

Thin Film Preparation and Characterization: Principles, Deposition Routes, Metrology, Process–Structure Relationships, and Emerging Directions

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Abstract: Thin films are engineered layers with thicknesses from less than nanometers to micrometer scale, their performance is influenced by surface and interfaces, defects and grain boundaries, residual stresses, confinement effects and material properties. This article introduces the way the performance of thin film is affected and a characterization overview is given through the application of the process-structure-property relationships in combination with the major fabrication technologies. Various PVD and CVD techniques like thin film sputtering, thermal evaporation, EB-evaporation, pulsed laser deposition PLD, CVD, PECVD, ALD as well as sol-gel, spin-coating, doctor-blade, spray-pyrolysis and chemical bath are discussed. Material's phases, size and orientation of grains, roughness, porosity, composition and quality of the interface between phases and between films are influenced by factors like surface condition, substrate preparation, nucleation, adatom migration and diffusion, vacuum condition or reagent composition, deposition rate, substrate temperature, plasma factors (e.g., electron density, plasma potential etc.) and heat treatment. Both approaches complement in determination of the desired structure and properties. XRD, XRR give structural properties and thickness. SEM, TEM and AFM explain structure and morphology of thin film. Composition is identified by EDS, XPS and bonding state by FTIR, Raman spectroscopy and so on. Optical properties (ellipsometry, UV-vis spectroscopy, photoluminescence) and electrical properties (4 points probe, Hall effect etc.) are found. Currently research focuses on low-temperature fabrication, plasma assistance, fabricating at an atomic scale, on line monitoring and optimization by the use of machine learning to enable scalable manufacturing with good yields, saving materials. Major remaining issues are reliability and scalability problems, precise control of interface, estimation of errors due to measurement inaccuracy and residual stress. Sustainable manufacture approaches are in investigation.

Keywords: Thin films; Thin-film deposition; PVD; sputtering; CVD; Atomic layer deposition; Optical properties; and electrical properties.

INTRODUCTION

Thin-film technology has a very special place and role in modern materials science. That is, with a very small amount of material, one can prepare a thin layer where the combination of the properties will be almost entirely different from that of the bulk solid. Electrical transport characteristics, optical absorption, catalytic activity, corrosion resistance, magnetic properties, mechanical characteristics and thermal properties may change quite unexpectedly when the thickness of the films is decreased and when one puts a material in contact with a substrate (by deposition). Interfaces play an increasing role as the thickness reduces and reaches characteristic length scales of the microstructures and the physical properties. The relevance of thin films from a technological perspective covers semiconductor devices, screens, solar cells, sensors batteries, optical coatings, parts for magnets, medical coatings, coatings that protect against mechanical wear, new quantum and ultra-thin material platforms. Very recently, comprehensive publications have been published

that describe thin films as a point where electronics, energy conversion & storage, sensor technology, biomedical engineering, and green production meet [Workie, A. B. *et al.*, 2023]. Material deposition and characterization are two things that should be seen not as separate acts, but rather they are complementary ones. Deposition is the process that sets a film's kinetic and thermodynamic history, while characterization gives proof of structure of the film and its properties through microstructural investigation. For instance, raising substrate temperature can mean an improved surface diffusion that could be a reason for enhanced crystallinity at the same time as it could result in undesirable interdiffusion, film roughness or loss of volatile components. In the same way, increasing the power in sputtering process will increase the deposition rate; yet, it could also affect the type of ions bombarding the surface, the compressive stress and the density of defects.

