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Transmission Electron Microscopy and Dynamic Light Scattering-Fundamental Perspective

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Abstract

Transmission Electron Microscopy (TEM) and Dynamic Light Scattering (DLS) are fundamental tools in nanotechnology and materials science, providing information about the structural and dynamic properties of nanomaterials and colloidal systems. This review provides a thorough examination of TEM and DLS, beginning with a discussion of their fundamental principles. It discusses the interaction of electron beams in TEM and the dynamics of scattered light in DLS, followed by an examination of instrumentation and experimental setups, including recent advances for increased resolution and sensitivity. The article then discusses the numerous applications of TEM and DLS in nanomaterial characterization. It includes analyses of nanoparticle size, shape, morphology, and distribution, as well as research into colloidal system dynamics such as nanoparticle aggregation, diffusion, and stability. The review concludes with a forward-looking perspective on emerging trends and future directions in nanomaterial characterization. It emphasizes the potential of TEM and DLS to overcome current challenges and advance our understanding of nanoscale phenomena. Overall, this comprehensive review provides researchers and practitioners with a solid understanding of the TEM and DLS principles, methodologies, and applications, as well as valuable fundamental insights.

Keywords: Transmission Electron Microscopy, Characterization, Nanomaterials, Dynamic Light Scattering (DLS).

Introduction

Transmission electron microscopy

Transmission electron microscopy (TEM) is a powerful technique that uses a beam of electrons to produce highly magnified and detailed images of thin specimens. Unlike light microscopes, which use light rays to form an image, TEMs use electrons, which have much shorter wavelengths and

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can reveal finer structures. TEMs can achieve a resolution of up to 0.2 nm, which is about 1000 times better than that of light microscopes. TEMs can provide information about the morphology, composition, crystal structure, and orientation of the sample, as well as the interactions between the electrons and the atoms. TEMs are widely used in various fields of science, such as biology, materials science, nanotechnology, and medicine(Zhang et al., 2009).

Principle of TEM

The basic principle of Transmission electron microscopy (TEM) is to use a beam of electrons to illuminate a thin specimen and form an image based on the transmitted electrons. The electrons have a much shorter wavelength than light, which allows them to reveal finer details of the specimen's structure. The electrons are generated by an electron gun and focused by a condenser lens into a coherent beam. The beam passes through the specimen and is scattered by the atoms in different directions. The scattered electrons are then collected by an objective lens, which forms an image of the specimen on a phosphor screen or a CCD camera. The image can be magnified by intermediate and projector lenses. The image contrast depends on the thickness and electron transparency of the specimen, as well as the use of optional apertures to block out high-angle diffracted electrons. TEM can also produce a diffraction pattern of the specimen by focusing the electrons scattered in the same direction into the back focal plane of the objective lens.

Instrumentation

The instrumentation of Transmission electron microscopy (TEM) consists of the following main components[1]:

- **Source of electron:** This is an electron gun that produces a beam of electrons by thermionic emission from a heated filament, usually made of tungsten or lanthanum hexaboride. The electron beam is accelerated by a high voltage, typically ranging from 40 to 400 kV.
- **Beam of electron:** This is a stream of electrons that travels in a vacuum chamber and is focused by electromagnetic lenses. The beam can be controlled by apertures, coils, and deflectors to adjust its intensity, size, shape, and position.
- Electromagnetic lenses: T**hese are devices that use magnetic fields to bend and converge** the electron beam. They act as the optical lenses in a light microscope. There are two types of lenses: condenser lenses and objective lenses. Condenser lenses focus the beam onto the specimen, while objective lenses collect the transmitted electrons and form the primary image.
- **Vacuum chamber:** This is a sealed enclosure that maintains a low pressure environment for the electron beam and the specimen. The vacuum chamber prevents the electrons from colliding with air molecules and losing energy. The vacuum chamber also protects the specimen from contamination and oxidation.
- **Sample holder and stag**e: This is a device that holds the specimen and allows it to be positioned and tilted in the beam. The specimen must be very thin, usually less than 100 nm, to allow the electrons to pass through. The specimen can be prepared by various methods, such as slicing, grinding, polishing, etching, or ion milling.
- **Image-producing system:** This is a system that consists of intermediate and projector lenses, which magnify the primary image formed by the objective lens. The image-

producing system can also include various apertures and filters to enhance the contrast and select the desired information from the transmitted electrons. The image-producing system can produce two types of images: bright-field images and dark-field images. Bright-field images show the areas of the specimen that transmit more electrons as brighter, while dark-field images show the areas that scatter more electrons as brighter.

