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CHAPTER 10

Comprehensive Characterization of Thin Films Using Advanced Microscopic and Spectroscopic Techniques

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Abstract: Usually found at the atomic or molecular scale, thin films have unique physical and chemical characteristics that are impacted by the size, shape, and spatial distribution of nanoparticles. Understanding these films' structure, surface characteristics, and functional behaviour requires accurate characterisation. Numerous spectroscopic and microscopic methods have been developed to gather comprehensive data on the composition and shape of thin films. Crystallinity, particle arrangement, and surface topography can all be understood by microscopic techniques like as X-ray diffraction (XRD), energy-dispersive x-ray analysis (EDAX), scanning electron microscopy (SEM), atomic force microscopy

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(AFM), and scanning tunnelling microscopy (STM). These methods create high-resolution pictures of individual particles and allow for visualisation and measurement at the nanoscale. Optical characteristics, molecular vibrations, elemental composition, and chemical bonding are all investigated using spectroscopic techniques like UV-visible spectroscopy, Raman spectroscopy, Fourier-Transform Infrared Spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS), and X-ray Fluorescence (XRF). Combining these techniques gives researchers a thorough grasp of thin film systems, which helps with material design and optimisation for uses in energy storage, electronics, sensors, and catalysis. The development of high-performance thin film nanomaterials is supported by the exact evaluation of structural and functional properties that is ensured by the combination of numerous approaches.

Keywords: Nanoparticles, Microscopy Thin film, Spectroscopy.

Introduction

Thin films refer to engineered layers of material formed at the atomic or molecular scale, typically with a thickness in the nanometre to microscale range. These materials often possess at least one dimension below 100 nanometres, making them integral to nanotechnology and material science. The unique properties of thin films arise due to their small size and surface effects, which differ significantly from those of bulk materials. Common examples observed in daily life include soap bubbles, anti-reflective coatings on lenses, and oil films on water surfaces [1].

Thin films are purposefully put onto substrates in material science to add or improve particular physical, chemical, or optical characteristics that the basic substrate cannot offer on its own [2]. High-purity materials and deposition methods including sputtering, evaporation, and chemical vapour deposition are used to create these films. Precursor gases, sputtering targets, and evaporation sources are materials used in the creation of thin films. Physical characteristics of thin films, such as particle size, shape, surface morphology, crystallinity, and dispersion, have a significant impact on their functionality and performance [3].To comprehend thin films' structural characteristics and interactions with their surroundings, characterisation is essential. For this, two main categories of procedures are used: spectroscopic and microscopic methods [4].Microscopic methods offer important insights into thin-film topography, size distribution, and morphology [3,4]. These consist of atomic force microscopy (AFM), scanning tunnelling microscopy (STM), transmission electron microscopy (TEM), high-resolution TEM (HRTEM), and scanning electron microscopy (SEM). High-resolution images produced by these techniques aid in the visualisation of individual thin film particles and the comprehension of their structural characteristics [5].Conversely, spectroscopic methods are employed to investigate how thin films interact with electromagnetic radiation at different wavelengths. These techniques make it possible to analyse the elements' concentration, bonding, and chemical makeup within the films [4,5,6]. Methods including X-ray photoelectron spectroscopy (XPS), X-ray photon correlation spectroscopy, Raman spectroscopy, ultraviolet-visible (UV-Vis) spectroscopy, zeta potential analysis, and attenuated total

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reflectance Dynamic light scattering (DLS) and Fourier-transform infrared spectroscopy (ATR-FTIR) are frequently employed [6]. All things considered, a thorough grasp of thin film characteristics via sophisticated characterisation methods is necessary to customise their performance in applications ranging from electronics and optics to healthcare and energy devices [5,6].

1. X-Ray Diffraction (XRD) Analysis of Nanomaterials and Thin Films

A popular non-destructive analytical method, X-ray diffraction (XRD) is mainly used to identify crystalline phases and to ascertain structural variations brought on by internal stresses or faults [6,7]. It works especially well for examining nanostructures, thin films, and bulk materials. Phase composition, average crystallite size, degree of crystallinity, texturing coefficients, lattice strain, and orientation parameters are among the vital details that XRD offers [7]. These characteristics are crucial for comprehending the performance and structural integrity of materials in a variety of physics, materials science, and engineering applications [7, 8].

In XRD, the material's crystal structure interacts with a collimated monochromatic X-ray beam. Since X-rays are electromagnetic waves, their interaction with atoms' electrons is the main cause of their elastic scattering. A diffraction pattern is created when these dispersed waves constructively interfere in particular directions. Bragg's Law [7-9] defines the prerequisite for constructive interference as follows:

$$2dsin\theta = n\boldsymbol{\lambda} \qquad \text{.................... (1)}$$

Where n is the diffraction order, θ is the diffraction angle, d is the interplanar spacing, and λ is the X-ray wavelength [8,9]. The tiny crystallite size and surface-induced stress in nanostructured materials cause noticeable peak position shifts and diffraction pattern broadening, which reveal information about particle size and strain [9].