Graphical Overview

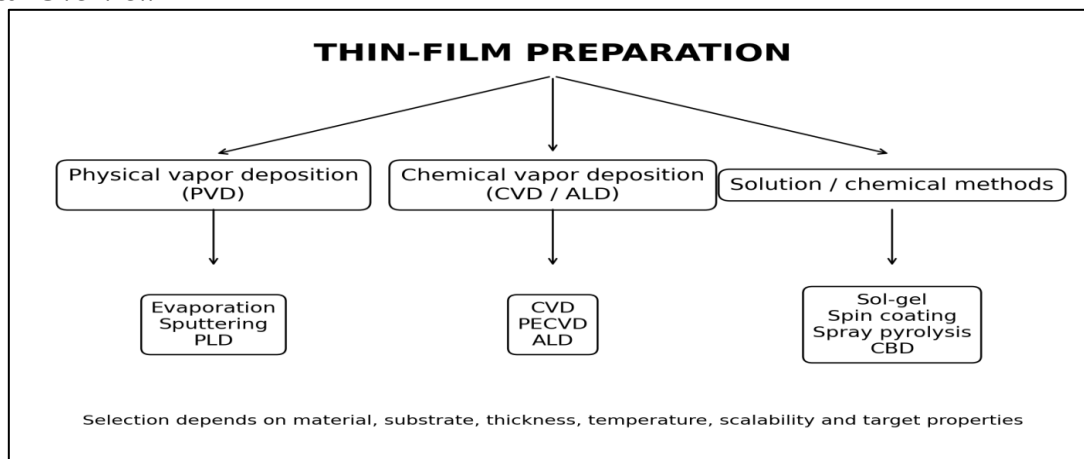


Fig. 1. Classification of major thin-film preparation routes. The choice of process is governed by material chemistry, substrate compatibility, thickness, temperature, scale and target properties.

The field has shifted to the point where coating has been replaced by a very controlled fabrication of objects with dimensions close to nanometers. The latest literature reviews highlight that ultimately the deposition parameters, nucleation, and interfacial processes dictate the structure of the film as well as its functional behavior [Garg, R. *et al.*, 2024]. So, this review aims to provide a link between the preparation parameters and the film characteristics which are actually measured. The topics of the paper include principal deposition technologies, growth mechanisms, process variables, post-deposition treatment, structural/ morphological/ compositional/ optical/ electrical/ mechanical characterization techniques, common causes of uncertainties, and application areas. Future avenues of research are also highlighted in the paper.

FUNDAMENTALS OF THIN-FILM GROWTH

Nucleation, Adsorption and Coalescence

Film growth starts when the molecules hitting the substrate surface become surface molecules or so-called species. The species that are adsorbed will react as a result to the surface energy, temperature of the substrate, supersaturation, and the mobility of the species. They may be diffusing; stable nuclei are the result of coalescence of adsorbed atoms/molecules or surface molecules/atoms. This is not a continuous monomolecular layer. In most cases, the different growth ways correspond to the Volmer Weber (island-like) mode, the Frank Kaplan (layer-by-layer) mode, and the Stranski Krastanov (layer-plus-island) mode. Real films often go from one regime to another based on the degree of coverage.

The interaction of arrival rate and surface diffusion strongly influences film growth. To give rough estimate only, the diffusional distance of an atom can be written in terms of

$$LD \approx (Ds\tau)^{(1/2)},$$

Where D_s denotes the diffusion coefficient on the surface and τ refers to the characteristic time that particles stay on the surface. Since D_s usually goes up rapidly as the temperature rises, heating the substrate would lead to larger crystallites sizes and higher degree of crystallinity. However, a too high temperature level can mean re-evaporation, interfacial reactions, or thermal stress.

Thermodynamic and Kinetic Considerations

The deposition of thin films is mainly controlled by a compromise between thermal driving forces and kinetic restrictions. Surface and interfacial energies mostly influence whether the formed islands will wet the substrate whereas deposition rate pressure temperature and reactive chemistry mainly affect how fast the equilibrium can be achieved. In PVD, vapour pressure is strongly affected by the pressure, in CVD and ALD, adsorption of precursor molecules and kinetics of surface reactions are fundamental, and in solution processes supercooling hydration complexation viscosity and solvent evaporation are of importance.

Besides these explanations, a very helpful idea is to relate the concepts to the Arrhenius formula $k = A \exp(-E_a/k_B T)$, where k represents the reaction or transport rate, A is a pre-exponent factor, E_a is an activation energy, k_B is Boltzmann constant and T is the absolute temperature. The equation clarifies why even a slight difference in the operation

temperature can drastically change the deposition kinetics, although many deposition processes do involve several simultaneously activated barriers that cannot be represented by a single kinetic expression.

THIN-FILM METHODS

PREPARATION

Physical Vapour Deposition

PVD is a method of forming films, and it works through the transportation of material to a substrate under vacuum or gas conditions in a controlled manner from a condensed source. Common PVD types include sputtering PLD electron-beam evaporation, thermal evaporation, etc. PVD is very appealing due to Truth is process monitoring allows for the easy control of the deposition rate, film thickness, and source purity. Current literature is pointing out sputtering as an ideal approach for various materials like metals oxides nitrides alloys carbonaceous films and ceramic coatings [Garg, R. *et al.*, 2024].

