanosheets and thin-film materials (e.g., graphene). These have marvelous mechanical strength, optical properties, and surface-to-volume ratio qualities that are best for them to be used in flexible electronics and optoelectronic devices.
3. **3D Nanostructures:** All those nanostructures with all three dimensions in the range of a nanometer. For example, nanodots and nanocubes are examples of 3D nanostructures. These give birth to the large surface area-to-volume ratio and are best fit in catalysis, biomedical applications, and creation.

II. 1D Nanostructures

1D nanostructures are defined as materials in which the size scale is essentially confined to only one direction, usually on the nanometer scale. The properties of these materials in the dimensions on the nanometer scale show some uniqueness and are, hence, very valuable for many scientific and technological applications.

A. Definition and Characteristics

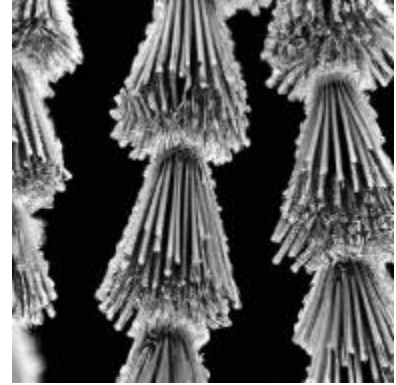
1D nanostructures are elongated structures, often with a high aspect ratio, wherein the length, which is one of the dimensions, is much larger compared to the other two dimensions (width and height).

B. Examples of 1D Nanostructures

1. **Nanowires:** These are long, thin structures with a diameter usually in the range of a few nanometers to a few hundred nanometers. Nanowires can be made from metals, semiconductors, and even oxides. Nanowires have high length-to-diameter ratios and make a good case for use in electronic and sensing applications.



2. **Nanorods:** Nanorods are the third type between nanowires and nanobelts and are rod-shaped with uniform diameters. Therefore, they have been widely used in the field of nanoelectronics, sensors, and optical devices due to their interesting optical and electrical properties.



C. Properties and Applications

1. **Electrical Conductivity:** 1D nanostructures show superior electrical conductivity, particularly in nanowires of conductive materials like gold, silver, and copper. It has been essential for nanoelectronics in developing nanowires acting as conductive channels in electronic circuits.
2. **Sensing Applications:** With their one-dimensional nature, nanowires have found utility in several sensing applications due to the high surface-to-volume sensitivity to exterior stimuli. For example, the nanowire can be used for high sensitivity in sensing biological molecules, gases, and chemicals because it has specificity receptors immobilized on their surfaces.
3. **Nanoelectronics:** Nanowires and nanorods are given importance in nanoelectronics. They provide the capability of miniaturization with improved performance over traditional electronic components. Further, several other applications use nanowires and nanorods to increase speed performance in transistors, diodes, and other electronic device applications, improve power dissipation capabilities, and increase functionality.

III. 2D Nanostructures

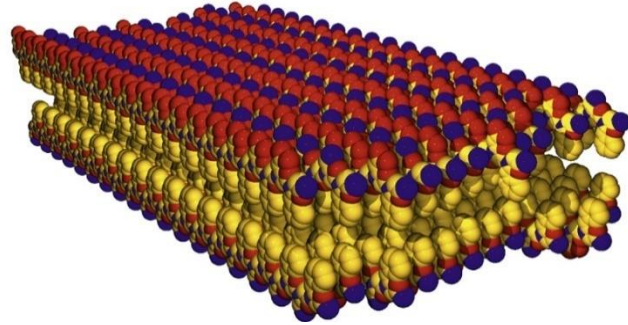
2D nanostructures are those materials in which most dimensions are two-dimensional, while one is far greater than the other. Consequently, these nanostructures have been the focus of attention due to the unique and exotic properties they exhibit and have potential applications in many scientific and technological fields.

A. Definition and Characteristics

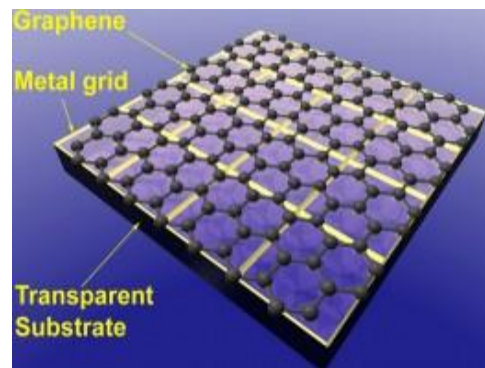
The 2D nanostructures are characterized by flat, sheet-like morphologies, whereby their thickness is within the nanometer scale, while the length and width project to higher dimensions, thus giving high aspect ratios and large surface areas relative to their volume.

B. Examples of 2D Nanostructures

1. **Nanosheets:** The term "nanosheets" evidently means those thin structures whose thickness is measured in nanometers. Nanosheets may be made of various materials: for example, graphene, transition metal dichalcogenides (TMDs), or even layered oxides. The nanosheets will have a planar structure and good electronic and mechanical properties.



2. **Thin Films (including Graphene):** "Thin film" represents a kind of continuous layer of material with thickness varying from several nanometers to a few micrometers. Among these is the well-known 2D nanostructure of one single layer of carbon atoms forming a hexagonal lattice: graphene. It has excellent mechanical, electrical, and thermal features, turning into a versatile material for most varied applications.



C. Properties and Applications

1. **Mechanical Strength:** One of the most significant and fundamental properties of 2D nanostructures is their mechanical strength and stiffness at an atomic scale. This property shows high tensile strength, making it applicable to structures requiring light but sturdy materials, such as aerospace components, structural reinforcements, etc.
2. **Optoelectronic Devices:** Two-dimensional nanostructures are vital elements in designing and developing high-performance photodetectors, light-emitting diodes (LEDs), and solar cells. The unique electronic band structures and optical characteristics, as mentioned above, point to their effectiveness in the absorption and emission of light and its manipulation; thus, they are expected to lead the sector of photonics and optical technologies.
3. **Flexible Electronics:** The high flexibility and transparency of 2D nanostructures, particularly graphene-based materials, make them ideal for use in flexible electronics applications. Such flexible 2D materials can be flexibly used in wearable devices, flexible displays, electronic skin, etc., to provide lightweight, bendable, and even transparent functionalities.

IV. 3D Nanostructures

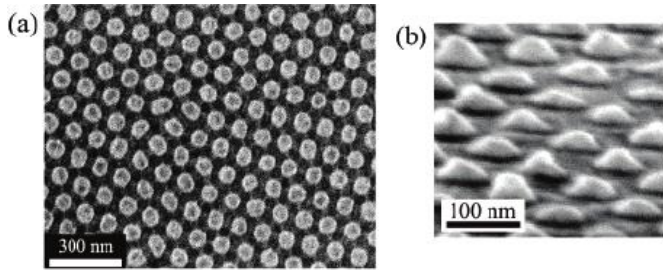
3D nanostructures are, in fact, dimensionally investigable material that has dimensions in all three spatial directions but is mostly limited within the sizes of the nanometer scale. Such material will have a few unique qualities from its complex 3D architecture. These nanostructures will hold tremendous promise for applications in science and technology.

A. Definition and Characteristics

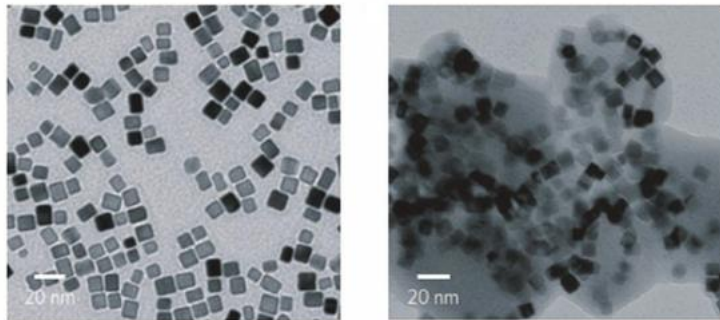
3D nanostructures are three-dimensional shapes and structures whose length, width, and height are all on the order of nanometer scale. While 1D and 2D nanostructures are either elongated or planar, 3D nanostructures have more complex forms and geometries.

B. Examples of 3D Nanostructures

1. **Nanodots:** Nanodots are particles of tiny size, usually between several and several tens of diameters. Most of them are spherical, the rest being cubic or taking some other geometric forms. Nanodots are widely used in nano-electronics, different sensing technologies, and numerous biomedical applications due to their minor size and colossal surface.



2. **Nanocubes:** It is a kind of nanostructured material that displays cubic shapes with dimensions in the nanometer range. Nanocubes present well-defined faces and edges; therefore, nanocubes are used in a wider field of applications for sensitive applications requiring control of precise surface properties, such as in catalysis, sensing, and materials science.



C. Properties and Applications

1. **Surface Area-to-Volume Ratio:** One of the prime characteristics of 3D nanostructures is the high surface-to-volume ratio. The property gives various advantages in applications with an increase in interaction with surrounding environments, such as high reactivity and increased efficiency, for example, accelerated processes in catalysis and sensing.
2. **Catalysis:** 3D nanostructures, e.g., nanodots and nanocubes can fill in for the catalysts at high temperatures concerning their surface area and unique surface properties. They could provide enhanced reaction rates and selectivity and lead to the development of entirely new

catalytic processes with applications in energy conversion, environmental remediation, and industrial chemistry.

3. **Biomedical Applications:** 3D nanostructures find immense applicability in biomedicine, especially for drug delivery, imaging, and tissue engineering. These structures can be functionalized with biomolecules or therapeutic agents: nanodots and nanocubes, for example, may be used for targeting specific cells or tissues by increasing contrast for imaging or providing a scaffold for tissue regeneration and repair.

V. Fabrication Techniques for Nanostructures

Nanofabrication requires exact methods to control dimensions, shapes, and properties at nanometer scales. This includes various techniques such as top-down, bottom-up, and hybrid approaches, which have become state-of-the-art in their unique ways for the fabrication of nanostructured materials.

A. Top-Down Fabrication Methods

Top-down methods are the controlled processes of larger structures to reduce them to nano-size. Examples of such a method are:

1. **Photolithography:** This method will be used when light is exposed on the substrate carrying photoresist material. The process of exposure followed by development occurs before etching the substrate to produce nano-sized features. It has, therefore, found great use in the manufacture of semiconductors for developing circuit patterns at the nanoscale on silicon wafers.

Detailed Process:

i. Substrate Preparation:

- Start with a clean silicon wafer. It will act as the substrate.
- Apply a coat of photoresist material (usually a polymer) to the silicon wafer by spin-coating. This will control the layer thickness of the photoresist film; that thickness controls the final detail size.

ii. Pre-bake:

- Prebake the photoresist-coated wafer on a hot plate or in an oven to drive off any solvent and evaporate excess moisture from the photoresist layer.

iii. Mask Alignment:

- Design a photomask to hold the desired pattern of the nanoscale features. The photomask is, in this case, a transparent plate with a pattern of opaque areas blocking light in such a way that the exposure of the light gives the pattern.

