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A Comprehensive Study of Development and Characterization of Thin Film Materials

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ABSTRACT

The material's efficiency and behavior in service can be influenced by its surface characteristics. A separate area of life has made extensive use of the possibility of modifying and altering these surface qualities to fulfill the unique requirement for improved performance. Applying a thin layer coating on the surface is one way to do this. Focusing on their structural formation, growth methods, and functional qualities, this study aims to design and characterize thin film materials. Thin film shape, composition, and behavior are investigated, along with the effects of process parameters, in this investigation of several deposition processes. The nucleation and growth processes, which impact the films' stability and performance, are given special attention. The films' thickness, surface structure, crystallinity, and elemental composition are examined using a variety of characterisation techniques, which allow for a thorough comprehension of their characteristics.

Keywords: *Thin film, Nucleation, Semiconductor, Photovoltaic, Coating.*

I. INTRODUCTION

"Thin films" refer to film layers that are deposited onto substrates and have thicknesses that vary between nanometers and a few micrometers. A thin film's thickness might be anywhere from a few micrometers to a nanometer. Modifying material surfaces or functional devices is made possible by thin films. Mechanical, optical, electrical, and thermal characteristics are significantly affected by the structure of a thin layer when examined at the nanoscale or micrometer scale. One defining feature of thin-film materials is the increasing number of technological applications for them. Materials science and many other fields of study, both theoretical and practical, have a better grasp of thin films. From a materials science perspective, thin films not only exhibit matter-like characteristics but also foretell how preparation methods will interact with material design. Numerous energy and electronics applications using thin-film materials have been investigated. These include electrical semiconductor devices, light-emitting diodes, optical coatings, thin-film solar cells, and thin-film batteries. Due to the material basis for both structure and processes, the elemental makeup of these films takes on added significance. The structure of a thin film is significantly impacted by the preparation procedure and the film's intrinsic growth elements. The thickness is the defining characteristic that differentiates thinner films from thicker materials. Alterations to the nano-to microscale range of thin-film characteristics may be brought about by seemingly little alterations to the growing process.

There are two main categories of thin films: organic and inorganic. It is possible to create thin films with desired characteristics by manipulating the growing process and the structure of the films. Vacuum technology is essential for the development of thin films, and many vacuum coating techniques have been developed to improve growth dynamics. The comparatively cheap manufacturing costs of low-temperature methods, the considerable application flexibility of these films, and the low cost of their ingredients, which may be changed during processing, are contributing to their rising popularity.

Significant analytical methods have been used to characterize thin films, with an emphasis on their role in promoting growth. It is necessary to identify the different properties of thin-film materials in order to link the characteristics of thin films to certain growth circumstances. Determining the size and composition of the material using these methods requires an in-depth familiarity with the thin-film data. Fundamentally important is the relationship between parameters and thin-film properties. It is worth mentioning that in this context, the features of a thin film are usually interpreted as the material's reaction to certain outside influences.

II. REVIEW OF LITERATURE

Toma, Fatema Tuz Zohora et al., (2021) By varying the processing settings and Cd/S ratio of the original precursors, the structural properties of CBD-produced CdS thin films have been studied in order to gain a better grasp of the growth conditions. An ammonium hydroxide and thiourea bath was utilized to fabricate a CdS thin film on a glass substrate using the CBD method. We employed X-ray diffraction (XRD) to examine the structure. The deposited CdS thin film was a cubic phase with small nano crystalline grains. Up to 60 degrees Celsius was applied to the film for a duration of two hours. Sintering the film at 300°C for one hour resulted in a dark yellowish tint and attained the desired thickness. At room temperature, the FTIR analysis, which encompassed the range of 350 cm-1 to 4500 cm-1, identified many functional groups in the material and provided clues as to where they likely originated. These investigations have established a baseline for the controlled laboratory fabrication of uniform, continuous, ultrathin CdS films. Due to its low cost, vast surface area, and ease of usage, this preparation method is ideal for producing thin films with exceptional efficiency.

