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Review on Characterization of Composite Materials with Mechanical Loading Using Different Microscopic Examinations for Engineering Applications

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Abstract: Composite materials and advanced systems including ceramics, metal matrix composites, and nano-structured materials possess intricate microstructures and multifunctional characteristics. Their performance is influenced by the individual components, as well as by factors such as interfacial bonding, filler distribution and microstructural evolution during use. Precise characterization is essential for : (i) Correlate structure with properties (ii) Identify flaws or defects (iii) Predict durability and failure modes (iv) Develop next-generation high-performance materials. Due to their heterogeneous and often anisotropic nature, composite materials require highly advanced characterization techniques. Microscopy has emerged as an indispensable tool for examining microstructural features, phase distributions, fibre/matrix interfaces, defect formations and damage progression across multiple length scales. Recent advances in instrumentation and imaging methods have elevated microscopy to unprecedented levels of resolution and analytical power, providing deeper insights and improving composite performance. This chapter emphasizes the pivotal role of advanced characterization techniques in optimizing materials for structural composites, energy storage systems and semiconductor technologies. Combining scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) greatly improves our ability to decode the complex chemical and structural properties of advanced materials. Additionally, thermos-gravimetric analysis (TGA) and differential scanning calorimetry (DSC) provide valuable information on thermal stability and how materials behave in real-world conditions. In particular, these characterization techniques have become vital for studying dynamic changes and degradation mechanisms in energy storage

devices during operation. These techniques are categorized based on their primary focus: topological, structural, and compositional analysis. This chapter discusses about how advanced characterization drives the design, synthesis, and application of composite materials across diverse technological fields.

Index-Terms: Composite materials and advanced systems, Microstructures, Characterization techniques, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR).

I. INTRODUCTION

In the rapidly evolving landscape of science and engineering, materials are no longer viewed as static building blocks. Instead, they are increasingly being designed to interact dynamically with their environment, adjusting their behavior and properties in real time. These materials, often termed smart functional composites or intelligent materials, represent a fundamental shift in how we think about functionality at the material level. Unlike traditional materials, which are passive and require external systems to monitor and control environmental changes, smart materials embed functionality within the material itself. This functionality includes the ability to sense stimuli, analyze data, and trigger specific responses all without the need for human intervention or complex external circuits. This marks the beginning of a new era in materials science, where intelligence is no longer confined to electronic devices but is integrated into the very matter that composes them [1]. This chapter explores the fundamental concepts, classifications, and mechanisms of smart composites focusing especially on polymer and fiber-based materials, which have garnered significant global interest due to their lightweight nature, ease of fabrication, and versatile functional characteristics. These smart composites are engineered at the molecular and microscopic levels to exhibit properties such as shape memory, conductivity, elasticity, and chemical reactivity, which are fine-tuned to respond to a variety of physical and chemical stimuli [2-3].

The concept of material intelligence may seem futuristic, but its origin is deeply rooted in one of nature’s oldest survival mechanisms the sensory-feedback loop found in biological organisms. In the natural world, organisms thrive by continuously monitoring their surroundings, processing sensory data, and responding appropriately to ensure survival. Whether it's a Venus flytrap snapping shut upon detecting prey, a chameleon adjusting its color based on the environment, or human skin responding to changes in temperature and pressure, nature has long demonstrated the sense-react-learn paradigm. This biomimetic philosophy where engineering solutions are inspired by biological processes has given rise to smart materials that emulate these natural behaviors. The foundational idea is to develop materials that are no longer passive but instead intelligent and adaptive, just like living systems. In this context, the term “material intelligence” refers to the capacity of a material to detect a stimulus, process the information, and respond functionally and efficiently, often in a reversible and repeatable manner [4]. These intelligent materials rely on molecular-level design and structural configurability, meaning that their physical or chemical properties change dynamically in response to environmental factors. For instance, some smart polymers can expand or contract when exposed to heat, alter conductivity in response to an electric field, or change color under different lighting conditions. Similarly, pH-sensitive hydrogels can swell or shrink based on the acidity of the environment, making them useful in drug delivery systems where timed and targeted release is crucial [5-8].

1.1 Classification and Mechanisms of Smart Composites

Smart composites are a fascinating class of materials engineered to exhibit dynamic behavior in response to external stimuli. Typically, they are hybrid systems composed of functional fillers such as nanoparticles, nanofibers, or specialized particles embedded within a polymeric or fibrous matrix. The matrix provides structural integrity, while the fillers introduce functionality, such as conductivity, magnetism, or responsiveness to environmental changes. It is the synergistic interaction between these two components that enables the composite to adapt its properties dynamically, offering capabilities far beyond those of traditional materials. The underlying mechanisms of smart composites often involve changes at the molecular or microstructural level, triggered by specific stimuli. These changes manifest macroscopically as variations in mechanical strength, shape, color, electrical conductivity, or permeability, among other properties. Understanding the classification of smart composites based on their stimuli-responsiveness is essential for their effective design and application in various fields, from aerospace to biomedical engineering [9-11].

1..1.1 Thermo-responsive materials

Thermo-responsive smart composites exhibit changes in shape, stiffness, volume, or mechanical behavior when exposed to variations in temperature. The mechanism is often rooted in phase transitions, such as glass transition or crystalline melting, within the polymer matrix or filler. A common example is shape memory polymers (SMPs), which can "remember" a programmed shape and recover it upon heating. Such materials find extensive applications in actuators, self-healing structures, and biomedical devices like stents and sutures [12].

1.1.2 Electro-responsive polymers

Electro-responsive, or electroactive, smart composites respond to electric fields by undergoing mechanical deformation (such as bending, stretching, or contracting) or by changing their electrical conductivity. These composites often utilize fillers like carbon nanotubes, conductive polymers, or metallic particles to achieve the desired response. They are pivotal in the development of artificial muscles, flexible electronics, sensors, and adaptive optics. The ability to control motion or conductivity with an applied voltage opens pathways for highly precise and programmable systems [13].

1.1.3 Magneto-responsive composites

Magneto-responsive smart composites alter their mechanical properties such as stiffness, damping, or orientation under the influence of a magnetic field. These composites typically incorporate ferromagnetic or superparamagnetic fillers like iron oxide nanoparticles into a flexible matrix. When exposed to a magnetic field, the fillers align or reconfigure, leading to macroscopic changes in the material's behavior. Such materials are ideal for applications requiring remote control, such as soft robotics, targeted drug delivery systems, and adaptive vibration dampers. 1.1.4 Photo-responsive smart composites [14-15].

Photo-responsive smart composites react to light stimuli usually in the UV, visible, or infrared ranges by undergoing changes in their optical, chemical, or physical properties. Mechanisms include photo isomerization, photo thermal effects, or photo crosslinking within the matrix or fillers. Light exposure can trigger shape changes, color transitions, or conductivity variations, offering a non-invasive and precise method to control material behavior. These materials are being explored for use in smart windows, optical switches, medical diagnostics, and light-driven actuators [16]. The responsive behavior of smart composites is typically characterized by rapid, reversible, and, in many cases, programmable transitions. This adaptability allows engineers and scientists to design systems with significant control over both form and function, enabling innovation across a wide range of industries. As research advances, the boundary between passive and active materials continues to blur, ushering in an era where materials are not just structural elements but active participants in sensing, actuating, and interacting with their environment.

1.2 Integration with Sensors and Actuators and Role of Smart Composites in Robotics and Automation

In the rapidly evolving fields of robotics and automation, the seamless integration of sensors (for data acquisition) and actuators (for mechanical response) is a fundamental requirement. Traditionally, this integration has relied on separate, often bulky, components, leading to limitations in system miniaturization, responsiveness, and flexibility. Smart composites, however, offer a transformative solution by embedding both sensory and actuation functionalities directly within the structural material itself. This innovation paves the way for more compact, efficient, and autonomous robotic systems [17-18]. Materials embedded with piezoelectric fibers, conductive nanoparticles, or responsive polymers are common examples of such smart sensing technologies. The benefits of self-healing smart composites extend far beyond mere convenience. By prolonging structural longevity, enhancing safety, and reducing maintenance costs, these materials are revolutionizing sectors where reliability and durability are paramount. In the automotive industry, self-healing paints and body panels are helping to minimize the need for frequent repairs. In aerospace applications, self-healing composites are critical for maintaining the structural health of aircraft and spacecraft, where even minor damage could otherwise have catastrophic consequences. Meanwhile, in civil infrastructure, incorporating self-healing materials into bridges, buildings, and pipelines promises to dramatically reduce the long-term costs and risks associated with structural degradation [19-22]. The compactness, sensitivity, and multi-functionality of smart composites deliver several crucial advantages for robotics and automation:

- Enhanced autonomy: Systems can sense, process, and react to stimuli without external controllers or extensive wiring.
- Lightweight design: Reduces the load and energy consumption, critical for wearable robotics and mobile systems.
- Greater flexibility and adaptability: Essential for soft robotics, where traditional rigid sensors and actuators are impractical.
- Improved reliability and durability: Embedded functionalities reduce mechanical failure points associated with separate components.

In applications ranging from wearable exoskeletons that assist human movement, to adaptive soft robots that navigate complex environments, to responsive control surfaces in aerospace, smart composites are rapidly becoming the backbone of next-generation robotics. Their ability to bridge the gap between sensing and actuation not only enhances system performance but also opens new frontiers for creating robots that are more intuitive, resilient, and intimately integrated with their environments. Few industries demand as much from materials as aerospace and defense, where weight reduction, strength, adaptability, and resilience are mission-critical. Smart composites are being integrated into aircraft wings, satellite components, and military gear, delivering multifunctional capabilities. Adaptive structures with morphing surfaces can change shape during flight to optimize aerodynamics, improving fuel efficiency and maneuverability. Embedded sensors within the material matrix enable real-time damage detection, allowing for predictive maintenance and enhancing safety. In defense applications, lightweight armor made from smart composites can offer enhanced impact resistance while maintaining flexibility and reduced bulk. These innovations are setting new standards for performance, durability, and survivability in some of the environments imaginable [23-26].

1.3 Mechanical Performance and Characterization of Smart Composites Under Loading

The mechanical performance of smart composites under cyclic and fatigue loading conditions has emerged as one of the most crucial areas of research in recent years. Unlike traditional materials, smart composites not only bear mechanical loads but also exhibit active responses to those loads, which adds an additional layer of complexity to their behavior. Researchers are particularly interested in how repeated mechanical stress over time affects critical material properties such as the elastic modulus (a measure of stiffness), damping capacity (the ability to absorb energy), and the onset of failure mechanisms like crack initiation, delamination, or permanent deformation [27-28]. To accurately predict how these materials will perform in real-world applications, extensive experimental studies are conducted, subjecting smart composites to cyclic loading over thousands or even millions of cycles. During these studies, scientists monitor changes in mechanical properties, the formation of microstructural damage, and the eventual point of material failure. The data gathered from such tests are invaluable for developing computational models and simulations that forecast the life expectancy of smart composites, predict degradation patterns, and identify critical thresholds under extreme operational conditions such as high-stress, high-frequency, or variable-temperature environments. In fields like robotics, prosthetics, and wearable technology, ensuring material durability is not just about enhancing performance. It can mean the difference between life and death. In a robotic prosthetic limb, for example, a sudden material failure due to fatigue could result in serious injury to the user. Smart composites help mitigate such risks by offering embedded sensing capabilities, allowing continuous real-time monitoring of stress, strain, and internal damage. Some advanced systems even enable adaptive responses, such as adjusting stiffness or initiating self-healing processes at early stages of damage, thereby significantly extending the service life of the component and improving user safety [29-31].

