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A Critical Evaluation of Structural and Growth Induced Defects in Thin Film Coatings

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ABSTRACT

Countless thin films have met their demise due to flaws. In the coating industry, flaws are inevitable and can manifest in a wide variety of sizes and shapes. These small foes have been a part of almost every thin film material-related technique and product. Vapors, uncompensated bonds, fissures, lumps, particles, air bubbles, inclusions, pinholes, and chemical flaws such contaminants and chemisorbed substances are all examples of defects. The formation mechanisms, morphological features, and effect on material performance of structural and growth-related flaws in thin film coatings are the primary foci of this work. The study emphasizes the importance of substrate properties, residual stresses, deposition conditions, and their impact on the creation and spread of defects. Growth flaws, such as protrusion and hole-type defects, that occur during deposition procedures and have a significant impact on the coating's dependability and integrity are given special attention.

Keywords: *Coatings, Defects, Stress, Pinholes, Surface.*

I. INTRODUCTION

With its extensive use in photovoltaics, LEDs, microelectronics, optical coatings, and sensors, thin film materials have emerged as a fundamental component of contemporary technology. Modern technology relies on these incredibly thin layers of material, which may be anywhere from a few nanometers to several micrometers thick. Despite thin films' technical significance, their structural and morphological quality greatly affects their dependability and functioning. Device performance can be severely compromised by even minute film flaws that impact electrical, optical, and mechanical characteristics.

Surface roughness, thickness uniformity, flaw presence, and grain size are all part of the film's morphology, which also includes the film's internal structural characteristics. Cracks, holes, dewetting patterns, precipitates, and thickness variations are common morphological defects that can occur during synthesis and deposition. Mechanical failure, lower conductivity, or poor optical performance might result from these flaws, which are more than just surface blemishes. Energy losses can be substantial due to even small flaws in materials used in applications where efficiency is highly dependent on quality, such as light-emitting devices and solar cells. As a result, producing high-performance thin film devices relies on managing and reducing these flaws.

Deposition methods, substrate characteristics, synthesis temperature, pressure, and growth rate are all factors that could lead to flaw creation in thin films. The film growth is affected by the energy and ambient conditions introduced by the various deposition processes, including sputtering, chemical vapor deposition, and physical vapor deposition. Surface imperfections and non-uniform film thickness might result, for example, from uneven deposition rates or inadequate substrate preparation. Similarly, delamination or cracking can occur as a result of thermal pressures caused by discrepancies in the expansion coefficients of the film and substrate. The intricate relationship between process parameters and defect development is underscored by these considerations, emphasizing the need for meticulous optimization of synthesis conditions. Imaging methods are among the most powerful tools for studying the morphology of thin films. Surface characteristics and fault distribution can be better understood using high-resolution pictures of thin films. Imaging film surfaces in great detail is a typical use of optical, scanning electron, and atomic force microscopy. The ease, quickness, and non-destructive nature of optical inspection make it a generally selected approach among them. Without harming the sample, researchers may swiftly detect fractures, dewetting areas, and thickness changes. This is why it shines in industrial environments that demand lightning-fast quality evaluation.

Nevertheless, when it comes to comprehensive quantitative analysis, eye examination and simple imaging techniques can only go so far in spotting blatant flaws. Because of our inherent subjectivity, human observers may fail to notice minute morphological differences that could have a major influence on performance. Researchers have resorted to quantitative image analysis approaches to circumvent this constraint. Surface roughness, film coverage, and defect density are some of the metrics that semi-manual tools like ImageJ and Gwyddion make possible to measure. These technologies make thin film image analysis more objective and repeatable, which improves comparisons between samples and experimental settings. Manual and semi-manual analytical approaches both have their benefits and drawbacks. They are quite labor-intensive and time-consuming, particularly when handling massive datasets. These days, automated experimental systems can quickly produce thousands of photographs, making them ideal for use in current research and industry. It becomes unfeasible and wasteful to manually or semi-manually analyze data quantities of this size. Moreover, these approaches frequently rely on thresholds and settings that the user specifies, which can lead to inconsistency and unpredictability. When precision and speed are of the utmost importance, as they are in high-throughput tests and industrial operations, this difficulty becomes readily apparent.

