



A Brief Overview of Basic Surface Morphology Characterization Techniques in Thin Films

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Thin films and nanoscale materials are the fundamental building blocks for many high-tech applications today. Understanding and optimizing the performance of these materials highlights the importance of fundamental surface morphology characterization techniques. The surface morphology of these materials at the atomic and molecular scale is studied by various characterization methods such as electron microscopes, scanning tunneling microscopes and surface analysis techniques. These techniques provide critical information on the material's surface topography, crystal structural properties and chemical composition. In thin film technologies, the uniformity and order of a material's surface morphology greatly affects its optical and electrical properties. Whereas in nanoscale materials, the determination of surface morphology plays an important role, especially in areas such as catalysis, sensor technologies and nanoelectronics. These characterization techniques provide researchers, engineers and industry in the fields of nanoscience and nanotechnology with critical information to optimize the design, fabrication and performance of nanoscale materials. For this purpose, scanning electron microscopy (SEM), atomic force microscopy (AFM), scanning tunneling microscopy (STM) and X-ray diffraction (XRD) techniques used in the characterization of the surface morphology of thin films were mainly investigated.

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1. Introduction

Thin films are sheets of materials with specific thicknesses whose atomic or molecular scale structures are controlled in the production process applied on any surface [1]. They are usually formed by depositing a layer of material on a substrate or on a specific surface, or by coating a surface. Thin films can be composed of atoms or molecules of different structures as well as totally different structures [2], [3]. For example, thin films can be produced from certain metals, organic materials or composite structures made of a mixture of these materials [2], [3]. Thin film characterization

techniques provide a complete understanding of the different properties of materials such as physical, chemical, mechanical, tribological, electronic, magnetic, etc. By processing the data obtained, it makes designing more efficient and advanced materials possible. In addition, optimizing, reducing, or increasing the specific properties of these materials is an important step in producing products that meet the needs of the industry. Advances in these areas contribute to the development of more efficient and effective solutions in many different fields such as semiconductor devices, solar cells, sensors, imaging

technologies, military technologies, electronic technologies, telecommunication technologies and biomedical applications [4]-[7].

Various structural characterization techniques are used to analyze the structure, atomic arrangement, surface morphology, constituents, and crystallographic structure of thin films. Among these characterization techniques, those that study and characterize the surface structure and morphology are of great importance. The surface is the most important part of a thin film. Surface properties can affect the physical, mechanical, optical, magnetic, electronic and tribological behavior of the thin films [8]-[10].

Successful characterization of the surface morphology of thin films is critical to understanding and optimizing these properties. Techniques such as electron microscopy, probe microscopy and diffraction techniques are used to study crystal properties and morphology. It is also important to measure mechanical and tribological properties such as surface porosity, hardness, etc. In this review we discuss microscopic techniques such as scanning electron microscopy, atomic force microscopy, tunneling electron microscopy and diffractive techniques such as X-ray diffraction and porosity measurement techniques used to characterize surface morphology. These techniques are the main techniques used to determine both the changes that occur at different stages of the thin film production process and the film properties. These techniques can be used to monitor, control and optimize the thin film production process.

2. Sample Preparation

Sample preparation is an important step in thin film characterization studies and, when performed correctly and efficiently, ensures the accuracy and reliability of measurement results. In fact, for many measurements and characterizations, the correct result cannot be obtained unless the correct sample preparation protocols are followed. However, the sample preparation process can vary depending on the thin film technique used, the sample type and the measurement objectives. Care should be taken in the sample preparation process and appropriate standards and guidelines should be followed where necessary. In addition, the surfaces of the samples to be examined must first be cleaned of organic and inorganic contaminants in order to preserve the life of the equipment and instruments used in the process. Ultrasonic cleaning or chemical cleaning methods are usually used for this process. Care should be taken with the chemicals used in the

decontamination process. If voluntary abrasion is not desired, care should be taken to ensure that the chemicals used do not have an abrasive effect on the surface. This can be detrimental to the formation of the surface and the surface properties of the material. Basically, the techniques that can be followed are listed below:

2.1. Selection of Sample

All characteristics of the material to be analyzed, such as microstructure, internal deformations, porosity, coating and coating thickness, heat treatment status, etc. must be available.

The number of samples to be selected should be of sufficient quality to provide information about investigated material without unnecessarily increasing the cost. In the selection of samples, care should be taken not to select from areas where there are visible deformations of the thin films.