Beyond performance enhancement, the use of smart composites reduces maintenance costs and downtime across industries. In sectors like aerospace, where maintenance schedules are strictly regulated and costly, the ability of smart materials to detect and potentially repair minor damages without requiring extensive human intervention represents a transformative shift. The combination of mechanical resilience, self-monitoring, and adaptive healing positions smart composites at the forefront of next-generation materials engineering. When subjected to an electric field, materials like electroactive polymers and specially designed smart fabrics exhibit mechanical deformation or conductivity changes. This electro-mechanical coupling can be harnessed for creating artificial muscles, soft robotics, or responsive clothing that adjusts fit or insulation properties based on electrical signals [32].

Magnetic fields elicit responses in magnetorheological elastomers and fluids, which can alter their stiffness, shape, or orientation almost instantaneously. Such materials are particularly useful for applications requiring rapid, tunable mechanical performance, such as adaptive vibration dampers or variable-stiffness joints in prosthetics and robotic systems. Under the influence of mechanical stress, smart composites embedded with sensors can detect deformation and convert it into measurable signals. Self-healing polymers go even further by autonomously responding to mechanical damage: the closing of cracks and reformation of material bonds occur without external repair efforts, significantly boosting the structural integrity and longevity of the component. By leveraging these diverse stimuli-responses, smart composites enable the development of products and systems that are not only responsive and adaptive but also self-sufficient and highly efficient. This versatility positions smart composites at the heart of future innovations across industries such as consumer electronics, healthcare, transportation, renewable energy, and defense [33].

1.3.1 Challenges and Future Directions on Smart Composite Materials

While the field of smart composites has achieved remarkable breakthroughs, it is important to recognize that several significant challenges continue to hinder their widespread adoption and commercial success. As with any emerging technology, the transition from laboratory innovation to real-world application is fraught with technical, economic, and logistical obstacles. One of the foremost challenges is scalability. Producing smart composites with consistent properties on an industrial scale remains a major hurdle. In laboratory settings, strict control over material composition, microstructure, and manufacturing conditions is relatively manageable. However, when moving to mass production, achieving the same level of uniformity and reliability across large batches becomes exponentially more difficult. Variations in the dispersion of functional fillers, inconsistencies in the matrix material, and manufacturing defects can lead to unpredictable performance, limiting the practical use of these materials in critical applications like aerospace, medical devices, and automotive structures. Closely tied to scalability is the issue of cost. Many high-performance smart materials rely on complex, multi-step fabrication techniques, the use of rare or expensive raw materials, or intricate post-processing treatments such as surface functionalization or Nano structuring. As a result, the final products are often prohibitively expensive for widespread use, especially in cost-sensitive industries. Reducing production costs without sacrificing functionality remains one of the primary goals for researchers and manufacturers alike. Advances in additive manufacturing (3D printing), self-assembly techniques, and scalable chemical synthesis methods are being actively explored to address this critical bottleneck. Another major obstacle is integration. Seamlessly incorporating smart composites into existing systems whether in robotics, biomedical devices, infrastructure, or consumer electronics without compromising the overall performance is a delicate balancing act. Traditional systems are often not designed to accommodate materials that actively change properties in response to stimuli. As a result, careful engineering is needed to ensure that the dynamic behavior of smart composites enhances, rather than destabilizes, the performance and reliability of the broader system. Furthermore, issues related to signal processing, power supply for active components, and long-term stability under operating conditions must also be carefully addressed during integration.

Another vital area is the push to enhance biocompatibility for medical applications. As smart composites find increasing use in fields like tissue engineering, bio-sensing, and implantable devices, ensuring that these materials are not only functional but also safe and harmonious with biological systems is essential. Researchers are exploring bioinspired materials, biodegradable smart composites, and the integration of natural polymers to create medical devices that are both high-performing and biologically inert or even capable of encouraging tissue regeneration. Despite the current challenges, the trajectory of innovation in smart composites remains incredibly promising. As fabrication techniques mature, costs decline, integration becomes more seamless, and intelligence is increasingly embedded at the material level, smart composites are poised to reshape the future of engineering, healthcare, robotics, energy, and beyond. The next decade is likely to witness a convergence of materials science, artificial intelligence, and biotechnology, leading to smart composite systems that are not only reactive but also predictive, autonomous, and capable of self-optimization truly ushering in an era of intelligent materials.

II. ADVANCED CHARACTERIZATION TECHNIQUES FOR COMPOSITE MATERIALS

From the figure 1, this chapter provides a detailed review of key characterization techniques for composite structures and nanoparticles, organized into three categories based on their analytical focus: topological, internal structural, and compositional investigations.

- ➢ Internal Structure Characterization:
  1. Scanning Electron Microscopy (SEM)
  2. Transmission electron microscopy (TEM)

3. Magnetic resonance force microscopy (MRFM)
4. X-ray diffraction (XRD)

- Topological Characterization:
  1. Field emission scanning electron microscopy (FESEM)
  2. Scanning probe microscopy (SPM)
  3. Optical microscopy methods
  4. Confocal microscopy and near-field scanning optical microscopy (NSOM)
  5. Dynamic light scattering (DLS)
- Compositional Characterization:
  1. X-ray photoelectron spectroscopy (XPS)
  2. Energy-dispersive X-ray spectroscopy (EDS)
  3. Secondary ion mass spectrometry (SIMS)
  4. Auger electron spectroscopy (AES)

2.1 Research Study: Application of Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) for Crystallographic and Microstructural Characterization

Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) are highly versatile and powerful techniques for characterizing materials in two and three dimensions. Offering exceptional spatial resolution from the nanoscale to the microscale—these methods provide detailed imaging and chemical analysis, making them ideal complements to the other nondestructive techniques covered in this book. This chapter introduces the fundamental principles of electron microscopy and outlines key SEM and TEM methodologies. It explores the signals produced by electron-matter interactions and their applications in various SEM/TEM techniques. Additionally, the chapter demonstrates how these methods are used to analyze critical microstructural features, such as surface topography, grain size, and local chemical composition, while highlighting current and emerging applications in materials science. The growing range of detectors and instruments available for materials characterization has equipped researchers with an increasingly diverse toolkit for investigating material behavior. Advances in instrumentation and analytical workflows have greatly improved our ability to capture and interpret complex, multimodal data, yielding deeper insights into material structure and performance. By integrating data across multiple length scales and combining in situ with ex situ analyses, researchers can develop a more holistic understanding of dynamic processes and structural properties. However, merging these diverse data modalities presents significant challenges due to differences in resolution, dimensionality, and underlying physical principles. Overcoming these challenges demands innovative approaches to accurately align and interpret heterogeneous datasets.

The papers in this special topic collectively examine the acquisition and integration of multimodal data to advance the study of structural materials. These contributions showcase various methodologies including segmentation frameworks, process monitoring tools, simulation improvements, and mechanical response assessments that highlight the critical role of multimodal data fusion in contemporary materials research [34–36] as sown in Figure 1.

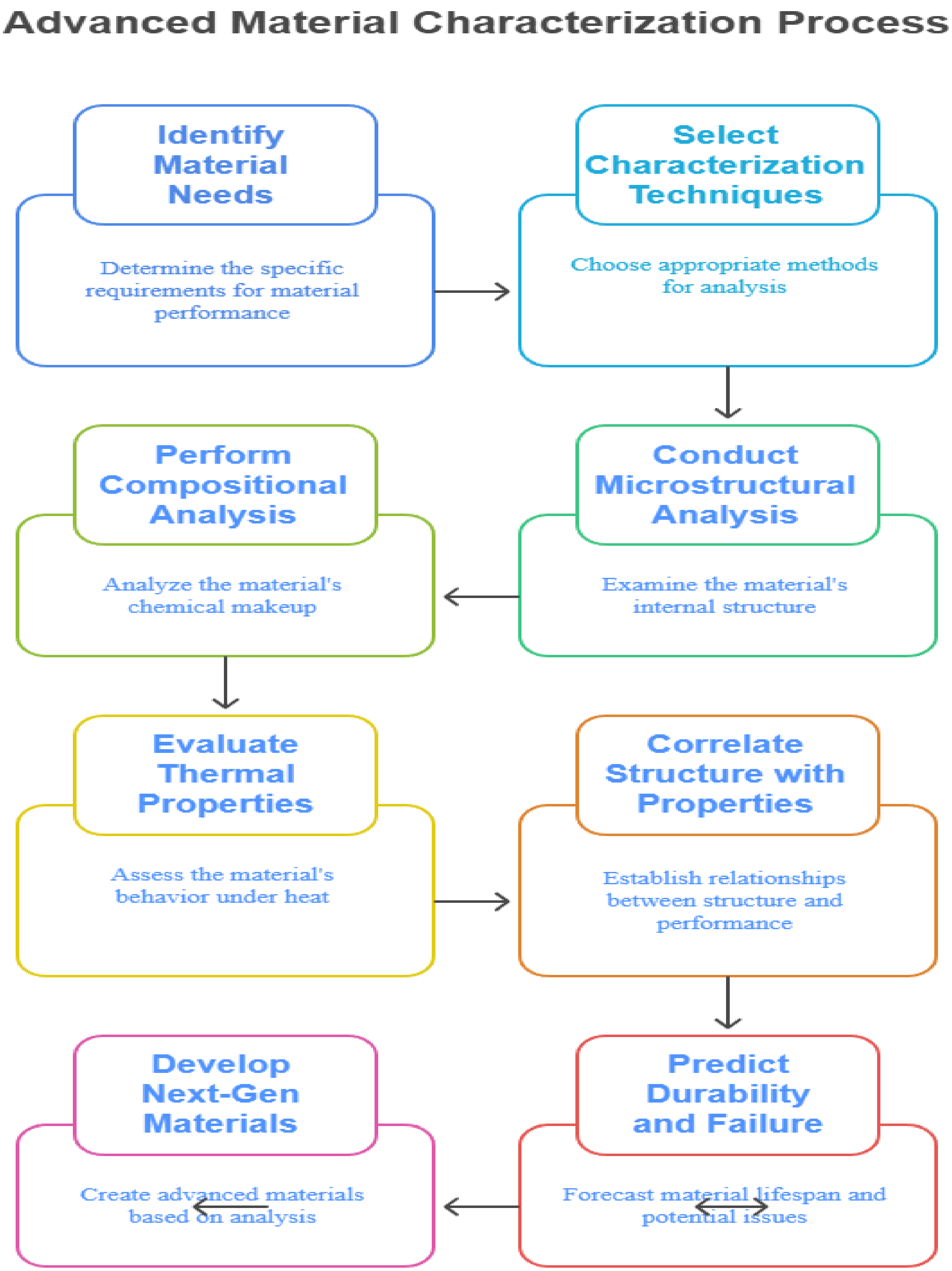


Figure 1: Steps involved in Advanced Materials Characterization Process

Understanding the crystallographic and microstructural features of materials is essential for predicting their mechanical, electrical, and thermal behavior as shown in Figure 2. SEM and TEM are two of the most widely used electron microscopy techniques in materials science, offering powerful tools for imaging and analysis. This research explores the strengths of both SEM and TEM in assessing key features such as grain boundaries, dislocations, phase interfaces, and local chemistry. This study investigates the application of Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) in the crystallographic and microstructural characterization of advanced engineering materials. By combining high-resolution imaging with analytical capabilities, SEM and TEM offer complementary insights into grain morphology, phase distribution, crystal orientation, and defect structures across multiple length scales [37-38].