- **Image-recording system:** This is a system that converts the electron image into a form that can be perceived by the human eye or stored digitally. The image-recording system can use a phosphor or fluorescent screen, which emits light when hit by electrons, or a CCD camera, which converts electrons into electrical signals. The image-recording system can also use a photographic film or plate, which records the electron image as a latent image that can be developed later.

Applications of TEM

Nanomaterial Characterization: TEM is widely used to characterize and study various types of nanomaterials such as nanoparticles, nanowires, quantum dots, and nanotubes. Researchers can analyze the size, shape, crystal structure, defects, and composition of these nanomaterials.

Material Science and Engineering: TEM is crucial for understanding the microstructure of materials, including metals, ceramics, polymers, and composites. It helps in studying grain boundaries, dislocations, defects, and phase transformations, providing insights into material properties and behavior(Shi, 2016).

Biology and Medicine: TEM is employed in biological research to study cellular and subcellular structures at high resolution. It enables visualization of organelles, protein structures, viruses, and cellular interactions, contributing to advancements in medical research and drug development.

Electronics and Semiconductor Industry: TEM is used to analyze semiconductor devices and integrated circuits, helping to optimize their performance and understand device failures. It aids in studying nanoscale features like transistors, interconnects, and dielectric layers.

Geology and Earth Sciences: TEM is applied in the study of geological samples to investigate minerals, rocks, and meteorites. It allows for the characterization of crystal structures, mineral composition, and the understanding of geological processes.

Catalysis and Chemistry: TEM is utilized in catalysis research to study catalysts at the nanoscale, facilitating the design of more efficient catalysts for chemical reactions. It is also used to analyze chemical reactions and structures of molecules.

Material Defect Analysis and Quality Control: TEM is crucial for detecting and analyzing defects, impurities, and structural irregularities in materials. It is used in quality control processes to ensure the integrity and performance of manufactured materials.

Environmental Sciences: TEM is used in environmental research to study pollutants, particulate matter, and airborne particles. It helps analyze their composition, size distribution, and potential impacts on the environment and human health.

Agricultural and Food Sciences: TEM is applied in agricultural and food research to study biological and structural aspects of plants, bacteria, fungi, and food components at the microscopic level, aiding in food safety and agricultural productivity. (Mukrimaa et al., 2016).

Crystallographic Analysis: TEM spectroscopy enables crystallographic analysis of materials, including crystal orientation, lattice defects, grain boundaries, and phase identification at the atomic level.

Advantages of TEM

TEM is a sophisticated analytical technique that provides detailed structural, morphological, and compositional information at the atomic and nanoscale. Here are the key advantages of using TEM,

Atomic Scale Imaging: TEM offers atomic-level resolution, enabling direct imaging of individual atoms and crystal lattices in a sample.

High Spatial Resolution: TEM provides extremely high spatial resolution, allowing for the visualization of sub-nanometer features and structures within the sample.

Crystallographic Information: TEM provides crystallographic information, including crystal orientation, defects, grain boundaries, and phase identification within a sample.

Detailed Microstructural Analysis: TEM allows for detailed microstructural analysis, providing information about particle size, shape, distribution, and arrangement.

In-situ and Operando Studies: TEM allows for in-situ and operando studies, enabling the observation of dynamic processes and reactions in real-time under various conditions.

Multi-Modal Imaging: TEM can be integrated with various imaging modes, such as Bright-Field (BF), Dark-Field (DF), and High-Resolution Transmission Electron Microscopy (HRTEM), providing a comprehensive view of the sample.