There are several ways to perform XRD, including grazing incidence, rocking curve, and the θ–2θ scan. In the θ–2θ scan mode, the detector travels at an angle 2θ at the same time that the incident X-ray beam strikes the sample at an angle θ, guaranteeing that reflected rays are always captured at the proper geometry [10]. A diffraction spectrum showing intensity as a function of 2θ is the end result. With this setup, reflection is only captured when the incident and reflected angles meet Bragg's condition [9–10].

$$Angle\ of\ Incidence\ (\theta_i) = Angle\ of\ Reflectance\ (\theta_r) \qquad \text{........... (2)}$$

The detector travels at twice the angular speed of the X-ray source in a standard X-ray diffraction (XRD) θ–2θ scan [5,7]. This satisfies Bragg's condition for constructive interference by guaranteeing that the angle of incidence (θ_i) and the angle of reflection (θ_r) are equal. Therefore, only when this geometric requirement is satisfied is the diffraction intensity reported. Significant internal stresses are caused by the existence of ultra-small crystallites and a high surface-to-volume ratio in nanomaterials [7-8]. When compared to conventional reference data, these stresses result in observable broadening and shifting of the diffraction peaks, which offers important information about the structural state of the material [6–8].

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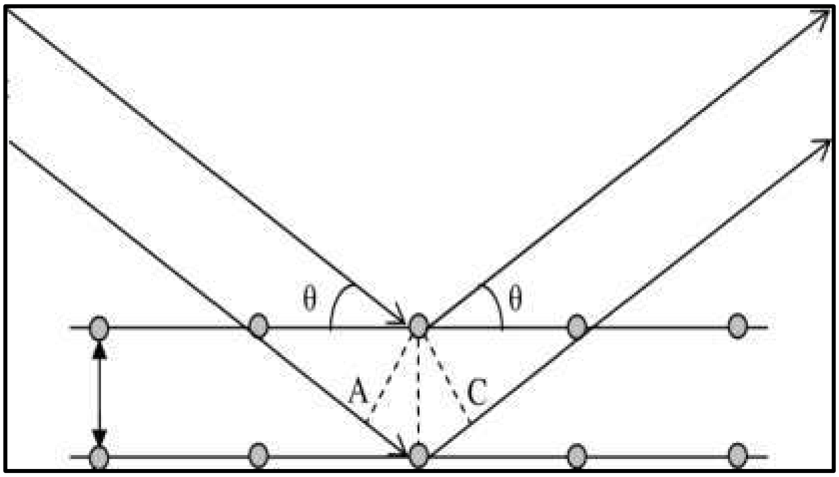

Fig. 1. Schematic diagram of X- Ray Diffraction [11]

In addition to revealing the crystalline structure of nanomaterials, XRD enables the assessment of internal lattice strain and the determination of crystallite size using the Scherrer equation [11]. The presence of discrete diffraction spots in the pattern corresponds to specific lattice planes, making XRD a vital technique for structural characterization of thin films and nanoscale materials [11-12].

Uses of XRD in Research and Other Fields

One essential non-destructive method for examining a material's crystallographic structure, phase composition, and physical characteristics is X-ray diffraction (XRD). It quantifies crystallite size and internal strain, identifies phases, and establishes crystallinity in materials research. XRD is used in nanotechnology to assess surface coatings, thin films, and nanostructures [13]. It is employed in the pharmaceutical sector to guarantee uniformity in medication formulations and to identify and track polymorphs [13–14]. XRD is used by geologists to identify minerals and analyse soil [14–15]. It aids in the study of phase changes, residual stress, and grain orientation in metals and alloys in metallurgy [14]. XRD is used in forensic science to examine trace evidence, such as paints and powders [15]. Because it provides accurate structural insights, XRD is therefore essential to the advancement of research and industrial applications [13–15].

2. Raman Spectroscopy: Principle and Applications

By using the inelastic scattering of monochromatic light, Raman spectroscopy is a potent analytical method for examining the molecular makeup and vibrational characteristics of materials [15]. Raman spectroscopy looks at the energy shift that happens when incident photons interact with the vibrational or rotational modes of molecules, as opposed to conventional absorption or emission spectroscopy, which depends on photon-induced electronic transitions. Special insights into molecular structures, chemical surroundings, and bonding properties are offered by this scattering process [13–15].