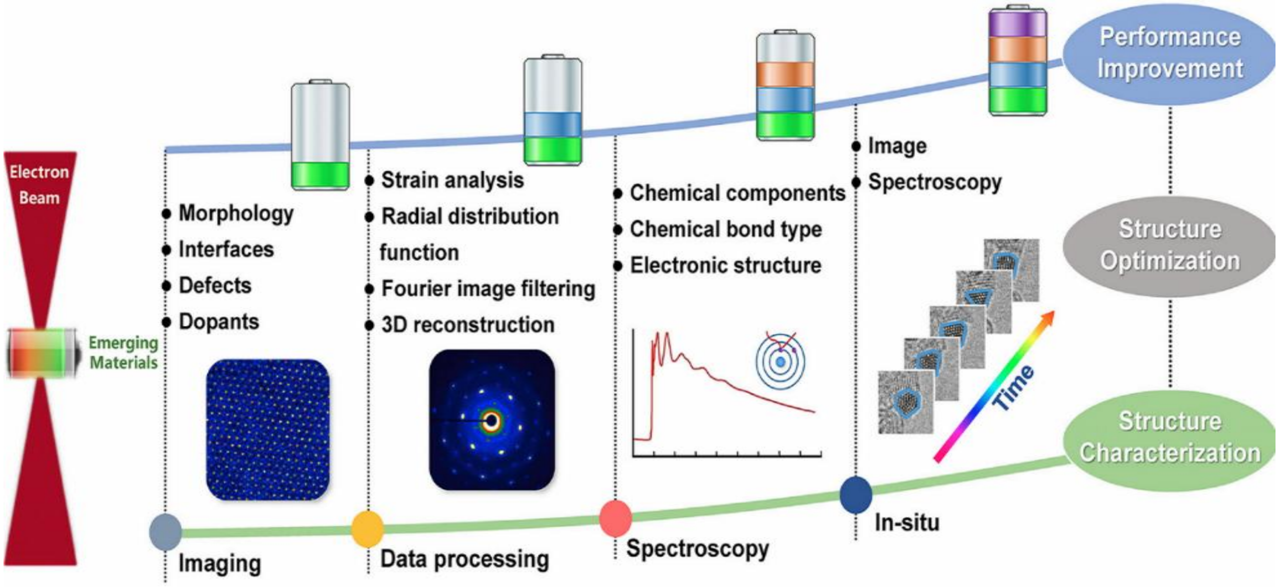


Figure 2. Schematic illustration of the analytical TEM techniques for emerging advanced materials (Courtesy: Yue Lin et al, https://doi.org/10.1016/j.matt.2021.05.005)

With significant advancements in spatial resolution and associated analytical capabilities, Transmission Electron Microscopy (TEM) has enabled detailed probing of materials at the atomic scale. However, a gap still exists that limits the broader application of TEM-based techniques in the characterization of emerging advanced materials. This review outlines the fundamental principles and functionalities of analytical TEM in this context, emphasizing not only its ability to visualize atoms and crystal lattices but also its capacity to extract in-depth insights from acquired data. We classify recent developments in analytical TEM into four main categories: advanced scanning TEM (STEM) imaging techniques, STEM-based data processing methods, electron energy loss spectroscopy (EELS), and in situ TEM techniques. This review aims to encourage wider adoption of analytical TEM in materials science and to facilitate a deeper understanding of structure–property relationships in next-generation materials. Advanced TEM techniques have played a crucial role in uncovering the origins of functional properties in emerging advanced materials at the atomic scale. However, the full potential of these techniques including their associated analytical methodologies has yet to be fully realized in practical materials research. This underutilization is likely due to disconnect between the materials science and electron microscopy communities. Drawing on our group's experience in both TEM and materials research, this review highlights the application of advanced analytical TEM techniques specifically tailored for characterizing emerging materials. While comprehensive overviews of general TEM methods already exist, our focus here is on advanced techniques that go beyond atomic imaging to extract meaningful insights from TEM data. From the figure 3, we categorize these techniques into four main areas: advanced scanning TEM (STEM) imaging, STEM-related data processing, electron energy loss spectroscopy (EELS), and in situ TEM. This review outlines the core principles and significant applications of these emerging approaches and aims to assist researchers in conducting deeper, more informed analyses of TEM data in the context of materials science [39-41].

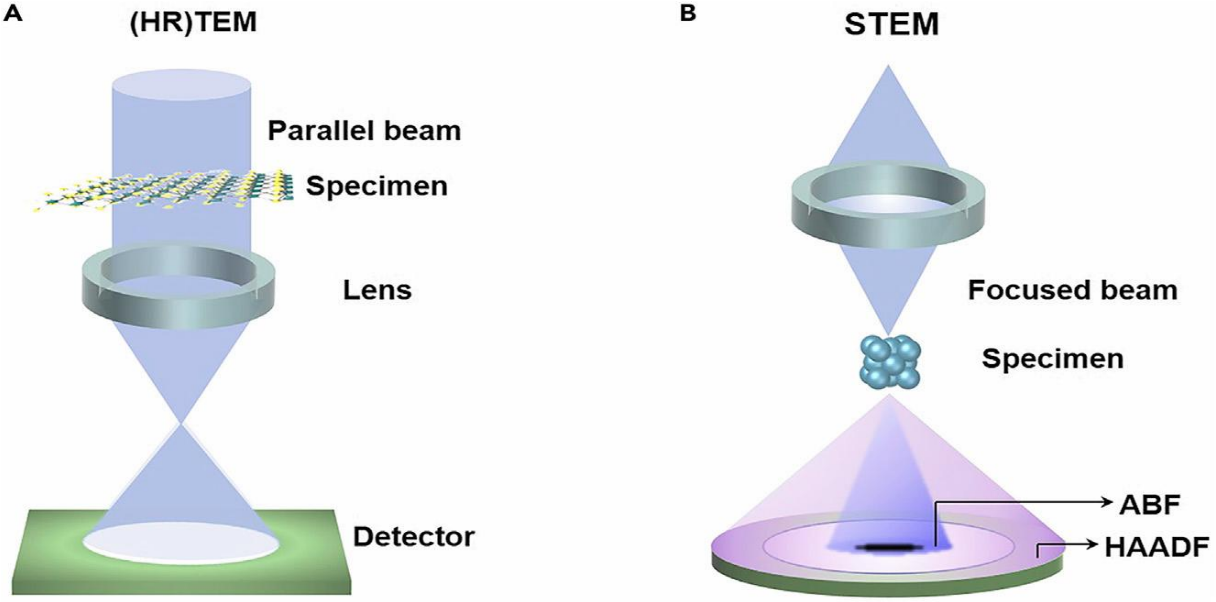


Figure 3. Typical imaging modes of (A) (HR) TEM and (B) STEM (Courtesy: Yue Lin et al, https://doi.org/10.1016/j.matt.2021.05.005)

Electron beam-sensitive materials, such as metal-organic frameworks (MOFs) and covalent-organic frameworks (COFs), have attracted significant attention due to their wide-ranging applications in gas adsorption and storage, photoelectric conversion, heterogeneous catalysis, and energy storage and conversion. Understanding their atomic structure is critical for elucidating structure–property relationships and for guiding the rational design of novel materials with enhanced performance. However, their high sensitivity to electron irradiation presents a major challenge for transmission electron microscopy (TEM), as conventional electron beams can cause substantial structural damage, hindering accurate structural characterization. Although annular bright-field scanning transmission electron microscopy (ABF-STEM) has been widely employed for imaging light elements, its effectiveness is limited by a low signal-to-noise ratio (SNR), contrast reversal due to defocus, and sensitivity to sample thickness variations. A more recent advancement, integrated differential phase contrast (iDPC) STEM, addresses many of these limitations. This technique employs a four-quadrant detector to capture differential phase contrast signals, allowing for physical analysis of electrons in the bright-field region. By integrating the x and y components of the DPC signal, a final image is reconstructed through two-dimensional integration. iDPC-STEM significantly enhances sensitivity to light elements and allows for imaging with an ultra-low electron dose (~40 $e^-/Å^2$), far lower than the dose typically required in high-resolution TEM (often $>10^3$ $e^-/Å^2$). Moreover, the higher SNR of iDPC-STEM images compared to ABF-STEM further supports its capability to achieve atomic-resolution imaging of beam-sensitive materials like MOFs and COFs, which are otherwise prone to damage under conventional high-energy electron exposure.

Advanced scanning transmission electron microscopy (STEM) has emerged as a remarkably powerful technique for directly visualizing the atomic structure and chemistry of a wide range of materials. Although electron microscopes have historically provided nanometer-scale resolution, the advent of aberration-correction technology in the early 21st century has dramatically improved imaging capabilities, pushing resolution to the sub-angstrom level [42]. This breakthrough in spatial resolution, combined with heightened imaging precision, has opened up new frontiers in electron microscopy. Utilizing an angstrom-sized electron probe, STEM allows researchers to directly observe atomic-scale structural and chemical information. For example, annular dark-field (ADF) STEM imaging leverages atomic number (Z) contrast to distinguish individual dopant atoms within bulk materials [43-44]. More recent advances, such as electron ptychography, have demonstrated resolutions as fine as 39 picometers, enabling visualization of interstitial atoms within a host matrix [45-46]. Furthermore, STEM can be seamlessly integrated with spectroscopic techniques like energy-dispersive X-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS), enabling detailed analysis of atomic-scale composition, electronic structure, and even vibrational properties [47]. Recent techniques such as electron ptychography [44], 4D-STEM [48], and ultra-high energy resolution electron energy loss spectroscopy (EELS) now facilitate the characterization of complex parameters including polarization, chemical and structural ordering, oxidation states, and electronic configurations at sub-nanometer scales. Despite the widespread availability of modern electron microscopes across both academic and industrial settings, a significant challenge remains: bridging the communication and knowledge gap between developers of electron microscopy techniques and researchers in materials science [49-50]. Nevertheless, it is essential for scientists from both fields to appreciate the expansive utility of STEM in the characterization of multifunctional materials. This perspective outlines recent innovations in STEM instrumentation and methods, highlighting their broad relevance in ferroic materials research. It also explores emerging opportunities for advanced data analysis to address fundamental and applied questions in materials science [51-52].

In the sections that follow, we outline various software tools developed to correct distortions in electron microscopy data and facilitate simplified post-processing. One widely adopted technique for mitigating sample drift and distortion involves capturing a sequence of images and aligning them during post-processing. Recent advancements in scanning and transmission electron microscopy (S/TEM) instrumentation have significantly improved usability and data acquisition efficiency. Modern aberration-corrected microscopes are now commonly equipped with automated tuning software, enabling rapid alignment and corrector adjustments within minutes. These improvements, combined with enhanced mechanical and thermal stability, have streamlined the operation of high-performance microscopes [53-54]. In many shared user facilities, electron microscopes are housed in carefully engineered environments that minimize external interferences such as mechanical vibrations, acoustic noise, temperature fluctuations, and magnetic fields. As a result, high-quality data can now be collected more efficiently, allowing researchers to devote more time to post-processing and analysis. Another method for correcting scan distortion involves calibration using a reference sample with a well-defined structure (e.g., silicon or strontium titanate). Measured distortions from the reference can then be corrected in experimental data via affine transformations. For non-linear drift correction, orthogonal image pairs rotated 90° relative to each other can be aligned by fitting contrast variations line-by-line along the slow scan direction. While only two images are required, incorporating additional orthogonal images can improve the correction's robustness. By employing these post-processing techniques, researchers can obtain STEM images free from artifacts caused by drift and scanning distortions, ensuring greater accuracy in subsequent quantitative analyses [55-56].

