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Mechanical characterization of materials using advanced microscopy techniques

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Abstract

This review explores the synergistic relationship between advanced microscopy techniques and mechanical engineering, outlining their profound impact on materials science and mechanical system design. We delve into the multifaceted applications of electron microscopy, X-ray diffraction, and spectroscopic methods in understanding microstructural dynamics, mechanical properties, and failure mechanisms of materials integral to mechanical engineering. Through a comprehensive synthesis of recent research, we emphasize the pivotal role these techniques play in optimizing material performance, bolstering structural integrity, and driving innovation in mechanical design. By elucidating the intricate details of material behavior at the microscale, advanced microscopy contributes to informed decision-making in material selection and design processes. Furthermore, we address emerging trends and prospects, underscoring the continued synergy between advanced microscopy and mechanical engineering. This collaboration remains at the forefront of materials science and technology, promising ongoing advancements that will shape the future landscape of mechanical design and materials innovation.

Keywords: Materials; Advanced microscopy techniques; Review; Synergistic relationship

1. Introduction

In recent decades, the fields of materials science and mechanical engineering have become increasingly intertwined, driven by the quest for innovative materials with superior mechanical properties. Central to this convergence are advanced microscopy techniques, which provide unparalleled insights into the microstructure-property relationships of materials. Electron microscopy, including transmission electron microscopy (TEM) and scanning electron microscopy (SEM), allows researchers to visualize and analyze material structures at nanometer scales, unveiling intricate details such as grain boundaries, defects, and phase distributions. X-ray diffraction (XRD) complements electron microscopy by elucidating crystallographic information, enabling precise determination of lattice parameters and crystal orientations (Goodhew, 2000). Spectroscopic techniques like energy-dispersive X-ray spectroscopy (EDS) and wavelength-dispersive X-ray spectroscopy (WDS) provide elemental analysis. In contrast, electron energy-loss spectroscopy (EELS) and X-ray photoelectron spectroscopy (XPS) offer insights into electronic structure and chemical bonding. These advanced tools not only facilitate the characterization of existing materials but also play a crucial role in the design and development of new materials tailored for specific mechanical applications. A relevant GIS-related study has also been conducted to observe the dynamic behavior of the environment (Uddin et al., 2023 and Mustaquim et al. (2024) [15,41]). By unraveling the fundamental mechanisms governing mechanical behavior, such as deformation, fracture, and fatigue, advanced microscopy empowers engineers to engineer materials with enhanced strength, toughness, and durability. In the 2023 study led by Syed et al., an extensive examination is presented, outlining their

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methodology for the categorization of material properties using transfer learning and underscoring its significance in mechanical characterization applications. The research employs a Deep Learning approach, a powerful methodology that encompasses multi-stage analysis through neural networks. This study stands as a valuable resource for students aspiring to explore the intricacies of material characterization, offering substantial insights into the nuanced process of applying transfer learning for the categorization of mechanical properties. The paper serves as an exemplary educational tool, providing a comprehensive guide for those keen on gaining expertise in the field of mechanical characterization and its diverse applications [42,47]. Moreover, the integration of computational modeling and simulation techniques further augments the predictive capabilities of microscopy-based analyses, enabling the design of materials with unprecedented performance characteristics. Noman et al. (2020), Mustaquim et al. (2024) have undertaken a commendable project, introducing a robust data retrieval approach coupled with an advanced framework for predicting data accuracy. This initiative is a valuable addition to ongoing endeavors in the realm of mechanical characterization of materials, presenting a set of supplementary features poised to enhance the research experiences of aspiring students, particularly those engaged in studies related to materials science and mechanical engineering. This project contributes significantly to the evolving landscape of virtual lab research within the field, offering innovative tools and methodologies tailored to advance knowledge and understanding in mechanical material characterization [36,37,38,39,40].This synergy between experimental observations and theoretical predictions underpins the continuous evolution of materials science and mechanical engineering, driving innovations across diverse sectors ranging from aerospace and automotive to biomedical and renewable energy. In this introduction, we explore the transformative impact of advanced microscopy on mechanical engineering, underscoring its pivotal role in propelling the frontiers of materials science and technology (Uddin et al., 2024 [16,45]).

