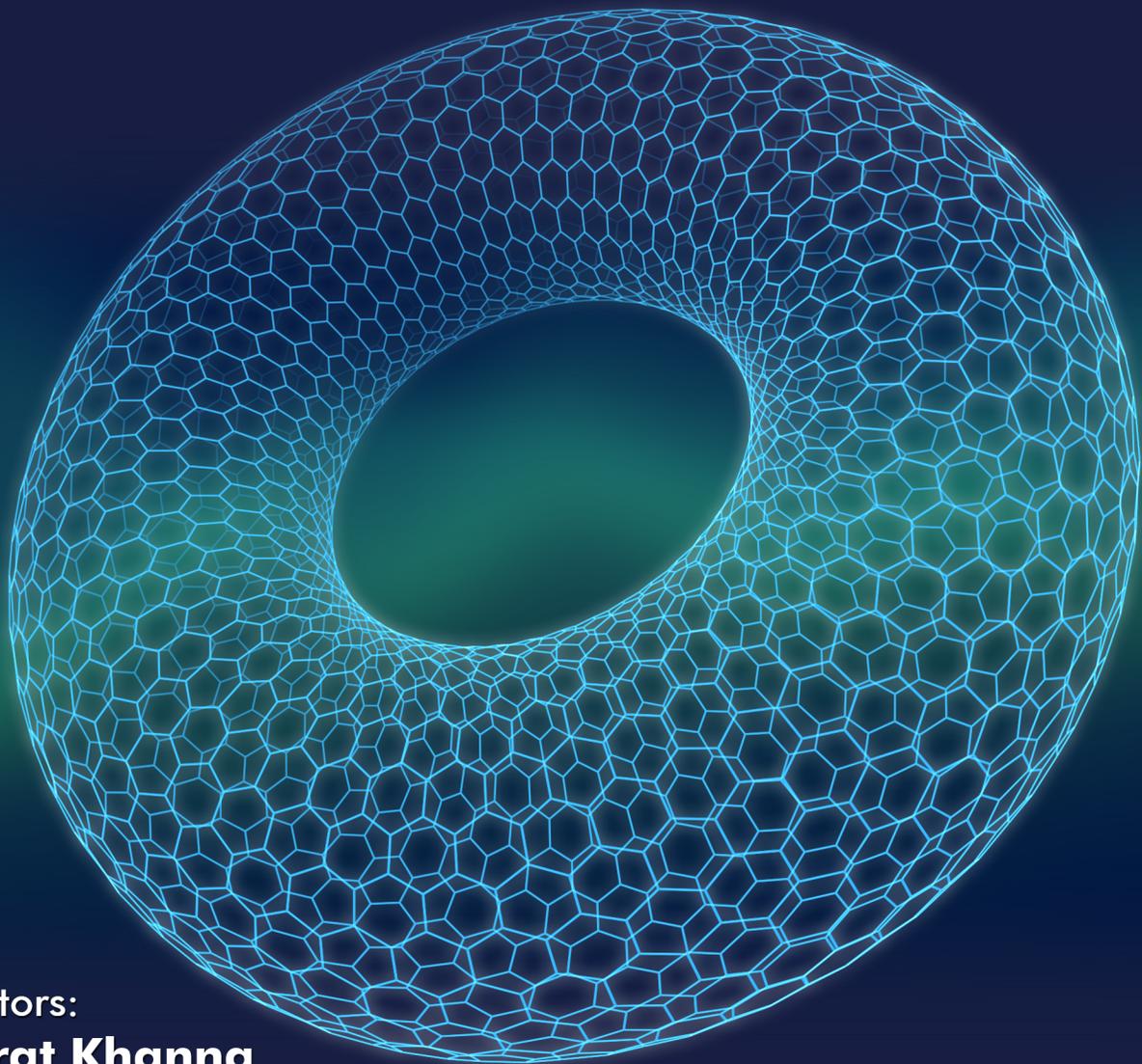


RECENT ADVANCEMENTS IN MULTIDIMENSIONAL APPLICATIONS OF NANOTECHNOLOGY



Editors:

Virat Khanna

Suneev Anil Bansal

Vishal Chaudhary

Reddicherla Umapathi

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Recent Advancements in Multidimensional Applications of Nanotechnology

(Volume 1)

Edited by

Virat Khanna

*University Centre for Research & Development
Chandigarh University
Punjab, India*

Suneev Anil Bansal

*ELFROU Inc
Gurgaon, India*

Vishal Chaudhary

*Bhagini Nivedita College
University of Delhi, Delhi
India*

&

Reddicherla Umapathi

*NanoBio High-Tech Materials Research Center
Department of Biological Engineering
Inha University, Incheon
South Korea*

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Editors: Virat Khanna, Suneev Anil Bansal, Vishal Chaudhary and Reddicherla Umapathi

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PREFACE

In this rapidly evolving field, nanotechnology has emerged as a powerful tool with endless possibilities. This book aims to provide a comprehensive overview of the latest advancements and applications of nanotechnology across various dimensions. It covers a wide range of topics, from electron microscopy to biogenic synthesis methods, from energy applications to agro-nanotechnology, and from nanotherapeutic strategies to nanosensors for virus detection.