2.2 CHARACTERIZATION OF X-RAY DIFFRACTION TECHNIQUES FOR EMERGING ADVANCED MATERIALS – RESEARCH STUDY

Magnetic Resonance Force Microscopy (MRFM) and X-ray Diffraction (XRD) serve as powerful, complementary techniques for crystallographic and microstructural analysis across different spatial scales. MRFM merges magnetic resonance imaging with atomic force microscopy to achieve ultra-high-resolution 3D subsurface imaging. Capable of detecting individual electron or nuclear spins, MRFM provides nanoscale mapping of magnetic environments, chemical composition, and spin-lattice interactions - offering unique insights into complex materials, especially low-dimensional or non-crystalline systems [57]. XRD, the established foundation of crystallographic analysis, delivers non-destructive, bulk-sensitive measurements of lattice parameters, crystal orientation, phase identification, and strain states. Advanced XRD methods like synchrotron diffraction and grazing incidence XRD enable precise characterization of crystallite size, micro strain, and texture in polycrystalline and thin-film materials. These techniques form a synergistic pair: XRD provides statistically robust bulk structural data, while MRFM reveals nanoscale heterogeneities. Their combined application has significantly advanced understanding of structure-property relationships in nanocomposites, semiconductors, and biomaterials, demonstrating the power of multimodal characterization in modern materials science. Specific topics discussed include the interpretation of XRD patterns to gain insights into crystalline structure, grain size and orientation, dislocation density, phase identification and quantification, as well as phase transformation, lattice parameters, residual stress and strain, and thermal expansion coefficients. By consolidating these critical discussions, this review aims to provide valuable insights for specialists and engineers working across the chemical, mining, metallurgy, iron, and steel industries [58-59].

X-ray diffraction (XRD) is a fundamental and widely utilized technique for material characterization. As advancements in materials science continue to lead to the development of novel and complex materials, there is a growing need to enhance existing analytical methods to effectively address emerging challenges. Despite being a well-established, non-destructive technique, XRD still demands improvements in its characterization capabilities, particularly when applied to materials with intricate mineral structures. This comprehensive review explores key aspects of XRD, including atomic crystal structure fundamentals, the principles of XRD, its diverse applications, potential sources of uncertainty in analysis, and necessary safety considerations. The review also outlines future directions for research, highlighting the integration of artificial intelligence (AI) and machine learning (ML) to enhance the precision, efficiency, and interpretability of XRD data in mineral characterization [60-62].

Analytical X-ray diffraction (XRD) continues to be a cornerstone technique for characterizing emerging advanced materials, offering insights into crystallographic structures, phase compositions, and microstructural properties. Recent research has expanded its capabilities, integrating advanced methodologies and technologies to address the complexities of novel materials.

2.2.1 Key Applications in Emerging Materials using X-ray diffraction techniques

Nanomaterials: XRD is pivotal in determining the crystalline structure and size of nanoparticles, aiding in the development of materials with tailored properties.

Thin Films: Utilized to assess the crystallographic orientation, phase purity, and stress in thin-film materials, which are critical in electronics and coatings.

High-Temperature Materials: Real-time XRD analysis at elevated temperatures helps in understanding phase stability and transformations, essential for materials used in extreme environments.

Complex Oxides and Minerals: Advanced XRD techniques, combined with AI, enhance the identification and analysis of complex mineral structures, facilitating the discovery of new materials.

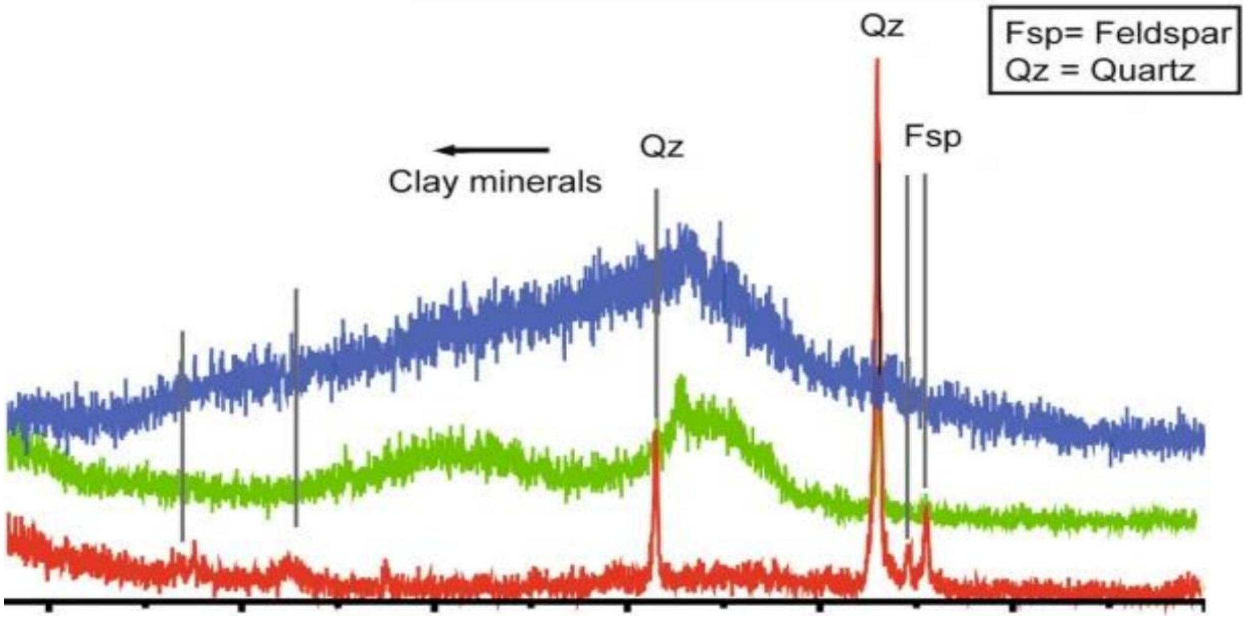


Figure 3. XRD was used for understanding the role of various treatments on the crystallinity of peat (a mixture of organic matter and minerals): (a) sample material without treatment (blue); (b) H_2O_2

chemical oxidation treatment (green); (c) combustion treatment (red) [Courtesy: Fultz et al, 2013 and Lamas et al 2017].

When X-rays interact with solid materials, they are scattered by the electrons orbiting atomic nuclei. These scattered waves propagate in various directions and can interfere either constructively or destructively, depending on their phase relationships and directions of travel. Diffraction specifically refers to the constructive interference of these scattered X-rays. This phenomenon is a direct result of the periodic atomic arrangement within crystalline materials, which facilitates coherent scattering. Therefore, accurate interpretation of XRD patterns relies on understanding the periodicity of the material's atomic structure. A key correlation exists: shorter periodicities produce higher diffraction angles, and vice versa [63]. Lamas et al. [64] illustrated the contrast in XRD patterns between amorphous and crystalline materials (Figure 3). In amorphous materials, lacking long-range order, the XRD pattern typically shows a broad hump or a single broad peak, representing average atomic distances. In contrast, crystalline substances display multiple sharp peaks, reflecting their well-defined atomic periodicity. XRD is also a powerful tool for assessing changes in crystallinity due to thermal or chemical treatments. For example, a recent study [65-66] used XRD to investigate the effects of different treatment methods on sample crystallinity. Their findings revealed that combustion treatment significantly enhanced crystallinity, as indicated by sharper and more defined XRD peaks (Figure 3). Additionally, XRD plays a critical role in studying phase transformations and constructing phase diagrams. While techniques such as transmission electron microscopy (TEM), electrical resistivity measurements, nuclear magnetic resonance (NMR), and differential scanning calorimetry (DSC) can aid in such analysis, XRD uniquely provides direct information about phase identity on both sides of a phase boundary. Phase transformations depend on parameters such as composition, time, and heat treatment temperature [67]. In alloy systems, XRD can map regions of varying crystal phases and is extensively used to investigate temperature-composition phase diagrams. By recording XRD patterns during controlled heating or cooling, shifts in peak positions can be observed, which correlate with changes in lattice constants—sensitive indicators of temperature and compositional variation [68]. As a practical example, Ashiri et al. synthesized tetragonal barium titanate ($BaTiO_3$) nanoparticles via the xerogel method. The xerogel method is a process used to create porous materials, often in the form of gels, which are dried to remove liquid while retaining their structural framework. Xerogels are widely used in applications like catalysis, drug delivery, sensors, and insulation due to their high surface area and porosity. Heating the xerogel from 800 °C to 1100 °C in 100 °C increments resulted in peak splitting near $2\theta = 45°$, as shown in Figure 14, signifying a polymorphic phase transformation. This demonstrates how XRD, as a non-destructive technique, enables precise monitoring of structural transitions during material synthesis [69-71].

2.3. ANALYTICAL AND CHARACTERIZATION OF FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR) FOR EMERGING ADVANCED MATERIALS – RESEARCH STUDY

Fourier-transform infrared spectroscopy (FTIR) is a widely adopted analytical technique that plays a vital role in the structural, chemical, and compositional analysis of emerging advanced materials. FTIR enables the identification and quantification of molecular species based on their vibrational modes, which are characteristic of specific chemical bonds and functional groups. As the field of material science progresses toward the development of novel and hybrid materials, including metal-organic frameworks (MOFs), covalent-organic frameworks (COFs), nanocomposites, biopolymers, and 2D materials, the demand for reliable and non-destructive spectroscopic techniques like FTIR has significantly increased [72-73]. In pharmaceuticals, FTIR aids in drug formulation analysis and the quality control of biopharmaceuticals. In biomedical research, Rubin et al. applied FTIR to distinguish between normal and aneurysmal human aortic tissues, attributing the differences mainly to protein composition [74-77]. Fourier-transform infrared (FTIR) spectroscopy is a versatile and widely adopted analytical technique based on the vibrational and rotational motions of atoms within molecules. As this technique relies on the interaction between infrared radiation and molecular dipole moments, FTIR is capable of identifying molecules through their unique vibrational fingerprints. Variations in atomic composition, bonding configurations, and spatial arrangements lead to distinct FTIR spectra for different substances. In principle, when a beam of continuous infrared light is directed at a sample, photons at specific wavenumbers corresponding to the energy difference between quantized molecular vibrational states are absorbed. This absorption manifests as an FTIR spectrum, revealing characteristic functional groups and molecular structures due to the constant vibrational motion of chemical bonds within the molecule [77-82].