New methods for automated picture analysis have been created. Feature extraction in conventional computer vision methods like edge detection, thresholding, and texture analysis is based on established algorithms. These techniques work well for particular kinds of flaws because they seek out particular patterns or characteristics in images. They are, however, typically modified to work with certain morphologies, materials, or imaging settings. This means they aren't adaptable enough to handle changes in imaging circumstances or systems. Their limited use is compounded by the fact that they necessitate meticulous parameter tweaking, which is both labor-intensive and demands specialized expertise. There are new possibilities for better fault characterisation and detection thanks to the proliferation of high-resolution imaging data and developments in computational approaches. Analysis methods that are efficient, scalable, and resilient enough to manage big datasets, change with the times, and consistently produce correct answers are in high demand.

II. REVIEW OF LITERATURE

Webb, Matthew et al., (2022) Functional, very stable thin film superlattices have the potential to revolutionize thermophotovoltaics and related thermal and optical applications. In this research, we look at defects that prevent epitaxial, low-miscibility oxide superlattices from growing layer by layer. Ba(Zr 0.5 Hf 0.5)O 3 /MgO 8 nm bilayer superlattices develop an inverted pyramid structure that is spatially phase offset from the matrix when strain relaxation causes 8 nm wide and 3 nm thick layer protrusions to propagate across the succeeding layers. Surface roughness plays a significant influence in the formation of these flaws, since their density and size increase with the sample's interface count. This defect causes phase breakdown of Ba(Zr 0.5 Hf 0.5)O 3 and superlattice decoherence, as shown by in situ high temperature transmission electron microscopy at 1000 °C in a vacuum. This research demonstrates that in order to create single-crystalline superlattices with sharp interfaces suitable for extreme environments, ideal growth conditions must be met.

Tang, Yunqing et al., (2021) In order to understand and control their functional properties, which are utilized in more applications and gadgets, it is necessary to expose the concentration of point defects in transition metal oxide thin films. Despite their evident relevance, there are few experimental techniques to evaluate the chemistry of defects and equilibrium constants in oxides at intermediate-to-low temperatures. This study uses a novel in situ spectroscopic ellipsometry technique on thin films to investigate the defect chemistry of La1−xSrxFeO3−δ (LSF) with x = 0.2, 0.4, and 0.5 (LSF20, LSF40, and LSF50). In order to find out how the concentration of LSF holes changes with temperature and partial pressure of oxygen, this technique links it to optical properties. This is the first all-encompassing account of the defect chemistry in LSF thin films from 350 °C to 500 °C, which expands our understanding of LSF20, LSF40, and LSF50 for low-temperature uses.

Li, Weiwei et al.., (2020) The intriguing properties of complex transition-metal oxides (TMOs), such as ferroelectricity, magnetism, superconductivity, (photo- and electro-)catalytic activity, ionic conductivity, etc., make them vital materials for state-of-the-art energy and electronics technologies. These characteristics are dictated by the partially occupied TM d orbitals and the local coordination environments, which can be affected by many factors such as compositions, borders between grains, surfaces, interfaces, etc. Recent developments in thin film epitaxy have piqued the interest of complex oxide researchers in defect management with the goal of improving or enhancing functional properties. Recent efforts in defect engineering to alter the functional properties of TMOs thin films are detailed in this article. Begin with a brief overview of the chemistry of TMO defects, discussing the many types of defects and how they affect electronic structure, electron configurations, and local atomic structure. Afterwards, we go over the latest findings in engineering flaws in TMOs that have the potential to bring about new functionalities like as ferroelectricity, magnetism, dielectricity, metal-insulator transitions, resistive switching, ionic conductivity, photo-electrocatalysis, and multi-ferroelectricity. Additionally, we provide an electrical structural perspective on the defect-structure-property relationship. Lastly, we emphasize the challenges of defects management and the opportunity for novel device design.

Taherimakhsousi, Nina et al., (2020) Due to the high susceptibility of thin-film materials and devices to defects, there has been much research on enhancing film morphology. This investigation might be expedited by using automated trials to test how different synthesis conditions affect film morphology. Incorporating optical imaging into automated experiments is a breeze, but addressing morphological defects in thin films requires automated analysis of the huge volumes of photos produced by these systems. Software experts are now required to tailor existing methods for automated film morphology analysis in optical images to specific applications since these approaches are not flexible to changes in image content or imaging parameters. Several defects may be detected and their severity quantified using our generalizable convolutional neural network (CNN) for thin-film image analysis. This network is flexible enough to handle a variety of materials and imaging settings. This CNN may be simply adjusted to perform additional tasks related to thin-film image processing, allowing automated thin-film research systems to make greater use of imaging.