- Setting the photomask on top of the wafer, all in line with an alignment system, helps ensure that the pattern from the photomask is aligned correctly relative to the photoresist layer applied on the wafer.

iv. Exposure:

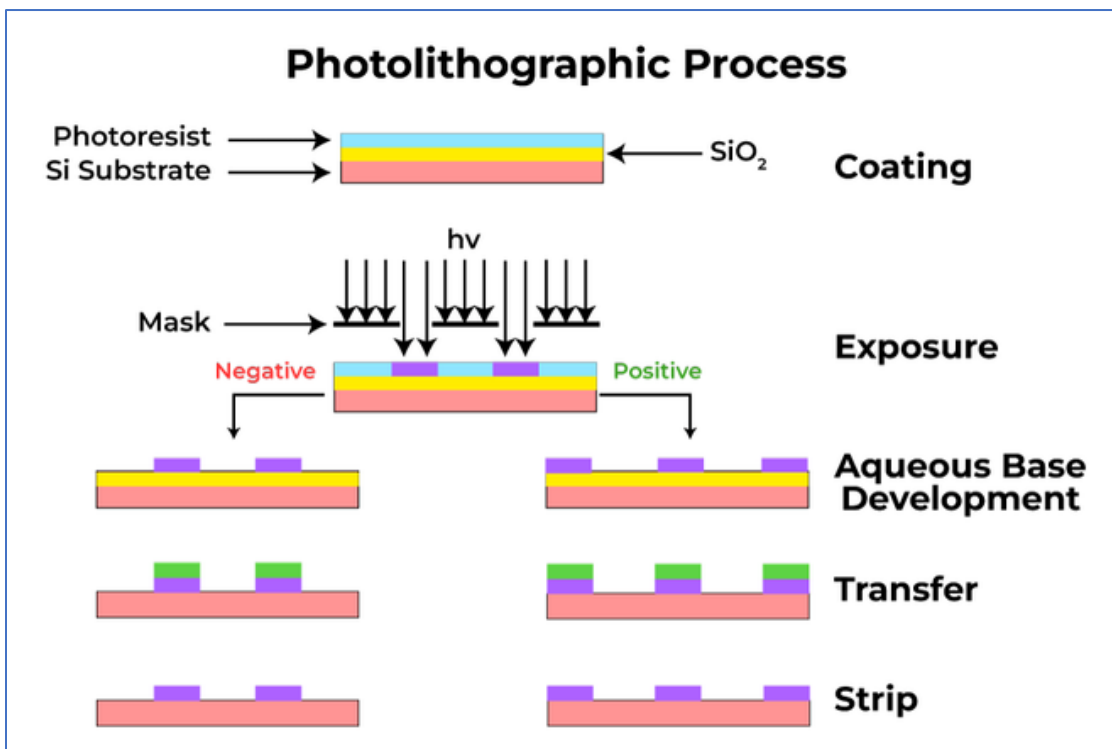
- Expose the aligned wafer and photomask to ultraviolet (UV) light in an exposure system. The areas of the photoresist layer exposed to UV light undergo a chemical reaction (photochemical change), becoming either more soluble (positive photoresist) or less soluble (negative photoresist) depending on the type of photoresist used.
- The UV light passes through the transparent regions of the photomask, exposing specific areas of the photoresist layer on the wafer according to the pattern on the mask.

v. Development:

- Transfer the exposed wafer to a developer solution that selectively removes either the exposed (positive photoresist) or unexposed (negative photoresist) regions of the photoresist layer.
- Agitate the wafer in the developer solution for a controlled time to ensure precise removal of the desired areas, revealing the patterned photoresist on the wafer.

vi. Post-bake:

- Perform a post-exposure bake on the developed wafer to harden the remaining photoresist pattern and stabilize its dimensions.



vii. Etching (Optional):

- If the patterned photoresist acts as a mask for subsequent etching steps, perform dry or wet etching on the exposed areas of the silicon wafer to transfer the pattern into the silicon substrate, creating nanoscale features.

Example Application:

Suppose we want to fabricate nanoscale transistor features on a silicon wafer using photolithography. We follow the above steps to pattern the photoresist layer, align the photomask to define transistor shapes, expose the wafer to UV light, develop the pattern, and then etch the silicon to create the transistor structures.

[A Step-by-Step Look at Photolithography](#)

2. **Electron Beam Lithography (EBL):** EBL utilizes a focused electron beam to directly write patterns on a substrate covered with a resist material. The exposed areas are then chemically or physically modified to create nanostructures. EBL is employed in research and semiconductor industries to fabricate nanoscale devices and structures with high resolution.

Detailed Process:

i. Substrate Preparation:

- Start with a clean substrate, typically a silicon wafer coated with a thin layer of electron-sensitive resist material.

ii. Electron Beam System Setup:

- Use an Electron Beam Lithography system equipped with an electron beam column, stage for sample positioning, and a computer interface for control.
- Load the substrate into the EBL system and ensure it is properly aligned and secured.

iii. Electron Beam Exposure:

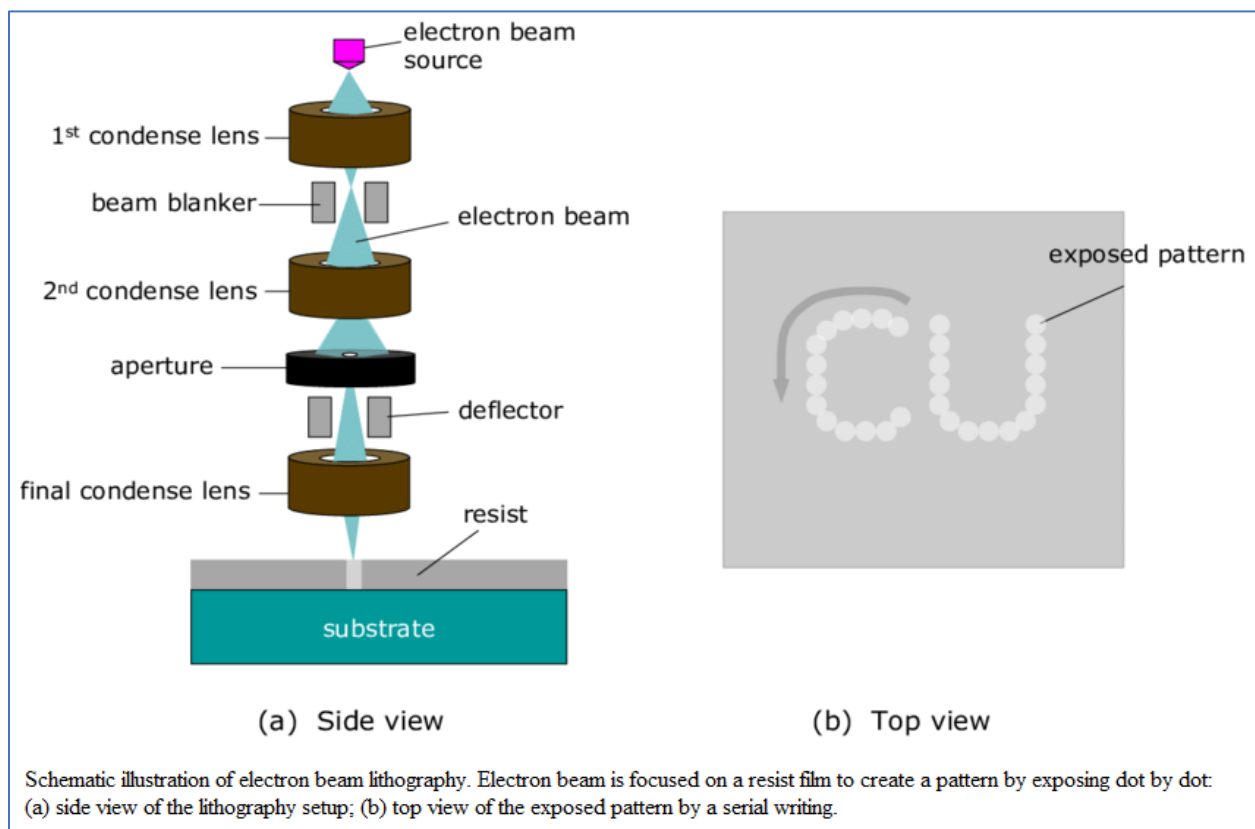
- Design a pattern or layout on a computer using EBL software. This pattern defines the nanoscale features to be written onto the resist layer.
- Configure the EBL system to control the electron beam parameters, such as beam intensity, spot size, and dwell time.
- Use the electron beam to expose specific areas of the resist layer according to the designed pattern. The electron beam interacts with the resist, causing localized changes in its chemical properties.

iv. Development:

- Transfer the exposed substrate to a developer solution that selectively removes the exposed or unexposed regions of the resist layer, depending on the type of resist used (positive or negative resist).
- Agitate the substrate in the developer solution to dissolve and remove the unexposed areas, revealing the patterned resist on the substrate.

v. Post-processing (Optional):

- Optionally, perform post-exposure baking or other treatments to further stabilize the developed resist pattern and enhance its durability.



Example Application:

Let us consider the fabrication of nanoscale structures, such as graphene nanoribbons, using Electron Beam Lithography (EBL).

- Substrate Preparation:** Start with a silicon wafer coated with a layer of electron-sensitive resist material.
- EBL System Setup:** Load the wafer into the EBL system and set up the electron beam parameters.

- iii. **Electron Beam Exposure:** Use the EBL software to design the pattern for graphene nanoribbons. Direct the electron beam to expose the resist layer, defining the nanoribbon shapes with nanometer precision.
- iv. **Development:** Transfer the exposed wafer to a developer solution to remove the unexposed resist areas, revealing the patterned graphene nanoribbons on the substrate.
- v. **Post-processing:** Optionally, perform post-exposure baking to stabilize the nanoribbon pattern.

The resulting substrate will have precise nanoscale features of graphene nanoribbons patterned using Electron Beam Lithography, demonstrating the high-resolution capabilities of EBL in nanofabrication processes.

[A Step-by-Step Look at Electron Beam Lithography \(EBL\)](#)

B. Bottom-Up Fabrication Methods

Bottom-up methods build nanostructures by assembling atoms or molecules into desired configurations. Examples include:

1. **Chemical Vapor Deposition (CVD):** In CVD, precursor gases are introduced into a chamber, where they react and deposit atoms or molecules onto a substrate, forming a thin film or nanostructure. For example, graphene can be synthesized on metal substrates using CVD by decomposing hydrocarbons like methane.

Detailed Process:

i. Substrate Preparation:

- Start with a clean substrate, often a copper foil or silicon wafer, which will serve as the growth surface for the deposited material.

ii. Deposition Chamber Setup:

- Introduce the substrate into a CVD (Chemical Vapor Deposition) chamber, which can be evacuated to low pressure and maintained at controlled temperatures.
- Develop a system with a gas inlet to facilitate the introduction of precursor gases into the chamber.

iii. Heating and Cleaning (Pre-treatment):

- Heat the substrate to a specific temperature, typically ranging from 600°C to 1000°C, depending on the material to be deposited.
- Perform a pre-treatment step to clean the substrate surface, remove any contaminants, and promote adhesion of the deposited material.

iv. Introduction of Precursor Gases:

- Introduce precursor gases into the CVD chamber. For graphene growth, precursor gases like methane (CH₄) or ethylene (C₂H₄) are used as carbon sources.
- Control the flow rates of precursor gases to achieve the desired deposition rate and thickness of the material.

v. Chemical Reaction and Deposition:

- The precursor gases undergo chemical reactions, which are either heat or plasma-activated, to cause the target material's formation on the heated substrate in the chamber.
- In the growth of graphene, the carbon atoms delivered from the gas precursor bond on the surface of the substrate to the partner carbon atom while creating a monoatomic layer.

vi. Cooling and Stabilization:

- After the deposition process, gradually cool down the substrate to room temperature to stabilize the deposited material.
- This cooling step helps prevent structural defects and ensures the integrity of the grown material.

vii. Removal of Substrate (if applicable):