Bedreddine, Maaoui et al., (2020) The structural, optical, and electrical characteristics were examined at various NiO concentrations (0.05, 0.10, and 0.15 mol.l−1) after nickel oxide was sprayed over a glass substrate in this study. The deposited film achieved a minimum crystallite size of 11.97 nm at 0.1 mol.l−1, and all sprayed films exhibited a cubic structure with a strong (111) preferred orientation. But at a concentration of 0.15 mol.l−1, α-Ni(OH)2 was detected. The band gap energy of the NiO thin films varied from 3.54 to 376 eV as a function of NiO concentration, and films made with 0.05 mol.l−1 NiO exhibited less disorder and fewer flaws, while having high transparency in the visible range. The NiO layer had an electrical conductivity of 0.169 (Ω.cm)−1 after being deposited at a concentration of 0.15 mol.l−1.

Mao, Samuel. (2013) The typical time it takes for a newly developed material to get from the lab to the market is over ten years. Thus, in order to address problems in fields as diverse as renewable energy and national security, it is essential to speed up the process of finding new materials. There is an urgent need to develop and implement high throughput screening and discovery methods to characterize and grow novel materials, as their discovery has lagged behind product design cycles in many industries. Several high-throughput characterisation methods developed in the author's lab are detailed in this paper, in addition to two unique forms of high-throughput thin film material development methodologies. Some of these methods include a "continuous" high throughput thin film material screening system that allows for the realization of ternary alloy libraries with continuously varying elemental ratios and a second-generation "discrete" combinatorial semiconductor discovery system that allows for the creation of arrays of individually separated thin film semiconductor materials of different compositions.

şen, Sevde et al., (2011) Thick phthalimide films are made utilizing the spin coating process with new materials such as p-phthalimidobenzoic acid (FIBA) and N-(phthalimido)-p-aminobenzoic acid (FIABA). The thin film deposition process of these novel materials is monitored using a combination of spin speeds and solution concentrations. Spectroscopic ellipsometry and ultraviolet-visible spectroscopy are used to examine the optical characteristics. The FIBA and FIABA films exhibited exponential behavior in their absorption as a function of spin speed. There was a shift from π to π *. The spectroscopic ellipsometry findings show that the FIABA thin film has a thickness of 12.99 nm and the FIBA thin film has a thickness of 15.86 nm at 2000 rpm.

Song, Junlong et al., (2009) The polymers frequently used to make synthetic fibers—cellulose, polypropylene, polyethylene, nylon, and polyester—were spin cast onto silica and gold wafers to create thin films. To observe the effects of a polymer solution on adsorption and friction after treatment (to imitate a textile finish), the thin films served as substrates for the quartz crystal microbalance and nano-indentation methods. We tuned the spin coating conditions based on the surface energy, layer thickness, and shape that we wanted. The films' physical and surface characteristics were investigated using atomic force microscopy, X-ray photoelectron spectrometry, ellipsometry, and contact angle. In sum, our thin films aid molecular level investigation into phenomena pertinent to textile and fiber production, such as swelling, degradation, and surfactant and polymer adsorption.