FTIR spectroscopy is based on the principle that molecules absorb infrared radiation at specific wavelengths corresponding to the vibrational transitions of their constituent atoms. These absorbance features produce a unique spectral "fingerprint" that can be used to identify both organic and inorganic compounds [83]. In advanced materials research, FTIR is especially effective for confirming the presence of functional groups, evaluating chemical modifications, monitoring polymerization or crosslinking reactions, and assessing interfacial interactions in composites or hybrid structures. For example, in MOFs and COFs, FTIR is employed to monitor the coordination bonding between metal nodes and organic linkers, or to detect the incorporation of functional groups designed to enhance adsorption or catalytic activity [84]. In polymeric nanomaterials, FTIR is crucial for tracking chemical changes during synthesis, such as the conversion of monomers to polymers or the introduction of dopants. Moreover, for graphene oxide and functionalized carbon nanotubes, FTIR provides insights into surface oxidation, reduction, and the success of functionalization processes. Recent advancements in FTIR instrumentation, such as attenuated total reflectance (ATR-FTIR) and micro-FTIR imaging, have further enhanced its application scope. ATR-FTIR allows for the direct analysis of solid, liquid, or gel samples with minimal preparation, while micro-FTIR enables spatially resolved chemical mapping, which is especially beneficial for heterogeneous or patterned materials [85-89].

Furthermore, FTIR spectroscopy is increasingly being integrated with computational modeling and machine learning to improve spectral interpretation and to predict material properties based on vibrational signatures. These developments make FTIR a powerful tool for in situ monitoring of chemical processes and real-time quality control in material synthesis and processing as shown in

Figure 4.

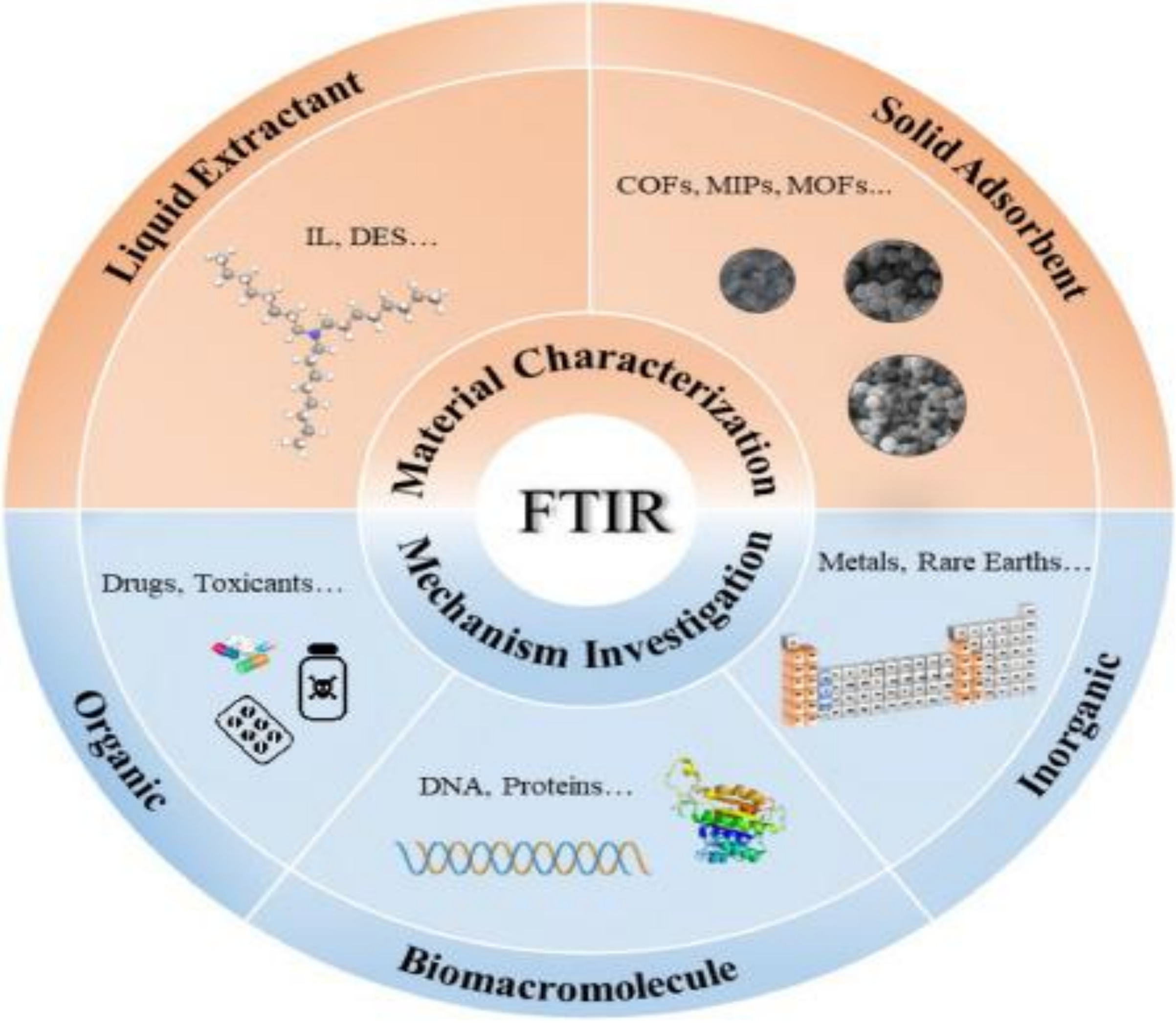


Fig. 4. Application scope of FTIR in sample preparation

Modern FTIR spectroscopy can be performed in several modes, including transmission, attenuated total reflection (ATR), and imaging-based modalities. These advancements have expanded the applicability of FTIR beyond traditional uses, enabling fast, accurate qualitative and quantitative assessments across diverse fields. Since the 1970s, FTIR has become a critical tool in disciplines such as food science, pharmaceutical analysis, life sciences, environmental science, traditional Chinese medicine, materials science, and forensics. In forensics, FTIR has proven capable of detecting subtle biochemical differences in decomposed tissues, aiding in post-mortem analysis, semen stain identification, and estimation of time of death. In metabolomics, FTIR enables rapid screening of plasma composition following protein precipitation using various solvents such as methanol, acetonitrile, and acetone. FTIR has also gained significance in sample preparation and material characterization, where it is employed to analyze the chemical structure of solid sorbents, liquid extractants, and separation media. It provides crucial insights into extraction mechanisms by identifying functional groups involved in interactions between target molecules and extraction matrices. These interactions may include hydrogen bonding, acid-base coordination, or water-mediated transport, depending on the physicochemical nature of the extractants [91-96].

Given its broad capabilities and non-destructive nature, FTIR continues to be an essential technique for understanding the molecular architecture of materials and intermolecular interactions. Its role in elucidating chemical bonding and facilitating the design of efficient extraction and separation systems underscores its importance in the development and characterization of emerging advanced materials. In conclusion, FTIR spectroscopy remains a cornerstone analytical technique in the characterization of emerging advanced materials. Its ability to provide rapid, non-destructive, and detailed chemical information makes it indispensable for understanding the structure–property relationships, guiding material design, and validating functional performance. Ongoing technological and methodological innovations continue to expand its applicability and resolution, reinforcing FTIR's role in next-generation material research

2.4 CHARACTERIZATION OF MAGNETIC RESONANCE FORCE MICROSCOPY (MRFM) FOR EMERGING ADVANCED MATERIALS

Magnetic Resonance Force Microscopy (MRFM) represents a highly sensitive and non-destructive analytical tool that bridges the spatial resolution of scanning probe microscopy with the chemical specificity of magnetic resonance techniques. This hybrid methodology is particularly relevant for the analytical characterization of emerging advanced materials, which often possess nanoscale features, complex architectures, and unique magnetic or electronic properties. MRFM operates by detecting the minute magnetic forces between electron or nuclear spins in a sample and a ferromagnetic tip attached to an ultra-sensitive cantilever. Under resonance conditions established via radio-frequency (RF) fields, these forces cause cantilever oscillations, which are measured to generate spatially resolved magnetic resonance data [97-98]. This allows for three-dimensional imaging with nanometer or even sub-nanometer resolution exceeding the capabilities of conventional MRI and often outperforming other nanoscale characterization tools in sensitivity and depth profiling. MRFM has proven especially valuable in materials where conventional techniques like TEM, SEM, or bulk NMR fall short such as non-crystalline, beam-sensitive, or spin-limited systems. For instance, MRFM enables the detection of single electron spins, paving the way for quantum-level insights into material behavior [99].

Scanning tunneling microscopy (STM), first introduced in 1981, enabled the direct imaging of individual atoms on a surface in real space. This breakthrough spurred a rapid expansion in scanning probe microscopy (SPM), leading to the development of various techniques that use a physical probe to scan across a sample while recording measurements at each point to generate surface images. In the field of magnetic imaging, some of the most sensitive and high-resolution techniques rely on scanning probes. Magnetism plays a crucial role in various biological systems, yet the typically weak magnetic forces involved require the development of highly sensitive tools for accurate detection [100-101]. In this context, magnetic force microscopy (MFM) provides outstanding lateral resolution and enables single-molecule investigations, similar to other single-probe microscopy (SPM) techniques. This comprehensive review explores the critical significance of magnetic forces in biological applications, emphasizing both the key advantages and inherent limitations of MFM. Alongside an in-depth explanation of its operational principles, the review highlights recent advancements in micro- and nanofabrication techniques for MFM tips that significantly improve the magnetic signal response from biomaterials compared to standard commercial probes. It also presents notable case studies demonstrating MFM's capability to quantitatively evaluate the magnetic behavior of biological nanomaterials, such as magnetotactic bacteria, cryptochrome flavoproteins, and magnetic nanoparticles interacting with animal tissues. Finally, the review outlines the most promising future directions in this field, addressing upcoming challenges and opportunities related to emerging quantum technologies [102-104].

