

NANOMATERIALS, METAMATERIALS, AND SMART MATERIALS: SYNTHESIS AND CHARACTERIZATION

Kamal I. M. Al-Malah

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Nanomaterials, Metamaterials, and Smart Materials: Synthesis and Characterization

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PREFACE

The rapid evolution of materials science has propelled us into an era where nanomaterials, metamaterials, and smart materials are becoming increasingly integral to technological advancement. These materials, with their extraordinary properties and abilities to interact with physical stimuli in novel ways, have reshaped our understanding of physics, chemistry, and engineering. As applications span from telecommunications to medicine, defense, energy, and environmental systems, the need for a comprehensive guide on their synthesis and characterization has never been more crucial.

This book, "**Nanomaterials, Metamaterials, and Smart Materials: Synthesis and Characterization**", aims to provide a detailed, interdisciplinary exploration of these exciting materials. It is structured to offer both a foundational understanding for newcomers and advanced insights for experienced researchers, with each chapter carefully designed to navigate the complex landscape of material synthesis, properties, and applications.

Starting with historical background and fundamental topics, the book builds a strong foundation, exploring electron arrangements, chemical bonding, and key material properties. It then delves into the quantum mechanics and solid-state physics that underpin the behavior of nano- and advanced materials, providing readers with the essential knowledge to understand the unique properties of these systems.

The subsequent chapters cover synthesis methods, from top-down to bottom-up approaches, as well as various characterization techniques used to probe the structural, mechanical, electrical, and optical properties of these materials. Special emphasis is placed on the challenges faced during the synthesis and integration of carbon nanostructures and their surface functionalization, offering practical insights into real-world applications and considerations.

The section on metamaterials introduces fascinating concepts such as negative index materials, cloaking, and perfect absorption. It guides readers through design principles, fabrication techniques, and applications across various fields. Additionally, the book explores smart materials, covering shape memory alloys, piezoelectric materials, hydrogels, electrochromic and thermochromic systems, and their potential uses in creating adaptive, responsive technologies.

By integrating theoretical concepts with experimental techniques and practical applications, this book serves as a valuable resource for students, researchers, engineers, and industry professionals alike. It encourages an appreciation of the interdisciplinary nature of this field and fosters a deeper understanding of the potential that nanomaterials, metamaterials, and smart materials hold for shaping the future.

I hope that this book will not only serve as an educational tool but also inspire future innovation in this exciting field. The journey through these pages will reveal the profound ways in which these materials can transform our world, one layer at a time.

The book is meant to be a textbook for non-specialist readers who would like to explore the world of non-conventional materials. It has the following four features:

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1. Pre-requisite definitions/concepts are highlighted whenever needed.
2. Relevant YouTube video links are selected for every synthesis characterization method to simplify or demonstrate the synthesis method/characterization method under focus.
3. For each written chapter, there is a large set of end-of-chapter problems in the form of multiple-choice questions and numerical (essay) problems. The end-of-chapter problems will help the course instructor by providing a large database of questions, and at the same time, the student will be tempted by such questions to gauge his/her level of comprehension.
4. Abundant citations of relevant literature.

Kamal I. M. Al-Malah

CHAPTER 1

Introduction to Nano- and Advanced Materials

HISTORICAL BACKGROUND

Nano- and advanced materials have a rich historical background that showcases the evolution of scientific understanding and technological progress. The development of these materials can be traced back to ancient civilizations, where humans began manipulating materials to enhance their properties and functionalities.

In ancient Egypt, for example, craftsmen developed methods to produce and utilize pigments with unique optical properties, such as the vibrant blue color of Egyptian blue, which was derived from a mixture of silica, copper, and calcium compounds. Similarly, in ancient Rome, the art of glassmaking led to the production of intricate glassware with enhanced mechanical strength and optical clarity.

During the Middle Ages, alchemists and metallurgists made significant contributions to the development of advanced materials. They experimented with various metal alloys, such as bronze and steel, to create materials with improved mechanical properties and durability. The discovery and refinement of techniques for producing iron and steel played a pivotal role in shaping the course of human civilization.

The Renaissance period witnessed remarkable advancements in materials science and engineering. Innovators like Leonardo da Vinci explored the mechanical properties of materials and invented new tools and machines. The emergence of the scientific method and the establishment of scientific societies further fueled the exploration and understanding of materials.

The Industrial Revolution, which began in the late 18th century, marked a turning point in the development of advanced materials. The invention of the steam engine and the subsequent boom in industrial manufacturing demanded materials that could withstand high temperatures, pressures, and stresses. This led to advancements in metallurgy, with the discovery of new alloys and heat treatment processes.

In the late 19th and early 20th centuries, the field of chemistry experienced significant growth and contributed to the development of advanced materials. Chemists discovered synthetic polymers, such as bakelite, formica, and nylon, which revolutionized industries such as plastics and textiles. These synthetic materials offered enhanced mechanical properties, versatility, and durability compared to their natural counterparts.

The advent of nanoscience and nanotechnology in the late 20th century brought about a paradigm shift in materials research. Scientists began exploring the unique properties of materials at the nanoscale, where quantum effects and surface phenomena dominate. This led to the emergence of nanomaterials as a distinct class of materials with exceptional properties and functionalities.

In the 1960s, the development of nanotechnology allowed for the production of nanomaterials, which are materials that have at least one dimension in the nanoscale (1-100nm). These materials have unique properties, such as high strength, high electrical conductivity, and high thermal conductivity, which make them attractive for use in a variety of applications.

The discovery of fullerenes in 1985 and subsequent advancements in nanoscale fabrication techniques, such as carbon nanotube synthesis and graphene isolation, opened up new possibilities for designing and engineering materials with unprecedented properties. Researchers began harnessing the potential of nanomaterials in various fields, including electronics, energy, medicine, and environmental remediation.

The historical journey of advanced materials and nanomaterials showcases the human quest for understanding and manipulating matter. From ancient civilizations to the modern era, the development of these materials has transformed various industries and paved the way for innovative technologies that shape our world today. As researchers continue to push the boundaries of materials science, the future holds exciting possibilities for even more advanced and transformative materials.