Ohlídal, Ivan et al., (2020) This article reviews the optics of thin films that are not uniform in thickness, featuring transition layers, overlayers, boundary roughness, uniaxial anisotropy, and other features. We give a quick review of the theoretical approaches that depict the optical characteristics of these nonuniform thin films and include these defects in their equations. These techniques follow the recursive and matrix formalities for the overlayers and transition layers. They use a polynomial formulation or the local thickness distribution to average the Mueller matrix elements in order to deal with thickness nonuniformity. They use either scalar diffraction theory or Rayleigh-Rice theory, or both, to deal with boundary roughness. Lastly, the Yeh matrix formalism is used for uniaxial anisotropy. We illustrate the theoretical findings by a few instances of optical characterization of nonstoichiometric silicon nitride thin films exhibiting boundary roughness and uniaxial anisotropy, as well as inhomogeneous polymer-like thin films exhibiting thickness nonuniformity and integration of transition layers. Such characterisation is achieved by means of spectroscopic reflectometry and variable-angle spectroscopic ellipsometry. There are a number of optical approaches that may be used to fully characterize the aforementioned thin films. Therefore, it is possible to determine the values of all the characteristics that characterize these films.

Wu, Fan. (2018). Deformation twins and phase interface are examples of microstructures and planar defects that have a substantial impact on the overall performance of a material system. Their influence is especially apparent in multi-layer thin-film heterostructures because of the influence on multi-layer structure creation and the small size of thin films. Our present understanding of the microstructure and defects in thin film heterostructures is summarized in this article to aid in the direction of future research in this field. The multilayer thin-film heterostructures that were the subject of this investigation were created using pulsed laser deposition. Microstructures and defects were examined using transmission electron microscopy.

Tolba, Ahmad et al., (2015) Some flat surface objects, such as thin films, paper, foils, steel slabs, aluminum plates, textiles, and glass sheets, can have their visual quality diminished as a result of production errors. The end outcome is less satisfied customers, more material squandered, and a damaged reputation. This research presents a novel application of image visual quality measurements, namely the multiscale structural similarity index (MS-SSIM). There is a new algorithm that can detect and localize defects in many different types of flat surface items very rapidly. In contrast to the current best practices, the proposed algorithm shows potential. A defect detection accuracy of 99.1% was achieved with a recall/sensitivity of 97%, a specificity of 100%, and a precision of 98.52%. The MS-SSIM's remarkable capacity to differentiate between normal and abnormal surfaces is shown by the discriminant power. Although it sacrifices processing speed, the MS-SSIM outperforms the single-scale SSI approach in terms of performance. The key advantages of the proposed method over the state-of-the-art approach based on Gabor filter banks are scale invariance, improved detection accuracy, processing speed approaching real-time, and the removal of the parameter selection problem.

Chen, Shiyou et al., (2010) When it comes to thin-film solar cells, quaternary absorber materials like Cu2ZnSnS4 are among the best options. A tiny stable chemical potential zone for the synthesis of stoichiometric compounds is shown by examining the thermodynamic stability of this quaternary molecule. Because its acceptor level is lower than the Cu vacancy, p-type CuZn antisites will predominate as defects under these circumstances. Various polytype structures of Cu2ZnSnS4 are formed by the dominating self-compensated defect pair in this quaternary compound, which is [CuZn−+ZnCu+]0. We suggest that, if kinetic barriers can be used to prevent the precipitation of ZnS, the development of Cu2ZnSnS4 under Cu-poor/Zn-rich conditions will optimize the efficiency of solar cells.

III. DEFECT FORMATION

In thin film systems, it is always expected that the film and substrate would adhere perfectly. But there are always imperfections, weakly bound regions, and even contaminants at the interface. These defects can be the result of a mismatch at the bond caused by the film material's and the substrate's differing characteristics. One area where there is a discrepancy is in the thermal coefficient of materials, which causes them to deform when exposed to changes in temperature. Due to their role as stress concentrators and influences on plastic deformation, preexisting defects in the thin film system are the primary failure sensitive locations.