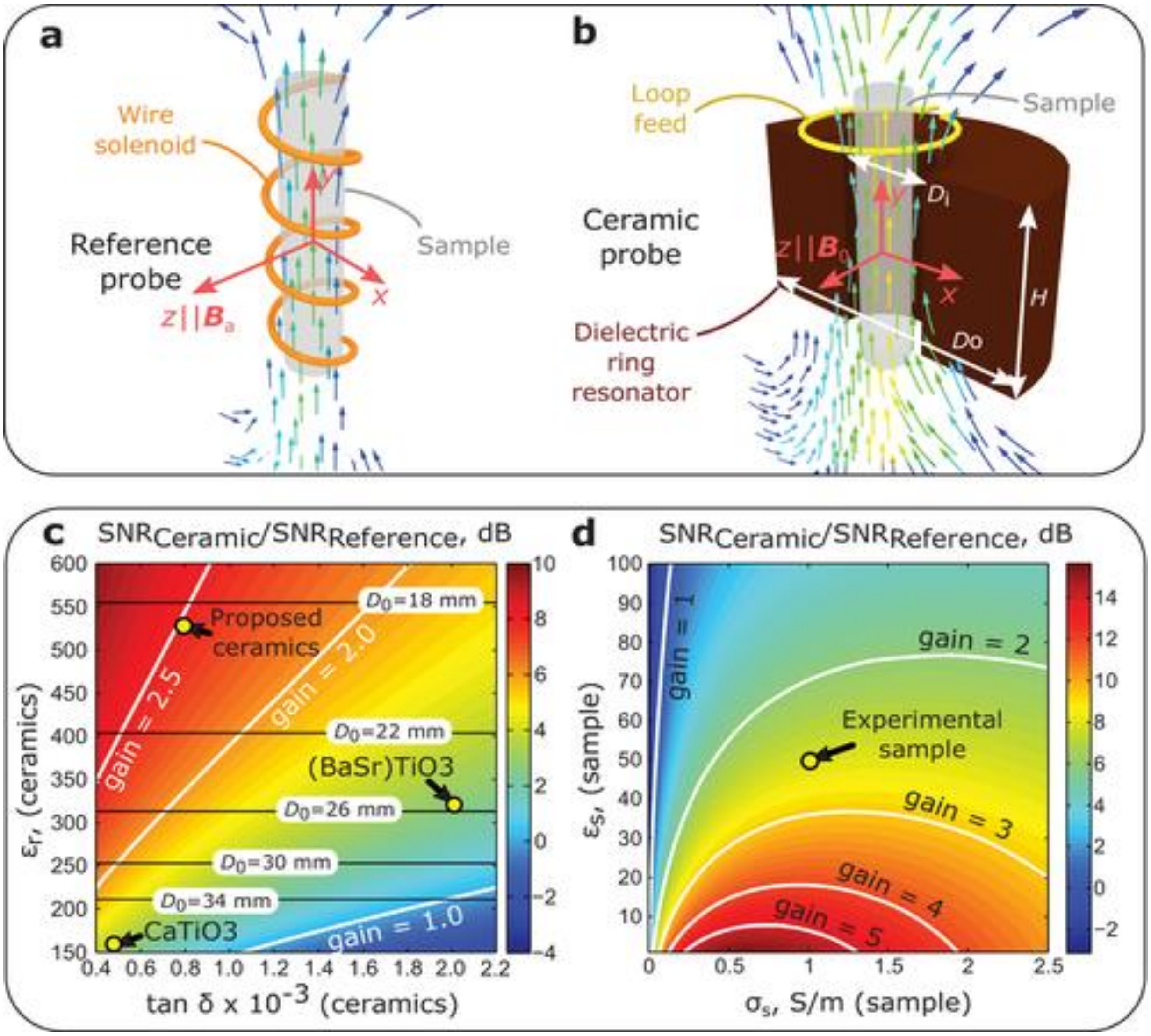


Figure 5: Characterization of magnetic resonance force microscopy (MRFM) for emerging advanced materials

This technique detects magnetic forces between nuclear spins in a sample and a magnetic tip, making it suitable for analyzing non-magnetic materials. It integrates the principles of magnetic resonance imaging (MRI) with magnetic force microscopy (MFM). Magnetic resonance force microscopy

(MRFM) operates by applying a radiofrequency (RF) pulse to polarize the nuclear spins in the sample. When the RF pulse matches the resonance frequencies of the nuclear spins and magnetic particles, they absorb energy and transition to higher energy states. The resulting mechanical movements from these nuclear spin transitions are sensed by the AFM tip, allowing for the reconstruction of three-dimensional images that reflect the spatial distribution of magnetism, aligned with the tip's position (Figure 5) [105-106].

The magnetic moment sensitivity (μ) of MRFM is expressed as:

$$\mu = \surd(1 / (G^4 \Gamma k^B T b)) \qquad (106)$$

where G represents the magnetic field gradient, Γ is the total friction on the cantilever oscillator, k^B is the Boltzmann constant, T is the operating temperature, and b is the detection bandwidth [106].

This equation highlights the key factors influencing sensitivity. Maximizing sensitivity (μ) requires using small magnetic particles to generate high magnetic gradients [107], reducing cantilever friction, and operating at lower temperatures. Furthermore, recent studies have shown that coating the sample with a thin layer of metal, such as gold, can reduce the signal-to-noise ratio by up to 20-fold. Sensitivity can be enhanced to approximately 10 μ by replacing the conventional RF coil with a microwave micro-strip resonator. MRFM also enables the observation of statistical spin fluctuations, which offer higher polarization levels and narrower distributions than thermal polarization [261]. This capability allows MRFM to achieve up to 10 times greater sensitivity than traditional electron paramagnetic resonance (EPR) setups, making it a highly promising technique for detecting weak magnetic signals from nanoscale volumes [107-108]

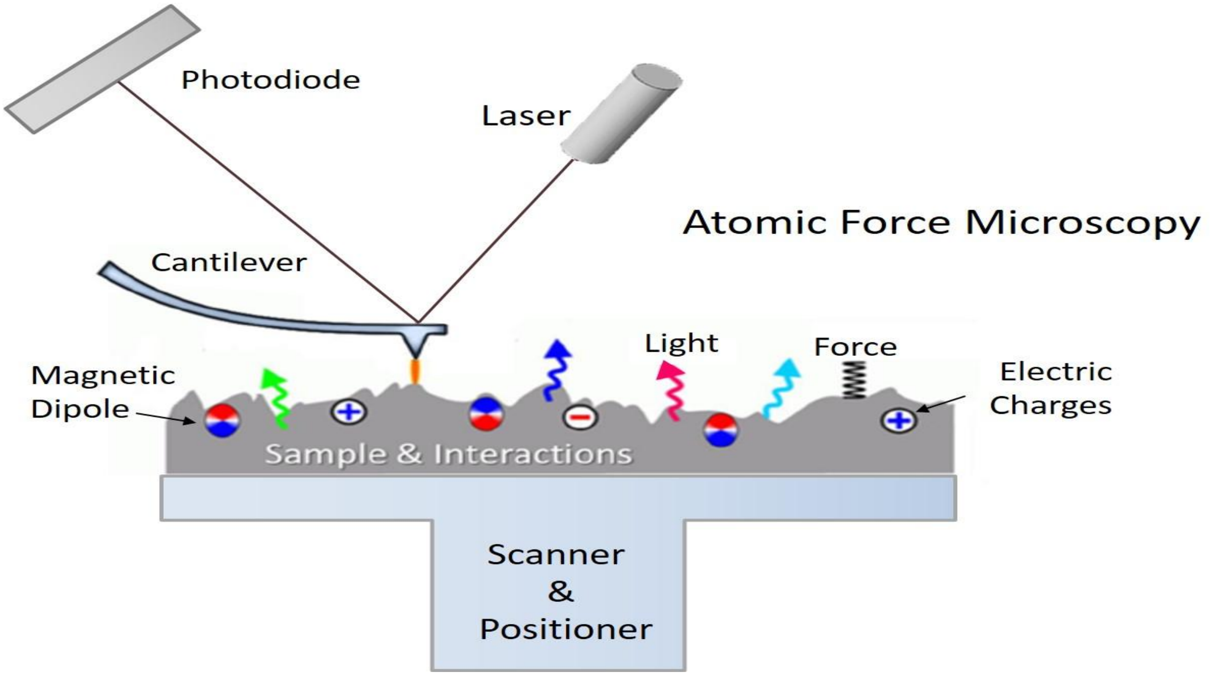
Each technique operates under different conditions and is best suited for specific sample types, but all are limited to detecting magnetic field or magnetization contrast at the surface. Because scanning with a physical probe is inherently slow, these methods are most effective over small fields of view (typically between 100 nm and 100 µm), with some capable of achieving atomic-scale resolution [109]. The spatial resolution is determined by both the size of the probe tip or sensor and its proximity to the sample surface. Their high sensitivity and fine spatial resolution make these techniques powerful tools for real-space imaging of features like uncompensated surface magnetization in anti-ferro-magnets or current distributions in ballistic, hydrodynamic, or superconducting regimes. Furthermore, scanning probes are especially useful for characterizing weakly magnetic materials with low magnetization or buried magnetic structures, such as encapsulated two-dimensional (2D) magnets.

2.5 TOPOLOGICAL CHARACTERIZATION OF ADVANCED COMPOSITE MATERIALS USING SCANNING PROBE MICROSCOPY (SPM)-RESEARCH STUDY

Scanning Probe Microscopy (SPM) encompasses a suite of techniques, including Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM), that are pivotal for the topological characterization of advanced composite materials. These methods provide nanoscale resolution, enabling detailed analysis of surface morphology, mechanical properties, and interfacial interactions within composites [110].

Numerous advanced and functional atomic force microscopy (AFM) techniques have been developed and applied across a wide range of research fields, from imaging surface morphology to quantifying various physical properties. The development and application of AFM-based physical characterization methods demand a solid understanding of fundamental natural sciences and electronic circuit principles. Future directions for AFM-based techniques include: (i) Overcoming the limitations of single-property measurements, especially since electrical properties are often interlinked with mechanical, thermal, and optical behaviors—thus, integrated methods that can probe these coupled properties are needed; (ii) Enhancing functional AFM technologies for operation under vacuum conditions; and (iii) Improving the ability to measure material properties under real working conditions, which is essential for optimizing device performance and advancing material research [111-112].

Atomic Force Microscopy (AFM) includes several fundamental operating modes, primarily used for acquiring surface topography. One such mode is the contact mode (Figure 2(b)), which operates in a static manner. In this mode, the probe tip remains in direct contact with the sample surface during scanning, and the interaction is dominated by repulsive forces. These forces are detected through the deflection of the cantilever. A z-axis feedback loop regulates the tip-sample force, ensuring it remains constant. The adjustments in the z-position required to maintain this constant force directly map the surface topography of the sample. The integration of X-Y scan position data with force feedback and thermal signals captured by a sensor positioned either at the tip or on the cantilever—yields the raw information necessary to generate both surface topography and thermal images. The thermal image contrast represents variations in the localized heat exchange between the tip and the sample. While the force feedback control system functions concurrently to maintain scanning stability, it operates independently from the thermal measurements. Meanwhile, the thermal control unit continuously analyzes thermal signals in real time.



SPM stands as a cornerstone in the topological characterization of advanced composite materials, offering unparalleled resolution and versatility. Its ability to provide comprehensive insights into surface morphology, mechanical properties, and interfacial dynamics makes it an indispensable tool in the development and optimization of next-generation composite materials [113].

2.6 TOPOLOGICAL CHARACTERIZATION OF ADVANCED COMPOSITE MATERIALS USING FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)-RESEARCH STUDY

Field Emission Scanning Electron Microscopy (FESEM) has proven to be an invaluable tool for the topological characterization of advanced composite materials. Its high-resolution imaging capabilities allow for detailed analysis of surface morphology, microstructural features, and interfacial interactions within composites [114]. García Santos et al [115] used FESEM to examine the microstructure of polypropylene fiber-reinforced cement composites. The study provided insights into the fiber-matrix interactions and the distribution of fibers within the cement matrix, highlighting the role of FESEM in assessing the homogeneity and potential weak points in composite materials. The microstructural characterization and mechanical evaluation of a cement composite reinforced with polypropylene fibers aimed at reducing weight and potentially adjusting setting times have been conducted. For the first time, detailed microstructural analysis was performed using both environmental scanning electron microscopy (ESEM) and field emission scanning electron microscopy (FESEM). The final surface morphology of the composite varies depending on the contact areas with the mold; however, the internal microstructure remains highly homogeneous, showing a uniform dispersion of fibers throughout the matrix. Brian Freeland et al studied the characterization of nanostructures and nanoparticles using FESEM. It emphasizes FESEM's capability to provide detailed topographical information, which is crucial for understanding the surface features and morphology of nanocomposite materials. Hiroshi Yao et al used FESEM to investigate the porous structures of silica nanoparticles derived from marine diatoms. The high-resolution images obtained provided valuable information on pore size, distribution, and overall morphology, demonstrating FESEM's effectiveness in characterizing porous composite materials. Three-dimensional gold nanoparticle super lattices were synthesized at the air/solution interface under highly acidic conditions. The surfaces of the gold nanoparticles were functionalized with N-acetyl-glutathione (NAG), which facilitated nanoparticle assembly through hydrogen bonding. Morphological analysis using FE-SEM revealed that the resulting super lattices exhibited fivefold symmetric structures—including pentagonal rods, decahedra, and icosahedra—likely formed through multiple twinning processes. High-resolution surface imaging demonstrated highly ordered nanoparticle arrangements featuring both close-packed and non-close-packed configurations. Given the complexity and breadth of fivefold twinned structures spanning cluster and surface sciences, these findings offer promising new insights and potential advancements in the field of nanoscience.