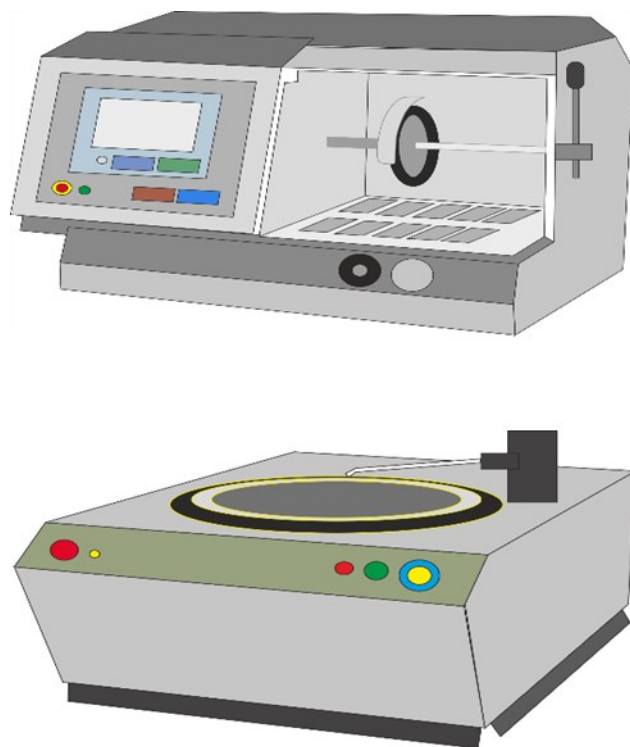


Figure 1: Images above showing the disks used for cutting (top) and the sanding machine used for sanding (bottom).

2.2. Cutting and Sanding

Many characterization techniques require samples to be of the appropriate size and shape. Therefore, specimens need to be cut and trimmed to the required dimensions using automated cutting tools wherever possible without damage. Damage to the surface during cutting can be removed during the grinding and polishing stage. The sanding process is carried out gradually from large to small grit sandpaper. Depending on the nature of the specimen and the type of characterization required, cooling is performed using water or appropriate chemicals to prevent the material temperature from rising too high during the cutting and grinding process. Grinding is rarely preferred in scanning tunneling microscopy and is not preferred in atomic force microscopy applications. This is because in these applications the aim is generally to get an idea of the morphological properties of the surface, such as depressions and protrusions. The grinding process can alter the certain properties of thin films. Therefore, attention should be paid to such details during sample preparation. Sectioning can be done by trimming, sawing, cutting with an abrasive disk.

This process is often used in techniques such as optical microscopy, scanning electron microscopy (SEM)

2.3. Cleaning

The surfaces of the samples must be properly cleaned before analysis. Physical cleaning removes dust, oil and residues. Chemical cleaning removes surface contamination with chemicals such as alcohol, acids, etc. Ultrasonic cleaning or plasma surface cleaning are also very effective methods. Surface cleaning is important in techniques such as X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). Care should be taken to avoid cavitation during the cleaning of soft materials. It should be ensured that the chemicals used during cleaning do not cause corrosion or deformation, especially in applications where surface topography is examined.

2.4. Thinning & Polishing

It is the final stage of the processes to obtain a smooth and even surface. In this stage, the samples are thinned and polished to obtain a smooth surface. Thinning can be performed by mechanical or chemical methods (Al_2O_3). It is carried out in 3 stages: coarse polishing to $3\mu\text{m}$, fine polishing to $1\mu\text{m}$ and final polishing to 50 nm . It is particularly

applicable in applications where the composition of the material is studied, or different layers of multilayer films are examined.

2.5. Sample Assembly

Some characterization techniques require the sample to be mounted in a specific way. For example, for scanning probe microscopy (SPM), the sample is mounted on a special carrier and then analyzed under the microscope. Similarly, different imaging and characterization applications may involve sample holders with different features and forms, and different mounting techniques can be sustained for these processes.

2.6. Sample Protection

Some characterization techniques require the sample to be protected from environmental factors. Therefore, samples must be protected by methods such as appropriate coating, storage under vacuum or storage in a gas atmosphere. This ensures that the material properties remain unchanged.

Sample preparation techniques are critical to the reliability and reproducibility of the characterization process. In addition, the same sample can be used for different characterization processes. In order to successfully carry out two or more different successive characterization techniques, the preservation of the samples needs to be taken into account. Correct and properly prepared samples help characterization techniques to produce accurate results.