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Raman spectroscopy has many benefits, even though it is intrinsically less sensitive than absorption or fluorescence methods because of the low likelihood of inelastic scattering events [15]. Utilising excitation wavelengths that are not absorbed by the medium is a significant advantage; this is especially helpful when examining samples that are aqueous or mineral-rich and show high levels of infrared (IR) absorption [15–17]. Additionally, because laser light can be precisely focused on sample locations the size of microns, the approach enables micro-scale spatial resolution [17]. Furthermore, vibrational modes that may be inactive or prohibited in IR absorption spectra can be detected using Raman scattering since it adheres to different selection principles than IR spectroscopy [17,18].

Working Principle

A polarised monochromatic laser source is focused onto the sample in a standard Raman spectroscopy setup. A high-resolution monochromator is then used to analyse the scattered light, which is frequently gathered at a 90° angle to the original beam. After setting the laser frequency, the scattered light is analysed to find frequency shifts that match the vibrational energy levels of the molecules in the sample [18]. A central Rayleigh peak, signifying elastically scattered light, and a series of sidebands, including Stokes lines at lower frequencies and anti-Stokes lines at higher frequencies in relation to the incoming light, are included in the resulting spectrum. Stokes lines are more frequently documented because they are typically more intense [18–20].

Raman spectroscopy is frequently thought of as an adjunct to infrared spectroscopy and is classified as a form of vibrational spectroscopy [19]. Although vibrational transitions are probed by both methods, the selection principles regulating transition activity are different. There are 3N–6 fundamental vibrational modes (normal modes) in a nonlinear polyatomic molecule with N atoms, each of which has a distinct symmetry and frequency [17–20]. When combined with infrared data, Raman spectroscopy makes it possible to identify these modes and, in many situations, to infer the molecular structure [20].

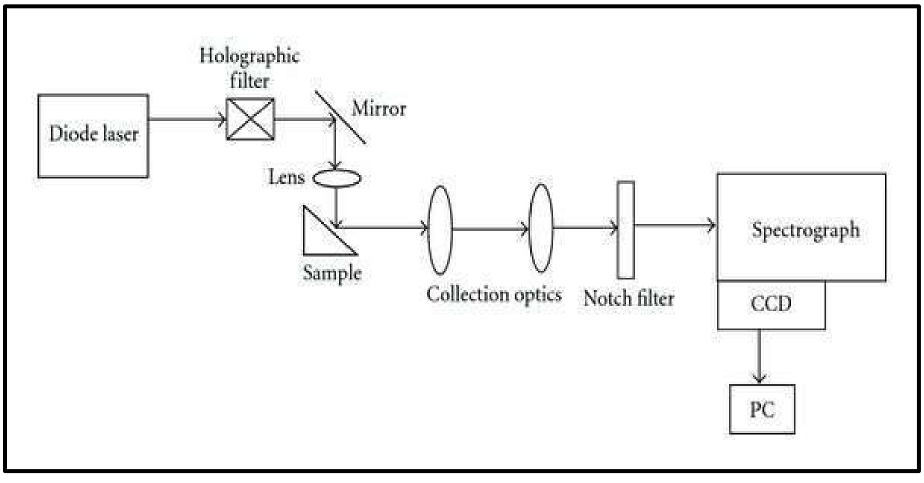

Fig. 2. Raman Spectroscopy [20]

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Depolarization and Molecular Symmetry

The depolarisation ratio (ρ), which characterises the polarisation properties of the scattered light, is a crucial quantity in Raman spectroscopy. The dispersed light may become depolarised or maintain the same polarisation when the incident beam is linearly polarised. The definition of the depolarisation ratio is as follows [19–20]:

$$\rho = \frac{I_{\perp}}{I_{||}}$$

Where the intensity of dispersed light polarised parallel to the incident beam are denoted by I_⊥ and I_(||) correspondingly [20–21]. The degree of depolarisation yields important details regarding the symmetry of the vibrational modes, which can then be utilised to deduce the molecule's structural characteristics.

Applications

Numerous disciplines, including chemistry, physics, materials science, biology, geochemistry, and forensic science, use Raman spectroscopy [19–21]. Without requiring a lot of preparation, it is frequently employed in the identification of chemical substances, molecular structure analysis, carbon-based material characterisation, stress and strain analysis in solids, and biological sample analysis [21].

3. Transmission Electron Microscopy (TEM): Principle and Applications

A sophisticated imaging method that is frequently used to examine the interior structure of incredibly thin objects at incredibly high resolutions is transmission electron microscopy (TEM) [22]. TEM creates two-dimensional images by passing a high-energy electron beam through a thin sample, in contrast to Scanning Electron Microscopy (SEM), which produces three-dimensional surface images [23]. The basic principles of this technique are the same as those of optical microscopy, but it employs electrons rather than light, enabling much higher spatial resolution—up to ~0.1 nm, which is almost 1000 times better than what is possible with traditional light microscopes [22,23].