Chapter 1 explores the remarkable capabilities of the electron microscope for qualitative and quantitative analysis of nano-materials. It sets the stage for subsequent chapters that delve into specific applications of nanotechnology. Advancements in perovskite nanomaterials for advanced energy applications are discussed in Chapter 2, emphasizing their significance in the quest for sustainable energy solutions. Chapter 3 explores the current developments in the use of copper oxide nanoparticles in the oil and gas industries, highlighting their potential for enhancing efficiency and performance. In Chapter 4, the application of nano-coatings to combat hot corrosion of metallic substrates is examined, presenting an innovative approach to protect materials subjected to extreme conditions. Agro-nanotechnology, discussed in Chapter 5, presents a promising pathway towards sustainable agriculture, where nanotechnology is harnessed to enhance crop production and mitigate environmental challenges. The impact of economic natural dyes on the performance and efficiency of TiO_2 nano-structure solar cells is explored in Chapter 6, uncovering new possibilities for greener and more efficient solar energy generation. Chapter 7 investigates the effect of annealing conditions on chemical bath-deposited CdTe thin films, offering insights into optimizing thin-film deposition processes. Chapter 8 highlights the biomedical necessities and green future of metallic nanoparticles, revealing their potential in various healthcare applications. Chapter 9 focuses on the production of silver nanoparticles with enhanced cytotoxicity and biological activity from *Kalanchoe Gastonis-Bonnieri* leaf extract, opening avenues for novel therapeutic approaches. Chapter 10 provides an overview of recent biogenic synthesis methods of metal nanoparticles and their applications, showcasing the potential of nature-inspired approaches in nanotechnology. Chapter 11 takes a step further by benchmarking different CNN architectures on a COVID-19 dataset, highlighting the role of nanotechnology in addressing public health challenges. Finally, Chapter 12 explores the application of novel nanotherapeutic strategies in treatment through herbal medicines, presenting an exciting fusion of traditional and modern medicine.

Readers of this book will gain a comprehensive understanding of the recent advancements in nanotechnology and its multidimensional applications. From the fundamentals of electron microscopy to cutting-edge developments in nanotherapeutics and biogenic synthesis methods, this book offers a broad perspective on the field. It equips readers with the knowledge to explore new possibilities, drive innovation, and contribute to the advancement of nanotechnology across various domains.

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We hope that this book serves as a valuable resource for researchers, scientists, academicians, and students interested in nanotechnology and its applications. It is our sincere belief that the knowledge shared within these pages will inspire further research, foster interdisciplinary collaborations, and contribute to the realization of a more sustainable and technologically advanced future.

Virat Khanna

University Centre for Research & Development
Chandigarh University
Punjab, India

Suneev Anil Bansal

ELFROU Inc
Gurgaon, India

Vishal Chaudhary

Bhagini Nivedita College
University of Delhi, Delhi
India

&

Reddicherla Umapathi

NanoBio High-Tech Materials Research Center
Department of Biological Engineering
Inha University, Incheon
South Korea

List of Contributors

Aquib Khan	Department of Polytechnic, Integral University, Kursi road Lucknow, Uttar Pradesh, India
Anand Singh	DST – Centre of Interdisciplinary Mathematical Sciences, Institute of Science, Banaras Hindu University (BHU)Varanasi, Varanasi, Uttar Pradesh, India
Ayushi Rastogi	Scitechesy Research and Technology Private Limited, Central Discovery Centre, BioNEST BHU, Banaras Hindu University, Varanasi – 2210035, India Department of Humanities and Applied Sciences, School of Management Sciences, College of Engineering, Lucknow – 226001, Uttar Pradesh, India
Che Azurahamanim Che Abdullah	Centre for Diagnostic Nuclear Imaging, Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia
Celin. S. R.	Department of Chemistry, WCC (Affiliated to MS University, Abishekapatti, Tirunelveli-627012), Nagercoil, Tamilnadu, India
Chesta Mehta	Department of Chemistry, M.L.Sukhadia University, Udaipur, Rajasthan, 313001, India
Deepshikha Verma	Department of Chemistry, M.B.S. College of Engineering and Technology, 181101, Jammu and Kashmir, India
Faria Fatima	Department of Agriculture, IIAST, Integral University, Kursi road Lucknow, Uttar Pradesh, India
Giriraj Tailor	Department of Chemistry, Mewar University, Chittorgarh, Rajasthan, 31290, India
Harsh Kumar Mishra	DST – Centre of Interdisciplinary Mathematical Sciences, Institute of Science, Banaras Hindu University (BHU)Varanasi, Varanasi, Uttar Pradesh, India
Jyoti Bhattacharjee	Department of Chemical Engineering, University of Calcutta, Kolkata 700009, India
Jyoti Chaudhary	Department of Chemistry, M.L.Sukhadia University, Udaipur, Rajasthan, 313001, India
Kumar Anurag	School of Energy Materials, Mahatma Gandhi University, Kottayam, Kerala-686560, India
Lankipalli Krishna Sai	School of Electronics Engineering, Vellore Institute of Technology, Chennai-600127, India
Muhammad Salman Habib	Department of Metallurgical & Materials Engineering, University of Engineering Technology G.T. Road Lahore, Pakistan
Muhammad Asif Rafiq	Department of Metallurgical & Materials Engineering, University of Engineering Technology G.T. Road Lahore, Pakistan
Mhd Hazli Rosli	Nanomaterial Synthesis and Characterization Lab, Institute of Nanoscience and Nanotechnology, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
Mohamed Abdelmonem	Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia

Manahil E. Mofdal	Qassim University, Faculty of Science, Department of Physics, Buraydah, KSA
Nur Farahah Mohd Khairuddin	Centre of Foundation Studies for Agricultural Science, Universiti Putra Malaysia, 43400, UPM Serdang, Selangor, Malaysia
Nada M. O. Sid Ahmed	Computer Engineering Department Computer Science and Engineering College, University of Hail, Hail, KSA
Nodar. O. Khalifa	Department of Physics, Sudan University of Science and Technology, Khartoum, Sudan
Nada H. Talib	Solar Energy Department, National Energy Research Center, Khartoum, Sudan
R. Ajitha	Department of Chemistry, WCC (Affiliated to MS University, Abishekapatti, Tirunelveli-627012), Nagercoil, Tamilnadu, India
Sunil Kumar Pradhan	School of Electronics Engineering, Vellore Institute of Technology, Chennai-600127, India
Santosh Kumar	Department of Mechanical Engineering, Chandigarh Group of College, Landran, Mohali, Punjab, India
Sudeshna Surabhi	Brindavan College of Engineering, Yelahanka, Bengaluru, Karnataka-560063, India
S.R. Kumar	Thin Film Laboratory, National Institute of Advanced Manufacturing Technology(Formerly NIFFT), Ranchi-834003, India
Subhasis Roy	Department of Chemical Engineering, University of Calcutta, Kolkata 700009, India
Saurabh Singh	M.L.V. Government College, Bhilwara, Rajasthan, 311001, India
Sumanta Bhattacharya	Maulana Abul Kalam Azad University of Technology, West Bengal, India
Tadisetti Taneesha	School of Electronics Engineering, Vellore Institute of Technology, Chennai-600127, India

CHAPTER 1

Electron Microscope: The Tool for Qualitative and Quantitative Analysis of Nano-Materials**Lankipalli Krishna Sai¹, Tadisetti Taneesha¹ and Sunil Kumar Pradhan^{1,*}**¹ *School of Electronics Engineering, Vellore Institute of Technology, Chennai-600127, India*

Abstract: An electron microscope is a highly advanced sophisticated tool where high energy electron beam is used as the source. Since an electron beam has a shorter wavelength than visible light photons, it may expose the structure of tiny objects and has a higher resolving power than a light microscope. While most light microscopes are constrained by diffraction to around 500 nm resolution and usable magnifications below 2000, a scanning electron microscope (SEM) may attain 5 nm resolution and magnifications up to roughly 10,000,000. Electromagnetic lenses, which are similar to the glass lenses of an optical light microscope, are used in electron microscopes to create electron optical lens systems. Large molecules, biopsy samples, metals, crystals, and other biological and inorganic specimens, among others, can all have their ultra-fine structure studied using electron microscopes. Electron microscopes are frequently used in industry for failure analysis and quality control. The images are captured using specialised digital cameras and frame grabbers by modern electron microscopes to create electron micrographs. To create an appropriate sample from materials for an electron microscope, processing may be necessary. Depending on the material and the desired analysis, a different procedure is needed. Transmission electron microscopes (TEM), scanning electron microscopes (SEM), reflection electron microscopes (REM), scanning tunnelling microscopes (STM), and other types of electron microscopes are commonly employed in academic and research institutions. The initial and operating costs of electron microscopes are higher and they are also more expensive to construct and maintain. High-resolution electron microscopes need to be kept in sturdy structures (often underground) with specialised amenities like magnetic field cancelling devices.

Keywords: Cryogenic transmission electron microscopy, Electron mapping, Energy-filtered transmission electron microscopy, Electron energy loss spectroscopy, Electron microscope, Environmental electron microscope, Low-voltage electron microscope, Magnification, Nano-materials, Scanning transmission electron microscope.

* **Corresponding author Sunil Kumar Pradhan:** School of Electronics Engineering, Vellore Institute of Technology, Chennai-600127, India; E-mails: sunilpradha@gmail.com, sunilkumar.pradhan@vit.ac.in

INTRODUCTION

Electron microscopy for nanotechnology is the use of electron microscopes to observe, analyse and manipulate materials at the nanoscale [1 - 5]. This field plays a crucial role in the development of modern nanotechnology and materials science, as it enables scientists and engineers to observe and study the structure, composition, and properties of nanoscale materials and devices [6, 7]. There are several types of electron microscopes used for nanotechnology, including Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Scanning Transmission Electron Microscopy (STEM), and Cryogenic Transmission Electron Microscopy (Cryo-TEM) [8, 9]. TEM works by passing a beam of electrons through a thin sample, producing an image of the internal structure of the material. SEM, on the other hand, uses electrons to scan the surface of a sample and produce a high-resolution image. STEM uses a beam of electrons to probe the sample and obtain chemical and structural information. Cryo-TEM operates at cryogenic temperatures, allowing for the study of delicate biological samples. Electron microscopy for nanotechnology has numerous applications, including the study of materials for electronic and energy applications, the development of new drug delivery systems, the investigation of cellular and molecular structures, and the characterization of nanoscale devices and materials [7 - 10]. The electron microscope, which uses electrons instead of light to magnify images, was first developed in the 1930s and revolutionized the field of microscopy [11, 12]. In the early days of electron microscopy, the technology was primarily used for imaging biological samples, but as the field developed, researchers began to apply the technology to the study of materials at the nanoscale. The development of electron microscopy was closely tied to advancements in the field of physics [13 - 16]. In the late 19th and early 20th centuries, scientists were exploring the properties of electrons and the way they interacted with matter. This research laid the foundation for the development of the electron microscope, which would use electrons to image samples and reveal their structure at an incredibly high level of detail [17]. In the 1930s, two German scientists, Max Knoll and Ernst Ruska, independently developed early versions of the electron microscope. These first-generation electron microscopes were large, complex devices that required a high level of expertise to operate, but they were capable of producing images of biological samples with a much higher level of detail than was possible with light microscopes [18 - 20]. In the decades that followed, advances in technology allowed for the development of smaller, more accessible electron microscopes. These microscopes made it possible for researchers to study a wider range of samples, including inorganic materials and materials at the nanoscale. In the 1960s and 1970s, the field of electron microscopy underwent a major expansion as researchers began to develop new techniques for imaging materials at the nanoscale [21]. This was a critical