Pelletier, H. et al., (2006) Film on substrate composites mechanical behavior during stiff tip penetration is investigated in this work utilizing finite element modeling (FEM). By comparing the strain gradient throughout the thickness of a layer formed on a harder substrate after being laid on a softer one, we may get a sense of the substrate effect's size. So long as the indentation depth (h) and film thickness (t) do not exceed a crucial ratio (h/t) of 0.35, the layer's mechanical behavior remains consistent. For $h/t > 0.35$, two distinct behaviors can be seen: (i) in the first case, where the film and substrate hardness values are Hf and HS, respectively, the total strain stays within the film thickness until the ratio h/t approaches 1, and (ii) in the second case, where the film and substrate hardness values are $Hf/HS \geqslant 1$, the total strain goes deep into the substrate. Based on these findings, it is clear that the empirical 10% rule does not apply, regardless of whether the film is hard or soft. The primary mistake stems from an inaccurate assessment of the indenter tip's contact depth with the film surface. It is true that the simulation runs show that pile-up can arise based on the ratios (h/t) and Yf/YS (where Yf and YS are the values of the film and substrate yield stresses, respectively). From the load-displacement curves that are derived by FEM, at least three different hardness variations may be observed, which are dependent on the model used to calculate the contact depth. Determining a weighting method to extract significant mechanical features of the thin film appears problematic under these conditions. Utilizing the loading phase is an additional approach to ascertain film characteristics. During loading, we look at how the indentation depth (h) relates to the applied load (P). Based on the preceding equation, we may calculate the film's yield stress when the film is soft on a tougher substrate ($Hf/HS \leqslant 1$). Amorphous Al2O3 films created experimentally by electron beam evaporation on a silicon substrate are treated using this method.

III. THIN FILM

Layers of thin materials, ranging in thickness from a few nanometers to one micron (10^{-6} meters), can be called a thin film. The thickness of the layers formed is the primary differentiator between thin film and thick coating depositions. While particles are dealt with in thick coating, atoms or molecules are deposited on a surface in thin film deposition. Materials may have their surface shape and physical and chemical characteristics changed with this method, all without affecting the material's bulk qualities. The desired qualities and use case dictate the type of thin film that may be created, which can be a single layer with a homogenous composition and microstructure, an inhomogeneous multilayer, or a composite. There are a few possible structures for the multilayer: periodic, pattern, or completely random. The four or five main sequential phases are common to nearly all methods for depositing thin films. The procedures that are specific to the thin film's characteristics as a whole are detailed below:

- It is decided where the pure substance will be placed. During deposition, this material source will serve as a target.
- A medium is used to transfer the target to the prepared substrate. The materials and deposition process dictate whether this medium is a fluid or a vacuum.

- A thin layer of the target is formed on the surface of the substrate by depositing it onto it.
- If annealing or other heat treatments are required to provide the required film characteristics, the thin film may undergo them.
- The characteristics of the film are examined. It is possible to adjust the deposition procedure by incorporating the analysis results if needed.

Thin Film Growth and Nucleation

The film's thickness and the deposition procedures used to deposit it can have a significant impact on the film's properties, which are in turn influenced by the substrate's underlying characteristics. The process of growing and nucleating thin films entails three main phases.

The process begins with the preparation of the deposition species, which include the substrate and the target material. Then, any deposition technique is used to transfer the target to the substrate. Finally, the target is grown on the substrate to create the thin film. The incoming atoms from the target can either condense on the surface of the substrate or instantly reflect off of it, returning to the gas phase after a specific amount of time has passed. The activation energy, substrate-target binding energy, adhesion coefficient, and many other parameters influence this process. The sticking coefficient is the ratio of the number of atoms that are either condensing or impinging on an object. Because of the energy loss, the atoms may not react with the substrate right away. "Adatom" is a portmanteau word meaning "absorbed atoms," and it describes the mobile atoms that will be present on the surface prior to condensation. During condensation, energy can be lost in one of many ways: by a chemical interaction with the atoms in the substrate, by colliding with the atoms that are diffusing on the surface, by discovering a favorable nucleation site, or by colliding with the species that have been absorbed off the surface. Each atom can act as a nucleation site for growth if the surface mobility is low and the adatom-surface interaction is strong; however, when the adatom-surface interaction is feeble, the condensing adatom's surface mobility is high, leading to condensation at preferential nucleation sites with stronger bonding. This could be because of an increase in the coordination number or a change in electronic or elemental chemistry.