Because electrons have a low penetrating power, TEM requires a very thin specimen, usually no more than a few hundred angstroms (1 Å = 10^{-10} m) [23, 24]. High Voltage Electron Microscopy (HVEM) can be used to enhance electron penetration when thicker specimens are required [24].

Working Principle of HRTEM

Electrons behave like waves, which is how High-Resolution Transmission Electron Microscopy (HRTEM) works. A filament emits electrons thermionically, accelerates them at high voltage, and focusses them onto the specimen [24]. A transmitted or diffracted beam is created when electrons interact with the material's atoms, and this beam is subsequently employed to create an image on a digital detector or fluorescent screen [23–25]. HRTEM is an effective method for nanoscale investigation because of the

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accelerated electrons' extremely small wavelengths, which allow for the visualisation of atomic groupings and crystallographic features [25].

Applications of TEM

TEM is widely used in research and industry for topographical, morphological, compositional, and crystallographic studies [26]. Some key applications include:

1. Structural analysis of materials and nanomaterials.
2. Characterization of defects, dislocations, and grain boundaries.
3. Investigation of semiconductors and integrated circuits.
4. Failure analysis in microelectronics and manufacturing industries.
5. Biological studies such as imaging bacteria, viruses, and cellular organelles.
6. Differentiation between plant and animal cells at the ultrastructural level.
7. Visualization of microbial features like flagella and plasmids.
8. Studying nanoparticles such as carbon nanotubes (CNTs) and metal nanoparticles.
9. Academic research in materials science, nanotechnology, and life sciences.

TEM thus plays a vital role in both fundamental research and technological innovation across disciplines [27].

4. Scanning Electron Microscopy (SEM): Principles and Applications

A concentrated beam of high-energy electrons scans a sample's surface to create incredibly detailed images using the Scanning Electron Microscope (SEM), a potent analytical instrument [28–30]. SEM uses electron beams to interact with the specimen's surface atoms, in contrast to optical microscopy, to disclose compositional, morphological, and topographical details at the nanoscale [27–29,31]. Under high vacuum, a highly concentrated electron beam—typically with a diameter of nearly one nanometer—scans the sample surface in a raster pattern. Depending on the device and use, electron beam energy can be as high as 200 kV [30].

The interactions between the input electron beam and the atoms in the sample, which result in the production of secondary electrons and backscattered electrons, are the main cause of the contrast in SEM images [29, 30]. The main purpose of secondary electrons, which are released from an atom's outer shell by inelastic scattering, is to create fine-grained surface pictures. Since the intensity of backscattered electrons, which are the product of elastic scattering, varies according to the atomic number of the constituent elements, they offer compositional contrast [30–32].

Working Principle of SEM

Electromagnetic lenses are used to concentrate and accelerate the electrons emitted by the electron source in SEM into a fine beam [31]. This beam is deflected both horizontally and vertically as it passes through a number of scan coils, allowing for a point-by-point scan of the material. Different signals, including

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secondary electrons, backscattered electrons, and X-rays, are produced as the electron beam interacts with the sample. These signals are picked up by detectors to create pictures or carry out elemental analysis [32].

On a monitor (formerly a cathode ray tube, or CRT), the output is shown. The brightness at each point is adjusted according to the strength of the signal that was detected [28–30]. Using the linear magnification formula [29–32], the magnification in SEM is calculated:

$$M=\frac{L}{l}$$

Where l is the raster length on the sample surface and L is the raster length on the display screen. A variety of materials may be seen in detail on their surfaces thanks to SEM's high magnification and resolution (down to about 1 nm) [32].

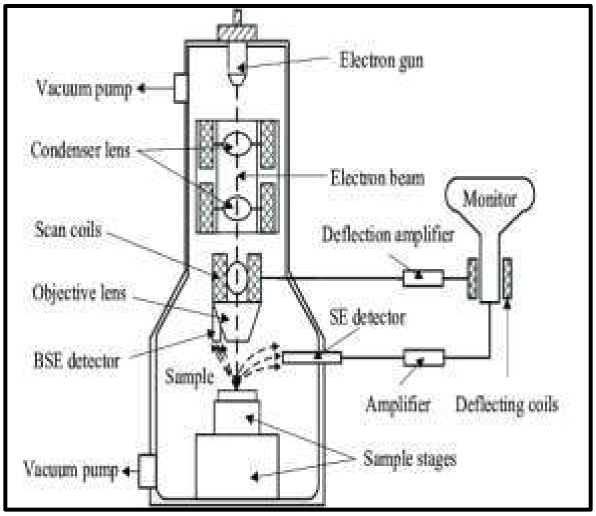

Fig. 3. Diagram for scanning electron microscope [62].