Buildup of residual strains is a result of ion/atom implantation effects during deposition. Delamination or fracture may occur if these residual stresses induce cracks to form with the film, in the substrate, or at the interface, and then emerge at the film. The formation of thin film residual stress can occur for a variety of reasons, including, but not limited to, epitaxy, precipitation, grain boundary relaxation, grain growth, impurities, and the shrinking of grain boundary voids.

Ibru et al. (2017) found that films are more prone to crack propagation and fracture susceptibility when subjected to tensile stresses; in contrast, films subjected to compressive stresses develop cracks at the film substrate interface, which result in delamination and buckling. Simplified schematics of common failure modes caused by compressive and tensile residual stresses are shown in Figures 1 and 2.

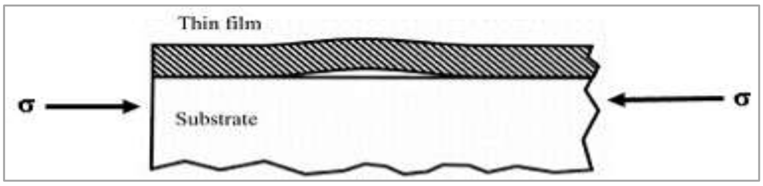

Figure 1: Bucking Initiated at Interface of Thin Film Due to Compressive Straining

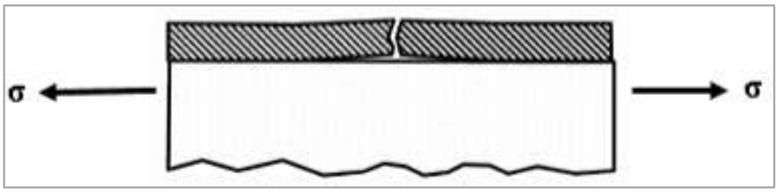

Figure 2: Cracks Formed Inside the Thin Film Due to Tensile Straining

Porosity is another flaw with thin films. The presence of gaps or pores in the film is a result of phase change and is independent of the thin film deposition process. Defects in earlier deposition processes, including cleaning, machining, and heat treatments, can also lead to porosity. The strength and ductility of a material are said to diminish as its porosity increases.

IV. GROWTH DEFECTS FORMED DURING DEPOSITION

Deposition transfers and amplifies all substrate surface morphological traits from mechanical preparation and ion etching onto the coated surface. Topographical anomalies and microscopic foreign particles on the substrate after cleaning or ion etching produce coating defects. This is due to shadowing. Coating produces a large fraction of seed particles that induce growth abnormalities. A TiN coating was created on a D2 tool steel substrate using BAI730, CC800/9 ML, and AIPocket evaporation. Figure 8 shows this coating's 3D profile. Growth fault density varies.

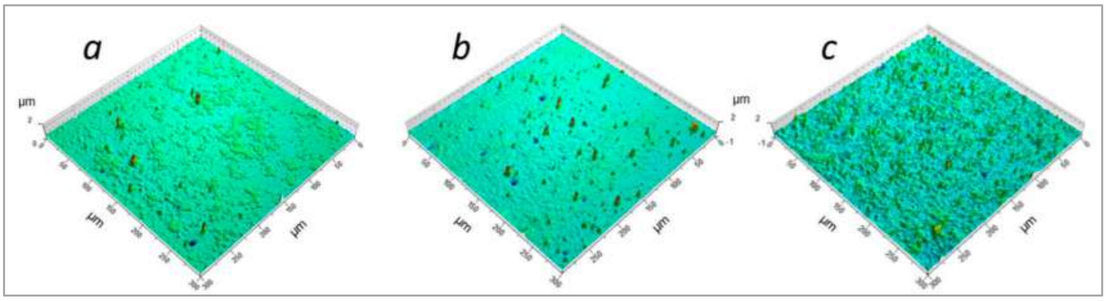

Figure 3: Three-Dimensional (3D)-Profilometry Image of TiN Coatings Deposited on D2 Tool

<u>Protrusion Defects</u>

- **Nodular Defects**

The most common growth problems, nodular flaws, exist in all PVD coatings. Seeds cause nodular deformities. Small particles including dust, foreign particles, coating material expulsions, and substrate protrusions can be seeds. Any irregularity, even a tiny one, may be a seed. Nodules can sprout from a seed put into the substrate. As it grows, the nodule becomes an inverted cone and expands until it reaches the film's surface as a dome. The nodule's seed is much smaller than the nodule. On its own, it's harmless. It develops differently from the covering but is the same material. Because the seed particle blocks some vapor flux and surface atoms have limited thermal mobility, the coating matrix has gaps at the nodule's edge. Externally, the nodule is distinct from the covering. This sharp boundary is fragile. Nodules often have apertures around them, and sometimes they detach and leave holes.