Atoms on the surface react with the condensing atoms and establish chemical bonds between them. In chemical bonding, the polarization of atoms can cause electrostatic attraction (van der Waals forces), atoms can share an orbital electron in metallic bonding (homopolar), or ions can be formed by electron loss or gain in electrostatic (coulombic heteropolar) bonding. Strong connection between the surface atom and the condensed atom as a result of the reaction indicates that the atom has undergone chemisorption. This adatom became a nucleus when it was surrounded by other adatoms, creating a continuous thin film. Deposition inherently produces thermodynamically unstable initial-stage nuclei, which, depending on the deposition settings, may desorb over time. We say that the nucleation problem is solved when the clustered nuclei get to a threshold safe size and become thermodynamically stable. A stable, critical-sized nucleus is created during the nucleation step, which entails going from a thermodynamically unstable state to a thermodynamically stable one. Until a saturation nucleation density is reached, the quantity and size of the stable critical nuclei increase throughout the deposition process.

A multitude of deposition parameters, including substrate temperature, working pressure, adhesion properties, binding energy between target and substrate, energy of impinging species, activation energies of adsorption, desorption, thermal diffusion, rate of impingement, topography, and chemical nature of the substrate, determine the nucleation density and average nucleus quantity. It is possible for a single deposition to undergo both the parallel and perpendicular growth modes of a nucleus, which are termed after the relative orientations of the substrate and target. The adsorbed atoms diffuse over the substrate's surface, causing parallel growth, whereas incoming species impact directly, causing perpendicular growth. In contrast to perpendicular growth, the rate of parallel development, also known as lateral growth, is substantially higher at this stage, and the developed nuclei are referred to as islands. The coalescence stage involves the gradual reduction of the substrate surface area and the subsequent formation of a thin film covering as tiny islands begin to coalesce with one another on the substrate's surface. This phenomenon, known as agglomeration, led to the creation of a larger island. Raising the substrate's surface temperature, which in turn increases the adatoms' surface mobility, improves the nucleation site's growth density and the agglomeration process. When new nuclei are formed in certain processes, they may also incorporate regions that were previously covered up due to coalescence. The growth of discontinuous film on the surface of the substrate results in a porous defect when larger islands grow in proximity to one another, leaving channels and holes of the exposed substrate between the islands. Agglomeration and the ongoing filling of the formed channels and holes are caused by the continued expansion of the bigger islands.

IV. APPLICATIONS OF THIN FILMS

Modern civilization owes a debt of gratitude to the material science and engineering community for their comprehension of new materials with highly variable physical, chemical, and mechanical characteristics. There has been a recent upsurge in interest from researchers studying two-dimensional solids and the optical characteristics of metallic films in the study of thin films. Here are some of the most common business applications for thin films:

1) Semiconductor

Thin film materials that make up electrical circuits for computers and other electronic devices are grown and deposited on flat, two-dimensional chips that the semiconductor industry has used as substrates. Flexible materials and electronics can be coated with this thin layer. While transferring the semiconductor film to the new substrate, it is possible to invert it so that additional components may be accessed from the other side. Because of this, the film has a greater capacity to incorporate more gadgets. Powerful, eco-friendly, three-dimensional electronics are possible by bonding layers of double-sided, thin-film semiconductors. Strapped silicon or silicon-germanium membranes in the form of single crystal sheets. Strain causes a change in the crystal's atomic arrangement, which in turn increases device speed while decreasing power consumption. Solar cells, smart cards, RFID tags, medical applications, and active-matrix flat panel displays are some of the non-computer applications that could benefit from this development. Wearable electronics or retractable computer screens similar to window shades would be possible with this technology, which would allow the integration of flexible semiconductors into fabric. The fact that silicon-germanium membranes absorb more light than silicon membranes makes them very interesting. Two to three orders of magnitude improvements in sensitivity can be achieved by using germanium without sacrificing material quality. Better low-light cameras or smaller, higher-resolution cameras could be made possible by harnessing this increased sensitivity.