Applications of SEM

SEM is widely used in numerous fields [30-33]:

1. **Materials science** – for failure analysis, microstructure examination, and quality control.
2. **Semiconductor industry** – for nanowire inspection and gas sensing applications.
3. **Forensic science** – in gunshot residue analysis, filament bulb examination in accident reconstruction, and bullet marking comparisons.
4. **Microelectronics** – for microchip assembly and defect analysis.
5. **Biomedical research** – in studying viruses, diagnosing diseases, analyzing biological samples, and testing vaccine effectiveness.

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5. Energy-Dispersive X-ray Spectroscopy (EDX): Principles and Application

For elemental analysis and chemical characterisation of materials, Energy-Dispersive X-ray Spectroscopy (EDX or EDS) is a microanalytical technique that is frequently used in conjunction with electron microscopy, such as Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) [34]. Characteristic X-rays are emitted as a result of the interaction between the incident electron beam and the atoms in the sample [34, 35].

Inner-shell electrons from atoms in the material are ejected when a high-energy electron beam hits the sample surface. In order to fill the resulting vacancy, electrons from higher energy levels move to the lower energy state . X-ray photons, which are unique to the element's atomic structure, are released as energy during this transition. Therefore, elemental identification may be done both qualitatively and quantitatively because each element produces X-rays with distinct energy levels [35, 36].

EDX detectors, typically integrated with SEM or TEM systems, collect these emitted X-rays and generate a spectrum. Peaks in the spectrum correspond to different elements present in the sample, providing direct insight into the sample's elemental composition [36]. This capability makes EDX a fundamental tool for analyzing the surface and subsurface features of materials [34-36].

Working Principle of EDX

The detection of distinctive X-rays released from a specimen after it has been excited by an electron beam is the basis for how EDX operates. An energy-dispersive detector subsequently analyses these X-rays and separates them according to their energy [37]. Peaks that represent the elements in the sample are shown in the resultant spectrum, and the strength of each peak indicates the relative concentration of that element [35–37].

A multi-channel analyser, a pulse processor, and a solid-state detector—typically silicon drift detectors—make up the EDX system [38]. This technique improves the spatial resolution of chemical distribution by enabling elemental mapping of a sample surface in conjunction with the scanning beam in SEM [39].

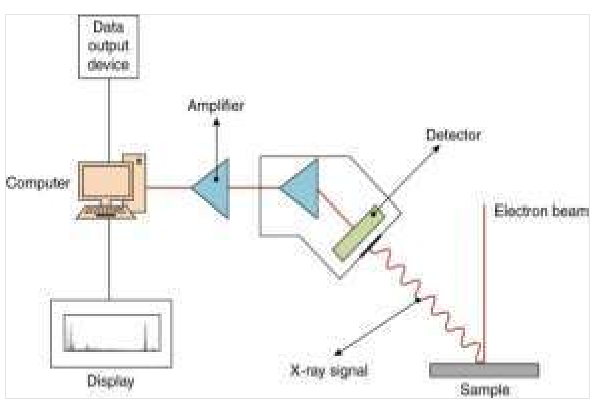

Fig. 4. Schematic diagram of EDX [63]

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Applications of EDX

EDX is used in many different scientific and industrial domains, Because of its effectiveness and adaptability. Key applications include [35-39]:

1. Quality control and process optimization in manufacturing environments.
2. Rapid detection of contaminants and unknown elements in materials.
3. Monitoring environmental emissions and controlling exposure to hazardous substances.
4. Troubleshooting production defects, identifying root causes of process failures.
5. Applications in agriculture and food industries, particularly in soil and fertilizer analysis.
6. Automotive and machinery sectors, for evaluating corrosion, coatings, and material interfaces.

6. Fourier-Transform Infrared Spectroscopy (FTIR): Principles and Applications

Fourier-Transform Infrared Spectroscopy (FTIR) is a widely used analytical technique based on the interaction of infrared (IR) radiation with matter [40]. FTIR measures how a sample absorbs light at different IR wavelengths, providing valuable information about the chemical composition and structure of the material [41]. The technique is particularly useful for identifying organic compounds and functional groups due to the unique absorption patterns of molecular bonds [40-42].

Certain wavelengths of infrared radiation are absorbed by a sample based on the vibrational transitions of molecular bonds [42]. A distinct absorption spectrum is produced by the absorption of infrared radiation at a particular frequency by each type of bond or functional group [41–43]. This enables the use of FTIR as a molecular identification fingerprinting method [40–42].