Nodular flaws on rotating substrates are different in shape and size from those on stationary substrates. The inverted cone-shaped nodules on stationary substrates differ from those on rotating substrates in that they have straight sides. The angle of vapor flow relative to the substrate plane greatly affects the size and shape of nodules on spinning substrates.

- **Flake Defects**

Flake flaws—growth defects that protrude from thin films—can also occur. Because seed particles emerge from diverse places, these errors are much larger and look different than nodular defects. Delamination on substrate and shield fixture holders in the vacuum chamber produces seed particles, large flake particles. The delamination happened in previous batches. When seed particles are heated and pressured before and during deposition, coatings delaminate. Flake fault seeds are huge, generally many tens of micrometers in diameter, and have an unusual shape. The flake seeds' exceptionally high diameter-to-thickness ratio gives the defects above them a flat top and step-like edges. The flake fault is caused by the larger seed protruding step-like. Arcing on substrate equipment like the substrate turntable can flake coating during ion etching and deposition. Overall, flake defects have less surface density than nodular faults. The main factors are coating thickness and substrate fixture and shield adherence. Cleaning all vacuum components after numerous batches reduces flake defect density. Despite their low concentration, flake defects can adversely damage the coating's performance if they penetrate the substrate and directly expose it to environmental hazards like oxidation and corrosion.

- **Droplet Defects**

Problems with droplets are common in coatings made via cathodic arc deposition. The contamination of the substrate surface by arc discharge droplets causes growth defects when metal ions are used for substrate etching. When droplet production and deposition occur simultaneously, coating growth defects start to appear. Metal droplets are formed as the target melts. The target releases liquid droplets of the substance of interest. It is possible for some of the droplets to settle on the substrate when the liquid solidifies. Defective coatings are deposited as a result of these drops.

Hole-Like Defects

In reference to the growing flaws under the thin film's surface, a wide range of terminology is utilized. Any growing fault below the thin film's mean surface level will be referred to as a hole for the purposes of this discussion. The origin and form of the hole-like flaws allow them to be recognized from protrusion faults.

- **Pinhole Defects**

One of the most common growing defects in photovoltaic (PV) thin films is pinholes, which are really discontinuities in the coating microstructure. From the substrate all the way to the coating's top surface, these holes extend with widths ranging from (sub)microns. There are a number of potential causes for pinholes. Pinholes typically form in imperfect substrates, such as pretreatment-induced pits or shallow depressions. Typically, pinholes begin in a geometrical structure, like a tiny but deep hole, where the shadowing effect prevents the film from developing on the walls. To be more precise, the coating is applied with more heaviness to the substrate's flat front side compared to the vacuum sidewall. Since the coatings are placed to the hollow walls, the shadowing effect makes them look columnar and porous.

Crater-Like Defects

The nodular defect, the surrounding matrix, and the seed particles all have weak connections. Because of the built-up stresses, a crater forms on the surface as some of the nodular defects split from the covering as it expands. It is conceivable that the crater that has developed is a "inverse" nodular or flake fault. Two outside forces that could induce the formation of such a crater are ultrasonic cavitation cleaning and wiping after removing the coated sample from the deposition chamber. If the spall-off happens during or after deposition, the substrate might be exposed at the base of these holes. The coating that is still in the process of developing will fill the ensuing crater if the nodular defect is removed from it while it is being deposited. Due to its microstructure, which is both highly porous and columnar, the overgrowing coating does not adhere effectively. After deposition is complete, a scanning electron micrograph of a crack reveals a crater-like defect that formed from the nodular flaw. There was probably already a lot of internal tension, and the nodular defects probably split when the material cooled.