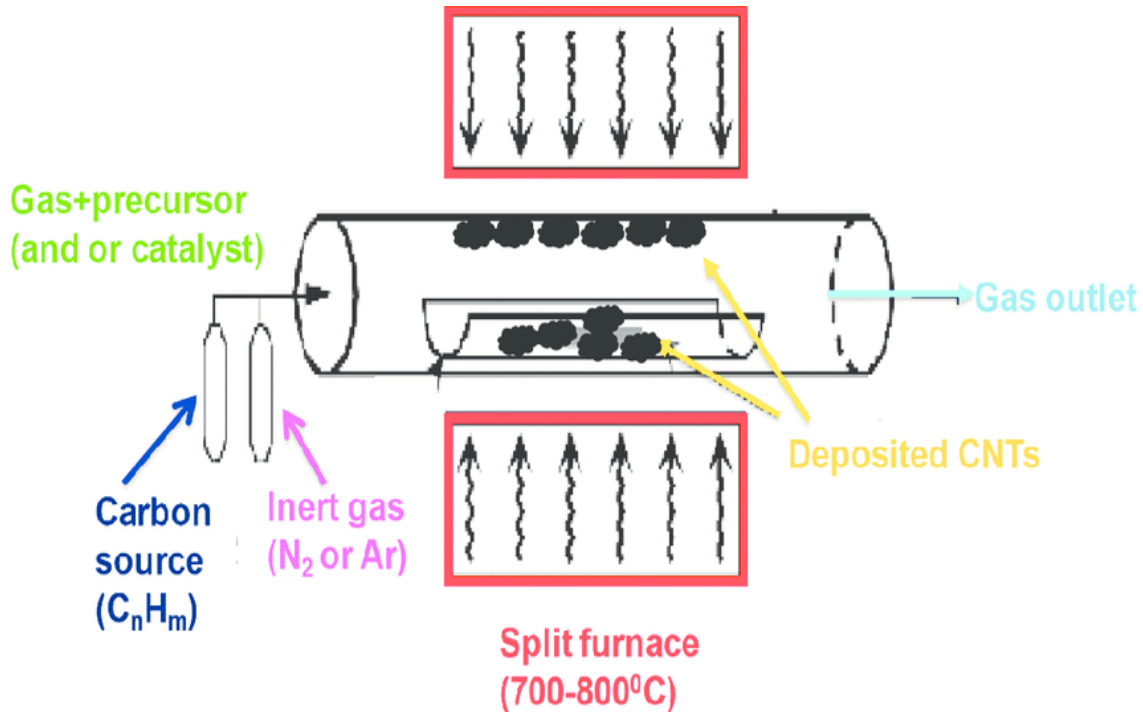
- In cases where the deposited materials need to be removed or separated from the substrate, use substrate removal techniques like etching or mechanical transfer.

Example Application: Growing Graphene Using CVD

1. **Substrate Preparation:** Use copper foil as the substrate since it has compatibility for graphene growth.
2. **CVD Chamber Setup:** A copper foil laid up in the CVD chamber with an inlet gas system and heating elements.
3. **Heating and Cleaning:** Heating of the copper foil was done to about 1000°C in a hydrogen atmosphere to clean the surface of the foil and help in adhering to the graphene.
4. **Introduction of Precursor Gases:** The precursor gas is introduced into the chamber, for example, methane (CH₄), as the source for carbon to grow graphene.
5. **Chemical Reaction and Deposition:** This initiates the CVD process, where carbon atoms from methane adsorb and recombine into a hexagonal lattice structure, depositing on the copper foil surface as a monolayer graphene.
6. **Cooling and Stabilization:** Slowly cool down the substrate so that the grown graphene layer stabilizes.

7. **Substrate Removal (Optional):** Remove the copper substrate by etching, leaving techniques including freestanding graphene.

The resultant product was high-quality graphene grown via Chemical Vapor Deposition (CVD), showing the versatile and scalable performance of CVD to a much higher degree for synthesizing nanomaterials.



[A Step-by-Step Look at Chemical Vapor Deposition \(CVD\)](#)

2. **Self-Assembly:** It is the process by which the supramolecular interaction of a molecular assembly results spontaneously in the organization of a given structure. An example is DNA nanotechnology; for instance, it allows the complementary base pairing of DNA strands to bring forth self-assembly into specific nanostructures, for example, nanoribbons or nanotubes.

Detailed Process:

i. Design and Selection of Building Blocks:

- Choose appropriate building blocks, such as molecules or nanoparticles, capable of self-assembly.
- For example, in the case of forming a lipid bilayer, phospholipid molecules are commonly used due to their amphiphilic nature.

ii. Dispersion or Dissolution:

- Disperse or dissolve the chosen building blocks in a suitable solvent or medium.
- For lipid bilayer formation, phospholipids are typically dissolved in water or an aqueous solution.

iii. Spontaneous Arrangement:

- Due to the inherent properties of the building blocks (e.g., amphiphilicity), they spontaneously arrange themselves into organized structures.
- In the case of lipid bilayers, phospholipid molecules align with their hydrophobic tails facing inward and hydrophilic heads facing outward, forming a double-layered membrane.

iv. Energy Minimization:

- The self-assembled structure minimizes its energy by maximizing favorable interactions and minimizing unfavorable interactions.
- For lipid bilayers, the hydrophobic effect drives the alignment of phospholipid tails away from water, while hydrogen bonding and electrostatic interactions stabilize the bilayer structure.

v. Structural Integrity and Stability:

- The self-assembled structure achieves structural integrity and stability through intermolecular forces and molecular interactions.
- Lipid bilayers, for example, are stable due to the cohesive forces between phospholipid molecules within each layer and the repulsion forces between water molecules and the hydrophobic tails.

Example Application: Lipid Bilayer Self-Assembly

i. Design and Selection of Building Blocks:

- Choose phospholipid molecules as the building blocks for the lipid bilayer due to their amphiphilic nature.

ii. Dispersion or Dissolution:

- Dissolve phospholipid molecules in an aqueous solution, where the hydrophilic heads interact with water molecules, while the hydrophobic tails cluster together.

iii. Spontaneous Arrangement:

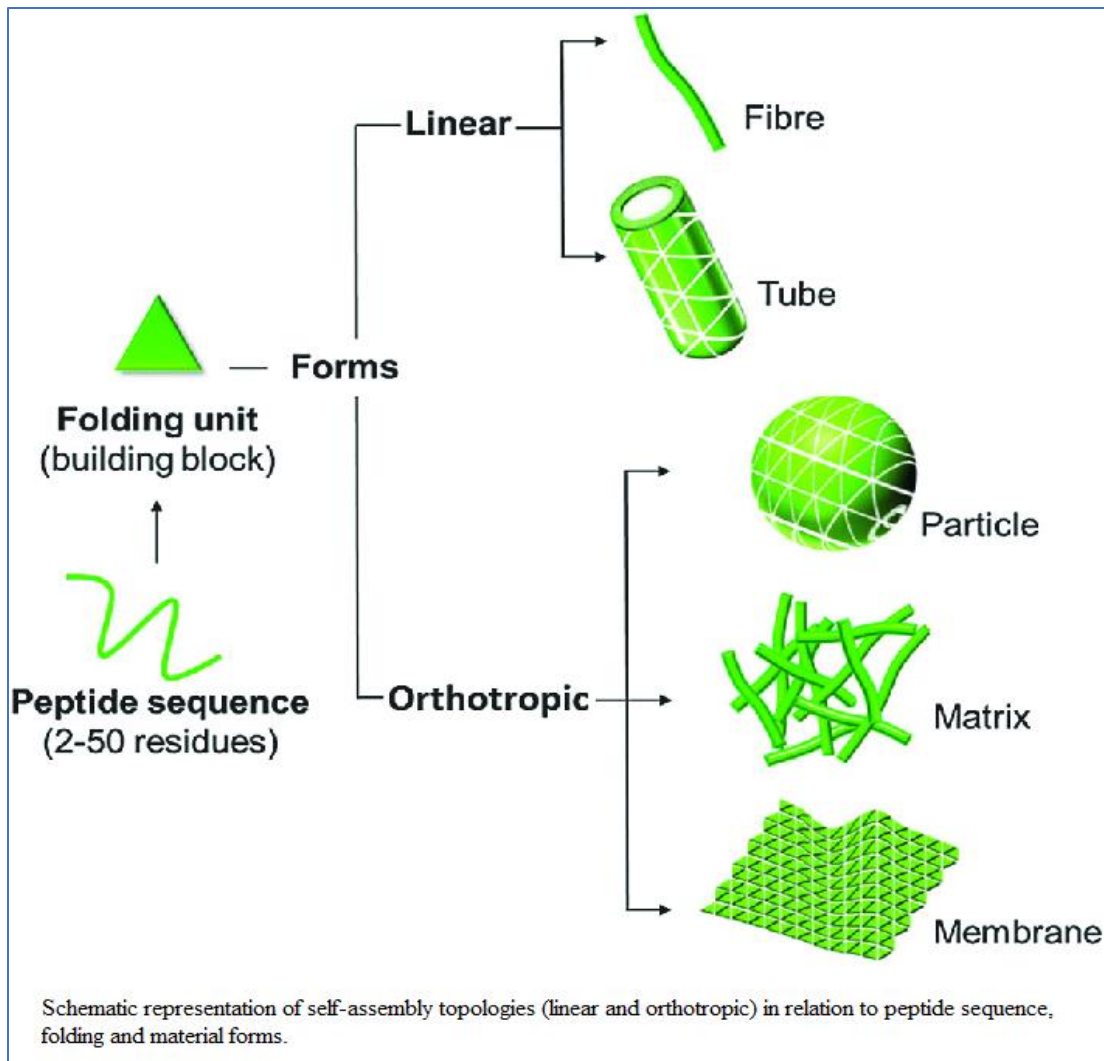
- Through its amphiphilic nature, phospholipids self-assemble as a lipid bilayer structure, whereby the tails of the heads of the parts that are lipophobic face inward and those of the heads that are lipophilic.

iv. **Energy Minimization:**

- The structure of the lipid bilayer minimizes the energy by maximizing hydrophobic interactions of tails and regions of hydrophobicity but also reduces the exposure of heads to the hydrophobic environment.

v. **Structural Integrity and Stability:**

- Structural integrity and stability of the lipid bilayer are maintained by cohesive forces acting between phospholipid molecules of each layer and repulsion forces of water molecules towards hydrophobic tails.
- This is an example of how self-assembly takes place between molecules, for example, the self-assembly of phospholipids into complex structures such as lipid bilayers through self-assembly processes brought about by intrinsic properties and intermolecular interactions.



C. Hybrid Approaches

These approaches are hybrids. The approaches that have both the characteristics of top-down and bottom-up approaches are generally used for flexible control and fine scalability. Examples are:

1. **Nanoimprint Lithography:** Nanoimprint lithography produces a pattern with the patterning capability of lithography and the self-assembly of materials. This process works by imprinting a structure of a desired nanostructure into a resist, followed by curing the resist. It, therefore, means that the structure of the desired nanostructure is formed, and its applications are in optics, data storage, and nanoelectronics.