Working Principle of FTIR

The basis of FTIR's operation is the absorption of infrared radiation, which results in molecular vibrations like chemical bond stretching or bending. The spectrum that results from the sample's absorption of frequencies that correspond to the vibrational modes of its bonds shows these interactions [41–43,45]. The FTIR spectrometer modulates the infrared light using an interferometer, producing an interferogram that encodes all frequency data as a function of optical path difference or time [45].

The Fourier Transform is then used to mathematically transform this interferogram and produce an IR absorption spectrum, which is usually displayed with transmittance or absorbance on the Y-axis and wave number (cm^{-1}) on the X-axis. Both qualitative and quantitative analysis are made possible by the fact that each peak represents a distinct molecular vibration [44].

Samples as thin as 20 microns can be analysed by modern FTIR instruments when coupled to microscopes, which is especially helpful for identifying residues, particles, coatings, and fibres [45].

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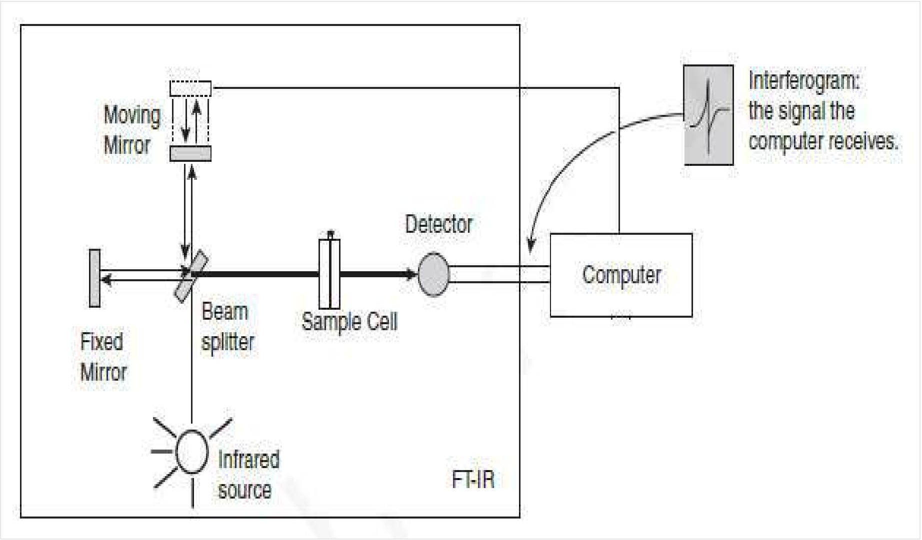

Fig. 5. Schematic representation of FTIR [64].

Applications of FTIR

FTIR spectroscopy is extensively used in various scientific and industrial fields. Key applications include [41-45]:

1. Identification of unknown substances and verification of product formulations.
2. Material characterization in polymers, rubber, and plastics through deformation or thermal analysis.
3. Microscale contamination analysis by detecting small fragments or particulates.
4. Thin-film and coating analysis, especially in quality control and failure analysis.
5. Monitoring of emissions in industrial processes, including automotive exhaust and smokestacks.
6. Real-time reaction monitoring by observing changes in IR absorption over time.

Because of its high sensitivity, quick analysis, low sample preparation requirements, and capacity to identify a broad variety of organic and inorganic chemicals, FTIR is beneficial [43–45].

7. X-ray Photoelectron Spectroscopy (XPS): Principle and Applications

X-ray A potent surface-sensitive analytical method for the quantitative and chemical examination of elements found on a material's surface, usually in the top 1–10 nm, is photoelectron spectroscopy (XPS). The elemental makeup, chemical states, and electronic states of the atoms on a sample's surface can all be determined with the help of XPS [46].

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The XPS Working Principle The basis of XPS is the photoelectric effect, in which atoms' core-level electrons are expelled when input X-ray photons, typically Al Kα or Mg Kα radiation, and assault the sample surface.

The kinetic energy $E_{kinetic}$ of the emitted electrons is measured using an energy analyzer, and the binding energy $E_{binding}$ is calculated using the equation:

$$E_{binding} = E_{photon} - (E_{kinetic} + \emptyset)$$

Where

- E_kinetic is the measured kinetic energy of the expelled electron,
- E_photon is the energy of the incident X-ray,
- The work function of the spectrometer is ∅.

Each element has characteristic binding energies, allowing XPS to identify the elements present and their chemical states. The depth of analysis is limited to a few nanometers, making it ideal for surface studies [47,48].