development, as it allowed scientists to study materials in much greater detail than was previously possible. With the ability to see materials at the nanoscale, scientists were able to discover new properties and behaviours that could not be observed at the macroscale. One of the key applications of electron microscopy in nanotechnology is imaging materials at the atomic scale. This allows researchers to study the structure and composition of materials at the smallest possible level, which can provide important insights into their properties and behaviour [22, 23]. In recent years, electron microscopy has also been used to study materials in various states, including liquids, gases, and even plasmas. Electron microscopy has also been critical in the development of other important technologies, such as nanolithography and nanofabrication. Nanolithography involves the patterning of materials at the nanoscale, and electron microscopy is used to verify the accuracy and precision of these patterns. Nanofabrication involves the creation of nanoscale structures and devices, and electron microscopy is used to study and refine these structures during the fabrication process [24, 25]. Today, electron microscopy is an essential tool in the field of nanotechnology and materials science, allowing researchers to study materials at the smallest possible scale and uncover new properties and behaviours. The field of electron microscopy continues to evolve and advance, with new techniques and innovations being developed all the time.

TYPES OF ELECTRON MICROSCOPY

Electron microscopes are a key tool in nanotechnology, allowing scientists and engineers to visualize, analyse and manipulate materials at the nanoscale. There are several types of electron microscopes, each with its advantages and limitations. Here are the most common types:

1. Transmission Electron Microscope (TEM): This type of microscope uses a beam of electrons to form an image of a thin sample. The electrons pass through the sample and are scattered, forming an image of the internal structure. TEMs are used for high-resolution imaging and analysis of a variety of materials, including metals, ceramics, and biological samples [26].

2. Scanning Electron Microscope (SEM): This type of microscope uses a beam of electrons to scan the surface of a sample, producing a topographical image. SEMs are used for surface imaging and analysis, and can also be used to obtain information about the chemical composition of a sample [27]. The schematic representation of the Field Emission Scanning Electron Microscope (FE-SEM) is illustrated in Fig. (1).

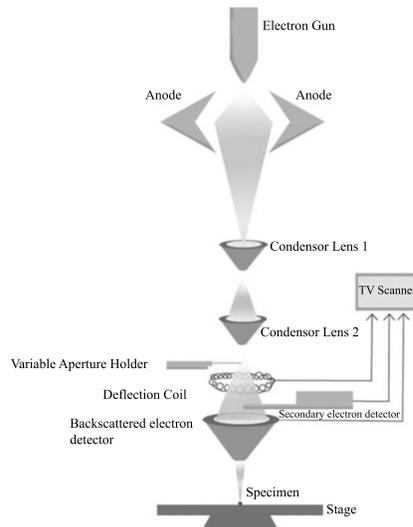


Fig. (1). Schematic diagram of Field Emission Scanning Electron Microscope (FE-SEM).

3. Scanning Transmission Electron Microscope (STEM): This type of microscope combines the features of a TEM and an SEM, allowing for high-resolution imaging of both the surface and internal structure of a sample. STEMs are often used for imaging and analysis of materials in the fields of electronics, materials science, and biology [28]. The schematic diagram of the Transmission Electron Microscope (TEM) is shown in Fig. (2) and the Schematic diagram of Energy Dispersive X-ray analysis in the TEM is illustrated in Fig. (4).

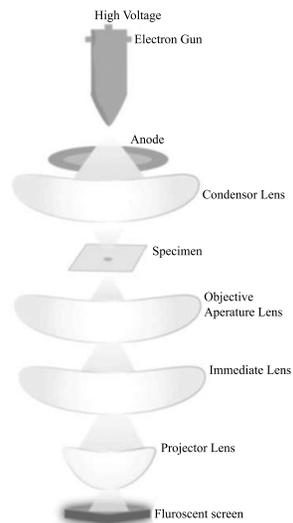


Fig. (2). Schematic diagram of Transmission Electron Microscope (TEM).

4. High-Resolution Transmission Electron Microscope (HRTEM): This type of TEM is specifically designed for high-resolution imaging, allowing scientists to visualize the internal structure of materials at the atomic scale. HRTEMs are used for a variety of applications, including the study of materials for electronic and optical applications, as well as the study of biological materials [29].

5. Environmental Electron Microscope (EEM): This type of electron microscope allows scientists to study materials under controlled environmental conditions, such as temperature, pressure, and gas composition. EEMs are used for a variety of applications, including the study of materials for energy and environmental applications, as well as the study of materials for biological and medical applications [30]. The schematic diagram of the Environmental Scanning Electron Microscope (ESEM) is shown in Fig. (6).