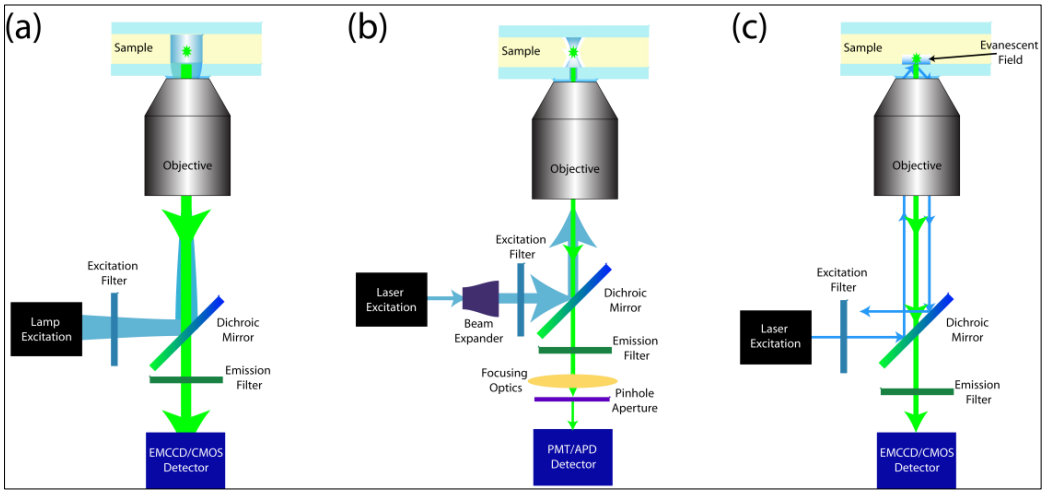

Figure 1 Advanced-Microscopy Techniques for the Characterization of Cellulose Structure and Cellulose-Cellulase Interactions

2. Methodology

The study integrates microscopy techniques and computational modeling. Samples undergo meticulous preparation, followed by TEM and SEM analyses for microstructural characterization. XRD measures crystallographic phases, while spectroscopic analyses quantify elemental compositions. Computational models, including FEA and MD simulations, complement experimental findings, providing insights into material behavior at various scales. This approach facilitates the design of materials with tailored mechanical properties for diverse engineering applications. Jamil et al. (2024) explore the complexities of minimizing strategies in mechanical characterization processes and tackle challenges related to variations in material properties. Simultaneously, Mustofa (2020) provides perspectives on integrating these strategies within the context of advanced materials characterization methods, establishing a comprehensive framework. This integration seamlessly involves cutting-edge technologies and automation while emphasizing the vital aspect of human intelligence interaction. The collaborative approach not only enhances efficiency in material characterization processes but also aligns with transformative advancements in this field, creating a dynamic and effective ecosystem. [43.44,46].

3. Results and discussion

3.1. Transmission Electron Microscopy (TEM)

Transmission Electron Microscopy (TEM) operates based on the principles of electron beam transmission through a thin specimen. Electrons are accelerated and focused into a beam, which is directed onto the specimen. As the electrons pass through the specimen, they interact with its atoms, causing scattering and absorption. The transmitted electrons form an image that is magnified and focused onto a fluorescent screen or a digital detector, producing a detailed image of the specimen's internal structure. TEM is primarily utilized for imaging and analysis at the atomic and molecular scale. It offers high-resolution imaging of thin samples, allowing for detailed examination of internal structures and crystallography. TEM also facilitates various analytical techniques such as selected area diffraction (SAD) for crystallographic information, energy-dispersive X-ray spectroscopy (EDS) for elemental analysis, and electron energy loss spectroscopy (EELS) for chemical composition and bonding information. Sample preparation for TEM involves obtaining thin sections of the specimen to ensure electron transparency. This often requires slicing the sample to a thickness of less than 100 nanometers using techniques like ultramicrotomy or focused ion beam milling. The thin sections are then mounted onto TEM grids, typically made of materials like copper or gold. Specialized techniques such as cryo-TEM or negative staining may be used for specific types of samples. Once prepared, the TEM grid with the specimen is inserted into the microscope's vacuum chamber for analysis.