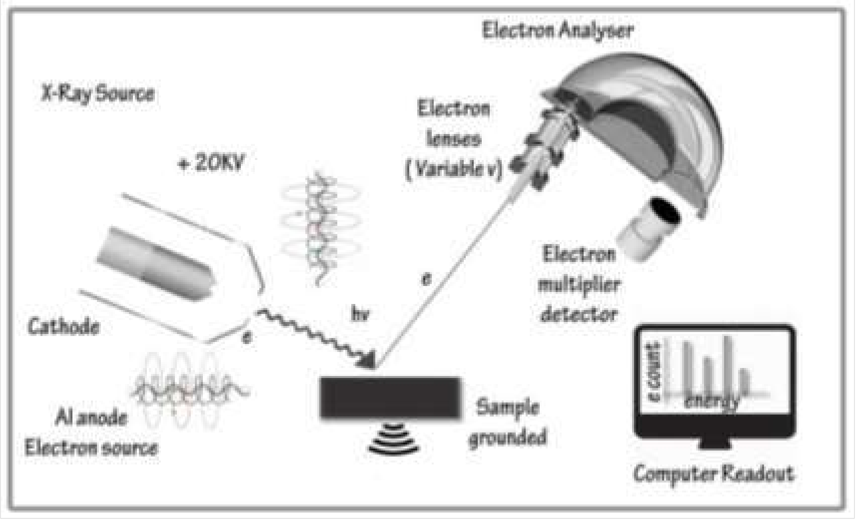

Fig. 6. Schematic representation of *XPS* [65].

Applications of XPS

XPS finds extensive applications in both academic and industrial research:

1. **Surface composition analysis** – identifying elements and their chemical states on the surface.
2. **Contaminant detection** – determining unwanted elements or compounds on material surfaces.

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3. **Depth profiling** – analyzing compositional changes across thin film layers using ion etching.
4. **Oxidation state identification** – distinguishing between different oxidation states of metals or compounds.
5. **Thin film characterization** – studying coatings, corrosion layers, and surface treatments.
6. **Failure analysis** – investigating surface failures due to chemical or mechanical processes.

Because of its high surface sensitivity and quantitative accuracy, XPS is widely used in fields such as materials science, nanotechnology, catalysis, corrosion science, and semiconductor research [49,50].

8. Atomic Force Microscopy (AFM): Principle and Applications

The high-resolution scanning probe microscopy (SPM) method known as "atomic force microscopy" (AFM) can image, measure, and work with materials at the nanoscale. The diffraction limit of traditional optical microscopes is much exceeded by AFM, which offers spatial resolution on the order of fractions of a nanometre [51]. By identifying the forces that interplay between a sharp probe tip and the sample surface, AFM is frequently used to map surface topography at the atomic level [52]. AFM is flexible for biological and soft material characterisation since it doesn't require sample coating or vacuum conditions, unlike methods like TEM or SEM [51–53].

Working Principle of AFM

A pointed probe set atop a flexible cantilever is how AFM works. Interatomic forces (such as van der Waals, electrostatic, capillary, or mechanical contact forces) between the tip and sample induce deflections in the cantilever as the probe moves across the surface. According to Hooke's Law, these deflections are proportional to the applied force [51-53,55]:

$$F = -k \cdot \Delta z F$$

Where:

- F = force acting on the cantilever
- k = spring constant of the cantilever
- Δz = deflection of the cantilever

In order to track cantilever motion as the tip scans the surface, a position-sensitive photodiode receives a laser beam that is focused on the rear of the cantilever and reflected into it. A topographical image is then created by reconstructing this data [53].

AFM can operate in several modes:

- **Contact Mode:** tip remains in contact with surface
- **Tapping Mode:** tip intermittently touches the surface

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- **Non-contact Mode:** tip hovers close to the surface detecting van der Waals forces

AFM can also perform force spectroscopy, which measures forces as a function of tip-sample distance to study mechanical properties such as stiffness and elasticity [54].

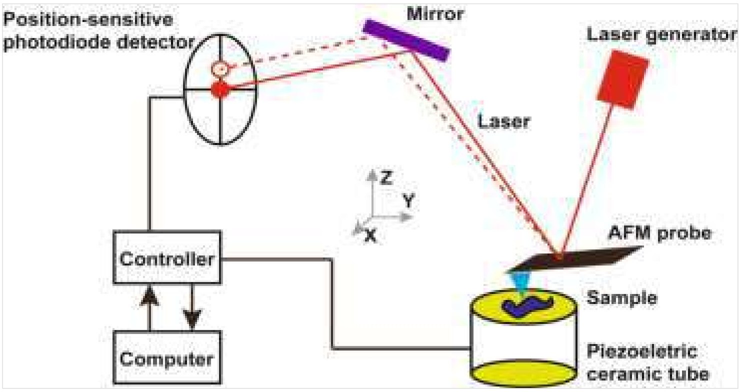

Fig. 7: Presentation of Atomic Force Microscope (AFM) [66]

Applications of AFM

AFM has a very range of applications across materials science, biology, and nanotechnology [51-55]:

1. Nanostructure imaging – High-resolution topographic imaging of surfaces including semiconductors, polymers, and biomolecules.
2. Thin film characterization – Studying surface roughness, morphology, and uniformity in coatings and photovoltaic materials.
3. Force measurements – Evaluating interaction forces and mechanical properties through force-distance curves.
4. Manipulation and nanolithography – Patterning surfaces using tip-induced forces (e.g., scanning probe lithography).
5. Biological applications – Imaging living cells and observing surface-bound proteins or DNA without damaging samples.