2) Photovoltaic Cells

Rigid solar panels use cells made of two thin layers of crystalline silicon to convert the energy from incoming photons into electricity. Amorphous silicon, rather than crystalline silicon, is used in high-solids slurries that may be applied to substrates using web-converting processes such slot die coating. This makes roll-to-roll manufacturing of flexible film solar products possible. The gas molecules have a far more difficult time passing through the film due to the many layer-to-layer interactions, which greatly improves its barrier properties. Photovoltaic applications rely on barrier layers to prevent performance deterioration caused by oxygen or moisture vapor infiltration, hence this is very necessary. Grid parity, the point at which the cost of electricity generated by a rooftop photovoltaic system is equal to that purchased from an electrical utility, is considered by many in the solar power industry and the investment community to be a watershed moment that will cause the market to accelerate its growth significantly. Rooftop photovoltaic systems have high upfront costs and a long return on investment, thus it's unlikely that solar system installations would spike suddenly, even when true grid parity arrives.

3) Photo Electrochemical Cells

A photocurrent is generated in photoelectrochemical experiments by exposing an electrode to light, which is then absorbed by the electrode material. Features of the photoprocess, including its kinetics and energetics, are explained by the relationship between photocurrent, wavelength, electrode voltage, and solution composition. Another possible source of photocurrents at electrodes is photolysis taking place in the solution immediately around the surface of the electrode. The characteristics of the electrode-solution interface can be better understood by photoelectrochemical investigations.

4) Super Capacitor

The Slander Oil Company of Cleveland, Ohio, proved in the 1960s that they understood how to store charges in the electric double layer at the solid-electrolyte interface. Back in 1982, the top research institution in the country created the first high-power double-layer capacitor specifically for military use. Capacitance, C = €A/d, is the electrostatic energy storage capacity of a standard capacitor, where €is the dielectric constant, d is the dielectric thickness, and A is the surface area. Two charge layers, spaced apart by a few angstroms, are formed in a double-layer capacitor when charges accumulate at the electrode-electrolyte interface. Supercapacitors are now known as devices that store electrochemical energy.

5) Optical Coatings

Applying a thin layer of material to an optical component, such a lens or mirror, changes the way light is reflected or transmitted. This process is called an optical coating. Optical coatings, such as antireflection coatings, are often used on lenses for photography and eyeglasses to reduce unwanted reflections. To make mirrors that reflect more than 99.99% of the light that hits them, another option is to use a high-reflector coating. Dichroic thin-film optical filters are made possible by modern optical coatings that are highly reflective at certain wavelengths and very anti-reflective at others.

6) Optoelectronic Lenses

Optoelectronic thin-film chips have radiation-emitting sites on at least some of their layers within the active zone. An optical element constructed from a section of the thin-film layer and placed downstream of the radiation-emitting region, with lateral dimensions greater than those of the radiation-emitting area. A series of epitaxially grown and partially removed layers on a growth substrate constitute a thin-film layer. This demonstrates a reduction in the substrate thickness. The entire growing substrate can also be removed from the thin-film layer. The thin-film layer could have an active region that emits electromagnetic waves. The layers that comprise the active zone can be a pn junction, a double heterostructure, a single quantum well structure, or even many quantum well structures. Within the active zone, ideally, there should be an area that emits radiation. The radiation-emitting region, for instance, is a section of the active zone. Within the specified active zone, electromagnetic radiation is produced as the optoelectronic thin-film chip operates.

7) Flat Panel Displays

Energies uses Mykrolis contamination control technology to offer many gas and liquid contamination control solutions for flat panel displays. Flat panel display manufacturing is competitive and technologically challenging worldwide. Device designers and manufacturers struggle to meet global demand for bigger screens, greater pixel resolution, and feature-rich performance. The necessity to reduce pollution in process gas, liquid, and air streams worries process engineers and designers. Entergy offers tough-condition survival tactics.

8) Data Storage

Magnetic data storage medium struggles to accommodate the growing data storage capacity of advanced microelectronic systems due to the superparamagnetic effect. This difficulty can be solved using thermomechanical data storage. An integrated transducer reads data indented on a surface by a nanoscale mechanical probe, which is heated to erase it. Data is stored on a polymer thin film in the IBM millipede. This study addresses thin film Ni-Ti shape memory alloy (SMA) as a thermomechanical data storage alternative. Previous research demonstrated that nanometer-scale indentations in thin films of martensitic phase Ni-Ti shape memory alloy recover greatly when heated. The frequency, depth, closeness, and thermomechanical cycling of indentations affect this method's feasibility. Both theory and experiment suggest that SMA thin films might store thermomechanical data despite these challenges.