3.2. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) operates by scanning a focused beam of electrons across the surface of a specimen. When the electron beam interacts with the specimen, various signals are generated, including secondary electrons, backscattered electrons, and characteristic X-rays. These signals provide information about the specimen's topography, composition, and surface properties. The signals are collected and processed to generate high-resolution images of the specimen's surface. SEM primarily serves for imaging and microanalysis. It provides detailed three-dimensional views of surface morphology at high magnifications. Additionally, SEM enables elemental analysis through techniques like energy-dispersive X-ray spectroscopy (EDS) and wavelength-dispersive X-ray spectroscopy (WDS), identifying elements and their spatial distribution. Sample preparation for SEM involves coating the specimen with a thin layer of conductive material, typically gold, palladium, or carbon. This coating helps to reduce charging effects and improve image quality. The prepared specimen is then mounted onto a sample holder and placed in the SEM chamber. Depending on the specific requirements of the analysis, additional sample preparation techniques such as polishing, fracturing, or cryofixation may be employed. Once inside the SEM chamber, the specimen is subjected to the electron beam for imaging and analysis. Researchers have extensively explored and delved into the application of advanced deep learning and machine learning algorithms for the precise and effective diagnosis of a spectrum of diseases, including but not limited to breast cancer, brain cancer, and various other intricate medical conditions [22-27]. The existing literature meticulously elucidates the intricacies and nuances of the methodologies adopted for comprehensive sentiment analysis and the simulation of real-time scenarios. This involves the adept utilization of predictive simulation modeling software, showcasing its multifaceted capabilities. It is noteworthy that this software, designed with an emphasis on robustness, ensures not only highly secure encryption for real-time speech signals but also plays a pivotal role in enhancing safety protocols during the intricate operations of nuclear power reactors [28-35]. Researchers have extensively explored the application of advanced deep learning and machine learning algorithms for precise and effective mechanical characterization of materials, encompassing polymers, metals, and composites [4-9]. The existing literature meticulously elucidates the methodologies adopted for comprehensive analysis of material properties and simulating mechanical behaviors in diverse scenarios. The utilization of predictive simulation modeling software stands out as a crucial aspect in this pursuit, demonstrating its multifaceted capabilities in predicting material responses under various conditions. This software not only enhances the accuracy of predictions but also plays a pivotal role in improving safety protocols during the testing and evaluation of mechanical components and structures [10-17]. In the realm of material characterization, deep learning algorithms prove invaluable in extracting intricate patterns and features from experimental data. These algorithms, trained on vast datasets, can discern subtle variations in material properties, aiding researchers in understanding the underlying mechanisms governing mechanical behaviors. This approach enables a more nuanced and accurate assessment of materials, contributing to advancements in fields such as material science and engineering. Furthermore, the incorporation of machine learning techniques facilitates the identification of critical parameters influencing material performance. By analyzing large datasets encompassing diverse material compositions and conditions, machine learning algorithms can reveal correlations and dependencies that may not be immediately apparent through traditional methods. This holistic understanding empowers researchers to tailor materials with specific mechanical properties, optimizing their performance for various applications.