AFM is particularly valuable for non-destructive analysis of soft, fragile, or insulating materials under ambient or liquid environments [55].

9. Brunauer–Emmett–Teller (BET) Analysis

One popular approach for determining the specific surface area of solid materials is the Brunauer–Emmett–Teller (BET) characterisation method [56]. In materials science, catalysis, nanotechnology, and adsorption research, where surface area is crucial in determining material performance, it is especially significant [56,57]. By expanding the Langmuir adsorption theory to multilayer adsorption, the BET

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approach works with both porous and non-porous substances. At liquid nitrogen temperature, the analysis usually entails the physical adsorption of a gas, usually nitrogen, onto the surface of a solid sample [57]. The obtained data offers important information about surface reactivity, adsorption characteristics, and material porosity [57].

Working Principle

The idea that gas molecules can adsorb in several layers on a solid surface is the foundation of the BET hypothesis. BET takes into account the development of multilayers prior to condensation, in contrast to the Langmuir model, which only assumes monolayer adsorption [56–58]. A solid sample is first exposed to an inert adsorbate gas at a steady temperature (about 77 K for nitrogen) to start the process. A single monolayer of molecules first forms on the gas's surface as the relative pressure rises, and then further layers are added [58]. An adsorption isotherm is created by recording the amount of gas adsorbed at various relative pressures [59].

The volume of adsorbed gas and the relative pressure are related by the BET equation in its linear version. The monolayer capacity is calculated from the BET plot's slope and intercept. The specific surface area of the sample can be determined using this value in conjunction with the adsorbate molecule's known cross-sectional area [58–60].

The method assumes:

1. The surface is energetically uniform [58].
2. Adsorption in each layer occurs the same heat adsorption beyond the first layer [59].
3. There is no interaction between molecules in adjacent layers [60].

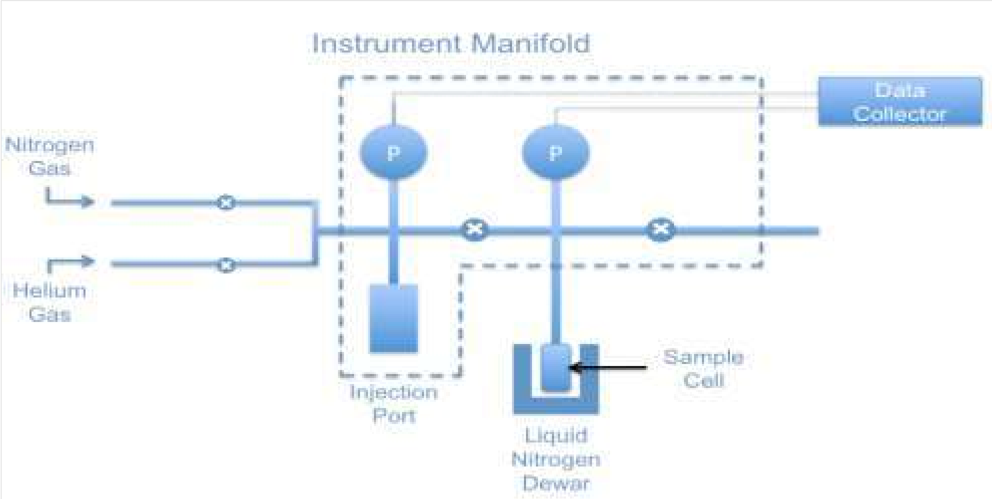

Fig. 8. BET Surface Area Analysis of Nanoparticle [66]

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Applications

The BET characterization technique is extensively used in research and industry due to its ability to provide reliable surface area measurements [59,60]. Key applications include:

1. **Catalysis** – In heterogeneous catalysis, surface area strongly influences reaction rates. BET analysis helps in designing catalysts with high surface exposure for better performance [59,60].
2. **Nanomaterials** – The surface area of nanoparticles is frequently associated with their size, shape, and surface activity. Understanding these parameters for use in biomedical devices, sensors, and energy storage is much easier with the use of BET analysis [56–58,61].
3. **Adsorbent Materials** – BET measurements evaluate adsorption efficiency for gas storage, separation, or purification for materials such as metal–organic frameworks (MOFs), zeolites, and activated carbon [61].

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