2. Dynamic light scattering (DLS)

Introduction

Dynamic light scattering: Dynamic Light Scattering (DLS), also known as Photon Correlation Spectroscopy (PCS) or Quasi-Elastic Light Scattering (QELS), is a technique for measuring the size distribution of small particles or molecules in a suspension or solution by analyzing scattered light. It is largely used to characterize particles' Brownian motion and diffusion coefficients, which provide information about their size and distribution

Principle of dynamic light scattering

Dynamic light scattering is a technique for determining the size distribution of particles in a suspension or solution. It operates by lighting the sample with a laser beam and then detecting the fluctuations in scattered light intensity caused by the particles' Brownian motion (Stetefeld et al., 2016). These fluctuations, known as the photon correlation function, are evaluated to determine particle size using autocorrelation principles.

Instrumentation

Some components of a Dynamic Light Scattering (DLS) system:

- **Laser Light Source:** DLS systems employ a laser as the light source, typically in the visible or near-infrared range. The laser emits a monochromatic beam of light.

- **Sample Dispersion:** The sample under investigation is usually in a liquid medium and should be well-dispersed to ensure accurate measurements. Proper sample preparation is essential.
- **Scattering Chamber:** The sample is placed in a scattering chamber, which is typically a cuvette or a cell with transparent walls. The chamber allows for the passage of the laser light and the collection of scattered light.
- **Scattering of Light:** When the laser beam interacts with the particles in the sample, it is scattered in all directions. The scattered light contains information about the particle sizes and their Brownian motion.
- **Photodetector:** A photodetector is used to detect the scattered light. It measures the intensity of the scattered light as a function of time.
- **Correlation Electronics:** DLS systems incorporate correlation electronics to analyze the fluctuations in the scattered light intensity over time. This analysis is based on the principles of photon correlation spectroscopy.
- **Auto-Correlation Function:** The correlation electronics generate an auto-correlation function, also known as a correlation curve, which provides information about the dynamics and motion of particles in the sample.
- **Particle Size Distribution:** The auto-correlation function is analyzed to determine the particle size distribution in the sample. It reveals information about the sizes of the particles or molecules undergoing Brownian motion.
- **Zeta Potential Measurement (Optional):** Some DLS systems are equipped with additional components, such as a phase analysis light scattering (PALS) detector, to measure the zeta potential of particles in the sample, providing information about their surface charge and stability.
- **Temperature Control (Optional):** Temperature control may be integrated into the system to perform measurements at different temperatures, which can affect the particle size and sample behavior.

Function Of Dynamic Light Scattering System

A light scattering system is an experimental setup or instrument designed to study the scattering of light by particles, molecules, or other structures in a sample. This technique provides valuable information about the size, shape, concentration, and other properties of the scatterers(Malvern Instruments Ltd., 2018). Here are the main functions and capabilities of a light scattering system:

Scattering Intensity Measurement: Measures the intensity of scattered light at different angles, providing data that can be analyzed to determine particle size and concentration.

Particle Size Analysis: Determines the size distribution and average size of particles or macromolecules in a sample based on the scattering pattern.

Particle Shape Analysis: Provides information about the shape or morphology of particles by analyzing the scattering pattern, especially for anisotropic scatterers.

Molecular Weight Determination: Utilizes light scattering in conjunction with other techniques to determine the molecular weight of macromolecules such as proteins, polymers, and aggregates.

Concentration Measurement: Estimates the concentration of particles or macromolecules in a solution or suspension based on the intensity of scattered light.

Zeta Potential Measurement: Determines the zeta potential of particles, providing insights into their surface charge and stability(Hackley & Clogston, 2011).

Application of Dynamic light scattering

Dynamic Light Scattering (DLS) is a technique used to measure the size distribution of particles in a suspension or solution by analyzing the fluctuations in the scattered light caused by Brownian motion(Hackley & Clogston, 2011). Some applications of Dynamic Light Scattering (DLS) systems:

Nanoparticle Characterization: DLS is widely used to measure the size distribution and hydrodynamic radius of nanoparticles and colloidal particles, which is essential in nanotechnology and materials science.

Protein and Biomolecule Analysis: DLS is used to measure the hydrodynamic size of proteins, nucleic acids, liposomes, and other biomolecules, aiding in the study of their structure, aggregation, and interactions.

Pharmaceuticals: In the pharmaceutical industry, DLS is applied to characterize drug formulations, micelles, liposomes, and other drug delivery systems, ensuring stability and efficacy.

Polymer and Polymer Nanoparticle Analysis: DLS helps in measuring the size and size distribution of polymer particles, polymer micelles, and other polymer-related materials.