9) Gas Sensors

Recent decades have seen remarkable advancements in sensor technology, leading to numerous scientific successes as a result of rigorous experimentation. However, this progress has also presented new obstacles and opportunities for the creation of smaller devices that can image and monitor diseased samples at the molecular level. The increasing need for health and environmental surveillance has redirected the scientific community's attention to macromolecules, especially for distant monitoring. Resistive metal oxide sensors make up a significant fraction of the gas detecting elements created, however gas sensors generally work on varied principles. However, for these sensing elements to work at their best, they often need to be heated. This leads to higher power use, which is inappropriate under inflationary circumstances. The remarkable electrical and mechanical properties of carbon nanotubes have recently brought them a great deal of interest. Due of their qualities, they may be used as building blocks for sensors, gas storage, field emission devices, nanoelectronics, and field emission devices. Several uses are greatly enhanced by the gas detection capabilities at ambient temperature.

V. THIN-FILM CHARACTERIZATION TECHNIQUES

Characterizing the features of sophisticated thin films is essential for comprehending their behavior and enhancing their performance across many applications. Various approaches have been devised to examine the structural, chemical, and physical aspects of these ultra-thin materials. A multifaceted approach employing many characterisation techniques is frequently necessary to achieve a thorough comprehension of thin film characteristics (see Fig. 1).