2.7 Topological Characterization of advanced composite materials using Optical microscopy and Confocal microscopy methods-Research study

Optical microscopy remains a fundamental and accessible technique for the topological characterization of advanced composite materials, offering valuable insights into their microstructural features. Optical microscopy enables the examination of fiber distribution, orientation, and the detection of voids or defects within composite materials. For instance, bright-field illumination has been utilized to identify unimpregnated areas and voids in fiber-reinforced composites, which are critical for assessing manufacturing quality.

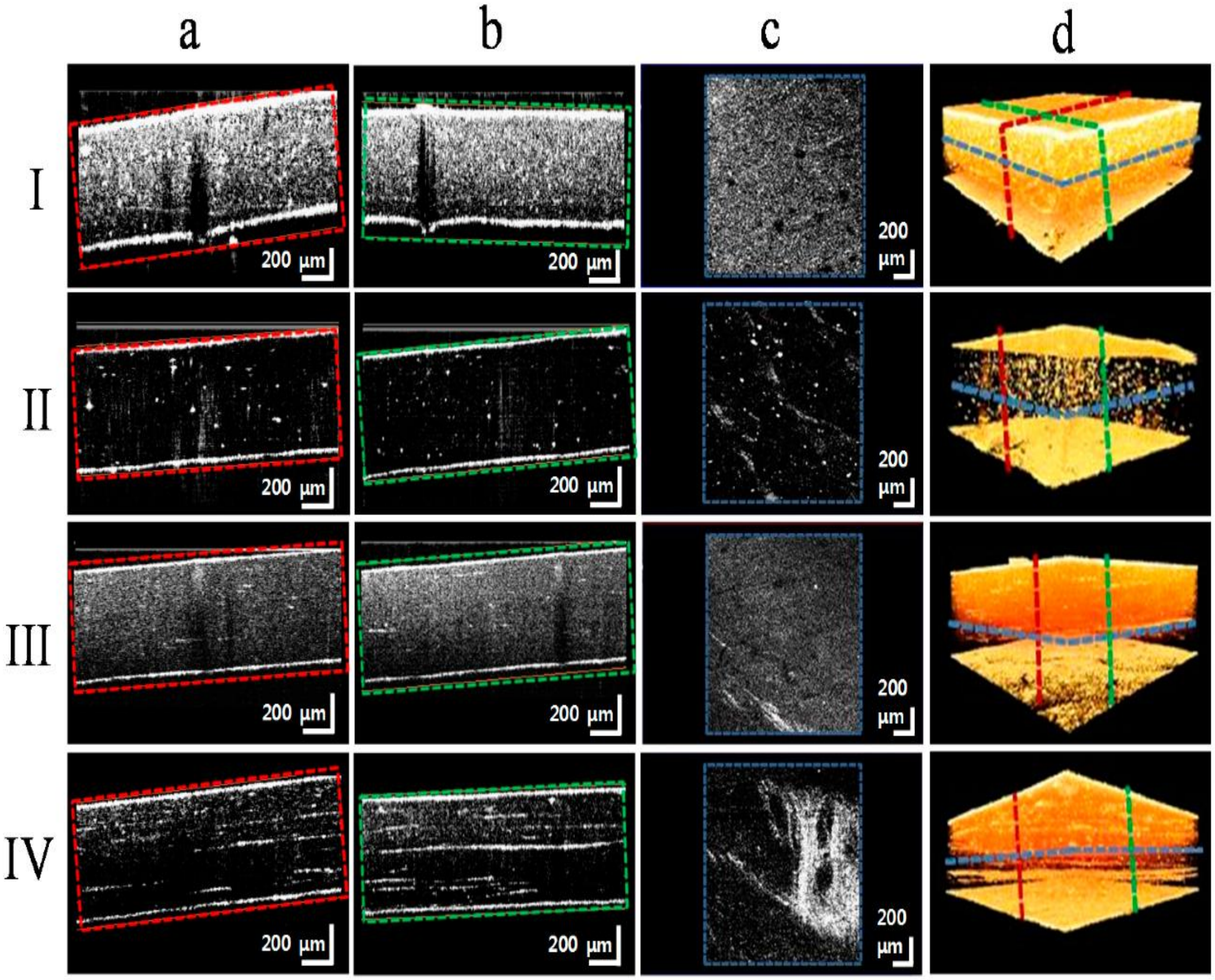


Figure 2. SD-OCT scan images for composites (I)–(IV) of (a) and (b) cross-sections with defects identified in lateral directions, (c) enface images with defects indicated, and (d) three-dimensional views with cross-sectional and enface image locations.(Courtesy: Muhammad Faizan Shirazi et al, Sensors 2017, https://doi.org/10.3390/s17051155)

Digital microscopy was utilized to examine the microstructure of fiber-reinforced composite tubes fabricated via filament winding. Optical microscopy was applied to characterize voids, while scanning electron microscopy (SEM) provided detailed insights into fiber orientation and layer structure. High-resolution image mosaics were constructed from the captured micrographs to visualize the internal architecture across various cross-sections of the samples. Image processing techniques were employed to identify and quantify voids and individual fibers, analyzing their size, shape, and spatial distribution.

Void distribution was evaluated using two approaches—local analysis and tessellation—highlighting varying patterns across different cross-sections. Automated detection of fiber layers allowed for measurement of average winding angles and their dispersion.

Additionally, structural characterization of nylon/graphene oxide (NY/GO) and polyetherblockamide/ trisilanolphenyl-polyhedral oligomeric silsesquioxane (PEBA/t-POSS) composites was conducted using spectral domain optical coherence tomography (SD-OCT). This non-destructive optical technique provided both cross-sectional and subsurface imaging, enabling precise detection of internal defects. A-scan analysis of SD-OCT images facilitated the measurement of defect depth and thickness, while volumetric imaging demonstrated SD-OCT's potential in composite material analysis and industrial quality control.

Micrographs aligned with the fiber direction were also assessed to estimate void fractions in manufactured plates. However, due to the inherent limitations of microscopy—particularly its sampling constraints—accurate void fraction calculation requires complete surface coverage. In this study, micrographs covered only a fraction of the component surface, introducing potential sampling bias. For simple plate geometries, samples were taken from statistically representative areas—center and edge—assuming these locations reflect the extremes of air entrapment during fabrication. For more complex geometries, it is crucial to acknowledge that micrograph-derived void data represents only localized characteristics.

2.8 Compositional Characterization of advanced composite materials using X-ray photoelectron spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS) is a pivotal technique for the compositional characterization of advanced composite materials, offering detailed insights into surface chemistry, elemental composition, and chemical states. X-ray Photoelectron Spectroscopy (XPS) has become a well-established analytical technique and is now widely used across the materials science community [124]. However, despite its broad application, XPS remains frequently misunderstood even in its basic use as highlighted by recent reviews documenting its misapplication in chemical analysis. X-ray Photoelectron Spectroscopy (XPS) has become a widely recognized and essential technique in materials science, commonly used to investigate the elemental composition, electronic structure, and chemical states at the surface of materials. Although peak fitting and deconvolution are standard practices in data interpretation, a foundational understanding of the method and proper data handling is often lacking [125-126].

To address this gap, a collaborative effort among leading experts in the XPS community has produced a series of invaluable resources aimed at guiding newcomers through the correct planning, execution, and interpretation of XPS experiments. These resources, published under the series Practical Guides for X-Ray Photoelectron Spectroscopy, include titles such as Practical Guides for X-ray Photoelectron Spectroscopy: First Steps in Planning, Conducting, and Reporting XPS Measurements and Practical Guide for Curve Fitting in X-ray Photoelectron Spectroscopy [127-128].

In addition, there are advanced XPS techniques and hybrid methodologies that many materials scientists may not be familiar with. A current area of development among XPS instrument manufacturers involves high-throughput analysis of individual sample areas using combinations of surface-sensitive tools and even non-surface-specific techniques like Raman spectroscopy. As these integrated, multi-technique systems become more widely available, there is an increasing need for broader awareness and understanding of the strengths, limitations, and complementary nature of these analytical approaches [129-130].

Advanced XPS methodologies, especially when combined with complementary surface-sensitive techniques, can provide valuable insights beyond the conventional capabilities of standard XPS. This review aims to introduce the broader materials audience to a range of non-traditional uses of laboratory-based XPS, along with combinatorial approaches incorporating techniques such as ion scattering, ultraviolet photoelectron spectroscopy (UPS), electron energy loss spectroscopy (EELS), and Auger electron spectroscopy all of which are often available within common lab-based systems [131-133].

Each peak in an XPS spectrum is accompanied by a characteristic stepped background, resulting from in elastically scattered photoelectrons. Accurate interpretation of XPS data requires proper modeling of this background. The most commonly used model is the Shirley background, introduced by D.A. Shirley [134-135], which uses an iterative method to estimate the number of in elastically scattered electrons based on the integrated signal around a given energy. While effective and widely adopted, its physical foundations remain somewhat vague. To address this, more refined models have been developed, particularly the Tougaard background method, which provides a physically grounded and quantitative description of inelastic scattering [136]. Although a comprehensive comparison of background models is beyond the scope of this review, we highlight how Tougaard's approach enables significantly improved accuracy in analyzing nucleation and growth behaviors in systematic sample studies [137-138]. Today, advanced XPS background analysis can be performed efficiently using specialized software like QUASES, making such refined interpretations both accessible and practical for routine materials characterization.