In recent years, the development of advanced materials and nanomaterials has continued to accelerate due to increased demand for new and improved products, as well as advances in technology. Examples of advanced materials and nanomaterials include carbon nanotubes, graphene, and metal-organic frameworks. These materials have been used to create products with improved properties, such as increased strength, electrical conductivity, and thermal conductivity.

Today, nanomaterials continue to drive advancements in materials science and technology. Scientists are exploring novel synthesis methods, such as bottom-up self-assembly and top-down lithography, to create nanomaterials with precise control over their size, shape, and composition. By manipulating these parameters, researchers can tailor the properties of nanomaterials to meet specific application requirements.

Baig *et al.* [1] explained that nanomaterials have emerged as an incredible group of materials encompassing a wide range of examples with at least one dimension measuring between 1 and 100 nm (or 0.1 μm). By employing a well-thought-out design, nanomaterials can achieve remarkably high surface areas. These materials possess exceptional magnetic, electrical, optical, mechanical, and catalytic properties that differ significantly from their bulk counterparts. It is possible to tailor the properties of nanomaterials according to specific requirements by precisely controlling factors such as size, shape, synthesis conditions, and appropriate functionalization. Their review specifically focused on advancements in nanomaterials such as fullerenes, carbon nanotubes, graphene, carbon quantum dots, nanodiamonds, carbon nano-horns, nano-porous materials, core-shell nanoparticles, silicene, antimonene, MXenes, 2D Metal-Organic Framework (MOF) nanosheets, boron nitride nanosheets, layered double hydroxides, and metal-based nanomaterials.

The development of advanced materials and nanomaterials is expected to continue to accelerate in the future due to increased demand and advances in technology. This will lead to the development of new and improved products with unique properties, such as increased strength, electrical conductivity, and thermal conductivity.

INTRODUCTORY TOPICS

The following outlined topics represent the basic knowledge needed to explore the world of nanotechnology or nanoscale materials in terms of knowing their behaviors as microscopic (tiny) or nanoscopic (extremely tiny) objects.

Electron Arrangement

Electrons whizz around the nucleus in “shells” at specific energy levels. In nanomaterials, where size plays a huge role, these electron arrangements become even more critical. Electrons in the outermost shell (valence electrons) are responsible for how atoms interact with each other through chemical bonds. By manipulating the size and structure of nanomaterials, we can control how these valence electrons interact, leading to unique properties. For example, gold at the bulk scale is a shiny yellow metal. However, at the nanoscale (as nanoparticles),

Carbon Nanostructures

PREREQUISITE DEFINITIONS

Work Function

The work function is essentially the energy required to remove an electron from a solid surface to a point just above the surface in a vacuum. It serves as a measure of how tightly the electrons are bound to the material. Let us imagine that we have a solid material exposed to a vacuum. Each electron within the material is at a certain energy level, represented by the Fermi level. The work function acts as a barrier to the escape of electrons from the material. If the work function is high, it means that a significant amount of energy is needed to remove an electron. Here are more details on the work function:

- **Positive Work Function as a Barrier:** A positive work function represents a barrier to the potential energy an electron can acquire above the Fermi level of the material. In simpler terms, it is like a hurdle that an electron needs to overcome to escape from the material into the vacuum.
- **Kinetic Energy of Emitted Electron:** When an electron is emitted from the material, it gains kinetic energy as it moves away. This kinetic energy is represented by the charge of an electron (q) multiplied by the potential difference between the Fermi level and the point of emission.
- **Electron Emission Mechanisms:** Electrons can be emitted from the material through various mechanisms, such as thermionic emission or field emission. Thermionic emission occurs when electrons gain enough thermal energy to escape, while field emission happens when an electric field pulls electrons from the surface.
- **Electron Emission and Positive Charge Buildup:** As electrons are emitted from the material, they lose negative charges and start to acquire a positive charge relative to the vacuum.
- **Electric Field and Equilibrium:** This positive charge buildup creates an electric field that points toward the material. This electric field acts in opposition to the force that ejects electrons, making it progressively harder to remove them.

- **Comparison of Materials:** When comparing different materials, it is essential to consider the work function and the depth of electron emission. If the electron emission depth is equal to the work function, it means electrons are right at the edge where they can be easily pulled out. This is ideal for some electronic devices. On the other hand, if the emission depth is not equal to the work function, then the electrons can be standing too close (shallower) or too far back (deeper). This will affect how easily electrons are emitted off the surface, impacting the device's performance.

The Lattice Constant

The lattice constants of a Hexagonal Close-Packed (HCP) structure depend on the specific material in question. However, in general, the lattice constants of an HCP structure are denoted as “a” and “c”, where “a” represents the basal plane lattice constant, which is the distance between two adjacent atoms in the same plane, and “c” represents the height of the HCP unit cell, which is the distance between two adjacent basal planes. For most HCP structures, the ratio of “c” to “a” is approximately 1.633. In other words, the height of the unit cell is about 1.633 times the length of the basal plane lattice constant. It is important to note that the specific values of “a” and “c” vary depending on the material. For example, in the case of pure titanium (Ti), the lattice constants are approximately $a = 2.95 \text{ \AA}$ (angstroms) and $c = 4.68 \text{ \AA}$. Other materials, such as magnesium (Mg) or zinc (Zn), will have different values for their lattice constants.

Bandgap

Let us think of electrons living in apartments. A solid material is like a giant apartment building for electrons. Each floor represents an energy level where electrons can live (energy bands). Electrons love to occupy the lowest available floors (lowest energy state). Normally, electrons fill the lowest floors first, following a “best deal” policy. There are two important levels of floors: The first level is the valence band, which is like the ground floor and the first few floors. This is where most electrons reside. It represents the energy levels of electrons in an atom's outermost shell (like their “home base”). The second level is the conduction band, which is like the top floors. Electrons in this band are free to move around the building (material) and conduct electricity. There is a gap between these two levels. In other words, there is an empty space between the valence and conduction bands called the band gap. It is like a locked set of floors that electrons cannot normally access. Let us classify materials based on the magnitude of the gap:

Conductors: In metals (like copper wires), the valence and conduction bands overlap, or the gap is very small. Electrons can easily move around, making them good conductors.