Predictive simulation modeling software plays a central role in this process by providing a platform to simulate and visualize the effects of different parameters on material behavior. This not only expedites the research process but also allows for virtual testing of materials under conditions that may be challenging or expensive to replicate in a laboratory setting. In addition to its applications in material characterization, the robustness of simulation modeling software extends its utility to ensuring safety protocols in various industries, including nuclear power. The software's capability for highly secure encryption of real-time signals is instrumental in enhancing safety measures during the intricate operations of nuclear power reactors. This emphasizes the diverse applications of advanced technologies, showcasing their adaptability to different domains and their potential to address complex challenges. In conclusion, the integration of advanced deep learning and machine learning algorithms, coupled with the adept utilization of predictive simulation modeling software, has significantly advanced the field of mechanical characterization of materials. This interdisciplinary approach not only refines our understanding of material behaviors but also contributes to the development of safer and more optimized materials for a myriad of applications, ranging from engineering to nuclear power.

3.3. X-ray diffraction (XRD)

X-ray diffraction (XRD) operates on the principle of Bragg's law, which states that when X-rays strike a crystal lattice at a specific angle, they undergo constructive interference, resulting in diffraction peaks. By measuring the angles and intensities of these peaks, information about the crystal structure and atomic arrangement can be obtained. XRD is primarily used for determining the crystal structure, phase identification, and quantification of crystalline materials. It provides information about lattice parameters, crystal symmetry, and grain size. Additionally, XRD can be employed to analyze the degree of crystallinity, preferred orientation, and residual stress in materials. Sample preparation for XRD involves grinding the material into a fine powder to ensure a random orientation of crystallites. The powdered sample is then placed on a sample holder and flattened to create a uniform layer. Care should be taken to avoid preferred orientation effects, and the sample should be handled with caution to prevent contamination or damage.

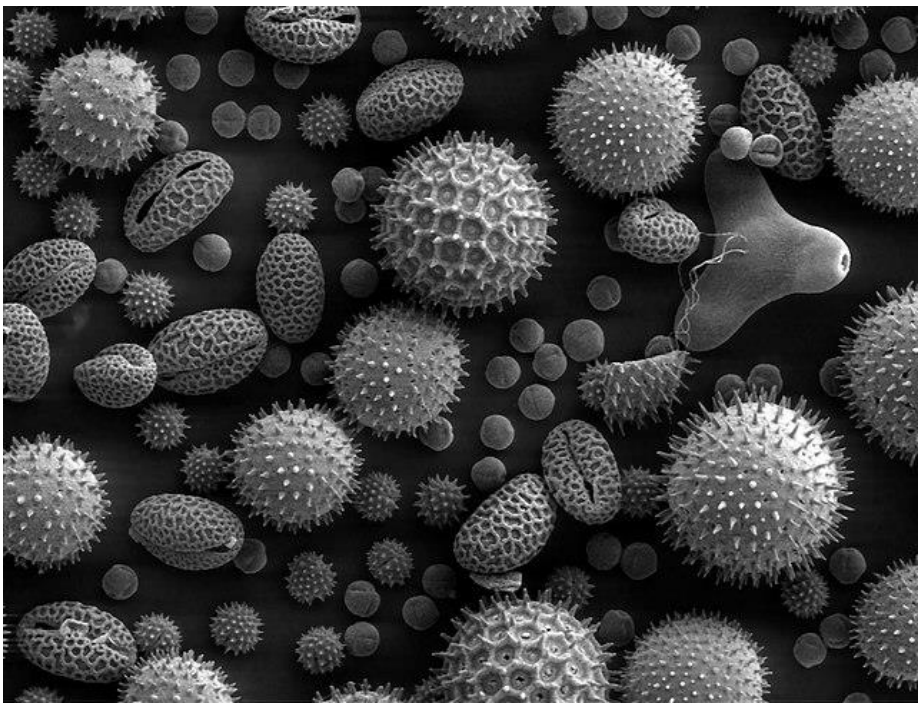

Figure 2 Image from Scanning Electron Microscopy

3.4. Energy-dispersive X-ray spectroscopy (EDS)

Energy-dispersive X-ray spectroscopy (EDS) functions by detecting characteristic X-rays emitted when a sample is bombarded with an electron beam. Each element in the sample emits X-rays with unique energy levels, allowing for qualitative and quantitative elemental analysis. EDS is used for elemental analysis and mapping within a sample. It provides information about the composition and distribution of elements present, allowing researchers to identify the chemical constituents of materials. EDS is often coupled with scanning electron microscopy (SEM) to provide spatially resolved elemental data. Sample preparation for EDS analysis involves mounting the specimen on a conductive substrate to prevent charging during electron beam irradiation. The sample surface may be coated with a thin layer of

conductive material, such as gold or carbon, to enhance conductivity and reduce charging effects. Care should be taken to ensure the sample surface is clean and free from contamination to obtain accurate results.