Thermal and Electron-Beam Evaporation

Thermal evaporation works by heating a source so much that its vapour pressure becomes high enough to carry material across the chamber. Electron-beam evaporation is a technique wherein a beam of focussed electrons replaces the resistive heater, thereby the use of materials with a high melting point. The way the deposition is done can

be fine-tuned by changing things like the power of the source, its shape, the pressure in the chamber, and where you put the substrate. The main drawbacks are that the deposition only happens in front of the line of sight, and it's hard to keep the right chemical balance when one of the components of the material is more volatile than the others. On top of this, it's a possibility of the source being contaminated.

Magnetron Sputtering

Ejecting sputtering works by launching the target atoms with the bombarding ions that are the driving force. Magnetic containment is applied in magnetron sputter processes to keep plasma dense at the area of the target which in turn increases the ionization efficiency. Direct current sputtering has been mainly applied for metallic targets while alternating current sputtering is suitable for non-metallic targets. Reactive sputtering is carried out by adding a reactive gas like oxygen or nitrogen to create oxides or nitrides. Important operational variables in process control are target power, gas pressure, gas composition, substrate bias, target, substrate distance and temperature. These controllable variables have a considerable effect on film properties like deposition rate density crystallinity texture, residual stress as well as the density of point and line defects [Garg, R. *et al.*, 2024].

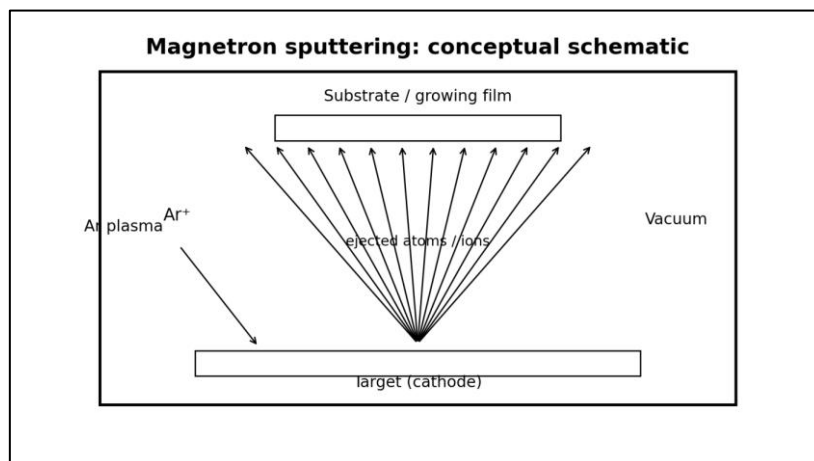


Fig. 2. Conceptual magnetron-sputtering geometry showing plasma generation, ion bombardment of the target and transport of sputtered species toward the substrate

Pulsed Laser Deposition

The laser pulsing ablation of the target followed by the transient plasma plume formation is called PLD. PLD works well with challenging oxides and compositionally complex materials since target composition can more readily be maintained than through equilibrium evaporation. Advances of PLD have toward larger area 2-D films

emphasizing uniformity of plume, particulate, and large-scale requirements of PLD.

Chemical Vapour Deposition

CVD relies on gas-phase precursors reacting or decomposing at, or very close to the heated surface of substrate. CVD is claimed to achieve a good conformity and large-area capability and have

wide ranges of applications for semiconductor films, carbon materials, oxides, nitrides and functional coating. Process parameters of CVD include precursors (gas state), feeding rate, pressure, carrier gas, reactive gas and temperature of substrate. In recent studies of field-enhanced CVD, plasma, photons, electric field and magnetic field can adjust the nucleation, grain growth, texture, density and phases formation of film.

Plasma-Enhanced CVD

PECVD uses the plasma to create species and thus lowers the temperature requirements to form the film. PECVD is useful for temperature sensitive substrates, and for material growth, as the radical atoms are used. The issue with PECVD is that ion bombardment and plasma chemistry can be

problematic; producing flaws or stress in surfaces or film if not optimized properly.

Atomic Layer Deposition

The ALD process uses the sequence of surface reactions that are mutually self-limiting. An ALD cycle is defined by exposure of precursors, A and B, and their purges (e.g., ABBA scheme where A and B are precursors). The inherent self-limiting process provides superior control over film thickness, profile, and conformity even for intricate structures such as three-dimensional objects. Modern review articles on ALD clarify that its real performance should rather be related with precursor dose, surface reactivity, purge efficiency, growth temperature and by-product clearance.