Semiconductors: In materials like silicon, the band gap is bigger. At very low temperatures, the conduction band is empty (*i.e.*, semiconductors will operate as insulators at absolute zero T). However, with a little push (thermal or light energy), electrons can jump the gap and become mobile, allowing them to conduct electricity.

Insulators: In materials like glass, the band gap is very large. It takes a lot of energy to move electrons across the gap, making them poor conductors (or good insulators).

If an electron receives a burst of energy (like sunlight) and can jump to a higher floor (higher energy level), this energy boost can be described by the equation $E = hv$ (where E is energy, h is Planck's constant, and v is frequency). This extra energy becomes the electron's "kinetic energy" (movement energy). If an electron jumps floors, it leaves an empty space behind in its original low-energy apartment. This empty space acts like a positive charge carrier (a "hole"). It is not a real positive particle, but it behaves like one. The behavior of this "hole" (positive charge carrier) is explained by hole theory, and it acts very much like a real positive charge moving around.

Tunable Bandgap

A tunable bandgap refers to the ability to control or adjust the energy difference between the valence band and the conduction band in a material. In traditional semiconductors, this bandgap is a fixed property determined by the material's atomic structure. In the previous section, we envisioned the bandgap as a locked set of floors in the electron apartment building. In a normal building, we cannot access those floors. However, with a tunable bandgap, it is like having a mechanism to unlock or adjust the access to those floors. This allows for more control over the electrical properties of the material. Below are some means to achieve a tunable bandgap:

- **Strain:** Applying mechanical stress to a material can change the spacing between atoms, which can, in turn, affect the bandgap.
- **Electric Field:** Applying an electric field can also influence the bandgap in some materials.
- **Doping:** Introducing impurities (dopant atoms) into a semiconductor can modify its electronic structure and bandgap.

Metamaterials

PREREQUISITE DEFINITIONS

Negative Index Metamaterials

The property of a negative refractive index in metamaterials refers to the phenomenon where light bends in the opposite direction of what is expected when it enters the material. This means that the refractive index of the metamaterial is negative, which is in contrast to the positive refractive index typically observed in natural materials like air, water, or glass.

In conventional materials, such as air or glass, light slows down as it passes through the material, resulting in a positive refractive index. In metamaterials with a negative refractive index, however, the behavior is different. Light still travels at its characteristic speed in a vacuum (the speed of light in a vacuum), but when it enters a metamaterial with a negative refractive index, it bends in the opposite direction compared to what would occur in conventional materials. Fig. (1) shows the difference in behavior between a positive and negative refractive index medium.

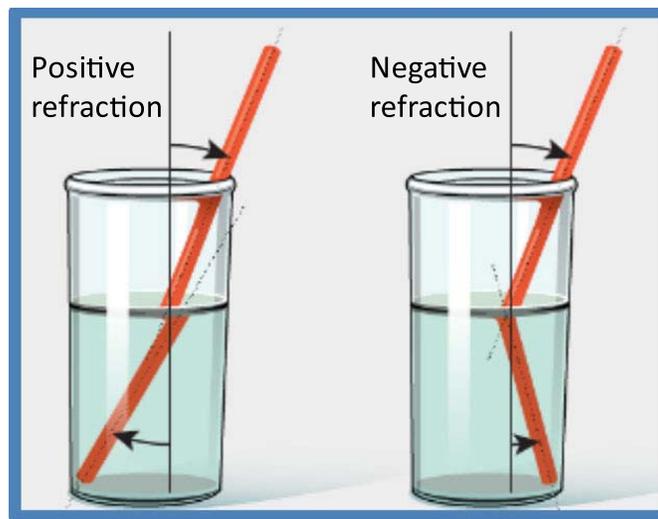


Fig. (1). Optical behavior of a positive and negative refractive indexed-material. [1].

Negative Index Metamaterials (NIMs) have unique wave propagation characteristics due to their negative permittivity (ϵ) and permeability (μ) properties.

Permittivity (ϵ) and permeability (μ) are crucial properties that describe how metamaterials interact with electric and magnetic fields, respectively:

Permittivity (ϵ)

It measures a material's ability to permit electric field lines. It affects how materials store and transmit electric energy. The permittivity is a measure of the electric polarizability of a dielectric medium. A material with a high permittivity (ϵ) polarizes more in response to an applied electric field, hence storing more energy in the material. This means its internal charges are displaced slightly, weakening the electric field within the material compared to free space (vacuum). In essence, permittivity reflects how much a material opposes the formation of an electric field.

It controls the response to electric fields, influencing dielectric properties and the behavior of electric charges within the material. Negative permittivity in metamaterials can result in unusual electromagnetic phenomena, such as negative refraction. Two points are worth-mentioning here:

- **Relative Permittivity:** Often, permittivity is discussed relative to the permittivity of free space (vacuum), known as the dielectric constant ($\kappa = \epsilon/\epsilon_0$), which is a dimensionless measure indicating how much a material can concentrate electric field lines compared to a vacuum.
- **Complex Permittivity:** In real materials, especially at high frequencies, permittivity can have a complex component ($\epsilon = \epsilon' - i\epsilon''$) where ϵ' represents the stored energy and ϵ'' represents the energy loss within the material. This is particularly relevant in understanding material losses in metamaterials.

Permeability (μ)

Permeability measures a material's ability to support magnetic field formation. It influences the material's magnetic properties and interactions. It controls the response to magnetic fields, affecting inductance and the behavior of magnetic fields. Negative permeability allows for unique magnetic properties, enabling applications like magnetic cloaking and novel wave manipulation. Higher permeability indicates a material's ability to concentrate magnetic field lines. When a material with high permeability is introduced into a magnetic field, the magnetic field becomes stronger within the material compared to free space.

Permeability essentially reflects how easily a material allows magnetic field lines to pass through it.