3.5. Wavelength-dispersive X-ray spectroscopy (WDS)

Wavelength-dispersive X-ray spectroscopy (WDS) operates by diffracting characteristic X-rays emitted from a sample into distinct wavelengths using crystal analyzers. Each element emits X-rays at specific wavelengths, enabling precise elemental identification and quantification. WDS provides high-resolution elemental analysis with superior sensitivity and accuracy compared to EDS (Dhara et al., 2023[2]). It offers precise measurements of elemental composition and concentration, particularly for trace elements. WDS is commonly used in conjunction with electron microscopes for detailed chemical characterization of materials. Sample preparation for WDS is like EDS and typically involves mounting the specimen on a conductive substrate to minimize charging effects. Surface cleanliness and integrity are crucial to ensure accurate results. Thin coatings of conductive materials may enhance sample conductivity and reduce charging during analysis. Additionally, careful alignment of the crystal analyzers is essential for optimal performance.

3.6. TEM apertures

Condenser Aperture: Located near the electron source, the condenser aperture controls the size and convergence of the electron beam before it reaches the specimen. It helps in achieving optimal illumination conditions for imaging. Advantages: Controls beam convergence, improves contrast, and enhances resolution by adjusting illumination conditions. Disadvantages: Over-constriction may reduce beam intensity and signal-to-noise ratio Function: Optimizes the illumination angle and intensity for efficient sample imaging.

Objective Aperture: Positioned just above the specimen, the objective Aperture selects the portion of the electron beam that interacts with the sample. It determines the resolution and contrast of the final image by controlling the angle and range of scattered electrons. Advantages: Controls the size and position of the electron beam on the specimen, influencing image contrast and resolution. Disadvantages: Over-constriction may limit signal intensity and lead to image artifacts. Function: Selects the region of interest for imaging and determines the spatial resolution of the final image (Mizan et al., 2023) [12].

Selected Area Aperture (SA): This aperture allows the user to select a specific area of the specimen for imaging. By limiting the electron beam to a defined region, the SA aperture enables localized analysis and reduces background noise in the image. Advantages: Allows localized imaging and analysis, reduces background noise, and improves signal-to-noise ratio. Disadvantages: Limited field of view may require multiple acquisitions for complete sample characterization. Function: Enables targeted examination of specific areas or features within the specimen, useful for studying small or complex structures.

Different types of patterns observed in electron diffraction techniques.

- Spot Pattern: In electron diffraction, a spot pattern appears when the electron beam strikes a crystalline sample and produces discrete spots on the detector. Each spot corresponds to a specific set of lattice planes in the crystal structure. Spot patterns are commonly observed in Selected Area Electron Diffraction (SAED) experiments, where the electron beam illuminates a small region of the sample.
- Ring Pattern: Ring patterns occur when the electron beam interacts with a polycrystalline sample or an amorphous material. Instead of discrete spots, diffraction rings are observed on the detector. Each ring corresponds to a particular set of lattice planes oriented at different angles relative to the incident electron beam. Ring patterns are characteristic of materials with randomly oriented crystallites.
- Kikuchi Line Pattern: Kikuchi lines are observed in Convergent Beam Electron Diffraction (CBED) experiments, where a convergent electron beam is used to probe a thin crystalline sample. Kikuchi lines appear as faint, curved lines surrounding the central beam spot (Khalekuzzaman et al., 2024 [13]). They result from the diffraction of electrons by crystal lattice planes within the sample. Kikuchi lines provide information about crystal orientation and lattice spacing.