V. PREVENTING DEFECTS IN THIN FILM

The end objective of any technique for depositing thin films is to cover a substrate with a homogeneous, high-quality film. You may not be able to get the video quality you seek if flaws like spits, particles, and gaps are present. Modifying a production procedure to remove these flaws and increase yield is possible if required. Because of this, producers may implement a more effective procedure, leading to enhanced performance in the long run.

Spits

Evaporation deposition is a technique for coating surfaces with a source material by gradually and uniformly evaporating it onto the substrate. When the film coating becomes encrusted with bigger particles of the target material discharged from the source, a phenomenon known as spitting occurs, it might disrupt the subsequent production step. Evaporation by thermal or e-beam means predominates in this case.

The efficiency of manufacturing and the performance of applications are both adversely affected by spitting. Because of the waste that results, this can significantly impact the return on investment for manufacturers. When indium bump deposition is nearing completion, it's possible that almost finished components may need to be scrapped because to spit flaws, which are typically bigger than the indium bumps. The worst spot to have a yield problem when thinking about cost of ownership is to trash components when they are at their most valuable.

There is a cost to reducing the deposition rate in order to lessen spits during evaporation: Investing in additional capital equipment to slow down the deposition rate and satisfy production demand increases the entire cost of manufacturing. On the other hand, with the correct setup, you can prevent spits. One such system that maximizes productivity and yield is Denton's indium bump system, which is engineered to avoid spitting even at high deposition rates.

Particles

The consistency of a layer, and therefore, the dependability and efficiency of its application, can be compromised when using thin film deposition due to particles, which are additional materials from the surrounding environment. In addition to particles that naturally occur in the environment (such as skin, hair, and flakes), particles can also originate from earlier stages of manufacturing. The chances of particle introduction rise when an application calls for applying several layers to a substrate.

Substrate or embedded surface particles will diminish device performance or render the product useless. For this reason, clean rooms are essential for the production of lasers and semiconductors, as they eliminate the possibility of environmental contamination.

The answer to the problem of particle prevention or removal in thin film coatings and applications might be different for each procedure. Appropriate shield design, cleaning frequency, and shield replacement are your primary weapons in the fight against particles. Depending on their proximity to the deposition zone, shields may require more regular cleaning or replacement. To lessen the likelihood of particle contamination, preventative maintenance is crucial. To succeed, you must have a firm grasp of the procedure.

Voids

Void, or vacancy, is a structural flaw in thin film deposition that occurs when material is absent from the film, like a hole. Several factors, like as temperature fluctuations and the kinetic energy of the deposition process, might influence void development. Void creation is more common in evaporation than in ion beam sputtering, but phase change can cause voids to arise in any deposition process.

The ability to adjust temperature is vital for managing void formation in thin film deposition. Raising the process temperature can decrease the quantity of voids; nevertheless, there is a cost to reducing voids: Substrates and existing layers can be damaged by high temperatures.

Void prevention can be achieved by the use of Ion Assisted Deposition (IAD). By using a low-energy ion beam that is focused on the substrate, IAD modifies conventional e-beam technology to avoid overheating the material. The beam's ions will cause the film to get denser. Environmental stability, mechanical endurance, and the absence of voids are all improved by the higher-density coating. When it comes to optimizing system design, Denton Vacuum is well-versed. No need to reduce your deposition rate—our application-specific method will help you achieve a spit-free setup. As a result, we are better able to regulate the process and satisfy production demands.

VI. CONCLUSION

The films' mechanical stability, longevity, and functional performance are greatly affected by flaws including pinholes, fractures, voids, and nodules, which are naturally present in thin film deposition methods. A significant factor in deciding the kind and degree of defects is the interplay between deposition parameters, substrate characteristics, and residual stresses. The results show that if there are growth faults, especially ones that start on the surface and are contaminated, they can spread into the film and cause it to fail or degrade in function. There is a need for more efficient and automated analytic methodologies since standard flaw detection methods, although beneficial, are sometimes inadequate for large-scale or high-throughput applications. Optimal deposition conditions, adequate surface preparation, and sophisticated process monitoring are necessary for effective defect control. Methods like ion-assisted deposition, temperature control, and cleanroom techniques can greatly decrease defect density and enhance film quality. In order to improve the efficiency and reliability of devices, it is crucial to reduce the number of flaws in thin films. The integration of intelligent analysis methods with enhanced characterization instruments should be the primary goal of future research in thin film production. This would allow for better process control and real-time fault identification.

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