6. Electron Spectroscopy for Chemical Analysis (ESCA): This type of electron microscope uses electrons to analyse the chemical composition of a sample. ESCA is used for a variety of applications, including the study of materials for electronic and optical applications, as well as the study of biological materials [31]. The schematic diagram of photoemission electron microscopy is illustrated in Fig. (3).

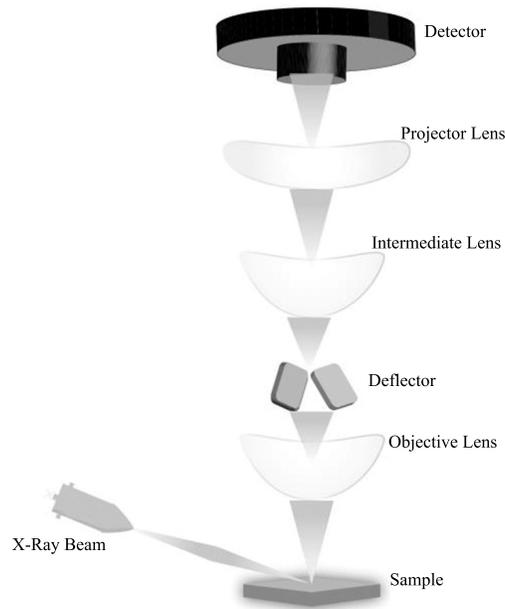


Fig. (3). Schematic diagram of photoemission electron microscopy.

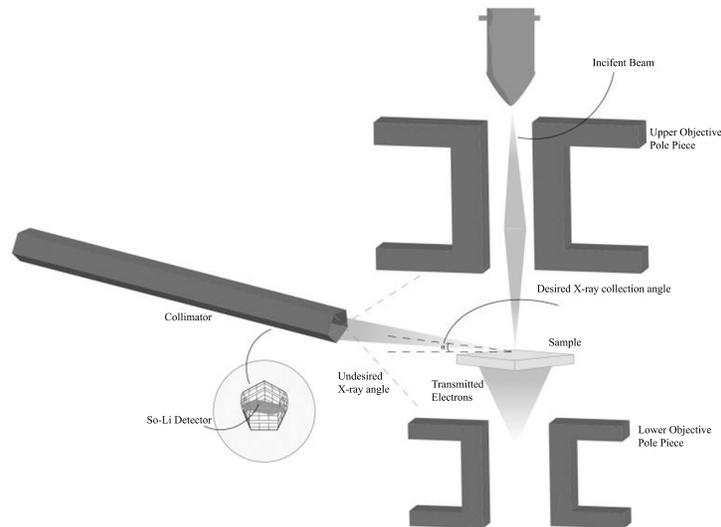


Fig. (4). Schematic diagram of energy dispersive X-ray analysis in the TEM.

7. Low-Voltage Electron Microscope (LVEM): This type of electron microscope uses a lower voltage electron beam, allowing for the imaging and analysis of biological materials without damage to delicate structures. LVEMs are used for a variety of applications in the fields of biology and medicine [32].

SAMPLE PREPARATION

Preparation of the sample is critical to obtaining accurate and high-resolution images with an electron microscope. This chapter will discuss various methods of preparing samples for electron microscopy, including thin-film preparation, embedding, and staining. It will also discuss the importance of controlling the sample's environment, including temperature and pressure, during imaging. Sample preparation is a critical step in the use of electron microscopes for nanotechnology [33]. The quality of the sample preparation will have a direct impact on the quality of the images and data obtained from the electron microscope. One of its kind is EPMA (Electron Probe Micro Analyser). The schematic diagram of the electron-electron probe micro analyser is illustrated in Fig. (5). Here are some key steps in the sample preparation process:

1. Sample Selection: The first step in sample preparation is selecting a suitable sample. The sample should be representative of the material being studied and should be of sufficient size and quality to allow for imaging and analysis in the electron microscope.

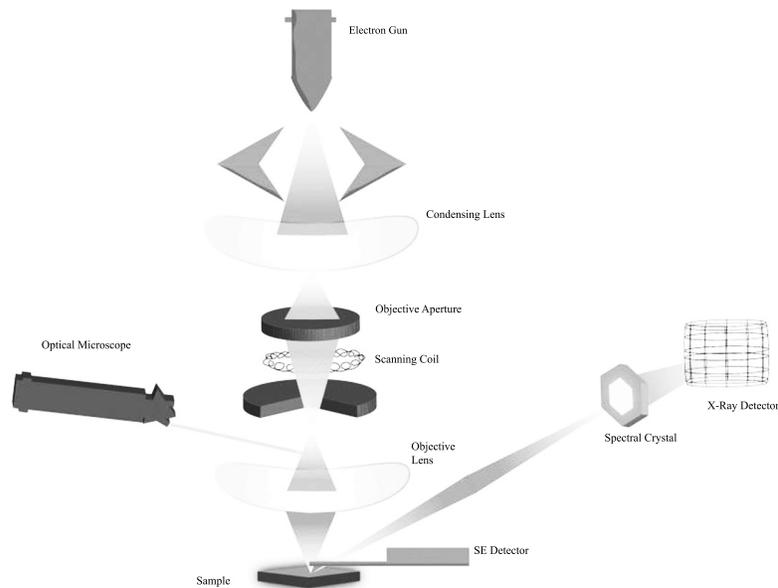


Fig. (5). Schematic diagram of electron probe micro analyser (EPMA).