Nanoimprint Lithography (NIL) Process:

i. Substrate Preparation:

- The substrate for the nanoimprint is to be cleaned before the nanoimprinting and could be either a silicon wafer or a glass slide.

ii. Preparing the Stamp:

- Design and fabricate a stamp or mold containing the desired nanopatterns. The stamp can be made of materials like silicon, quartz, or polymers.
- Use lithography techniques such as electron beam lithography (EBL) or focused ion beam (FIB) milling to create the nanopatterns on the stamp.

iii. Applying Release Agents (Optional):

- Release agents or coatings may optionally be applied to the surface of the stamp to ease separation once imprinted.

iv. Nanoimprint Process:

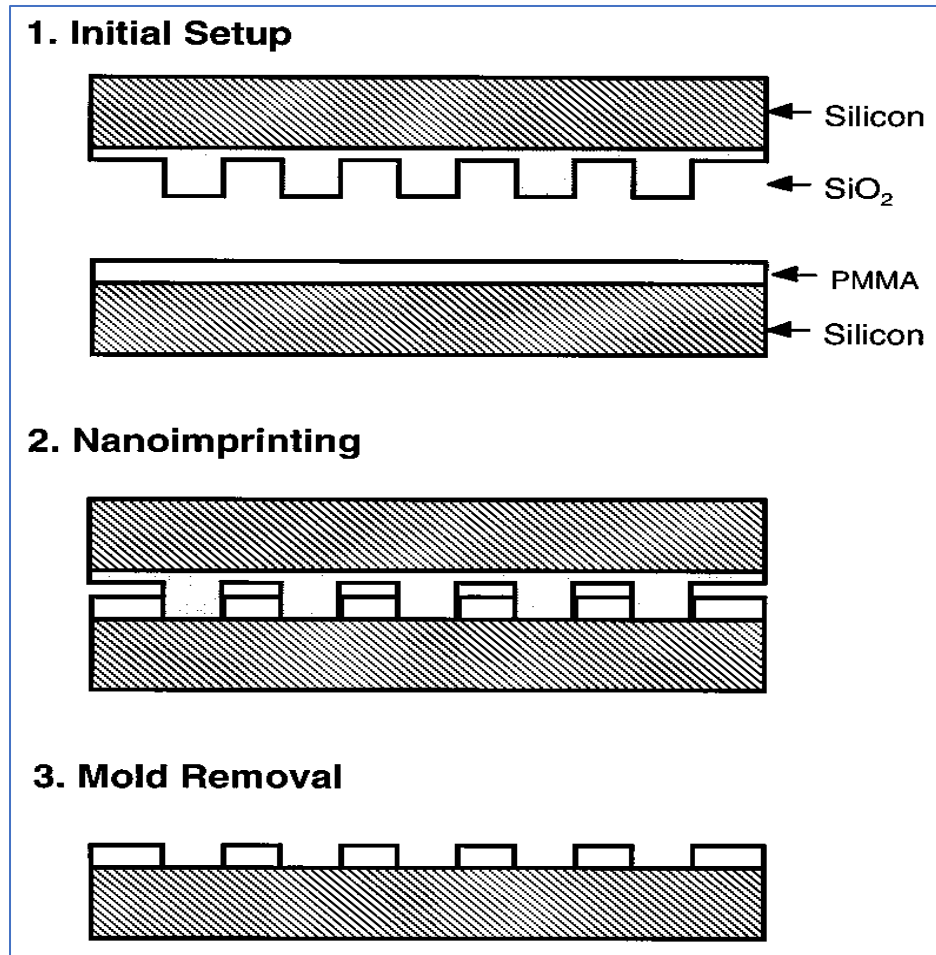
- The substrate and the stamp should be heated to a suitable temperature, usually above the glass transition temperature of the imprint material.
- Make firm contact of the stamp with the substrate so that the nano-patterns on the stamp are transferred to the substrate.
- Allows the imprinting process to occur for a constant pressure and temperature for the time specified, usually from a few seconds to minutes.

v. Cooling and Separation:

- Cool down the stamp and substrate to solidify the imprint material and fix the nanopatterns.
- Take out the stamp from the substrate with minimum force to avoid displacement of the nanopatterns from the substrate surface.

vi. Post-Imprint Processing:

- If required, post-imprinting processes should be performed either to further cure the imprint material, clean the surface of the substrate, or even additional etching for sharpening the nanopatterns.



Example Application: Nanoimprinting Nanopatterns on Silicon Wafer

i. Substrate Preparation:

- Use a clean silicon wafer as a nanoimprinting substrate.

ii. Preparing the Stamp:

- Fabricate a stamp with nanopatterns using lithography techniques like EBL. For example, create an array of nano-sized pillars on the stamp.

iii. Applying Release Agents:

- Apply a release agent on the stamp's surface to assist in easy de-molding during nanoimprinting.

iv. **Nanoimprint Process:**

- Transferring heat to the stamp and to the substrate material at a temperature higher than the glass transition temperature of the imprint material.
- Press the stamp onto the substrate with pressure, transferring the nanopatterns onto the surface of the silicon wafer.

v. **Cooling and Separation:**

- Cool the stamp and the substrate to solidify the imprint material, hence fixing the nanopatterns on the silicon wafer.
- Gently separate the stamp from the substrate, leaving behind the nanopatterns on the wafer.

vi. **Post-Imprint Processing:**

- Optionally, carry out post-imprint processes such as curing the imprint material with UV light, cleaning the substrate, or etching to improve the definition of nanopatterns.

This is an example of how precise nanopatterns on the surface of a substrate can be realized using Nanoimprint Lithography, providing a cost-competitive and scalable production method for application areas such as semiconductor devices, optical components, and nanoelectronics.

[A Step-by-Step Look at Nanoimprint Lithography \(NIL\)](#)

2. **Atomic Layer Deposition (ALD):** ALD is one of the most precise thin-film deposition techniques, growing layer by layer through sequential chemical reactions. It allows bottom-up growth with atomic-level control, providing exact thickness and composition control. Applications of ALD are in the semiconductor and electronics industries for fabrication purposes, like in the formation of nanoscale coatings, barriers, and high-k dielectrics.

Detailed Process:

i. Substrate Preparation:

- Start with a clean substrate, typically a silicon wafer or glass slide, on which the thin film deposition will occur.

ii. Load Substrate into ALD Chamber:

- Place the substrate into the ALD chamber; this chamber is an ultrahigh vacuum environment equipped with precursor and deposition stages.

iii. Purge and Pump Down:

- Evacuate the ALD chamber to achieve a high vacuum by pumping out residual gases and impurities.
- Then, purge the chamber with inert gas (e.g., nitrogen) to remove any remaining traces of atmospheric gases.

iv. First Precursor Pulse:

- Introduce the first precursor gas into the chamber, which reacts with the substrate surface to form a monolayer of the desired material.
- For example, in depositing alumina (Al_2O_3), the first precursor could be trimethylaluminum (TMA) $[(\text{CH}_3)_3\text{Al}]$.

v. Purge and Remove Excess Precursor:

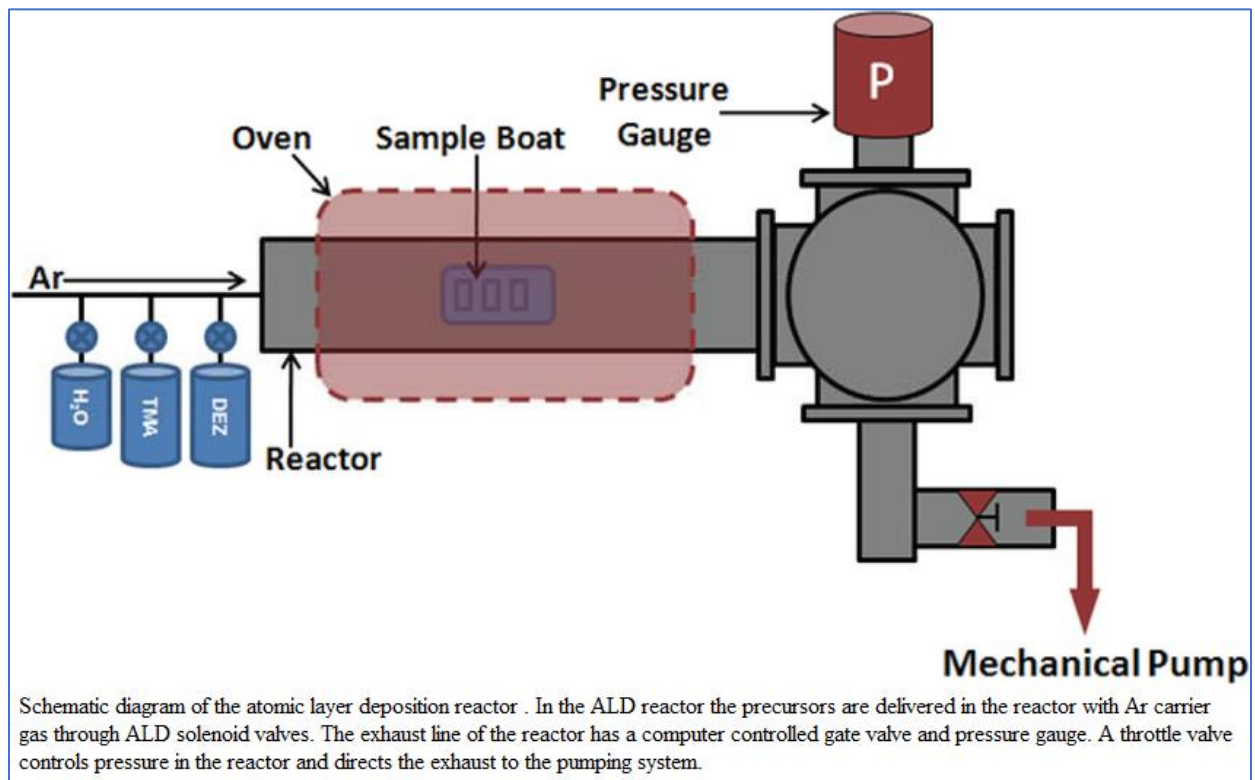
- After the first precursor pulse, purge the chamber again to remove excessive precursor gas and reaction by-products.
- This step ensures that only the desired monolayer is deposited on the substrate without contamination.

vi. Second Precursor Pulse:

- Introduce a second precursor gas, usually a reactive oxygen source like water vapor (H_2O) or ozone (O_3).
- Thin film deposition is completed when this precursor reacts with the monolayer that was created by the first precursor.

vii. Purge and Repeat Cycles:

- Until the thin layer reaches the required thickness, repeat the purging and precursor pulse processes several times.



viii. Post-Deposition Annealing (Optional):

- To enhance the crystallinity, density, and adhesion of the deposited thin film, consider post-deposition annealing.

Example Application: ALD Deposition of Alumina (Al₂O₃) Thin Film

i. Substrate Preparation:

- The substrate to be used for the deposition of alumina thin films by ALD has to be a clean silicon wafer.

ii. Load Substrate into ALD Chamber:

- Place the wafer made of silicon in the ALD chamber and close the chamber lid.

iii. Purge and Pump Down:

- Evacuation of the chamber to a high vacuum level and purging with an inert gas to remove the residual gases.

iv. First Precursor Pulse:

- Introduce the trimethylaluminum (TMA) gas into the chamber; it reacts with the silicon wafer surface to give a monolayer of aluminum.

v. Purge and Remove Excess Precursor:

- Excess TMA gas and reaction byproducts are then purged through the chamber to leave a uniform aluminum monolayer on the surface of the substrate.

vi. Second Precursor Pulse:

- Introduce water vapor (H₂O) into the chamber, and this will react with the aluminum monolayer to form a monolayer of alumina (Al₂O₃).

vii. Purge and Repeat Cycles:

- Excess water vapor from the reaction and all reaction byproducts were purged in the chamber, and then, the TMA-water vapor cycle was repeated a few more times to grow the alumina thin film up to the desired thickness.

viii. Post-Deposition Annealing (Optional):

- In some cases, the deposited alumina thin film may even be annealed to improve its properties, such as adhesion and crystallinity.
- This example illustrates that Atomic Layer Deposition (ALD) is capable of depositing a well-controlled, conformal, and uniform alumina (Al₂O₃) thin film on the substrate for various applications in microelectronics, optics, and surface coatings.

[A Step-by-Step Look at Atomic Layer Deposition \(ALD\)](#)

D. Examples of Fabrication Techniques for Each Type of Nanostructure

1. **Nanowires (Top-Down):** Among the latest and most advanced techniques in the world for the production of nanowires, electron-beam lithography (EBL) stands out. It is based on the ability of EBL to pattern trenches on the nanoscale.
2. **Graphene (Bottom-Up):** Graphene can be synthesized using CVD, where a metal catalyst, e.g., copper, is exposed to hydrocarbon gases at high temperatures, which will result in the growth of graphene on the substrate.
3. **DNA Nanotubes (Hybrid):** DNA nanotubes can be fabricated by a combination of nanoimprint lithography, patterning DNA origami template structures, and self-assembling DNA strands together to form nanotubes.