3. Surface and Morphological Characterization Techniques

Surface and morphological characterization techniques are important research tools used in fields such as materials science and nanotechnology. These techniques enable us to understand the performance, reactivity and functionality of materials by studying the structure, morphology, topography and properties of material surfaces. Surface and morphological characterization techniques include a variety of microscopic and analytical methods and play a critical role in materials research.

3.1. Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is a characterization technique used to obtain high-resolution images of sample surfaces to study surface morphology and properties. Under vacuum, an electron gun can produce electrons in a variety

of ways. In primitive electron microscopes, cathode ray tubes were used as electron sources, but with the development of technology, Schottky diode electron guns with high efficiency and low dispersion angle have been produced. The purpose of these guns is to produce the electrons used in electron microscopy. The electrons from the electron gun pass through magnetic lenses, which allow the electrons to be accelerated and the electron beams to be properly directed. The accelerated electrons have a shorter wavelength and higher discrimination power. The magnetic lenses bend and narrow the path of the electron beam, focusing it on the sample. The electrons hit the sample quickly. Scanning electron microscopy also works on the principle that when the accelerated electron beam is focused on the sample, electrons scattered or reflected from the sample surface are collected by the detectors.

Some electrons are scattered back as a result of elastic collisions between the incoming (primary) electrons and the nuclei in the sample. Secondary electrons are scattered from the sample surface as a result of inelastic collisions between incoming (primary) electrons and sample electrons. And these are low energy electrons.

Scanning coils direct the electron beam to scan the sample in a raster pattern (X-Y scan). This is the process of scanning over a grid that represents the sample surface in pixels or dots. Flexible collisions between incoming (primary) electrons and atomic nuclei in the sample cause some electrons to be scattered back. These are very important for image

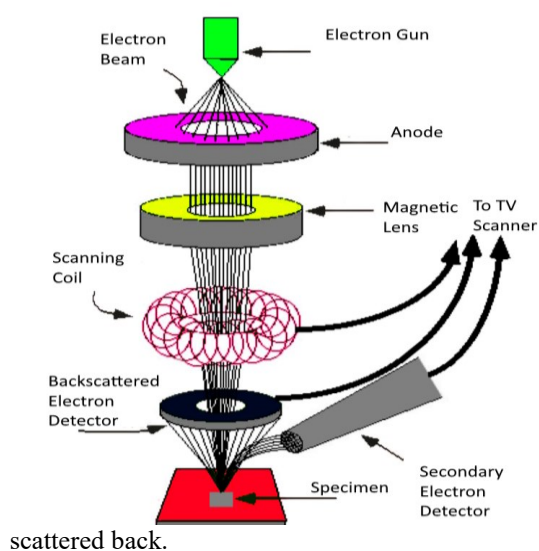


Figure 2: Image showing the basic components of scanning electron microscopy (SEM).formation (also called backscattered electrons).

Inelastic collisions between incoming (primary) electrons and sample electrons result in secondary electrons being scattered from the sample surface. These are low energy electrons. The backscattered electrons and secondary electrons are collected by two separate detectors and an image is formed. The images from the backscattered electron detector or the secondary electron detector can be used separately or combined to observe different properties of the surface under investigation.

Because the electron beam in EM can be focused down to the nm range using magnetic lenses, resolution is high and surface morphology can be observed in detail. The large area scanning capability also allows surface topography to be analyzed over a large area. As the electrons also penetrate the interior of the sample, it provides cross-sectional or sectional images of the sample for depth profiling. In addition, it has disadvantages such as needing for a vacuum environment, high maintenance requirements and the cost of the equipment. In addition, materials must have conductive properties to be visualized in the SEM and to obtain high resolution. Special sample preparation techniques are used to visualize nonconducting materials.

SEMs have a wide range of applications. It is used in many fields such as materials science, nanotechnology, semiconductors, metallurgy, biomedical research, archaeology and materials engineering. The SEM is used to characterize the surface morphology, particle size, layer thickness, surface defects, film coatings, microstructure and composition of samples.

3.2. Atomic Force Microscopy

Atomic force microscopy (AFM) was developed after scanning tunneling microscopy (STM). Because AFM uses force as an interaction, it can examine all types of samples, from conductors to insulators, but it cannot produce atomic images with the same precision as STM.