Relative permeability ($\mu_r = \mu/\mu_0$) shows how a material concentrates magnetic field lines compared to a vacuum. Complex permeability ($\mu = \mu' - i\mu''$) accounts for magnetic energy storage and loss, particularly at high frequencies. High permeability materials can reach magnetic saturation, where further increases in field strength do not increase magnetization.

These two properties can be modeled using the Drude model in the time domain. Here is how different combinations of ϵ and μ affect wave behavior:

Forward Waves (Both ϵ and μ are Positive)

The material behaves like conventional materials. Electromagnetic waves propagate forward with energy flow and phase velocity in the same direction.

Band Gap (ϵ and μ are with Different Signs)

One of the parameters (either ϵ or μ) is negative, while the other is positive. This creates a band gap where no wave propagation occurs because the medium does not support propagating solutions. The wave is thus attenuated and does not travel through the material.

Backward Waves (Both ϵ and μ are Negative)

The material exhibits negative refractive index properties. Electromagnetic waves propagate backward, meaning the energy flow (Poynting vector) and phase velocity are in opposite directions. This results in unusual phenomena like reverse Doppler effect and negative refraction.

Notice that for most metals at optical and infrared frequencies, the permittivity (ϵ) is generally negative, and metals may exhibit behaviors akin to plasmonic materials, sometimes creating conditions similar to band gaps or other complex electromagnetic responses. On the contrary, in the microwave and lower frequency ranges, metals exhibit a positive permittivity (ϵ). On the other hand, the magnetic permeability is generally positive and close to the permeability of free space (μ_0) for non-magnetic metals like copper, aluminum, and silver. For ferromagnetic metals like iron, nickel, and cobalt, the permeability can be significantly higher but is still positive. Thus, at microwave and lower frequencies, metals exhibit both positive permittivity and permeability, thus supporting forward wave propagation.

Smart Materials

PREREQUISITE DEFINITIONS

The Martensite Phase

Shape memory materials (SMMs) possess the unique ability to revert to a pre-defined shape when subjected to specific thermal or mechanical stimuli, a property known as the shape memory effect. The martensite phase is a crucial aspect of SMMs. This phase is the low-temperature phase, where the material exhibits a distorted or non-equilibrium crystal structure.

In shape memory alloys (SMAs), which are a common type of SMMs, the martensite phase is formed through a solid-state phase transformation. This transformation typically occurs when the material is cooled below a critical temperature called the martensite start temperature (M_s). As the temperature decreases further to the martensite finish temperature (M_f), the transformation completes, and the crystal lattice of the material undergoes a rearrangement, leading to the formation of the martensite phase.

The martensite phase in SMAs is characterized by specific crystal structures, which depend on the alloy's composition. The most common martensitic crystal structures observed in SMAs are body-centered tetragonal (BCT) and body-centered orthorhombic (BCO). The specific crystal structure of the martensite phase determines the unique shape memory behavior of the material.

Upon heating the shape memory material above a certain temperature called the austenite start temperature (T_{AS}), the material begins to revert back to its original austenite phase. This transformation completes at the austenite finish temperature (T_{AF}), allowing the material to recover its initial shape. This reversible transformation between the martensite and austenite phases enables the shape memory effect, making SMMs useful in various applications such as actuators, medical devices like stents, and aerospace components like coupling devices and fasteners.

On the other hand, shape memory polymers (SMPs) are a unique class of smart materials capable of undergoing significant and reversible changes in shape when exposed to specific external stimuli such as heat, light, or changes in pH. Unlike shape memory alloys (SMAs), SMPs are composed of polymeric materials that exhibit this remarkable property through the interplay of their molecular structures and thermomechanical properties.

Here is the key to understanding the behavior of SMP shape changes:

1. **Original Permanent Shape:** This is the inherent shape of the SMP before any external stimuli are applied.
2. **Temporary Shape Formation:** When SMPs are heated above their glass transition temperature (T_g) or melting temperature (T_m), they become soft and deformable. In this softened state, the material can be molded into a new, temporary shape.
3. **Temporary Shape Fixing:** This temporary shape is then fixed when the material is cooled below T_g or T_m . As the polymer chains become rigid again, they lock in the **deformed (temporary)** shape.
4. **Recovery to Original Permanent Shape:** Finally, when the SMP is reheated above T_g or T_m once more, the polymer chains regain their mobility, and the material undergoes a reversible phase transition. This allows the SMP to return to its original, permanent shape due to its inherent elasticity.

SMPs offer several advantages over traditional shape memory materials, including lower density, greater flexibility, and the ability to be processed into complex shapes and sizes. Their unique properties make them highly desirable for a variety of applications across different industries. In the biomedical field, SMPs are used to develop minimally invasive medical devices such as stents, sutures, and drug delivery systems. Additionally, SMPs find applications in aerospace and automotive industries for morphing structures, self-healing materials, and adaptive components. The versatility and tunability of SMPs, combined with their cost-effectiveness and ease of processing, continue to drive research and innovation, expanding their potential uses in numerous advanced technologies.

The Austenite Phase

The austenite phase is the high-temperature phase of a shape memory material (SMM). In shape memory alloys (SMAs), which are a common type of shape memory material, the austenite phase is the stable phase at temperatures above a critical temperature called the austenite start temperature (T_{AS}). When a shape memory alloy is heated above this temperature, it undergoes a solid-state phase transformation from the martensite phase (the low-temperature phase) to the aus-

tenite phase. This transformation is reversible, allowing the SMA to transition back to martensite upon cooling.

The austenite phase in SMAs is characterized by a specific crystal structure that depends on the alloy's composition. The most common austenitic crystal structure observed in SMAs is face-centered cubic (FCC), although other structures like body-centered cubic (BCC) can also occur. In the austenite phase, the crystal lattice of the SMA is more symmetric and regular compared to the distorted or non-equilibrium structure of the martensite phase. This transition between the martensite and austenite phases is responsible for the unique shape memory effect observed in these materials.