Comparison of Thin Film Deposition Techniques

1. Principle of Operation:

- **NIL:** Utilizes physical deformation of a stamp or mold to transfer patterns onto a substrate.
- **CVD:** Involves chemical reactions of precursor gases on a substrate surface to deposit thin films or coatings.
- **Self-Assembly:** Relies on the spontaneous organization of building blocks into ordered structures based on their inherent properties.
- **ALD:** Utilizes alternating cycles of precursor gas pulses to deposit thin films atom-by-atom in a layer-by-layer fashion.

2. Resolution and Feature Size:

- **NIL:** Offers high-resolution and nanoscale featured capabilities depend on the design of the stamp.
- **CVD:** Can achieve nanoscale thickness but may have limitations in defining precise lateral dimensions.
- **Self-Assembly:** Can produce nanoscale features but may have limited control over specific feature sizes and arrangements.
- **ALD:** Offers atomic-level control and can achieve precise nanoscale thickness and dimensions.

3. Materials Compatibility:

- **NIL:** Compatible with diverse classes of materials like polymers, metals, and semiconductors.
- **CVD:** Suitable for depositing thin films of various materials, such as metals, oxides, and nitrides.

- **Self-Assembly:** Suitable for organic and biomolecular materials but may have limitations with inorganic materials.
- **ALD:** Compatible with a wide range of materials, including oxides, nitrides, metals, and semiconductor materials.

4. Scalability and Throughput:

- **NIL:** Good for research and prototypes on a smaller scale but might not hold up well in mass production.
- **CVD:** Scalable for industrial production with relatively high throughput.
- **Self-Assembly:** Can be scalable for certain applications but may have throughput limitations.
- **ALD:** Suitable for both research and industrial-scale production with moderate throughput.

5. Cost and Equipment Complexity:

- **NIL:** Depending on the level of detail and size needed, moderately difficult and expensive technology may be necessary.
- **CVD:** Moderate to high equipment complexity and cost due to gas handling, vacuum systems, and precursor sources.
- **Self-Assembly:** Generally low equipment cost but may require specialized equipment for specific applications.
- **ALD:** Moderate to high equipment complexity and cost due to precise control of gas pulses, vacuum systems, and precursor sources.

6. Applications:

- **NIL:** Used in nanoelectronics, photonics, and biomimetic structures.
- **CVD:** Commonly used in semiconductor manufacturing, thin film coatings, and nanomaterial synthesis.
- **Self-Assembly:** Applied in nanotechnology, biomolecular engineering, and surface functionalization.
- **ALD:** Widely used in microelectronics, optics, protective coatings, and nanomaterial synthesis.

Feature	NIL	CVD	Self-Assembly	ALD
Principle of Operation	Physical deformation of a stamp/mold	Chemical reactions of precursor gases	Spontaneous organization of building blocks	Alternating gas pulses for atom-by-atom deposition
Resolution and Feature Size	High resolution, nanoscale features achievable	Nanoscale thickness, limitations in lateral dimensions	Nanoscale features, limited control over size/arrangement	Atomic-level control, precise nanoscale features
Materials Compatibility	Wide range (polymers, metals, semiconductors)	Metals, oxides, nitrides	Organic and biomolecular materials (limited inorganic)	Oxides, nitrides, metals, semiconductors
Scalability and Throughput	Limited for large-scale production	Scalable for industrial production	Scalable for certain applications (throughput limitations)	Suitable for research & industrial production (moderate throughput)
Cost and Equipment Complexity	Moderate (depends on scale/precision)	Moderate to high (gas handling, vacuum systems)	Generally low (may require specialized equipment)	Moderate to high (precise gas control, vacuum systems)
Applications	Nanoelectronics, photonics, biomimetic structures	Semiconductor manufacturing, thin film coatings, nanomaterials	Nanotechnology, biomolecular engineering, surface functionalization	Microelectronics, optics, protective coatings, nanomaterials

Thus, each process of fabrication has its advantages and limitations, which make them suitable for the requirements and applications of fabrication in nanostructure fabrication. The most suitable process, in terms of different factors of resolution, material compatibility, scalability, cost, and specific needs of applications, is considered for the choice of researchers and engineers.

VI. Characterization Techniques for Nanostructures:

A. Scanning Electron Microscopy (SEM):

- **Principle:** SEM uses a focused beam of electrons to scan the surface of a sample, generating high-resolution images.
- **Application:** SEM is used to visualize the morphology, size, and surface features of nanostructures.
- **Example:** An SEM image can reveal the nanoscale structure of carbon nanotubes, providing information about their diameter, length, and arrangement.

Step by Step Process:

1. Sample Preparation:

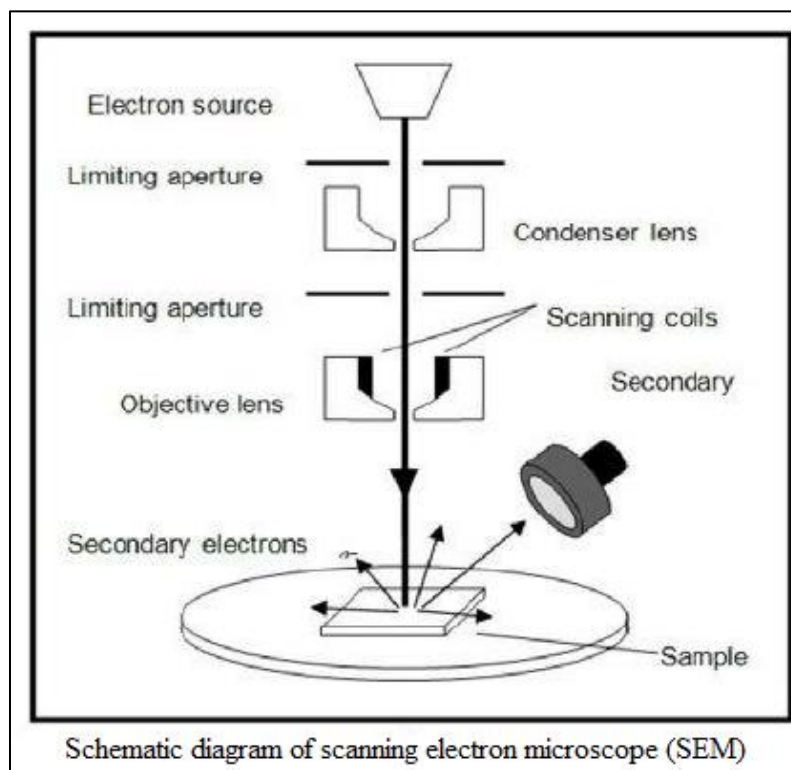
- Start by preparing the sample for SEM analysis. The sample to be analyzed is usually mounted on a metal stub using conductive adhesive or carbon tape.
- If the sample is of non-conductive nature, then it might have to be coated with a very thin layer of conductive material (e.g. gold, platinum) using techniques like sputter coating to avoid charging effects while imaging.

2. Chamber Preparation:

- Load the prepared sample into the SEM chamber and ensure proper vacuum conditions inside the chamber.
- Adjust the chamber settings, including vacuum level, beam energy, and aperture size, based on the sample type and desired imaging resolution.

3. Electron Beam Generation:

- Activate the electron beam source within the SEM, typically a tungsten filament or field emission gun (FEG), to generate a focused beam of electrons.
- Control the beam energy based on the sample's properties and desired imaging mode (e.g., low voltage for surface imaging, high voltage for deep imaging).



4. Sample Imaging:

- Position the electron beam to scan across the sample surface systematically in a raster pattern.
- As the electron beam interacts with the sample, various signals are emitted, including secondary electrons (SE), backscattered electrons (BSE), and characteristic X-rays.

5. Signal Detection and Image Formation:

- Use a suitable detector in an SEM, allowing the acquisition of different signals that might be given off from the sample:
 - Secondary Electron Detector: Captures secondary electrons emitted from the sample's surface, providing high-resolution images of surface morphology and topography.
 - Backscattered Electron Detector: Detects backscattered electrons, which are useful for imaging compositional variations and contrast differences in materials.
 - X-ray Energy Dispersive Spectroscopy (EDS) Detector: Detects characteristic X-rays emitted during electron-sample interactions, enabling elemental analysis and mapping.

6. Image Processing and Analysis:

- Acquire SEM images at various magnifications and angles to capture detailed information about the sample's structure, morphology, and composition.
- Use image processing software to enhance contrast, adjust brightness and sharpness, and annotate features of interest within the SEM images.
- Perform quantitative analysis, such as measuring particle sizes, surface roughness, and elemental composition using SEM-EDS data.

7. Interpretation and Reporting:

- Analyze the SEM images and interpret the analysis results to make inferences about the characteristics, properties, and structural features of the sample.
- Create a comprehensive report or presentation that provides a concise summary of the SEM observations, incorporating image annotations, measurements, and elemental mapping data.

Example Application:

- For instance, SEM analysis can image and characterize the surface morphology of synthesized nanoparticles for catalysis. It can display the particle size distribution, shape, aggregation state, and surface characteristics that can be very useful not only for understanding but also for optimization of catalytic activity.

[A Step-by-Step Look at Scanning Electron Microscopy \(SEM\)](#)

B. Transmission Electron Microscopy (TEM):

- **Principle:** TEM transmits electrons through a thin sample, forming an image based on electron interactions, offering high-resolution imaging.
- **Application:** TEM is used to examine internal structure, crystallography, and defects in nanostructures.
- **Example:** TEM can reveal the atomic arrangement and lattice defects in gold nanoparticles, aiding in understanding their crystalline structure.

Step by Step Process

1. Sample Preparation:

- Begin by preparing a thin sample for TEM analysis. Samples are typically ultra-thin sections (100 nm or less) obtained through techniques like ultramicrotomy or focused ion beam (FIB) milling.
- Mount the thin sample on a TEM grid, which is a small support grid made of materials like copper or gold with a thin carbon or copper film.

2. Chamber Preparation:

- Load the TEM grid with the mounted sample into the TEM instrument's vacuum chamber. Ensure that the chamber is under high vacuum conditions to prevent electron scattering and loss of resolution.
- Stabilize the sample and grid in the holder to minimize vibrations during imaging.

3. Electron Beam Generation:

- Activate the electron beam source within the TEM, typically a tungsten filament or field emission gun (FEG), to generate a focused beam of electrons.
- Adjust the beam energy (accelerating voltage) based on the sample's thickness and desired imaging resolution. Higher voltages are used for thicker samples, while lower voltages provide better resolution for thin samples.

4. Electron Beam Transmission and Sample Interaction:

- Direct the electron beam through the sample on the TEM grid. As the electrons pass through the sample, they interact with its atoms, leading to various scattering phenomena.
- Two primary types of interactions occur:
 - **Elastic Scattering:** Electrons are scattered by atomic nuclei without losing energy, providing information about sample structure and crystal lattice.

- Inelastic Scattering: Electrons lose energy due to interactions with sample electrons, resulting in phenomena like electron energy loss spectroscopy (EELS) and inelastic scattering imaging.