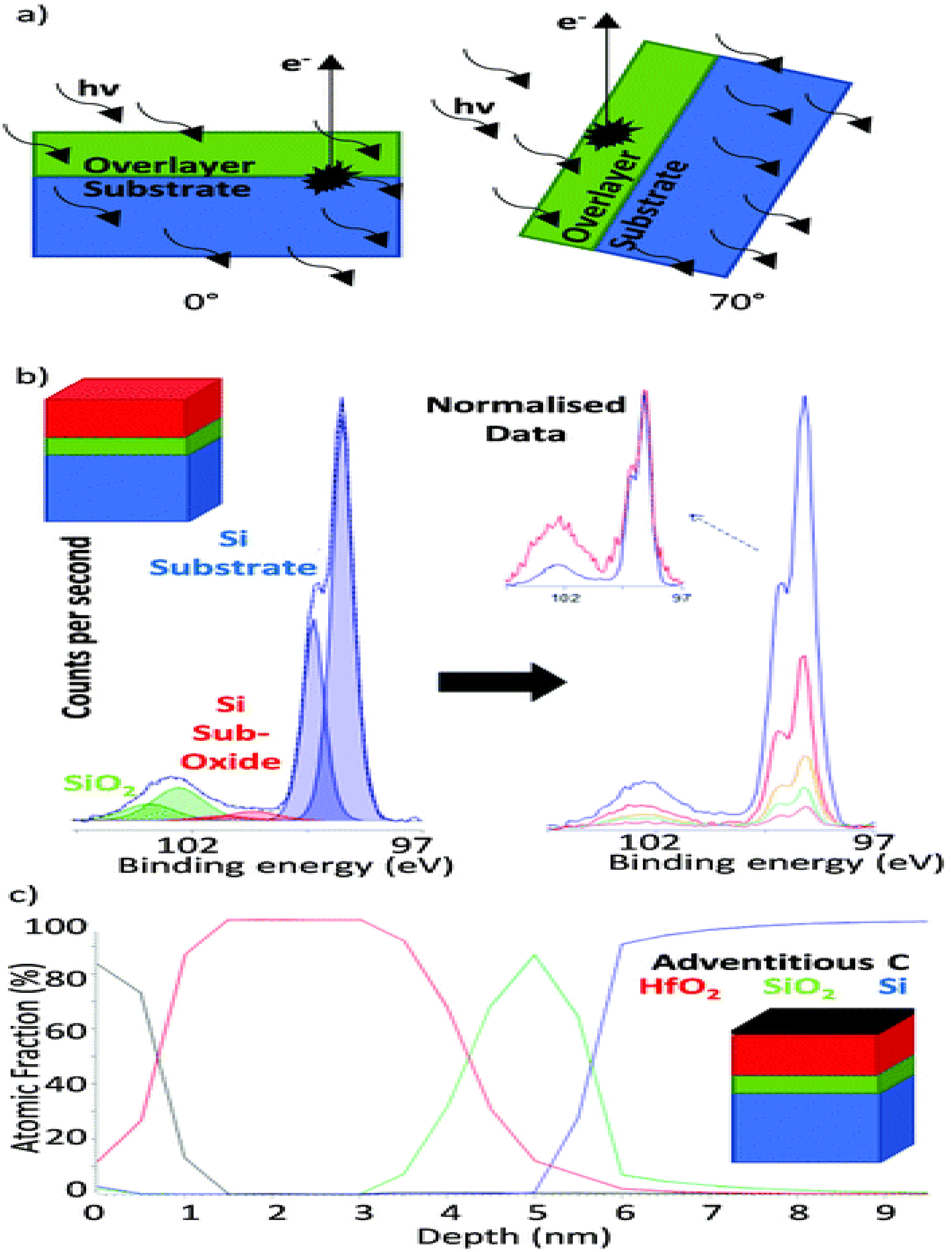


Figure: Compositional Characterization of advanced composite materials using Energy-dispersive X-ray spectroscopy (EDS)

2.9 COMPOSITIONAL CHARACTERIZATION OF ADVANCED COMPOSITE MATERIALS USING SECONDARY ION MASS SPECTROMETRY (SIMS ANALYSIS)

Secondary Ion Mass Spectrometry (SIMS) is a powerful analytical technique used to determine the surface composition, chemical structure, and elemental distribution of materials. It operates in two primary modes: Dynamic SIMS (DSIMS) and Static SIMS (SSIMS). DSIMS is particularly effective for depth profiling, allowing the analysis of impurity and trace element distributions across thin films and interfaces. In contrast, SSIMS is ideal for surface-sensitive applications, such as the analysis of polymeric materials, since it targets only the outermost molecular layers without significant surface erosion [140-141]. In SIMS analysis, a focused beam of high-energy primary ions—typically from an ion gun—is directed at the sample surface. This bombardment causes atoms and molecules to be sputtered from the surface. Some of these ejected species become ionized (secondary ions) and are subsequently analyzed based on their mass-to-charge ratio (m/z) using a mass spectrometer. These spectrometers may be of various types, including magnetic sector, quadrupole, time-of-flight (ToF), or hybrid systems. SIMS offers both high sensitivity and sub-micron spatial resolution, enabling the detection of elemental and isotopic compositions as well as chemical structures and molecular fragmentation patterns at the nanoscale. This makes it an invaluable tool across multiple fields such as materials science, surface chemistry, and the semiconductor industry—especially for analyzing thin films, coatings, and complex surface structures. In static SIMS, the primary ion beam is pulsed to limit surface damage, which is suitable for analyzing delicate or surface-stable materials. Meanwhile, dynamic SIMS uses a continuous beam that progressively sputters the surface, enabling in-depth compositional analysis through layered structures [142].

A series of Nicalon SiC fibers coated with carbon and silicon carbide (SiC) through Chemical Vapor Deposition (CVD) were characterized using Secondary Ion Mass Spectrometry (SIMS). The results (i) provided insight into the material's composition, (ii) revealed compositional heterogeneity, and (iii) highlighted the limitations of applying SIMS to fiber analysis. Complementary analyses using SIMS and Electron Energy Loss Spectroscopy (EELS) were conducted on a segment of the coated fiber. For elements detected by both techniques, the results showed reasonable consistency. The SiC-CVD coatings were found to be compositionally non-uniform—carbon concentrations were higher near the fiber surface, gradually approaching stoichiometric SiC after a few microns. The coated fibers also exhibited elevated levels of oxygen and free carbon. Further analysis of a glass-ceramic matrix composite reinforced with Nicalon SiC fibers using SIMS confirmed the compositional variations, particularly the carbon-rich regions near the fiber interface and the presence of oxygen in the coatings [143-144].

Modern SIMS laboratories are capable of delivering detailed, high-resolution data on the atomic and molecular composition of a wide range of materials—including metals, ceramics, and polymers—making SIMS essential for advanced material characterization and nanotechnology research.

2.10 COMPOSITIONAL CHARACTERIZATION OF ADVANCED COMPOSITE MATERIALS USING AUGER ELECTRON SPECTROSCOPY (AES)

Auger Electron Spectroscopy (AES), also known as Scanning Auger Microscopy (SAM), is a surface-sensitive analytical technique that utilizes a focused electron beam to induce the emission of Auger electrons from the near-surface region of a solid sample. These emitted electrons possess energies that are characteristic of the elements present and, in some cases, provide insight into their chemical bonding states. AES enables elemental mapping of the sample surface and, when combined with ion sputtering, facilitates compositional depth profiling, offering elemental and chemical information as a function of depth. Due to the characteristics of the Auger process, all elements except hydrogen and helium can be detected, with detection limits as low as 0.1 atomic percent. The typical sampling depth ranges from 15 to 30 monolayers (approximately 50–100 Å), and the technique can resolve features down to 0.1 µm, making it particularly effective for analyzing unknown conductive materials. In SAM mode, the AES system performs a raster scan of the electron beam across the sample surface to generate images representing the lateral elemental distribution. Auger spectra, which reveal the surface composition or identify specific surface features, can be collected in either point analysis mode (with the beam focused on a fixed location) or area-averaged mode (with the beam scanned over a region). The spectral data is typically recorded in N (E) format, where the electron energy analyzer steps through a selected range and measures the signal at each energy level. Repeated scanning allows enhancement of the signal-to-noise ratio. Data processing routines such as differentiation, smoothing, expansion, and background subtraction can be applied to enhance interpretation. A multiplexing approach is also available, focusing on specific energy ranges containing peaks of interest for automated data acquisition and quantification. This process often uses elemental sensitivity factors derived from standard materials [145-146].

When AES is performed in conjunction with ion sputtering, it yields compositional depth profiles that detail the variation in elemental composition beneath the surface as a function of sputtering time or depth, providing comprehensive insight into the layered structure of materials.

As composite materials are increasingly adopted for critical structural applications, it becomes essential to understand their behavior under load, particularly in identifying and interpreting failure mechanisms. While optical microscopy and scanning electron microscopy (SEM) remain primary tools for such investigations, the insights they offer can occasionally be limited or ambiguous. To complement these conventional methods, Auger Electron Spectroscopy (AES) offers valuable surface-sensitive analytical capabilities [147].

In one example, AES depth profiling enabled a more comprehensive analysis of the fracture surface of a titanium metal matrix composite reinforced with boron-carbide-coated boron fibers. The technique revealed detailed chemical and morphological information at the failure interface, which would otherwise remain elusive with standard microscopy. AES was also effectively used to characterize the solid electrolyte interphase (SEI) in composite electrode materials, providing insight into the heterogeneous nature of SEI layer growth on both graphite and silicon nanoparticles. The findings demonstrated that SEI layers are not uniform in thickness: graphite exhibited a mean SEI thickness of approximately 13 nm, while silicon nanoparticles showed SEI thicknesses ranging from 20 to 25 nm, with the particles themselves having crystalline cores of 50–70 nm in diameter. A "sticky-sphere" model was proposed to explain the observed variability, suggesting that SEI layers form by replacing polymer binders initially surrounding the silicon nanoparticles [148-150].

Additionally, SiC fiber/silicate glass matrix composites, proposed as candidate materials for jet engine components, have been extensively studied using scanning AES. Analyses of fiber surfaces and matrix troughs (regions associated with fiber pull-out) revealed that fracture toughness correlates with the formation of a 200+ Å carbon-rich interfacial layer during fabrication. In contrast, strong fiber-matrix bonding, which lacks this carbon layer, was linked to brittle failure and diminished mechanical performance. The sensitivity of AES to surface chemistry within the first few nanometers makes it indispensable for analyzing nanostructures, where even minor surface modifications can significantly impact bulk properties. Conventional techniques such as energy-dispersive spectroscopy (EDS) and electron energy loss spectroscopy (EELS), though commonly paired with SEM and TEM, often fall short in detecting these subtle surface alterations—unless under highly favorable conditions [151-152].

Modern scanning AES instruments offer nanoscale structure of lateral resolution and depth profiling capabilities within a few nanometers, making them ideal for nanotechnology, metallurgy, and semiconductor applications. This review underscores the distinct advantages of scanning AES and its growing importance in advanced material characterization.

III. CONCLUSIONS

Based on the research studies on the characterization of composite materials under mechanical loading using various microscopic techniques, the following key conclusions for engineering applications:

- The combined use of optical microscopy, scanning electron microscopy (SEM), Auger electron spectroscopy (AES), and X-ray photoelectron spectroscopy (XPS) provides a comprehensive understanding of microstructural changes and failure mechanisms in fiber-reinforced and polymer matrix composites under mechanical stress.
- AES and XPS have proven especially valuable in identifying interfacial chemical states, contamination, and degradation layers that influence the mechanical performance, particularly at fiber/matrix interfaces and failure surfaces. These techniques offer nanometer-scale depth sensitivity, essential for high-performance engineering materials.
- Optical microscopy and digital image processing effectively detect voids, fiber alignment, and layer structure. Their statistical analysis—such as tessellation and local density mapping—enables quantification of void distributions and fiber orientation, critical for assessing manufacturing quality and structural reliability.
- Techniques like spectral domain optical coherence tomography (SD-OCT) provide non-destructive, depth-resolved imaging, enabling real-time analysis of internal defects and sub-layer structures, making them particularly useful for quality assurance and in-situ monitoring in polymer composites.
- SIMS and AES depth profiling reveal heterogeneity in chemical composition across coatings and interfaces, which significantly affects the composite's mechanical integrity. In titanium

matrix and SiC fiber composites, such profiling has been essential in linking interfacial chemistry to crack propagation and failure behavior.

- Observations from SEM and AES indicate that microstructural features—such as carbon interlayers or localized oxidation—at the fiber-matrix boundary can govern composite toughness and fracture mode, underlining the importance of nanoscale control during fabrication.
- Engineering applications increasingly demand high-fidelity, reproducible materials. Integration of microscopy techniques enables accurate modeling of composite behavior under load, aiding in failure prediction, material optimization, and lifespan estimation.

These conclusions support the growing need for comprehensive microscopic and spectroscopic analysis protocols to characterize advanced composites used in aerospace, automotive, energy, and biomedical engineering applications.

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