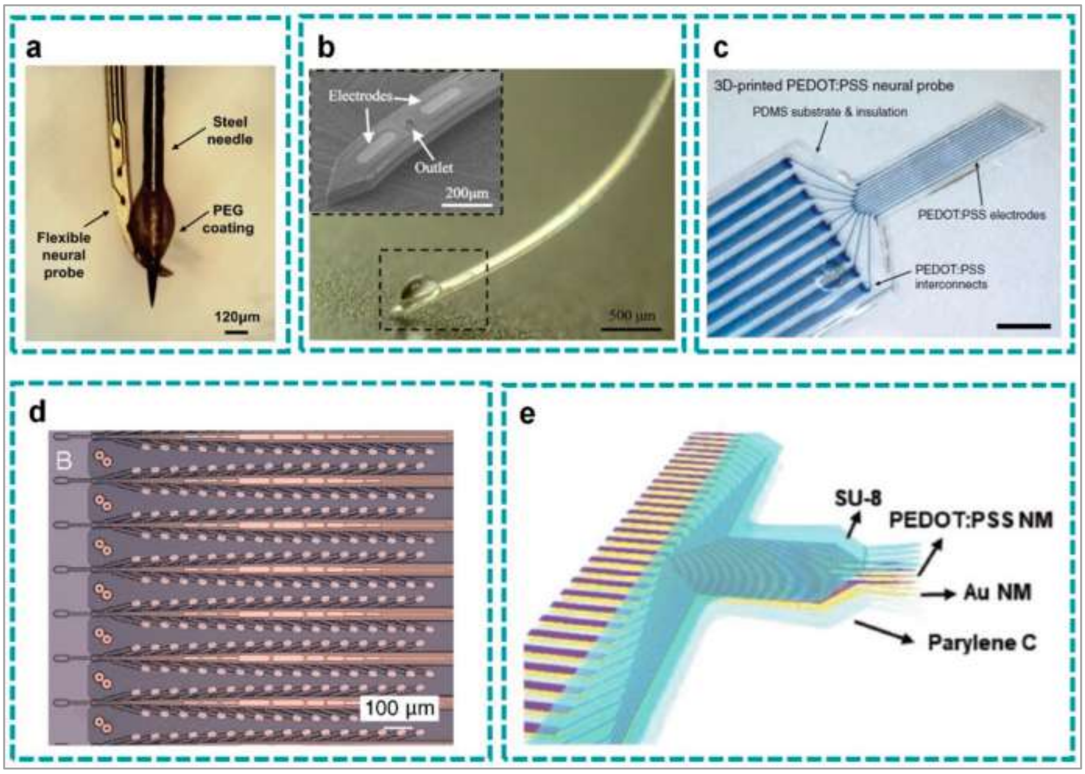

Figure 1: Thin Film Neural Probe Structures

1. X-ray Reflectometry (XRR)

Even for constructions with several layers, this non-destructive method can ascertain the density, thickness, and surface roughness of the films. X-ray reflectivity spectroscopy (XRR) is a useful tool for analyzing the vertical structure of films by measuring the intensity of X-rays reflected from their surfaces at grazing incidence angles.

2. Spectroscopic Ellipsometry (SE)

The refractive index and other dielectric characteristics of thin films may be rapidly and accurately measured using ellipsometry, a robust optical method. This technique is both non-destructive and extremely sensitive since it relies on studying the change in polarization state of light reflected off the film surface.

3. Energy-Dispersive X-ray Spectroscopy (EDS)

This method of analysis involves subjecting a sample to a focussed electron beam and then examining the resulting X-ray emissions to determine the elemental composition of the film. By using EDS, one may get numerical data on the distribution of elements in the film.

4. Quartz Crystal Microbalance (QCM)

The mass gain during film growth may be monitored in real-time using QCM, a very sensitive approach that allows for the calculation of film thickness. A quartz crystal's resonance frequency changes when material is deposited onto its surface; this is the basis of the device.

5. Scanning Electron Microscopy (SEM)

Imaging thin films in cross-section with a scanning electron microscope (SEM) allows one to see the film's shape and thickness in a visual format. This method creates high-resolution, extremely sharp pictures by scanning the surface of the material with a concentrated electron beam.

6. Time-of-Flight Elastic Recoil Detection Analysis (TOF-ERDA)

One very precise method, TOF-ERDA, can identify every constituent in a thin film and give distribution depth profiles. To do this, high-energy ions are blasted into the sample and their energy and time-of-flight are measured.

7. X-ray Diffraction (XRD)

One of the most effective methods for studying the phase and crystalline structure of thin films is XRD. To determine the atomic structure and crystallographic characteristics, it is necessary to measure the strength of X-rays that are diffracted off the film at certain angles.

8. Low-Energy Electron Diffraction (LEED)

The atomic ordering of crystalline thin films may be studied in depth using LEED, which examines the diffraction patterns of low-energy electrons scattered from the film bottom. Surface structures and epitaxial growth are two areas where it shines.

9. X-ray Photoelectron Spectroscopy (XPS)

Through the analysis of the kinetic energy and intensity of photoelectrons released from the sample when exposed to X-rays, XPS is able to ascertain the chemical bonding environment of elements in thin films. It tells you a lot about the film's chemical composition and condition.

Depending on the growth method and deposition conditions, thin films can have amorphous, polycrystalline, or epitaxial structures and morphologies. For a comprehensive understanding of the thin film's characteristics and to guarantee their best performance in different applications, it is sometimes necessary to combine many characterisation techniques in an in-depth examination.

VI. CONCLUSION

The capacity of thin film materials to greatly enhance the performance of a variety of gadgets has propelled them to a central role in contemporary science and technology. Findings from this study highlight the fact that growing circumstances, film thickness, and preparation techniques all have a significant impact on thin film characteristics, with seemingly little adjustments producing large behavioral shifts. Achieving stable and high-quality films requires an understanding of the nucleation and growth processes, while sophisticated characterisation techniques shed light on their structure and composition. Thick films are becoming increasingly important in today's world due to their many uses in energy systems, optical devices, sensors, and electronics. Further study into thin film technology will help bring forth technical solutions that are more efficient, dependable, and environmentally friendly, and it has great promise for future developments.

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