HISTORY OF SMART MATERIALS

The concept of using smart materials dates back to the 19th century, and the development and utilization of such materials have evolved over time. Here is a brief history of the milestones in the field of smart materials:

- **Early Discoveries:** The groundwork for smart materials was laid in the 19th century with the discovery of various phenomena and materials that exhibited unique properties. For example, in 1820, Thomas Seebeck discovered the thermoelectric effect, which is the basis for the generation of electricity from temperature differences. In the 1880s, Pierre and Jacques Curie discovered the piezoelectric effect in certain crystals.
- **Shape Memory Alloys (1950s):** The development of shape memory alloys (SMAs) in the 1950s marked a significant milestone in the field of smart materials. SMAs, such as Nitinol (Nickel-Titanium), possess the ability to “remember” their original shape and recover it when subjected to specific stimuli, typically heat. This discovery paved the way for numerous applications in industries such as aerospace, medical devices, and consumer electronics.
- **Electroactive Polymers (1960s-1970s):** The field of electroactive polymers (EAPs) emerged in the 1960s and 1970s. EAPs are polymers that exhibit changes in shape, size, or mechanical properties when subjected to an electric field. They provided new possibilities for actuators, sensors, artificial muscles, and other applications.
- **Development of Responsive Materials (1980s-1990s):** In the 1980s and 1990s, significant progress was made in the development of various smart materials. This included advances in areas such as piezoelectric materials, electrochromic materials, magnetostrictive materials, and shape memory polymers. Researchers explored new material compositions and fabrication techniques to enhance the responsiveness and adaptability of these materials.

CHAPTER 5**Nanomaterial Synthesis and Surface Functionalization****BOTTOM-UP AND TOP-DOWN APPROACH FOR NANOMATERIAL SYNTHESIS**

The bottom-up and top-down approaches are two general methods for synthesizing nanomaterials. The bottom-up approach starts with individual atoms or molecules and builds them up into larger structures, while the top-down approach starts with a bulk material and reduces it in size to the nanoscale.

Bottom-up Approach

The bottom-up approach is often used to synthesize nanoparticles. In this approach, atoms or molecules are first brought together in a controlled environment. The atoms or molecules then interact with each other through chemical or physical forces, and they eventually form larger structures. The bottom-up approach can be used to synthesize a wide variety of nanoparticles, including metals, semiconductors, and polymers.

Some of the most common bottom-up methods for synthesizing nanoparticles include:

1. **Chemical Vapor Deposition (CVD):** A gaseous precursor is decomposed into its constituent atoms or molecules, which then condense to form nanoparticles. It is widely used for producing thin films and nanoparticles of various materials.
2. **Sol-Gel Synthesis:** A solution of metal salts or metal alkoxides is hydrolyzed to form a gel. The gel is then dried to form nanoparticles. It is commonly used for producing metal oxide nanoparticles and coatings.
3. **Template-Assisted Synthesis:** A template is used to control the shape and size of the nanoparticles. The template is then removed, leaving behind the nanoparticles. It is useful for creating nanoparticles with specific shapes and sizes.

4. **Hydrothermal Synthesis:** Chemical precursors react in an aqueous solution under controlled temperature and pressure conditions to form nanostructures. It is effective for synthesizing a wide range of inorganic nanomaterials.

5. **Biological Synthesis:** Biological materials or processes are used to create nanostructures. It is utilized for the eco-friendly synthesis of nanoparticles using plants, bacteria, or fungi.

6. **Supercritical Fluid Synthesis:** Supercritical fluids, which have properties of both liquids and gases, are used as solvents to produce nanoparticles. It enables controlled nanoparticle formation with unique properties.

7. **Spray Pyrolysis:** A precursor solution is atomized into fine droplets, which are carried by a gas stream into a high-temperature furnace where pyrolysis occurs, forming nanoparticles. It is used for producing a wide variety of nanoparticles and nanostructured materials.

8. **Thermal Decomposition:** A precursor compound is decomposed at high temperatures in the presence of a stabilizing agent, resulting in nanoparticle formation. It is commonly used for synthesizing metal and metal oxide nanoparticles.

9. **Microwave-Assisted Synthesis:** Microwave radiation is used to heat the reaction mixture, leading to rapid and uniform heating and the formation of nanoparticles. It enhances reaction rates and improves nanoparticle quality. This method is covered in Chapter 2 | “Bottom-up Carbon Nanostructure Synthesis Methods” | “Microwave-Assisted Synthesis” section.

10. **Sono-chemical Synthesis:** Ultrasonic waves induce chemical reactions in solution, leading to nanoparticle formation. It produces nanoparticles through high local temperatures and pressures generated by ultrasound. This method is covered in Chapter 2 | “Bottom-up Carbon Nanostructure Synthesis Methods” | “Ultrasonic Synthesis (Sono-chemical Synthesis)” section.

11. **Electrochemical Deposition:** An electric current reduces metal ions from a solution onto a substrate, forming nanoparticles. It allows precise control over nanoparticle size and composition. This method is covered in Chapter 2 | “Bottom-up Carbon Nanostructure Synthesis Methods” | “Electrochemical Cell Method” section.

Chemical Vapor Deposition (CVD)

In CVD, the precursor gases are decomposed, and the resulting atoms or molecules undergo chemical reactions on the substrate surface, leading to the

growth or deposition of thin films or nanostructures. The process is driven by the self-assembly of the atoms or molecules, which leads to the formation of larger structures. The CVD process begins with a gaseous precursor, which is usually a reactive gas or a mixture of gases. These precursors can be organic or inorganic compounds, such as metalorganic compounds, halides, or hydrides. Examples of common precursors include silane (SiH_4) for silicon-based films, trimethylaluminum (TMA) for aluminum-based films, or tungsten hexafluoride (WF_6) for tungsten-based films.

To initiate the CVD process, the precursor gas is introduced into a reaction chamber containing a heated substrate. The substrate is the material onto which the thin film or nanostructure will be deposited. It is typically a solid material, such as silicon, glass, or metal.