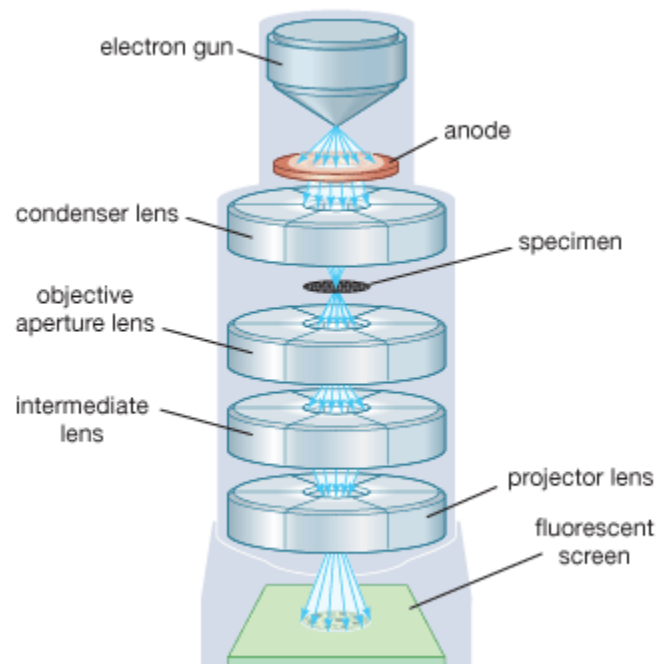
5. Imaging Modes and Contrast Mechanisms:

- Choose the appropriate imaging mode based on the desired contrast and information to be obtained:
 - Bright-Field Imaging (BF-TEM): Captures electrons transmitted through the sample, producing contrast based on variations in sample thickness, density, and composition.
 - Dark-Field Imaging (DF-TEM): Uses scattered electrons to form images, enhancing contrast for features like defects, interfaces, and strained regions.
 - High-Resolution TEM (HR-TEM): Provides detailed imaging of atomic structure and lattice spacing, suitable for studying crystallography and nanoscale features.



6. Image Formation and Recording:

- Use specialized detectors and lenses within the TEM instrument to capture transmitted electrons and form images on a fluorescent screen or digital camera.
- Acquire TEM images at various magnifications and beam conditions to capture different aspects of the sample's structure, including fine details and atomic arrangements.



7. Image Processing and Analysis:

- Process TEM images using software to enhance contrast, adjust brightness and sharpness, and annotate features of interest.

- Perform quantitative analysis, such as measuring lattice spacings, particle sizes, and crystal orientations, using TEM imaging and diffraction data.

8. Interpretation and Reporting:

- Interpret the TEM images, diffraction patterns, and analysis results to characterize the sample's structure, morphology, defects, and crystalline properties.
- Prepare a detailed report or presentation summarizing the TEM observations, including image annotations, measurements, and crystallographic information.

Example Application:

- An example of TEM analysis could involve studying the atomic structure and defects in semiconductor nanowires. TEM imaging would reveal the nanowire's diameter, length, crystal orientation, and any dislocations or defects present, aiding in understanding their electronic and optical properties.

[A Step-by-Step Look at Transmission Electron Microscopy \(TEM\)](#)

C. Atomic Force Microscopy (AFM):

- **Principle:** AFM uses a sharp probe to scan the surface of a sample, measuring forces between the probe and sample to create a topographic map.
- **Application:** AFM is used to visualize surface roughness, measure height variations, and characterize mechanical properties at the nanoscale.
- **Example:** AFM can map the surface roughness of graphene sheets, providing insights into their flatness and quality.

Step by Step Process

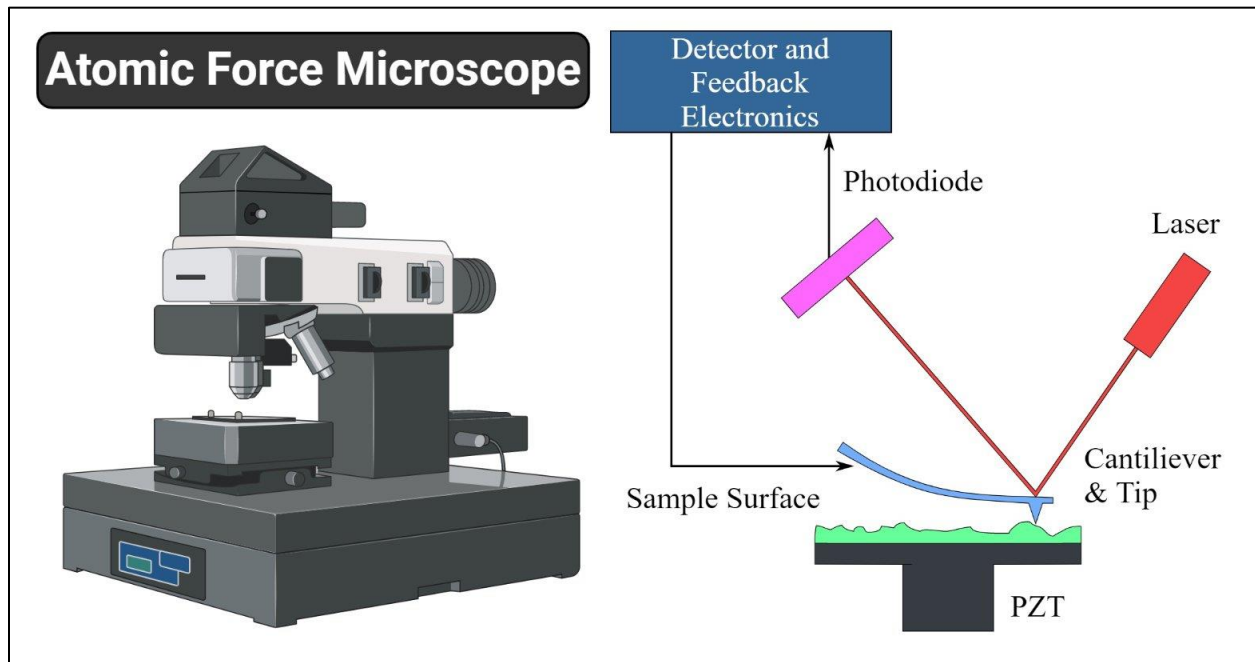
1. Sample Preparation:

- Let us start this sample preparation for setting up the AFM analysis. A sample can be, for instance, a thin film deposited on a flat substrate or biological particle solid mounted on a flat substrate.
- Ensure that the surface of the sample is clean and free from all contaminants, which may affect AFM measurements.

2. AFM Probe Selection:

- Choose the appropriate AFM probe (cantilever), depending on the sample chosen and the imaging mode. It comes in different sizes, shapes, and composition materials.

- Common probe types use silicon nitride tips; special types of probes are involved with uses for magnetic force microscopy or nanomechanical measurements.



3. AFM Instrument Setup:

- Affix the selected AFM probe to the scanning head of the AFM instrument. It has a flexible cantilever to which the probe is mounted.
- After calibration, calibrate the sensitivity of the AFM instrument, the spring constant of the cantilever, and the imaging parameters of the size and speed of the scan.

4. Approach and Contact:

- Place the AFM probe over the sample surface through piezoelectric actuators controlled by the AFM software.
- Lower the probe carefully to the surface of the sample and finally let the tip of the probe make light contact with the surface of the sample. Use feedback mechanisms through deflection of the cantilever or by optical interferometry for the onset of contact.

5. Surface Scanning and Imaging:

- Commence the surface scanning procedure by systematically moving the AFM probe across the sample surface in a grid-like pattern.

- As the AFM probe scans, it interacts with the sample surface, leading to deflection of the cantilever due to surface forces such as van der Waals, electrostatic, or magnetic interactions.

6. Tip-Sample Interaction Modes:

- AFM can operate in different imaging modes based on the type of surface interaction being probed:
 - Contact Mode AFM: The probe maintains constant contact with the sample surface during scanning, providing topographic images based on probe-sample deflection.
 - Tapping Mode AFM (Intermittent Contact): The probe oscillates near its resonant frequency, intermittently tapping the surface, reducing lateral forces and minimizing sample damage.
 - Non-contact Mode AFM: The probe hovers just above the sample surface, detecting forces without direct contact, suitable for imaging delicate samples or studying long-range forces.

7. Data Acquisition and Image Analysis:

- Capture AFM images with high-resolution topographic information about the sample surface, including features like height variations, roughness, and nanoscale structures.
- Use AFM software to process and analyze the acquired data, including image flattening, line profiles, surface roughness calculations, and three-dimensional (3D) rendering of surface topography.

8. Interpretation and Reporting:

- Interpret the AFM images and analysis results to characterize the sample's surface properties, nanoscale features, mechanical properties (e.g., stiffness), and interactions with the AFM probe.
- Prepare a detailed report or presentation summarizing the AFM observations, including image analysis results, measurements, and conclusions about the sample's structure and properties.

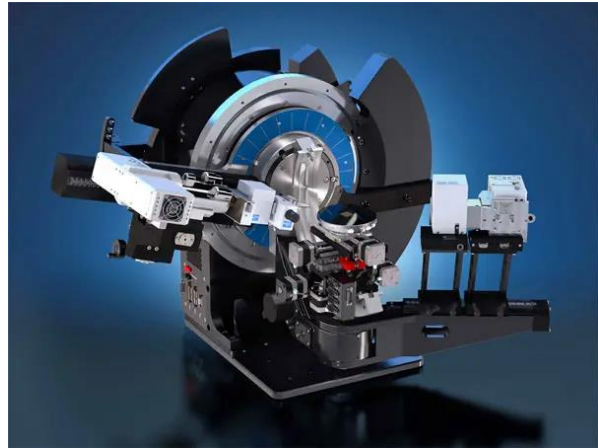
Example Application:

- An example of AFM analysis could involve imaging and characterizing the surface roughness and morphology of a graphene thin film. AFM images would reveal the atomic-scale structure, layer thickness, defects, and quality of the graphene layer.

[A Step-by-Step Look at Atomic Force Microscopy \(AFM\)](#)

D. X-ray Diffraction (XRD):

- **Principle:** XRD measures the diffraction pattern of X-rays interacting with a crystalline sample, providing information about crystal structure and phase composition.
- **Application:** XRD is used to determine crystallographic orientation, phase purity, and crystallite size in nanostructures.
- **Example:** XRD analysis can identify the crystal structure and phase transitions in semiconductor nanocrystals, aiding in material characterization.



Step by Step Process

1. Sample Preparation:

- Begin by preparing the sample for XRD analysis. The sample should be in a powdered form or a thin film to allow X-rays to penetrate and interact with the crystal lattice.
- Ensure the sample is finely ground and homogenized to eliminate grain boundaries and obtain a representative bulk structure.

2. Instrument Setup:

- Load the prepared sample onto a sample holder or mount it on a flat substrate compatible with the XRD instrument.
- Align the sample holder within the XRD instrument's goniometer stage, which positions the sample at specific angles for X-ray analysis.

3. X-ray Source and Beam Generation:

- Activate the X-ray source within the XRD instrument, typically a sealed tube or rotating anode generator, to produce a monochromatic X-ray beam.
- Filter and collimate the X-ray beam to ensure a narrow and well-defined wavelength suitable for diffraction experiments.

4. Bragg's Law and Diffraction Angle Calculation:

- X-ray diffraction analysis is based on Bragg's Law, which states that monochromatic X-rays are diffracted in such a manner that they interfere constructively to form the diffraction peaks at an angle when they impact the crystal lattice of the studied material.

- Calculate the diffraction angle (θ) using Bragg's Law: $2d\sin(\theta) = n\lambda$, wherein d is the lattice spacing, λ is the X-ray wavelength, and n is the order of the obtained diffraction.