The AFM consists of a flexible lever, usually made of silicon or silicon nitride (Si_3N_4), and a sharpened thin tip that scans the surface. The scanning process is performed by the piezoelectric mechanism on demand, using the ability of piezoelectric materials to move mechanically with electrical impulses. Electrical voltages applied to the mechanism cause

the cantilever to make small and precise movements.

The operation of AFM is based on the principle of placing the tip very close to the surface of the sample and measuring the interactions with the surface. Depending on the type of measurement, surface characterization is carried out under the influence of electronic, magnetic, van der Waals or frictional forces; the tip and surface interact through these effects in the characterization process.

When the distance between the tip and the surface is large (10-100 Å) the surface attracts the tip, when the distance is equal to the bond length (1-2 Å) the push and pull forces are balanced, at smaller distances the push force increases. As the lever approaches the sample surface it is stretched by the atomic forces and returns, similar to the behavior of a spring-like system. This vibrating or bending motion is detected by a photodiode and a three-dimensional image of the surface topography is obtained. Depending on the scanning pattern, AFM can operate in 3 different modes.

How does AFM work?

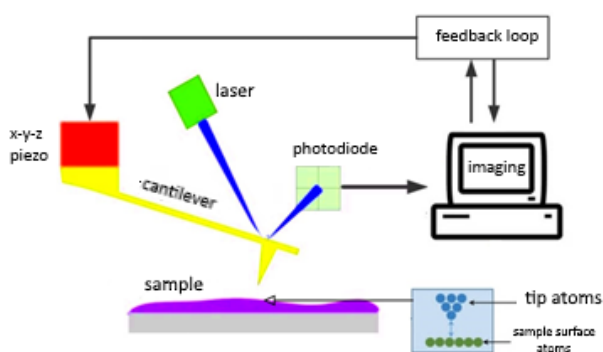


Figure 3: An illustration showing the working principle of an atomic force microscope.

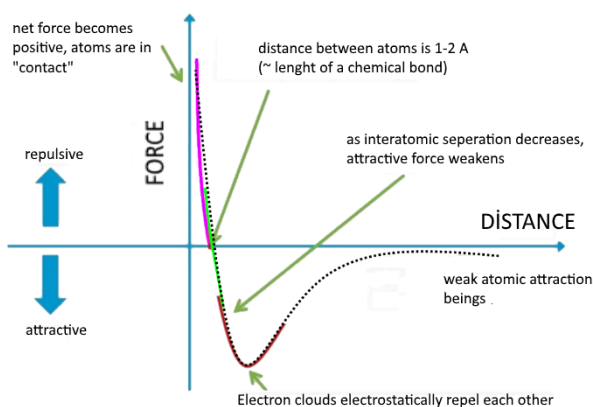


Figure 4: A graph showing the force acting on the AFM tip and the types of force according to the distance between the AFM tip and the surface.

The appropriate AFM modes, such as contact mode, non-contact mode and tapping mode (semi-contact mode or hybrid mode), determine the tip-to-surface distance. These modes are used to measure and characterize the topography and physical properties (roughness, etc.) of the sample surface in different ways. Each mode may be suitable for different samples and different types of characterization. For example, the non-contact mode may be preferable when examining soft surfaces or when no damage is to be left on the surface. Similarly, if the mechanical or tribological properties of the surface are to be investigated, the contact mode should be preferred. In contact mode the tribological properties of the surface can be characterized as direct contact is made with the surface.

In contact mode, under the influence of the driving force, the tip is in contact with the surface. In non-contact mode, the tip vibrates without touching the surface, the distance between tip and surface is maximal, only the interaction forces are measured. In tapping (semi-contact) mode, the lever tip touches the surface slightly at the bottom of the vibration and moves away again. This is the most preferred mode of operation. Each mode has different advantages and uses. It is selected according to the characteristics of the sample.

AFM is characterized by its high resolution, high sensitivity, damage-free and real-time imaging capabilities. In addition, AFM can map the surface in 3D, as close to reality as possible. It is for this reason that AFM is used for the determination of surface topography, surface roughness, particle size, film thickness, molecular organization and interactions.

AFM has a wide range of applications. It is used in many fields such as materials science, nanotechnology, semiconductors, biology, polymers, surface coatings and nanomaterials. In addition, AFM can also be used to characterize mechanical properties (hardness, elasticity, etc.) as well as electrostatic, magnetic and chemical interactions.