As the precursor gas enters the reaction chamber, it undergoes decomposition, either thermally or with the assistance of a plasma. During thermal decomposition, the precursor molecules are heated, causing them to break apart into their constituent atoms or smaller molecules. Alternatively, plasma-enhanced CVD (PECVD) uses plasma, which is an ionized gas with high energy, to dissociate the precursor molecules into reactive species.

Once the precursor is decomposed, the resulting atoms or molecules are highly reactive and can readily react with each other or the substrate surface. These reactive species then condense or react on the surface of the heated substrate, forming a solid product. The deposition occurs through chemical reactions between the precursor and the substrate surface.

The temperature of the substrate is a critical parameter in CVD, as it influences the reaction kinetics and the properties of the deposited film. Heating the substrate promotes the chemical reaction by providing sufficient energy for the reactants to overcome activation barriers and form the desired product. By controlling the process parameters such as precursor concentration, temperature, pressure, and deposition time, it is possible to precisely control the thickness, composition, and morphology of the deposited film. This versatility makes CVD a widely used technique in various industries, including semiconductor manufacturing, surface coating, and nanotechnology research.

The substrate can be a variety of materials, such as a metal, semiconductor, or polymer. CVD is a versatile technique that can be used to synthesize a wide variety of nanomaterials. It is often used to synthesize nanoparticles, thin films, and nanowires. CVD is also a scalable process, which means that it can be used to produce large quantities of nanomaterials.

CHAPTER 6**Nanomaterial Characterization Techniques: Part 1**

Before we delve into the world of nanometer characterization techniques, we have to emphasize the fact that it is of immense importance to well prepare the sample and ensure that it is free of any traces of impurities other than its constituents. Moreover, the sample should be prepared, stored (if applicable), and placed inside the sample chamber without being subjected to any harsh environment that may result in a distortion or cause any structural defects prior to testing.

SAMPLE PREPARATION IMPORTANCE

Sample preparation and handling techniques play a crucial role in nanomaterial characterization as they significantly impact the quality and reliability of the data obtained from various characterization methods. Proper sample preparation ensures that the nanomaterials are representative of their actual properties, free from artifacts, and suitable for the chosen analytical technique. Here is an explanation of the importance of sample preparation and handling techniques in nanomaterial characterization:

Sample Homogeneity

- **Importance:** Nanomaterials often exhibit size and shape-dependent properties. To accurately characterize these properties, samples must be homogeneous. Aggregates or agglomerates can lead to misleading results.
- **Techniques:** Ultrasonication, sonication, vortexing, or mechanical stirring can be used to disperse nanoparticles and break up agglomerates. Care must be taken to choose the appropriate method to avoid damaging or altering the nanomaterials.

Contamination Control

- **Importance:** Contaminants from the environment or sample-handling equipment can interfere with measurements. For instance, trace amounts of dust or other particles can affect the accuracy of nanomaterial characterization.
- **Techniques:** Work in clean and controlled environments such as cleanrooms or laminar flow hoods. Use clean glassware and disposable tools, and wear appropriate personal protective equipment to prevent contamination.

Sample Stability

- **Importance:** Many nanomaterials are sensitive to environmental factors like moisture, oxygen, and light. Sample degradation or structural changes can occur if not handled properly.
- **Techniques:** Store nanomaterial samples in inert atmospheres, such as nitrogen or argon, and use airtight containers. Protect samples from light if they are light-sensitive. Perform experiments quickly to minimize exposure to external conditions.

Instrument Compatibility

- **Importance:** Different characterization techniques may require specific sample formats or sizes. Samples must be prepared to meet the requirements of the chosen instrument.
- **Techniques:** Size reduction (*e.g.*, milling) or dilution may be necessary to adapt samples to the instrument's specifications. Ensure that the sample format is compatible with the instrument's sample holder or stage.

Avoiding Artifacts

- **Importance:** Sample preparation can introduce artifacts or changes in the nanomaterials' properties. It is essential to minimize these alterations.
- **Techniques:** Be mindful of the choice of solvents, dispersants, or chemicals used during sample preparation, as they can interact with nanomaterials. Perform control experiments to assess the impact of sample preparation on the properties of interest.

Sample Quantity

- **Importance:** Some characterization techniques require specific sample quantities, and nanomaterials can be limited in supply. Efficient sample use is essential.
- **Techniques:** Optimize sample sizes to meet the instrument's requirements while conserving precious nanomaterials. For techniques like electron microscopy, where beam damage is a concern, use minimal sample amounts.

Reproducibility and Standardization

- **Importance:** To ensure the reliability of results and facilitate comparisons between studies, sample preparation and handling methods should be standardized and documented.
- **Techniques:** Develop standard operating procedures (SOPs) for sample preparation and handling. Record details of the methods used, such as sonication

duration, temperature, and concentrations. Share these methods with others in the field to promote reproducibility.

Safety Considerations

- **Importance:** Nanomaterials may pose health and safety risks. Proper handling and disposal procedures are essential.
- **Techniques:** Follow safety guidelines for nanomaterial handling, including the use of personal protective equipment (PPE), fume hoods, and waste disposal protocols. Ensure that researchers are adequately trained in safety procedures.

In summary, sample preparation and handling techniques in nanomaterial characterization are critical for obtaining accurate and meaningful data. Researchers must carefully plan and execute these steps to ensure that the nanomaterials under investigation are representative of their true properties and that results are reliable and reproducible. Proper handling also helps ensure the safety of researchers and minimizes environmental contamination.

Nanomaterials have unique properties that make them useful in a wide range of applications, from electronics and medicine to energy and environmental protection. Nanomaterials characterization is the process of measuring and analyzing the physical, chemical, and structural properties of nanomaterials. Nanomaterials are materials that have at least one dimension of less than 100 nanometers. Due to their small size, nanomaterials have unique properties that make them useful in a wide range of applications. However, their small size and complex structure can also make them difficult to characterize.