2. Sample Preparation: Once a suitable sample has been selected, it must be prepared for analysis in the electron microscope. This often involves cutting, polishing, and thinning the sample to create a thin, electron-transparent section that can be imaged in the electron microscope.

3. Specimen Preparation for SEM: For samples to be imaged in a Scanning Electron Microscope (SEM), they must be coated with a conductive material, such as gold or platinum, to prevent charging and to improve the quality of the images.

4. Specimen Preparation for TEM: For samples to be imaged in a Transmission Electron Microscope (TEM), they must be prepared as ultra-thin sections that are electron transparent. This often involves preparing a “shadow” replica of the sample using a variety of techniques, such as the replication of the surface of the sample with a thin layer of material or the use of a focused ion beam to mill a thin section from the sample.

5. Specimen Preparation for STEM: For samples to be imaged in a Scanning Transmission Electron Microscope (STEM), they must be prepared as ultra-thin sections that are electron transparent. This often involves preparing a “shadow” replica of the sample using a variety of techniques, such as the replication of the surface of the sample with a thin layer of material or the use of a focused ion beam to mill a thin section from the sample [34].

6. Specimen Preparation for EEM: For samples to be imaged in an Environmental Electron Microscope (EEM), they must be prepared in a way that allows for the controlled exposure of the sample to specific environmental conditions, such as temperature, pressure, and gas composition. This often involves the use of specialized sample holders and the preparation of the sample in a controlled environment.

7. Specimen Preparation for LVEM: For samples to be imaged in a Low-Voltage Electron Microscope (LVEM), they must be prepared as thin sections that are electron transparent, but are also able to withstand the low-energy electron beam used in LVEMs. This often involves the use of special sample preparation techniques, such as the use of low-energy beam damage mitigation techniques, to minimize damage to delicate biological structures [35]. Fig. (6) represents the schematic of Environmental Scanning Electron Microscope (ESEM)

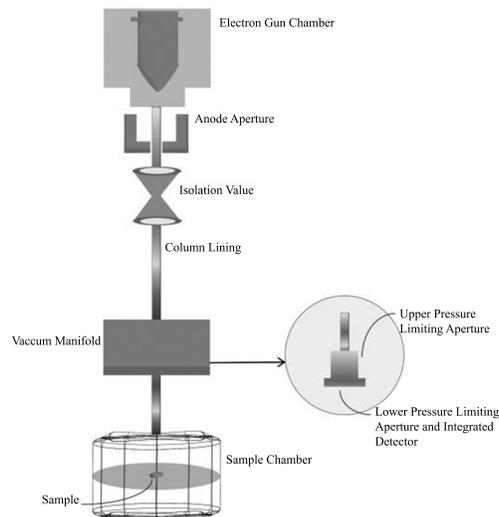


Fig. (6). Schematic of Environmental Scanning Electron Microscope (ESEM).

IMAGING TECHNIQUES

We will discuss various imaging techniques that can be used with electron microscopes, including bright-field imaging, dark-field imaging, and annular dark-field imaging. It will also cover advanced imaging techniques, such as electron tomography and holography. Image techniques play a crucial role in the use of electron microscopes for nanotechnology. The schematics of low-energy electron microscopy are shown in Fig. (7). Electron microscopes provide high-resolution images that can reveal the detailed structure and composition of materials at the nanoscale [36]. Here are some of the key image techniques used in electron microscopy for nanotechnology:

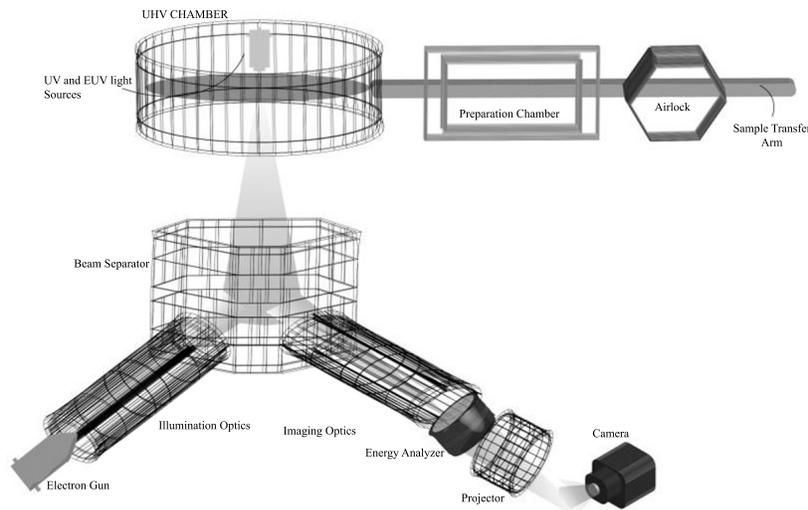


Fig. (7). Schematics of low-energy electron microscopy.