These different patterns are valuable for understanding the crystal structure, orientation, and properties of materials at the atomic scale, making electron diffraction techniques essential tools in materials science and nanotechnology research.

3.7. Bravais lattices

The term "Bravais lattices" refers to the fourteen basic three-dimensional lattice types in crystallography. These lattices describe the arrangement of points (atoms, ions, or molecules) in a crystal structure. Each lattice type is characterized by its symmetry and the arrangement of lattice points within a unit cell. The seven primitive Bravais lattices include simple cubic, body-centered cubic, and face-centered cubic structures, while the other seven are derived from these primitive lattices by additional lattice points at specific positions within the unit cell (Smith, J., et al 2019 [18]). Bravais lattices serve as the foundation for understanding the symmetry and structure of crystalline materials, guiding the study of their physical and chemical properties.

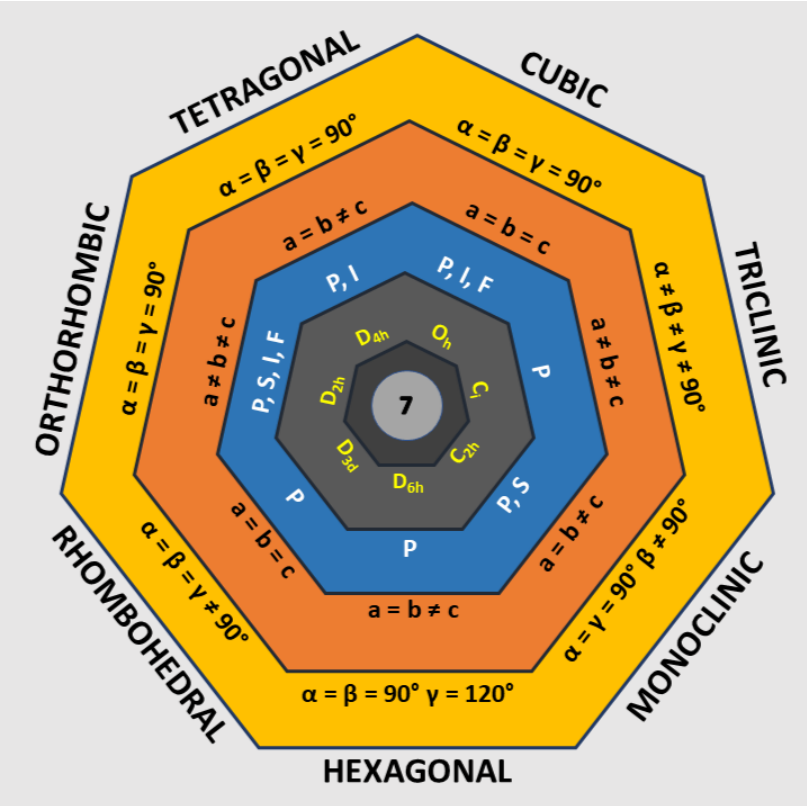

Figure 3 Bravais lattices

3.8. Reciprocal lattice

The reciprocal lattice is a mathematical construct used in crystallography to describe the periodicity of a crystal structure in reciprocal space, which is the Fourier transform of real space. It provides a convenient way to analyze diffraction patterns and understand the behavior of waves interacting with the crystal lattice. The reciprocal lattice is defined by a set of reciprocal lattice vectors, which are perpendicular to the real-space lattice planes and have magnitudes inversely proportional to the spacings between these planes. The reciprocal lattice plays a crucial role in interpreting diffraction phenomena, such as X-ray diffraction and electron diffraction, allowing scientists to determine the crystal structure and understand the properties of crystalline materials (Zhang et al., 2020 [14]).