The specific techniques used to characterize a nanomaterial will depend on the specific properties of interest and the type of nanomaterial being characterized. For example, if we are interested in determining the size and shape of a nanomaterial, we might use DLS or SEM. On the other hand, if we are interested in determining the elemental composition of a nanomaterial, we might use ICP-MS. Nanomaterials characterization is an important tool for developing and using new nanomaterials in a variety of applications. By understanding the properties of nanomaterials, scientists and engineers can design nanomaterials with specific properties for specific applications.

In this chapter, we will basically cover the microscopic or, I would rather say, nanoscopic techniques. We will continue with the rest of the techniques in the next chapter.

Nanomaterials Characterization Techniques: Part 2

SPECTROSCOPY TECHNIQUES

Spectroscopy techniques use light or other forms of electromagnetic radiation to interact with the nanomaterial and produce a spectrum that can be used to identify its chemical composition and structure. Common spectroscopic techniques for nanomaterials characterization include UV-Vis (Ultraviolet-Visible), FTIR (Fourier-Transform Infrared), and Raman spectroscopy, which are powerful tools for characterizing nanomaterials. These techniques offer insights into the electronic, vibrational, and chemical properties of nanomaterials.

Ultraviolet-Visible (UV-Vis) Spectroscopy

UV-visible spectroscopy: UV-visible spectroscopy measures the absorption or transmission of light in the ultraviolet and visible wavelength ranges. This technique can be used to identify the type and concentration of different functional groups in a nanomaterial.

The basic principle of UV spectroscopy is governed by the Beer-Lambert law, which states that the absorbance of a solution is proportional to the concentration of the absorbing species and the path length of the light through the solution. The Beer-Lambert law can be expressed mathematically as follows:

$$A = \epsilon bc$$

Where:

- A is the absorbance
- ϵ is the molar absorptivity coefficient
- b is the path length
- c is the concentration

The molar absorptivity coefficient is a measure of how strongly a molecule absorbs UV light. It is specific to each molecule and depends on the wavelength

of the light. The path length is the distance that the light travels through the solution. It is typically measured in centimeters.

The concentration is the molar amount of absorbing species in the solution. It is typically measured in moles per liter. The Beer-Lambert law can be used to calculate the concentration of an absorbing species in a solution by measuring the absorbance of the solution at a specific wavelength.

Example 1:

Suppose that we have a solution of a dye with a molar absorptivity coefficient of $1000 \text{ M}^{-1} \text{ cm}^{-1}$. If we measure the absorbance of the solution at a wavelength of 250 nm and get a value of 0.5, what will be the molar concentration of the dye for $b=1 \text{ cm}$?

Solution:

We can use the Beer-Lambert law to calculate the concentration of the dye in the solution as follows:

$$c = A / \epsilon b$$

$$c = 0.5 / 1000 \text{ M}^{-1} \text{ cm}^{-1} * 1 \text{ cm}$$

$$c = 0.5 \text{ mM} = 500 \text{ }\mu\text{M}$$

Purpose

- UV-Vis spectroscopy is primarily used to study the electronic properties of nanomaterials, especially their absorption and scattering of light in the UV and visible regions of the electromagnetic spectrum.
- It provides information about the presence of electronic transitions, bandgap energies, and optical properties.

How it works

- UV-Vis spectroscopy involves shining light with a range of wavelengths (typically in the UV and visible regions) through a sample of the nanomaterial.
- The nanomaterial absorbs specific wavelengths of light, leading to changes in absorbance.
- The resulting absorption spectrum, often displayed as a UV-Vis absorption spectrum, shows the relationship between the intensity of absorbed light and its wavelength.
- Peaks in the spectrum correspond to electronic transitions within the nanomaterial.

Applications

Determining Optical Properties

- UV-Vis spectroscopy helps identify the absorption maxima and bandgap energies of nanomaterials, which are critical for various optical applications.
- It is used to characterize the color, transparency, and opacity of nanomaterials.

Quantifying Concentration

UV-Vis spectroscopy is employed to determine the concentration of nanomaterials in solution through Beer's law, which relates absorbance to concentration.

Monitoring Surface Plasmon Resonance

This technique is used to study plasmonic nanomaterials (*e.g.*, gold and silver nanoparticles) by observing shifts in absorbance related to surface plasmon resonance (SPR).

Talam *et al.* [1] examined the effect of nanoparticle size on their properties. UV-Vis absorption spectroscopy is a common way to measure the size of nanosized particles. The authors examined the absorption spectrum of ZnO nano-powder completely dispersed in water, which showed a strong absorption band at about 355 nm and a sharp absorption peak at about 258 nm, as shown in Fig. (1). A band in a UV-Vis spectrum is a broad region of absorbance, while a peak is a sharp point of absorbance. The band at 355 nm in the dispersed ZnO nanoparticle UV-Vis spectrum is due to the absorption of photons by the valence electrons of the ZnO nanoparticles. On the other hand, the peak at 258 nm is due to the absorption of photons by excitons, which are excited electrons and holes that are bound together.

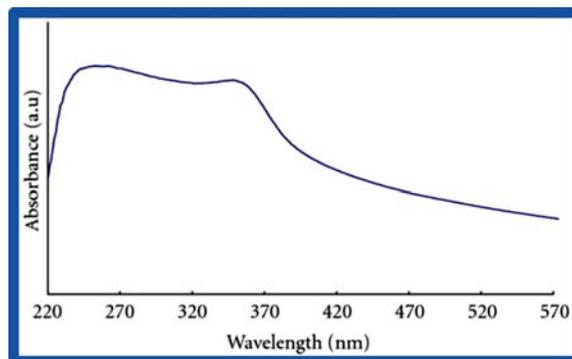


Fig. (1). UV-Vis absorption spectrum of dispersed ZnO nanoparticles in water [1].

APPENDIX

Table 1. Nanometer characterization techniques and corresponding molecular properties [1].