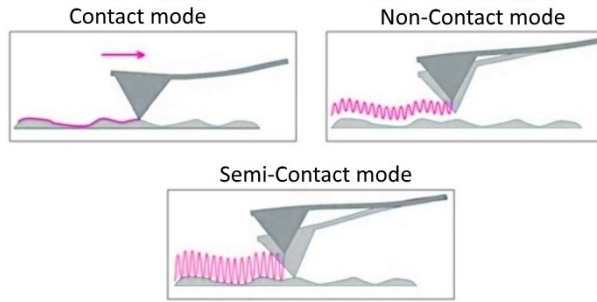


Figure 5: The modes used in the AFM characterization process.

3.3 Scanning Tunneling Microscope (STM)

The Scanning Tunneling Microscope (STM) enables imaging and inspection at the nanometer scale. It has a resolution of about 2-10 angstroms. It can show individual atoms. Conductive materials are required for its operation. Changes in conductive materials can be imaged. A very sharp and pointed metal tip scans and analyses the surface of the sample at the atomic level.

STM works on several principles, one of which is the "tunneling effect" based on quantum mechanics, which allows us to see the surface. If the distance between two points separated by energy barriers is small enough, the wave-like electron can travel from one end to the other by tunneling. In STM, when the atomic-scale tip is brought very close to the sample surface (up to 1 nm), electrons create a tunnel current between the tip and the surface. The tip is made of a conductive material such as tungsten or platinum-iridium. For the current to flow, the sample must also be conductive. The intensity of the current decreases exponentially with the distance between the tip and the sample surface.

Another principle is the 'piezoelectric effect'. Piezoelectric materials can be made to move mechanically by electrical impulses. The typical piezoelectric material is PZT (lead zirconium titanate). The spike is placed on top of a piezoelectric tube, which controls it at the angstrom level, allowing it to scan the surface with great precision.

The final principle is the 'feedback loop'. By continuously monitoring the tunnel current, it co-ordinates the current and the position of the spike.

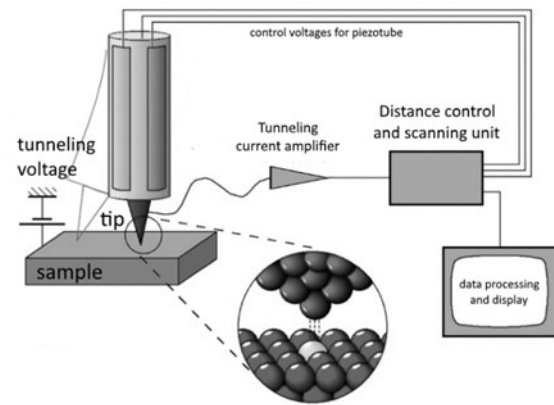


Figure 6: An illustration showing the working mechanism of the STM.

Two different modes are used to create a 3D image of the topography. In constant current mode, the feedback loop makes adjustments at the tip to maintain a constant tunneling current. In this mode, voltage and/or position adjustments are made. These adjustments are processed by the computer and displayed as an image in the software. The constant height mode is preferred for very flat surfaces. The feedback loop is closed and the image is generated based on the measured current as the tip is moved across the surface.

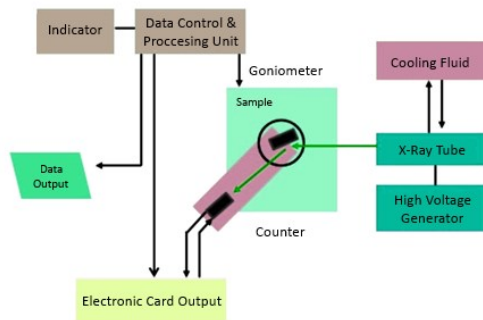
Thanks to the ability of the STM to manipulate atoms at the atomic level, individual atoms or molecules can be manipulated and patterns can be created at the atomic level. This is of great importance in fields such as nanotechnology and the production of nanomaterials. The applications of STM are quite broad. It is used in a wide range of fields such as materials science, nanotechnology, semiconductors, catalysis, surface chemistry, molecular physics and biology. STM is an important research tool that is used to study a wide range of surface properties such as the arrangement of surface atoms, surface roughness, crystal structures, surface reactivity, molecular arrangements and nanomaterials.

3.4. X-ray Diffraction

The structure of materials can be studied at the atomic level using high-resolution electron microscopy. However, X-ray diffraction (XRD) is often used to identify different structures and determine the properties of the crystal structure. This is because the distance between atoms in crystalline material is about 0.15-0.4 nm, which is the wavelength of X-rays. In addition, X-ray scattering does not damage the sample. Electron

scattering is often used for thin films. However, XRD is a more suitable technique when precise measurements of crystal lattice parameters are required. When electrons are scattered, the X-rays follow a straighter path and penetrate much deeper into the material. This enables elemental analysis not only of thin films but also of bulk materials.