1. Bright Field Transmission Electron Microscopy (TEM): Bright field transmission electron microscopy (BF-TEM) is a widely used imaging technique in the field of nanotechnology. BF-TEM works by transmitting electrons through a thin sample and imaging the electrons that are transmitted through the sample. The resulting image provides information about the sample's composition and structure. In BF-TEM, a beam of electrons is directed through a thin sample, and the electrons that are transmitted through the sample are collected by a detector. The electrons that are transmitted through the sample are the ones that are not absorbed or scattered by the sample's atomic structure. This results in a bright background and dark contrast for the atomic columns in the sample, hence the name "bright field." BF-TEM provides high-resolution images of materials and is commonly used for imaging materials in the nanoscale, including metals, ceramics, polymers, and biological materials. BF-TEM is particularly useful for imaging materials with a high atomic number, as the electrons are more likely to be absorbed by the material, providing better contrast in the image. One of the main advantages of BF-TEM is its high resolution, which allows researchers to observe the details of the atomic structure of a sample [37]. This high resolution is possible because of the small wavelength of electrons, which is much smaller than the wavelength of light used in other imaging techniques, such as optical microscopy. BF-TEM also provides information about the chemical composition of the sample, which is particularly useful for the study of materials at the nanoscale. Another advantage of BF-TEM is that it is a non-destructive technique, meaning that the sample is not altered or damaged during the imaging process. This is important for the study of delicate and sensitive materials, such as

biological samples or materials with complex structures. BF-TEM also provides information about the three-dimensional structure of the sample, as the electrons are transmitted through the entire thickness of the sample, rather than just the surface. However, BF-TEM also has some limitations. One of the main limitations is that it requires a thin sample to be prepared, as electrons can only be transmitted through thin samples. This can be a time-consuming and complex process, particularly for samples that are not naturally thin or that have complex structures. Additionally, BF-TEM requires the sample to be in a vacuum, as the electrons cannot be transmitted through air. This can also present challenges for the study of biological samples, as they are sensitive to the vacuum conditions required for BF-TEM.

2. Dark Field Transmission Electron Microscopy (TEM): Darkfield transmission electron microscopy (DF-TEM) is an imaging technique used in the field of nanotechnology to study the structure and composition of materials. It is similar to bright field transmission electron microscopy (BF-TEM), but instead of imaging the electrons transmitted through the sample, DF-TEM images the electrons that are scattered by the sample's atomic structure [38]. In DF-TEM, the electrons are directed towards the sample at an angle and those that are scattered by the sample's atomic structure are collected by a detector, while the electrons transmitted through the sample are blocked by an aperture. This results in a dark background and bright contrast for the atomic columns in the sample. DF-TEM provides high-resolution images of materials and is commonly used for imaging materials in the nanoscale, including metals, ceramics, polymers, and biological materials. DF-TEM is particularly useful for imaging materials with low atomic numbers, as the electrons are less likely to be absorbed by the material, providing better contrast in the image. One of the main advantages of DF-TEM is its ability to image materials that are not visible in BF-TEM, such as interfaces between two materials, grain boundaries, or defects in the sample. DF-TEM provides a clearer representation of the sample's structure and can provide information about the sample's composition and crystal structure. Another advantage of DF-TEM is its non-destructive nature, which is particularly important for the study of delicate and sensitive materials, such as biological samples. DF-TEM also provides information about the three-dimensional structure of the sample, as the electrons are scattered through the entire thickness of the sample [39].

However, like BF-TEM, DF-TEM also has some limitations. One of the main limitations is that it requires a thin sample to be prepared, as electrons can only be scattered through thin samples. This can be a time-consuming and complex process, particularly for samples that are not naturally thin or that have complex structures. Additionally, DF-TEM also requires the sample to be in a vacuum, as electrons cannot be scattered through air [40].

3. High-Angle Annular Dark Field Scanning Transmission Electron Microscopy (HAADF-STEM): High-Angle Annular Dark Field Scanning Transmission Electron Microscopy (HAADF-STEM) is a type of imaging technique used in nanotechnology to study the structures of materials at the nanoscale [41]. The technique involves the use of a transmission electron microscope (TEM) to probe the sample with a beam of electrons and image the resulting scattered electrons. In HAADF-STEM, the scattered electrons are collected using an annular detector, which captures electrons that are scattered at high angles with respect to the incident electron beam. HAADF-STEM is particularly useful for imaging heavy atoms in a sample, as these atoms scatter electrons more strongly than lighter atoms. This makes it possible to produce high-resolution images of material structures that contain heavy atoms, such as interfaces and grain boundaries in metals and alloys. The technique is also useful for imaging materials with low atomic numbers, such as carbon-based materials, where other imaging techniques, such as bright field TEM, may not provide enough contrast for clear imaging. HAADF-STEM imaging can be performed in either high-resolution or low-resolution mode, depending on the desired imaging goal. High-resolution mode provides images with high spatial resolution, making it possible to resolve fine details in the material structure. Low-resolution mode provides images with lower spatial resolution but a higher signal-to-noise ratio, making it possible to image larger structures or material distributions. The HAADF-STEM imaging process starts with preparing a thin sample of the material to be imaged. This can be done using a variety of techniques, including mechanical thinning, chemical etching, or ion beam milling. Once the sample has been prepared, it is loaded into the TEM and positioned in the electron beam. The incident electron beam is then focused onto the sample, and the resulting scattered electrons are collected using the annular detector. The detector collects electrons scattered at high angles with respect to the incident electron beam, providing a high-resolution image of the material structure [42]. HAADF-STEM images can be analysed to determine the chemical composition and crystal structure of the material, as well as its electrical and mechanical properties. This information is critical for understanding the properties of materials at the nanoscale and for developing new materials with improved properties. In addition to imaging, HAADF-STEM can also be used for spectroscopic analysis, such as energy-dispersive x-ray spectroscopy (EDS) and x-ray fluorescence (XRF), which provide information on the chemical composition of the sample. EDS and XRF use X-rays emitted from the sample to determine the elements present and their distribution. This information can be combined with the HAADF-STEM imaging data to produce a more complete picture of the material structure and properties. One of the major advantages of HAADF-STEM is its ability to provide high-resolution imaging of materials with low atomic numbers, such as carbon-based

materials. This is particularly important in the field of nanotechnology, where the development of new materials with improved properties is a key goal. By providing detailed information on the structure and properties of these materials, HAADF-STEM can help researchers develop new materials with improved properties and performance.