Technique	Derived molecular information
XRD (group: X-ray-based techniques)	Crystal structure, composition, crystalline grain size
XAS (EXAFS, XANES)	X-ray absorption coefficient (element-specific) – chemical state of species, interatomic distances, Debye-Waller factors, also for non-crystalline NPs
SAXS	Particle size, size distribution, growth kinetics
XPS	Electronic structure, elemental composition, oxidation states, ligand binding (surface-sensitive)
FTIR (group: further techniques for structure/composition/main properties)	Surface composition, ligand binding
NMR (all types)	Ligand density and arrangement, electronic core structure, atomic composition, influence of ligands on NP shape, NP size
BET	Surface area
TGA	Mass and composition of stabilizers
LEIS	Thickness and chemical composition of self-assembled monolayers of NPs
UV-Vis	Optical properties, size, concentration, agglomeration state, hints on NP shape
PL spectroscopy	Optical properties – relation to structure features such as defects, size, composition
DLS	Hydrodynamic size, detection of agglomerates
NTA	NP size and size distribution
DCS	NP size and size distribution
ICP-MS	Elemental composition, size, size distribution, NP concentration
SIMS, ToF-SIMS, MALDI	Chemical information (surface-sensitive) on the functional group, molecular orientation and conformation, surface topography, MALDI for NP size
RMM-MEMS, ζ -potential, pH, EPM, GPC, DSC, <i>etc.</i>	Please check the relevant parts of the manuscript
SQUID-nanoSQUID (group: magnetic nanomaterials)	Magnetization saturation, magnetization remanence, blocking temperature
VSM	Similar to SQUID through M–H plots and ZFC-FC curves

Technique	Derived molecular information
Mössbauer	Oxidation state, symmetry, surface spins, magnetic ordering of Fe atoms, magnetic anisotropy energy, thermal unblocking, distinguish between iron oxides
FMR	NP size, size distribution, shape, crystallographic imperfection, surface composition, M values, magnetic anisotropic constant, demagnetization field
XMCD	Site symmetry and magnetic moments of transition metal ions in ferro- and ferri-magnetic materials, element-specific
Magnetic susceptibility, magnetophoretic mobility	Please check the relevant parts of the manuscript
Superparamagnetic relaxometry	Core properties, hydrodynamic size distribution, detect and localize superparamagnetic NPs
TEM (group: microscopy techniques)	NP size, size monodispersity, shape, aggregation state, detect and localize/quantify NPs in matrices, study growth kinetics
HRTEM	All information by conventional TEM but also on the crystal structure of single particles. Distinguish monocrystalline, polycrystalline, and amorphous NPs. Study defects
Liquid TEM	Depict NP growth in real time, study growth mechanism, single particle motion, superlattice formation
Cryo-TEM	Study complex growth mechanisms, aggregation pathways, good for molecular biology and colloid chemistry to avoid the presence of artifacts or destroyed samples
Electron diffraction	Crystal structure, lattice parameters, study order-disorder transformation, long-range order parameters
STEM	Combined with HAADF, EDX for morphology study, crystal structure, and elemental composition. Study the atomic structure of hetero-interfaces
Aberration-corrected (STEM, TEM)	Atomic structure of NP clusters, especially bimetallic ones, as a function of composition, alloy homogeneity, phase segregation
EELS (EELS-STEM)	Type and quantity of atoms present, chemical state of atoms, collective interactions of atoms with neighbors, bulk plasmon resonance
Electron tomography	Realistic 3D particle visualization, snapshots, video, quantitative information down to the atomic scale
SEM-HRSEM, T-SEM-EDX	Morphology, dispersion of NPs in cells and other matrices/supports, precision in lateral dimensions of NPs, quick examination–elemental composition
EBSD	Structure, crystal orientation, and phase of materials in SEM. Examine microstructures, reveal texture, defects, grain morphology, deformation

(Table 1) cont....

Technique	Derived molecular information
AFM	NP size and shape in 3D mode, evaluate the degree of covering of a surface with NP morphology, dispersion of NPs in cells and other matrices/supports, precision in lateral dimensions of NPs, quick examination–elemental composition
MFM	Standard AFM imaging together with the information of magnetic moments of single NPs. Study magnetic NPs in the interior of cells. Discriminate from non-magnetic NPs

Table 2. Molecular properties and recommended characterization techniques [1].

Molecular property	Recommended characterization techniques
Size (structural properties)	TEM, XRD, DLS, NTA, SAXS, HRTEM, SEM, AFM, EXAFS, FMR, DCS, ICP-MS, UV-Vis, MALDI, NMR, TRPS, EPLS, magnetic susceptibility
Shape	TEM, HRTEM, AFM, EPLS, FMR, 3D-tomography
Elemental-chemical composition	XRD, XPS, ICP-MS, ICP-OES, SEM-EDX, NMR, MFM, LEIS
Crystal structure	XRD, EXAFS, HRTEM, electron diffraction, STEM
Size distribution	DCS, DLS, SAXS, NTA, ICP-MS, FMR, superparamagnetic relaxometry, DTA, TRPS, SEM
Chemical state–oxidation state	XAS, EELS, XPS, Mössbauer
Growth kinetics	SAXS, NMR, TEM, cryo-TEM, liquid-TEM
Ligand binding/composition/density/arrangement/mass, surface composition	XPS, FTIR, NMR, SIMS, FMR, TGA, SANS
Surface area, specific surface area	BET, liquid NMR
Surface charge	Zeta potential, EPM
Concentration	ICP-MS, UV-Vis, RMM-MEMS, PTA, DCS, TRPS
Agglomeration state	Zeta potential, DLS, DCS, UV-Vis, SEM, Cryo-TEM, TEM
Density	DCS, RMM-MEMS
Single particle properties	Sp-ICP-MS, MFM, HRTEM, liquid TEM
3D visualization	3D-tomography, AFM, SEM
Dispersion of NP in matrices/supports	SEM, AFM, TEM
Structural defects	HRTEM, EBSD

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Dr. Al-Malah graduated from Oregon State University in 1993, and his area of specialty during his M.S. and Ph.D. programs dealt with protein interactions and behavior at interfaces in biological systems. He is currently studying the modeling, simulation, and optimization aspects of physical/biophysical systems and the characterization of molecular properties within the dome of chemical, biochemical, pharmaceutical, and food engineering.

Dr. Al-Malah is the sole book author with renowned publishers, as shown below:

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