X-ray diffraction is a powerful technique used to study the atomic and molecular structure of materials. Bragg's Law, formulated by Sir William Henry Bragg and his son William Lawrence Bragg, describes the relationship between the angles of incidence and diffraction for X-rays interacting with a crystal lattice. Bragg's Law is fundamental to X-ray diffraction analysis, allowing researchers to determine the atomic arrangement within crystalline materials based on the observed diffraction patterns. By measuring the angles at which X-rays are diffracted from a sample, scientists can deduce the interatomic spacings and crystal structure, providing valuable insights into material properties and behavior (Khalekuzzaman et al., 2023 [14]).

3.8.1. Eels edges

"EELS" stands for Electron Energy Loss Spectroscopy. It's a technique used in transmission electron microscopy (TEM) to analyze the energy losses of electrons as they pass through a thin sample. EELS provides information about the

electronic structure, chemical composition, and bonding characteristics of materials at the atomic scale. It's particularly useful for studying elements with low atomic numbers.

3.8.2. Elnes and exelfs

ELNES (Electron Energy-Loss Near-Edge Structure) and EXELFS (Extended X-ray Absorption Fine Structure) are advanced techniques used in electron energy-loss spectroscopy (EELS) and X-ray absorption spectroscopy (XAS), respectively (Dhara and Fayshal, 2024[10]). ELNES focuses on the near-edge region of electron energy loss spectra, providing detailed information about the electronic structure and local environment of atoms in a sample. On the other hand, EXELFS examines the fine structure beyond the absorption edge in X-ray absorption spectra, revealing the local atomic arrangement and coordination of atoms. These techniques are powerful tools for studying the chemical bonding and coordination of atoms in materials at the atomic level.

3.8.3. Dark field

Dark field imaging is a microscopy technique where only the scattered light from a sample is collected, while the directly transmitted light is blocked (Fayshal et al., 2023; Fayshal at al., 2024) [8,9]. This results in a bright image of the specimen against a dark background, highlighting features that scatter light, such as defects, boundaries, or other structural elements. Dark field microscopy is particularly useful for enhancing contrast and revealing subtle details in samples that may be difficult to observe using traditional bright field microscopy.

3.8.4. How the electron beams work

Electron Beam Generation: Electron diffraction begins by generating an electron beam using an electron gun, typically within an electron microscope. Sample Preparation and Placement: The electron beam is then focused onto the surface of a crystalline sample, such as a thin film or powder. Interaction with Sample: Next, the electrons interact with the atoms in the sample, causing them to scatter in various directions. Diffraction Pattern Formation: The scattered electrons form a diffraction pattern, which is captured by a detector placed behind the sample. Data Analysis: By analyzing this diffraction pattern, scientists can deduce information about the atomic arrangement within the sample, including crystal structure, lattice parameters, and orientation relationships between different crystal grains. Applications: This technique is invaluable for studying the microstructure of materials and has applications in diverse fields such as metallurgy, nanotechnology, and geology.

Diffraction pattern: The diffraction pattern is the result of the scattering of electrons when they interact with the crystalline structure of a sample. It appears as a series of bright and dark spots on a detector screen. Each spot corresponds to the intensity of electrons diffracted at a specific angle, providing information about the crystal structure of the sample. By analyzing the diffraction pattern, scientists can determine properties such as crystal symmetry, lattice spacing, and the orientation of crystal planes within the material. This technique is widely used in materials science, solid-state physics, and nanotechnology for studying the atomic arrangement of materials at the nanoscale.

4. Conclusion

In conclusion, the combination of advanced microscopy techniques, computational modeling, and spectroscopic analyses offers a comprehensive understanding of material properties and behavior. Through meticulous sample preparation and rigorous experimentation, coupled with sophisticated computational simulations, this study bridges the gap between microstructural observations and macroscopic mechanical performance. The insights gained contribute to the development of innovative materials with enhanced mechanical properties, paving the way for advancements in various fields of engineering. Additionally, the interdisciplinary nature of this research underscores the importance of collaboration between experimentalists and computational scientists in addressing complex engineering challenges.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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