5. Sample Rotation and Data Collection:

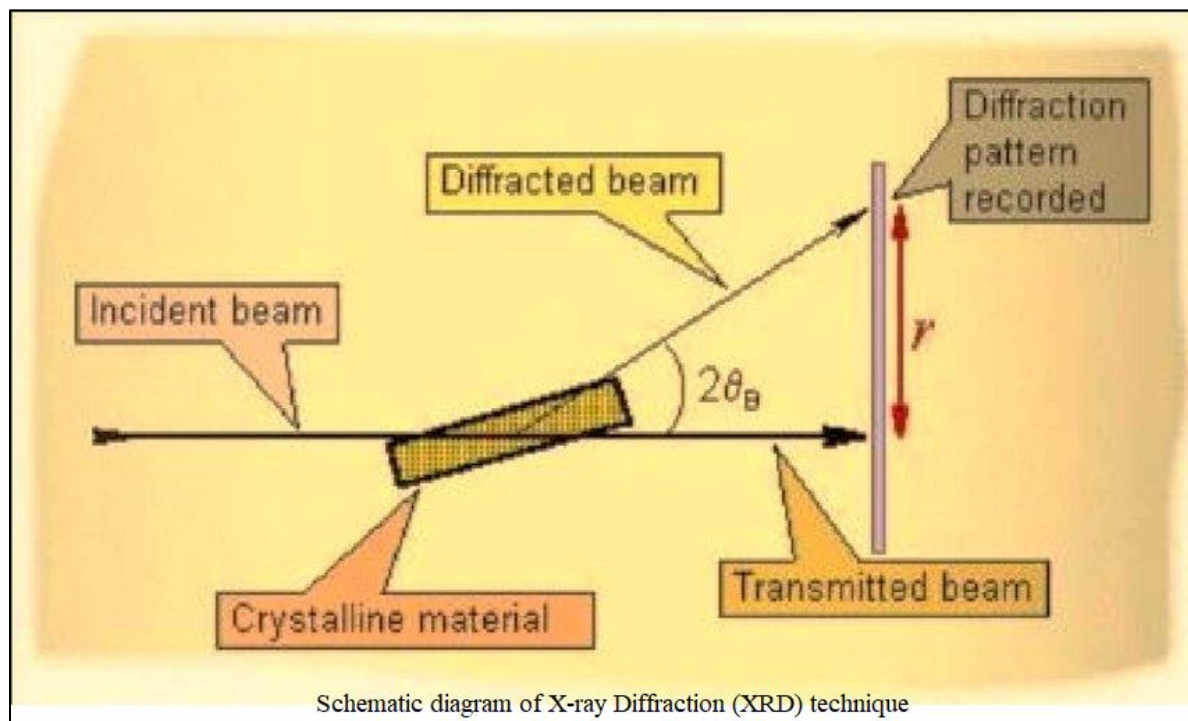
- Rotate the sample holder at varying angles (θ) using the goniometer stage while the X-ray beam interacts with the sample.
- Collect diffraction data by measuring the intensity of diffracted X-rays at different angles (2θ) using a detector, such as a scintillation counter or semiconductor detector.

6. Diffraction Pattern Analysis:

- Analyze the collected diffraction data with the aim to find characteristic diffraction peaks representing the crystal planes and lattice structure in the collected sample.
- Determine the position, intensity, and width of the diffraction peaks that provide information related to crystallography orientation, phase composition, lattice parameters, and crystallite size.

7. Data Interpretation and Structural Analysis:

- Analyze the X-ray diffraction pattern to determine the crystal structure, phase purity, crystal symmetry, and preferred orientation of the sample.
- Therefore, quantitative analysis should be carried out for peak fitting, Rietveld refinement, and lattice parameter calculations, allowing further extraction of more detailed structural information from the diffraction data.



8. Reporting and Applications:

- Prepare a detailed report or presentation with an overview of results received from XRD analysis, including diffraction patterns, peak positions, crystallography data, and other structural parameters.
- The wide applications of XRD analysis in materials science, geology, chemistry, and pharmaceuticals include phase identification, crystallographic texture analysis, quantitative phase analysis, grain size analysis, and identification of phases.

Example Application:

- For example, it is possible to use XRD analysis for the characterization of a ceramic with respect to its crystal structure and phase composition. The characteristic peaks on the XRD diffraction patterns of different crystallographic planes of various phases present in this ceramic sample are used during the process of characterization in material quality control.

[A Step by Step look at X-ray Diffraction \(XRD\)](#)

Comparison of Characterization Techniques

Technique	Principle	Application	Example
Scanning Electron Microscopy (SEM)	Uses a focused electron beam to scan the sample surface, generating high-resolution images.	Visualize morphology, size, and surface features.	Reveals nanoscale structure of carbon nanotubes (diameter, length, arrangement).
Transmission Electron Microscopy (TEM)	Transmits electrons through a thin sample, forming an image based on electron interactions.	Examine internal structure, crystallography, and defects.	Reveals atomic arrangement and lattice defects in gold nanoparticles (crystalline structure).
Atomic Force Microscopy (AFM)	Uses a sharp probe to scan the sample surface, measuring forces to create a topographic map.	Visualize surface roughness, measure height variations, and characterize mechanical properties.	Maps surface roughness of graphene sheets (flatness, quality).
X-ray Diffraction (XRD)	Measures the diffraction pattern of X-rays interacting with a crystalline sample.	Determine crystal structure, phase composition, and crystallite size.	Identifies crystal structure and phase transitions in semiconductor nanocrystals (material characterization).

E. Examples of How Each Technique is Used:

- **SEM:** Characterizing the surface morphology of nanowires, nanoparticles, and thin films.
- **TEM:** Investigating the internal structure and defects in nanomaterials like quantum dots and nanotubes.
- **AFM:** Measuring the thickness and mechanical properties of nanomembranes, polymers, and biological structures.
- **XRD:** Determining the crystal structure and phase transformations in nanocrystalline materials such as metal oxides and nanocomposites.

Questions and Answers

- ❖ Q: What is a nanostructure?
A: A nanostructure is a structure with dimensions in the nanometer range, typically between 1 to 100 nanometers.
- ❖ Q: Name a fabrication technique used for creating nanostructures by physical deformation.
A: Nanoimprint Lithography (NIL).
- ❖ Q: What is the primary principle behind Chemical Vapor Deposition (CVD)?
A: CVD involves chemical reactions of precursor gases on a substrate surface to deposit thin films or coatings.
- ❖ Q: Which characterization technique is suitable for studying internal structure and defects in nanomaterials?
A: Transmission Electron Microscopy (TEM).
- ❖ Q: What is the function of a cantilever in Atomic Force Microscopy (AFM)?
A: The cantilever in AFM is used to detect surface forces and produce high-resolution images of sample topography.
- ❖ Q: Define Bragg's Law as applied in X-ray Diffraction (XRD).
A: Bragg's Law relates the diffraction angle to the spacing between crystal lattice planes and the wavelength of X-rays.
- ❖ Q: What is the primary purpose of Self-Assembly in nanostructure fabrication?
A: Self-Assembly is used to spontaneously organize building blocks into ordered structures based on their properties.

- ❖ Q: Name a common top-down fabrication method for nanostructures.
A: Electron Beam Lithography (EBL).
- ❖ Q: What type of microscopy is suitable for visualizing surface morphology and topography at the nanoscale?
A: Scanning Electron Microscopy (SEM).
- ❖ Q: Which technique is used to deposit thin films atom-by-atom in a layer-by-layer fashion?
A: Atomic Layer Deposition (ALD).
- ❖ Q: How does XRD help determine crystallographic orientation in materials?
A: XRD measures diffraction patterns to identify crystal planes and their orientation relative to the incident X-ray beam.
- ❖ Q: What is the primary advantage of using AFM over optical microscopy for imaging nanoscale features?
A: AFM offers higher resolution and the ability to image surfaces in three dimensions.
- ❖ Q: Name a hybrid fabrication approach combining top-down and bottom-up methods.
A: Nanoparticle-assisted Lithography (NAL).
- ❖ Q: Which technique is suitable for analyzing surface roughness and mechanical properties at the nanoscale?
A: Atomic Force Microscopy (AFM).
- ❖ Q: What is the primary application of X-ray Diffraction (XRD) in materials science?
A: XRD is used for phase identification, crystallographic texture analysis, and quantitative phase analysis in materials.
- ❖ Q: Which microscopy technique uses secondary electrons to produce high-resolution images of sample surfaces?
A: Scanning Electron Microscopy (SEM).
- ❖ Q: Name a common bottom-up fabrication method for nanostructures.
A: Self-Assembly.
- ❖ Q: What is the purpose of post-deposition annealing in thin film fabrication?
A: To improve the crystallinity, density, and adhesion of deposited thin films.
- ❖ Q: Which characterization technique is suitable for studying surface roughness and height variations at the nanoscale?
A: Atomic Force Microscopy (AFM).

- ❖ Q: What is the primary advantage of ALD in depositing thin films compared to other techniques?
A: ALD offers precise control over film thickness and uniformity at the atomic level.
- ❖ Q: How does TEM differ from SEM in terms of sample interaction and imaging?
A: TEM transmits electrons through a thin sample for internal imaging, while SEM uses secondary electrons for surface imaging.
- ❖ Q: Name a technique used for creating nanoscale patterns on surfaces using physical deformation.
A: Nanoimprint Lithography (NIL).
- ❖ Q: What information can be obtained from XRD diffraction patterns?
A: Crystal structure, phase composition, lattice parameters, and crystallite size.
- ❖ Q: Which technique is suitable for high-resolution imaging of atomic-scale features and lattice spacings?
A: High-Resolution Transmission Electron Microscopy (HR-TEM).
- ❖ Q: What is the primary function of a sputter coating in SEM analysis?
A: To deposit a thin conductive layer on non-conductive samples to prevent charging effects during imaging.
- ❖ Q: Name a technique used for creating nanowires and nanorods through chemical reactions on a substrate surface.
A: Chemical Vapor Deposition (CVD).
- ❖ Q: How does SEM differ from TEM in terms of sample preparation and imaging mode? A: SEM and TEM have similar operating modes, and they both have the same requirements in terms of electron sources and beams. However, they differ in imaging mode and sample preparation. In SEM, the sample calls for the use of conductive coating since surface imaging is needed, while the TEM requires the use of a thin sample for internal imaging.
- ❖ Q: What is the primary application of AFM in nanotechnology?
A: AFM techniques are useful for surface property research, nanomaterial characterization, and nanomechanical measurement.
- ❖ Q: Name a technique used for precise control of film thickness at the atomic level.
A: Atomic Layer Deposition (ALD).

- ❖ Q: What is the primary advantage of using XRD for materials characterization?

A: XRD is used to extract detailed crystal structures, phase composition, and atomic arrangements within materials.

- Explain the concept of nanostructures and their significance in modern technology.

Nanostructures are a structure in which at least one of its dimensions is in the size between 1 to 100 nanometers. These properties at the nanoscale have made this an indispensable component of modern technology, thanks to their unique characteristics like quantum effects, high surface area-volume ratio, and enhanced mechanical, optical, and electrical properties. Such nanostructures are of supreme importance to many fields, including nanoelectronics, photonics, catalysis, biomedicine, and energy storage.

- Discuss the difference between top-down and bottom-up fabrication methods for nanostructures. Provide examples of each.

Top-down fabrication involves shrinking bulk materials to create nanostructures. There are various techniques that can be used to achieve this, including lithography methods like electron beam lithography. Utilizing bottom-up fabrication techniques involves the meticulous assembly of atoms or molecules to create intricate nanostructures. Some examples of these technologies include self-assembly and chemical vapor deposition (CVD). Nanoimprint lithography (NIL) is a form of top-down fabrication, while the self-assembly of nanoparticles showcases bottom-up fabrication.

- Explain the principle of operation and application of Transmission Electron Microscopy (TEM) in nanotechnology.

TEM functions simply through the beaming of electrons through a thin sample to form the image. It may be used to study the internal structure, defects, crystallography, and features of materials at the nanometer level. Therefore, the TEM provides specific information on atomic arrangements, lattice parameters, or even phase composition, things which are mostly required during the characterization of such type of materials.