X-ray diffraction (XRD) is based on the principle that X-rays are bent as waves when they pass



through a crystal or a thin film of material. The resulting diffraction patterns provide information on the interaction and distribution of light with atoms in the crystal. X-rays are generated in a cathode ray tube, filtered to be monochromatic, concentrated, and directed at the material. At small angles of incidence, X-rays are totally reflected from the surface of the crystal and scattered by the parallel planes of atoms in the crystal, a phenomenon known as diffraction. The diffraction pattern appears as bright spots or rings that form patterns due to the wave character of X-rays. These patterns are measured by a detector and used for XRD analysis.

Figure 7: An illustration showing the structure of the X-ray diffraction device.

This analysis is based on Bragg's law. According to Bragg's law, the structure is intensely reflected by X-rays at a certain angle. The intense reflection points are realized by parallel arrangement of atoms in the structure. The incident rays must be parallel to each other, and the scattered rays must be parallel to each other. The law describes the condition of constructive interference of scattered rays from lattice points. If X-rays with wavelength " λ " scattered from crystal planes parallel to each other and with a distance " d " between them form an angle " θ ", the distance between the planes (d) must be zero or equal to one full wavelength (λ) to obtain constructive interference between the X-rays and to ensure intense reflection.

$$n \cdot \lambda = 2 \cdot d \cdot \sin \theta$$

n : diffraction constant

λ : wavelength

d : cage spacing

θ : refraction angle

From this perspective, constructive and destructive interferences may occur depending on the crystal structure of each material. These interferences can be detected by the detector at certain angles during the analysis of the material. The interference patterns observed at specific angles are used to estimate the crystalline properties of the material.

XRD has a wide range of applications. It is used in many fields, including materials science, mineralogy, crystallography, metallurgy, semiconductors, polymers, and chemistry. XRD is used to determine the crystal structure of a sample, analyze crystal orientations, determine phase composition, assess crystal quality, examine grain size and crystal structural deformations.

3.5. Surface Area and Porosity Measurement

Porosity is the proportion of pores in a material. Surface area and porosity measurements are analytical methods that characterize materials. These techniques are used to evaluate the properties of the surface of the material, the porous structures that it contains and the degree of porosity. The material's surface area is determined by its porous structures and the types of porosity present, such as the micropores (smaller than 2 nm), mesopores (between 2 nm and 50 nm) and macropores (larger than 50 nm).

Surface Area Measurement is a technique used to determine the effective surface area of a material's surface. This includes both the actual surface area and the increased surface area due to the distribution of its internal structure. The porosity of a material directly affects its effective surface area. Gas adsorption methods, such as the Brunauer-Emmett-Teller (BET) method, are commonly used to measure surface area. The surface area is calculated using the BET method, which involves adsorption of a large number of molecules on the material's surface. The gas penetrates the voids in the material and is absorbed under a certain pressure. The Boyle-Mariotte law describes the pressure-volume relationship that underlies the gas absorption method. Micropores begin to fill first at

low pressures, with the first layer filling at low pressures. The completion of the first layer is indicated by the knee on the graph.

After completion of the first layer, the rate of layer formation is slower. High pressure causes the filling of mesopores. The gas used in the investigation was used to determine the size of the pores. Nitrogen gas (N_2) provides information on pores between 3.5-4 nm, while carbon dioxide gas (CO_2) provides information on micropores. The density of the gas increases upon adsorption on the material, ensuring a homogeneous distribution of gas molecules over the surface area. The BET method analyzes the adsorption isotherm, providing information about porosity and explaining the multilayer adsorption process.

Surface area and porosity measurement are essential research tools for understanding the structural properties and performance of materials. These techniques are commonly used in characterization of nanoporous materials, evaluating catalyst effectiveness, controlling surface coating quality, biomedical applications, and determining gas storage capacity. Surface area and porosity measurements are of critical importance in the design of materials, quality control, performance improvement, and applied research.

4. Conclusion

Surface morphology is crucial in determining properties of thin films and nanomaterials. Our study focuses on basic characterizing techniques, including STM, AFM, SEM and XRD, which are commonly used to characterize surface morphology.

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