Colloidal Stability Studies: DLS is used to monitor and analyze the stability of colloidal suspensions and emulsions over time, providing crucial insights for industrial applications.

Quality Control in Manufacturing: DLS is used for quality control of products, ensuring the desired particle size and size distribution in various industries such as cosmetics, paints, and coatings.

Food and Beverage Industry: DLS helps in analyzing the size distribution of particles in food and beverage products, ensuring product quality and stability.

Environmental Analysis: DLS is applied to study and measure particle sizes in environmental samples like aerosols, sediments, and colloidal suspensions, aiding in environmental monitoring.

Agriculture: DLS is used to analyze the size distribution of particles in agrochemicals, fertilizers, and agricultural formulations, optimizing their efficiency and performance.

Research in Physics and Chemistry: DLS is a fundamental tool for researchers studying colloids, soft matter, and complex fluids, providing insights into particle dynamics and interactions.

Biotechnology and Medical Research: DLS is utilized in various biomedical and biotechnological studies, including drug development, diagnostics, and biomolecular interactions.

Cosmetics and Personal Care Products: DLS is employed to analyze the particle size distribution of ingredients in cosmetics and personal care products, ensuring product stability and performance.

Education and Research: DLS systems are used in academic institutions for teaching and research in physics, chemistry, biology, and related fields.

Dynamic Light Scattering is a versatile technique with applications spanning across research, industries, and various scientific disciplines, providing valuable insights into particle size and distribution in diverse sample types.

Advantages of Dynamic light scattering system

Dynamic Light Scattering (DLS) is a technique used to measure the size distribution of particles in a suspension or solution based on the analysis of scattered light(Malvern Instruments Ltd., 2018). Some advantages of using a Dynamic Light Scattering (DLS) system:

Particle Size Measurement: DLS provides rapid and accurate measurement of particle sizes in a sample, including nanoparticles and colloidal particles, without the need for extensive sample preparation.

Wide Range of Particle Sizes: DLS can measure a wide range of particle sizes, from a few nanometers to several micrometers, making it suitable for a broad array of applications.

Real-Time Analysis: DLS allows for real-time monitoring of changes in particle size and size distribution, enabling the study of dynamic processes and reactions.

High Sensitivity: DLS is highly sensitive, capable of detecting small changes in particle size and concentration, making it suitable for analyzing low concentrations of particles.

Non-Invasive Technique: DLS is a non-invasive and non-destructive technique, preserving the integrity of the sample and allowing for multiple measurements on the same sample.

Sample Flexibility: DLS is versatile and can analyze various sample types, including liquids, suspensions, emulsions, proteins, polymers, and nanoparticles.

Ease of Use: DLS systems are user-friendly, with straightforward setup and data acquisition processes, making them accessible to both novice and experienced users.

Cost-Effective: DLS systems are relatively cost-effective compared to some other particle characterization techniques, providing valuable data at a reasonable price.

Quality Control: DLS is used for quality control in manufacturing to ensure consistent particle sizes and distributions in manufactured products.

Zeta Potential Calculation: In some DLS systems, zeta potential can be calculated, providing additional information about particle surface charge and stability.

Research and Development: DLS is widely used in research and development for the characterization of nanoparticles, colloids, polymers, and biomolecules.

Biological and Biomedical Applications: DLS is valuable in biological and biomedical research, aiding in the analysis of biological nanoparticles, liposomes, proteins, and drug delivery systems.

Educational and Research Tool: DLS systems are utilized in academic institutions for teaching and research purposes, providing insights into particle size and dynamics.

Environmental Monitoring: DLS can be used for environmental monitoring to analyze particles and colloids in environmental samples.

Dynamic Light Scattering is a versatile and essential technique, providing critical insights into particle size and size distribution, making it applicable in various scientific, industrial, and technological domains.

Conclusions:

TEM spectroscopy finds applications in various research areas, including materials science, nanotechnology, biological sciences, physics, chemistry, and semiconductor technology. TEM spectroscopy is a powerful analytical tool that offers detailed insights into the electronic and structural properties of materials at the nanoscale, contributing to advancements in various scientific and technological fields. Dynamic Light Scattering (DLS) is a technique used to measure the size distribution of particles in a suspension or solution based on the analysis of scattered light.

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