4. Scanning Electron Microscopy (SEM): Scanning electron microscopy (SEM) is a type of electron microscopy that generates images of a sample surface by scanning the sample with a focused beam of electrons and detecting the electrons that are emitted from the sample. It is widely used in nanotechnology for imaging and analysis of materials at a nanoscale resolution. In SEM, the electrons interact with the sample to produce a signal that contains information about the sample's composition and topography. This signal is then processed to produce an image. SEM images typically have a high resolution, with the ability to resolve features as small as a few nanometres. There are different imaging modes in SEM that are used to generate images with different information content, such as secondary electron imaging, backscattered electron imaging, and X-ray energy-dispersive spectroscopy (EDS). Secondary electron imaging provides information about the sample's surface topography, while backscattered electron imaging provides information about the sample's composition and bulk structure. EDS provides a chemical analysis of the sample, with the ability to identify the elemental composition and chemical state of the sample. SEM is a powerful tool for imaging and analysing materials at the nanoscale and has wide applications in materials science, biology, electronics, and many other fields. The schematics of wavelength dispersive spectroscopy (WDS) is shown in Fig. (8).

5. Low-Voltage Electron Microscopy (LVEM): Low-voltage electron microscopy (LVEM) is a type of electron microscopy used for imaging samples at low electron beam energies, typically less than 10 kV. This allows for the imaging of delicate or biological samples without damaging them. The imaging techniques used in LVEM include:

a) Bright Field Imaging: This is the most basic form of LVEM imaging, where the electrons are transmitted through the sample to form an image based on the transmission and absorption of electrons by the sample.

b) Dark Field Imaging: This is an alternative to bright field imaging, where electrons that are scattered by the sample are collected to form an image. This can be useful for detecting structures that are not visible in bright-field imaging.

c) Z-Contrast Imaging: This imaging technique is used to determine the composition of the sample based on the contrast between different elements. This

can be useful for imaging samples with different atomic numbers or for imaging samples with light elements.

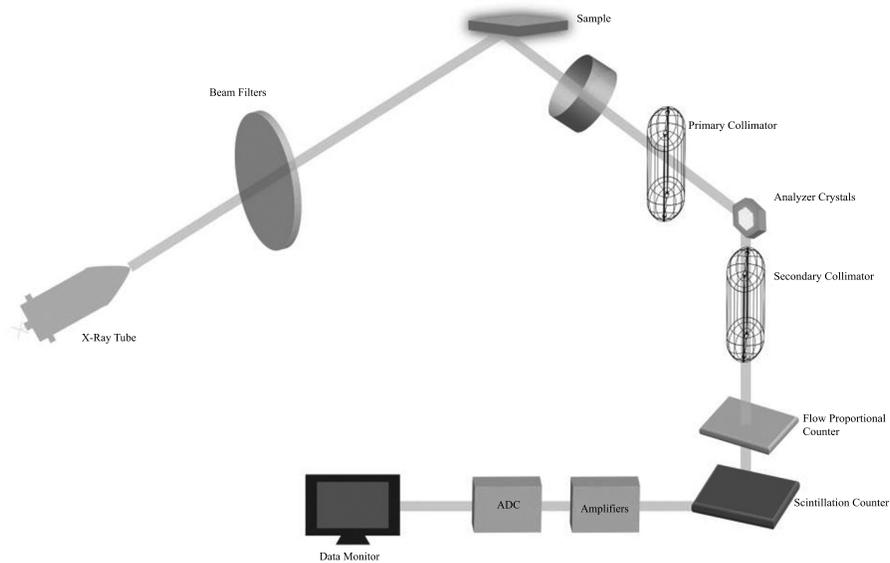


Fig. (8). Schematics of wavelength dispersive spectroscopy (WDS).

6. Scanning Transmission Electron Microscopy (STEM): This is a type of LVEM that uses a focused electron beam to scan across a sample and collect the electrons that are transmitted through the sample. STEM can provide high-resolution images of the sample structure, including atomic arrangements.

7. Energy-Dispersive X-Ray Spectroscopy (EDS): This is a technique used in conjunction with LVEM to determine the elemental composition of the sample. EDS measures the energy of the x-rays emitted by the sample as the electron beam is scanned across it. These techniques allow LVEM to provide high-resolution images of delicate or biological samples with minimal damage, making it a valuable tool for nanotechnology research and development.

8. Energy-Filtered Transmission Electron Microscopy (EFTEM): Energy-filtered transmission electron microscopy (EFTEM) is an imaging technique used in nanotechnology to study the chemical composition of materials at the nanoscale. It works by filtering the transmitted electrons through a monochromator and detecting the electrons that have lost a specific amount of energy due to interactions with the sample [43]. This energy loss information is then used to produce an elemental map of the sample, providing information about the distribution of different elements in the material. EFTEM can be combined with other imaging modes, such as dark field and bright field imaging, to obtain