- Describe the steps involved in Atomic Force Microscopy (AFM) for imaging nanoscale features.

The procedure followed in imaging with AFM involves sample preparation, probe selection, instrument setup, approach, and contact with the sample surface, sample scanning using the cantilever, surface force detection, data acquisition, and, finally, image processing and data analysis. AFM is used to visualize surface topography, determine surface roughness, and characterize mechanical properties of surfaces at the nanometer level.

- Discuss the significance of X-ray Diffraction (XRD) in materials characterization and provide an example of its application.

XRD turns into vital in some applications that involve the identification of crystal structure, phase composition, lattice parameters, and size of crystallite in materials. This is applied to a wide range of material science, geology, chemistry, and pharmaceuticals. For example, it may be used to identify the crystal phases of nanoparticle semiconductor in thin films and be used for quantitative studies of crystallographic texture to gain insight into material attributes and performance.

- Explain the fabrication process of nanowires using Chemical Vapor Deposition (CVD) and discuss its applications.

In the CVD process, nanowires form from the chemical reaction of precursor gases when applied to the substrate. Among the different ways of nanowire formation in synthesis, one includes the CVD process of forming nanomaterials. All these point to the fact that the potential for the development of versatile applications in the area of nanoelectronics, sensors, catalysis, and synthesis of different types of nanomaterials is immense. For example, the CVD technique has been used to grow semiconductor nanowires for electronic device applications, among others. They range from the renewable energy source to the creation of nanocomposites for innovative materials.

- Compare and contrast Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) in terms of sample preparation, imaging mode, and applications.

SEM provides the imaging of surfaces using a conductive coating for non-conductive samples, while TEM provides the imaging of internal structures of samples thin enough to transmit electrons. The possibility of checking the internal structure, crystallography, and defects of internal structure was given by the TEM technique. In SEM, information is given for the surface or morphology while in TEM, it provides the information on the morphology structure for the internal or bulk structure and crystallography. Both of these techniques are quite indispensable to the characterization of the nanostructure and material in the subject of nanotechnology.

- Discuss the advantages and limitations of Atomic Layer Deposition (ALD) in thin film fabrication. Provide examples of ALD applications.

ALD provides atomic-level precise thickness, uniformity, and conformality but is a slow deposition process compared to others. ALD is of wide use in many applications, but not limited to, those that consist of microelectronics, optics, protective coatings, and syntheses of nanomaterials. For example, ALD has been used in the deposition of thin films in the semiconductor devices and growth of nanocomposites with controlled properties.

- Explain the concept of self-assembly in nanostructure fabrication and provide examples of self-assembled structures.

Self-assembly is a spontaneous organization of building blocks into ordered structures due to properties such as molecular forces or surface interactions. Self-assembly has been used to create functional materials, sensors, and nano-devices, for instance, in self-assembled monolayers (SAMs), in assemblies of nanoparticles, or in structures made of DNA.

- Discuss the importance of characterization techniques such as AFM, XRD, and SEM in nanotechnology research. Provide specific examples of their applications.

AFM plays an important role in imaging characteristics with dimensions of a nanometer, and it allows probing the surface properties of the materials, including the measurement of the roughness of thin films. On the other hand, XRD assumes critical importance in the case of nanomaterials for the analysis of the crystal structure and phase; it helps in the identification of the polymorph in nanoparticles. SEM is handy in visualizing surface morphology and topography, with a good example being the study of the occurrence of nanoparticle aggregates. It is very beneficial to the development of nanotechnology and materials science.

- Describe the concept of lithography in nanostructure fabrication. Explain the difference between photolithography and electron beam lithography.

Lithography is the technology of under patterning surfaces on a nanoscale, including both surface mask and source radiation technologies. Photolithography is patterned transfer technology to the substrate and is suitable for mass production. Another high-resolution, high-quality photoresist-patterning suitable technology for fine and complex nanostructures is electron beam lithography (EBL).

- Explain the role of sputter coating in Scanning Electron Microscopy (SEM) analysis. How does sputter coating affect sample imaging?

Sputter coating deposits a fine conductive layer on non-conducting samples to avoid sample charging during the SEM analyses. It also improves the conductivity of the samples, thus helping in decreasing image distortion for better contrast enhancement and high-resolution imaging of the surfaces under study.

- Discuss the application of Self-Assembly in the fabrication of nanocomposites. Provide an example of a self-assembled nanocomposite and its properties.

Nanocomposites with controlled structures and properties can be fabricated by self-assembly. For example, graphene oxide sheet-reinforced polymer matrix can be applied in self-assembly, and its mechanical strength, electrical conductivity, and barrier properties can be increased in the nanocomposite.

- Explain the working principle of High-Resolution Transmission Electron Microscopy (HR-TEM) and its advantages in imaging nanomaterials.

HR-TEM is a technique for imaging by the transmission of electrons through a sample at atomic resolution. The method gives full information on atomic arrangements, crystal defects, and surface structures and is thus of great value in the research of nanomaterials, catalysts, and semiconductor devices.

- Discuss the fabrication process of nanodots using top-down lithography techniques. Explain the challenges and advantages of nanodot fabrication.

Techniques including electron beam lithography (EBL) for patterning dots on a substrate make the nanodots. These would display uniform size and distribution, enabling easy and simple control of the dimension of the dots as well as the arrangement suitable for applications of quantum dots and data storage devices.

- Describe the operation of Energy Dispersive Spectroscopy (EDS) in Scanning Electron Microscopy (SEM). How is EDS used for elemental analysis in nanomaterials?

EDS detects characteristic X-rays emitted from sample interactions between conduction band electrons during SEM. It provides data on the elemental composition from the X-ray spectra and, therefore, assists in identification of contained elements in the nanomaterial, stoichiometry development, and mapping of the elements in the samples.

- Explain the principles of Contact Mode and Tapping Mode Atomic Force Microscopy (AFM). Compare their advantages and disadvantages in imaging biological samples.

Contact Mode AFM maintains constant contact with samples, offering high-resolution imaging but risking sample damage. Tapping Mode AFM oscillates the probe, reducing lateral forces and preserving sample integrity. Tapping Mode is preferable for imaging soft and biological samples due to minimal force exertion and reduced sample deformation.

- Discuss the fabrication process of nanorods using Chemical Vapor Deposition (CVD). Explain how CVD parameters influence nanorod growth and properties.

Nanorods are synthesized via CVD by controlling precursor gas flow, temperature, and substrate properties. CVD parameters affect nanorod size, aspect ratio, crystallinity, and surface morphology, impacting their optical, electronic, and catalytic properties in applications such as sensors and photovoltaics.

- Explain the mechanism of electron diffraction in Transmission Electron Microscopy (TEM). How is electron diffraction used to analyze crystal structures in nanomaterials?

Electron diffraction occurs when electrons interact with crystal lattices, leading to diffraction patterns that reveal crystal symmetry, lattice parameters, and orientation in nanomaterials. TEM uses electron diffraction for crystallographic analysis, identifying phases, defects, and grain boundaries in nanoparticles and thin films.

- Discuss the advantages of Hybrid Approaches in nanostructure fabrication. Provide an example of a hybrid approach and its applications.

Hybrid Approaches combine top-down and bottom-up methods, offering versatility, precision, and scalability in nanostructure fabrication. An example is Nanoimprint Lithography (NIL) combined with self-assembly to create ordered nanostructures for photonic devices, biosensors, and nanoelectronics, showcasing enhanced performance and functionality.

- Explain the concept of surface area-to-volume ratio in nanostructures. How does this ratio affect the properties and applications of nanostructured materials?

The surface area-to-volume ratio increases significantly as the size of a material decreases to the nanoscale. This increased ratio results in enhanced surface interactions, reactivity, and properties such as catalytic activity, sensitivity, and surface plasmon resonance, making nanostructured materials suitable for applications in catalysis, sensors, and biomedical devices.

- Describe the principles and applications of Dynamic Light Scattering (DLS) in nanoparticle characterization. How does DLS measure particle size in suspensions?

DLS measures particle size in suspensions by analyzing fluctuations in scattered light caused by Brownian motion. It provides information about nanoparticle size distribution, aggregation, and stability, making it valuable for studying colloidal systems, drug delivery carriers, and biomolecular interactions in nanomedicine.

- Discuss the role of nanowires in nanoelectronics and photonics. Provide examples of nanowire-based devices and their applications.

Nanowires are used in nanoelectronics as building blocks for transistors, sensors, and memory devices due to their high aspect ratio, conductivity, and tunable electronic properties. In photonics, nanowires enable light manipulation and energy conversion in devices such as nanolasers, photodetectors, and solar cells, offering advantages in miniaturization, efficiency, and integration.

- Explain the concept of strain engineering in nanomaterials. How can strain engineering enhance material properties and device performance?

Strain engineering involves intentionally introducing mechanical strain into nanomaterials to modify their electronic, optical, and mechanical properties. This technique can enhance carrier mobility, bandgap engineering, and device performance in semiconductor nanowires, quantum dots, and 2D materials, leading to improved device efficiency and functionality.

- Describe the working principle and applications of Scanning Tunneling Microscopy (STM) in nanotechnology. How does STM image atomic-scale features on surfaces?

STM operates by scanning a sharp metal probe over a sample surface, measuring tunneling current between the probe and surface atoms. It images atomic-scale features, defects, and electronic states on conductive surfaces, enabling studies of surface chemistry, adsorption, and surface reconstructions in nanomaterials, catalysis, and surface science research.

- Discuss the concept of Quantum Confinement in nanostructures. How does Quantum Confinement affect electronic and optical properties in semiconductor nanoparticles?

Quantum Confinement occurs when electron wave functions are confined within nanoscale dimensions, leading to quantized energy levels and size-dependent properties. In semiconductor nanoparticles, Quantum Confinement alters bandgap energy, exciton dynamics, and optical absorption, influencing device performance in photodetectors, quantum dots, and light-emitting diodes (LEDs).

- Explain the significance of bandgap engineering in semiconductor nanostructures. How can bandgap engineering be utilized to design optoelectronic devices with tailored properties?

Bandgap engineering involves modifying the energy bandgap of semiconductor materials through size, composition, or strain engineering. It allows the design of optoelectronic devices with specific emission wavelengths, absorption spectra, and electronic properties,

enabling applications in lasers, photovoltaics, and light-emitting devices (LEDs) with enhanced efficiency and performance.

- Describe the working principle and applications of Raman Spectroscopy in nanomaterial characterization. How does Raman Spectroscopy provide structural and chemical information in nanoscale systems?

Raman Spectroscopy analyzes scattered light from molecular vibrations to provide structural, chemical, and compositional information in nanomaterials. It identifies functional groups, defects, and crystalline phases, making it valuable for studying carbon nanotubes, graphene, and nanocomposites in materials science, chemistry, and biomedicine.

- Discuss the challenges and advantages of scaling down materials to the nanoscale. How does nanoscale confinement impact material properties and behavior?

Scaling down materials to the nanoscale poses challenges such as size-dependent properties, surface effects, and fabrication complexity, but it offers advantages like enhanced reactivity, quantum effects, and novel functionalities. Nanoscale confinement alters electronic, mechanical, and thermal properties, influencing material behavior and applications in nanotechnology, energy, and healthcare.

- Explain the concept of Quantum Dots and their applications in optoelectronics. How do Quantum Dots emit light, and what properties make them suitable for displays and imaging?

Quantum Dots are semiconductor nanoparticles with size-tunable optical properties due to quantum confinement effects. They emit light through bandgap recombination, enabling applications in displays, LEDs, and biomedical imaging with advantages such as narrow emission spectra, high brightness, and photostability, contributing to advances in lighting technology and medical diagnostics.

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