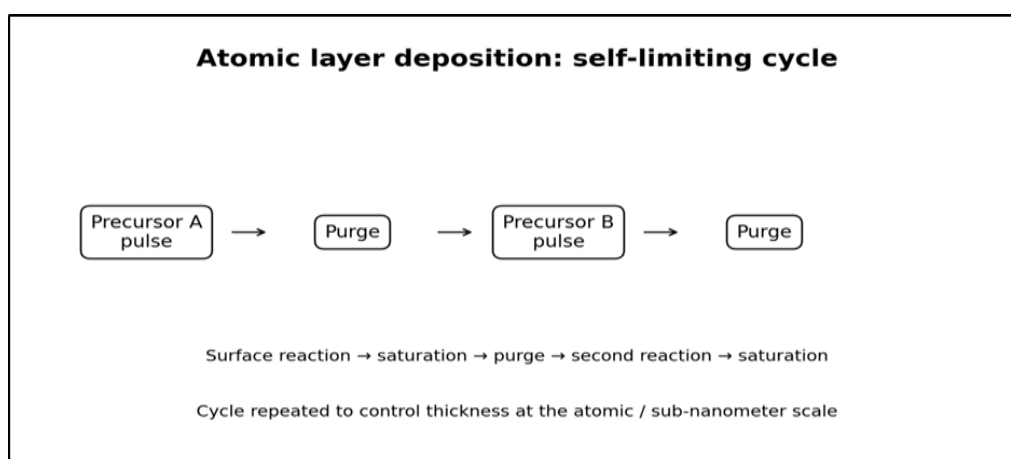


Fig. 3. Schematic ALD cycle. Sequential precursor exposures and purges generate self-limiting surface reactions and enable precise thickness control.

Solution-Based Methods

Solution processing provides benefit of low equipment cost, large area fabrication and relatively lower processing temperature. Major fabrication methods include sol-gel, spin coating, dip coating, spray pyrolysis, chemical bath deposition. Such methods are particularly suitable for the fabrication of oxides, chalcogenides, transparent conducting films, solar cell absorbers and so on. The drawbacks are related to the solvent handling, induced stress while drying, cracking tendency of the film, solution chemistry sensitivity of the precursors, precursor stability etc [Emrie, D. B. 2024; Workie, A. B. *et al.*, 2023; Krishna, S., & Vasu, V. 2024].

Sol-Gel And Spin Coating

The transformation from molecular precursors into a colloidal sol and then to a gel (sol-gel processing) is based on a sequence of hydrolysis and condensation reactions. In sol-gel processing, the precursor layer is formed on the substrate by

spin coating or dip coating and is then dried and calcined. Research on sol-gel technology up to present still mainly utilizes its characteristic features such as low temperature processing and composition control, though it is very sensitive to concentration of precursors, rate of hydrolysis, chemistry of solvents, moisture, and temperature treatment [Emrie, D. B. 2024].

Spray Pyrolysis

Spray Pyrolysis sprays a suspension of solution to the substrate that reaches certain temperature for it to release the solvent and the decomposition takes place to form film [Emrie, D. B. 2024]. The application for scalable coatings or for photovoltaic, or photocatalytic materials is appealing. Major affecting parameters for the film morphology and phase as identified by recent reviews include substrate temperature, solution precursor concentration, atomization of the solution, carrier gas flow and the spray rate [Workie, A. B. *et al.*, 2023].

Chemical Bath Deposition

Controlled precipitation at or near the substrate surface, forms the basis of the CBD process. Availability of ions for precipitation-free ion concentration is governed by the presence of complexing agents, pH, temperature and precursor concentration, deposition rate depends on these

factors and others such as current density. CBD holds advantages particularly over large area semiconductor films, CdS type buffer layers, although the conditions need stringent control- Solution stability and parasite precipitation are major issues.

COMPARATIVE ASSESSMENT OF PREPARATION METHODS

Table 1: Qualitative comparison of common thin-film preparation methods

Method	Typical environment	Key strengths	Main limitations	Thickness control	Representative uses
Thermal evaporation	High vacuum	Simple, high purity, high rate	Line-of-sight; multicomponent stoichiometry	Good	Metals, optical coatings
Magnetron sputtering	Vacuum + plasma	Adhesion, uniformity, composition flexibility	Equipment/parameter complexity	Good	TCOs, oxides, nitrides, coatings
PLD	Vacuum / reactive gas	Complex compositions, epitaxy	Particulates, area/scale limitations	Good	Oxides, 2D materials
CVD/PECVD	Reactive gases	Conformal, scalable	Precursor chemistry, temperature	Good	Semiconductors, graphene, coatings
ALD	Sequential precursors	Atomic-scale control, conformality	Slow growth, precursor cost	Excellent	Dielectrics, 3D devices
Sol-gel / spin	Solution	Low cost, compositional control	Cracking/shrinkage	Moderate-good	Oxides, sensors
Spray pyrolysis	Aerosol + heated substrate	Large area, low cost	Roughness/process sensitivity	Moderate	Oxides, photovoltaics
CBD	Aqueous/solution bath	Simple, low temperature	Parasitic precipitation	Moderate	Chalcogenides, buffer layers

PROCESS PARAMETERS AND POST-DEPOSITION TREATMENT

Substrate Preparation

Another commonly underestimated yet critical process affecting nucleation and adhesion-cleaning. Surface treatments such as native oxide formation and native oxide formation and / or etching and contamination (particulates, organic residues, hydration, hydroxylation) all affect wettability and surface energies. The sequence of processes typically involved are solvent cleaning, rinsing, drying and, where necessary, a plasma/UV-ozone treatment, with the actual recipe dependent on compatibility of the substrate and film chemistry.

Substrate Temperature

Temperature affects surface diffusion, desorption, reaction rate and the mobility of defects. Often

there is an optimum temperature window in which both crystallinity and density increase without resulting in interfacial reactions or decomposition, thus one size 'higher is better' approach isn't applicable.

Pressure, Gas Composition and Plasma Power

The addition of reactive gas, as discussed, modifies stoichiometry and phase stability. Similarly, the pressure influences the mean free path and energy spectrum of the arriving species, which become critical to plasma-processes. As noted above, the power of plasma systems strongly influences their density and rate; high ion bombardment can lead to increased compressive stress, or increased defect formation. Review articles of sputtering systems from the last decade tend to discuss the coupling of energy and

chemistry as a vital tool in designing and scaling-up systems [Garg, R. *et al.*, 2024].

Solution Chemistry

With wet-chemical techniques, pH, precursor concentration, complexing agents, volatility, viscosity and bath temperature control nucleation and growth; even slight variation affects the particle structure, roughness, porosity and preferred orientation; hence careful control over the preparation of solution, aging, solution volume and its constituents is a must for reproducibility.

Annealing

Thermal annealing provides increased crystallinity, removal of residual solvent and/or ligands and can result in an enhancement of grain growth and alterations of defect populations. Other effects include altered oxidation states, interdiffusion, and creation of thermal stress. Therefore, temperature of annealing atmosphere, time, and ramp rate must be explicitly detailed.

THIN-FILM CHARACTERIZATION

It is clear that no single technique will fully describe a thin film. A thorough characterization scheme involves measurements of different length scales and physical variables, which must be correlated with one another. The analysis of structural, morphological, chemical, electrical, optical and mechanical data sets should provide self-consistency. More recent reviews on the characterization of films have highlighted the role of the techniques described. XRD, SEM, AFM, ellipsometry, and Raman spectroscopy in relation to films are discussed in [Soumya, S. S. 2024; Bhutia, S. Z. *et al.*, 2024; Karakuş, E., & COŞKUN, B. 2023; Brinker, C. J., & Scherer, G. W. 1990].

X-Ray Diffraction

The fundamental uses for determining crystalline phase, lattice parameters and preferred orientation using XRD are given. Using Bragg's Law:

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

where n is the order of diffracted intensity, λ is the X-ray wavelength, d is the interplanar spacing and θ is the Bragg angle. In the case of the peak-broadening analysis, crystallite size, D , can often be calculated using the Scherrer relation:

$$D = K\lambda/\beta\cos\theta$$

where K is a shape-dependent constant, λ is the instrumental-corrected Full Width at Half Maximum, β measured in radians, and θ is the Bragg angle. It is imperative not to interpret result automatically as grain size.

X-Ray Reflectivity

This is also particularly good for thin/multilayer films as the interference fringes allow thickness, density, and interface/surface roughness to be calculated. These can become non-unique (Inverse Problem) hence physically constrained models or other independent thickness measurements.

Scanning Electron Microscopy

SEM can display the surface and cross-section morphology of high spatial resolution. The morphology of the grains, of cracks, pores, pinholes and the continuity of the film can thus be studied. Direct assessment of the film thickness from cross-sectional SEM would need the interface film/substrate to be distinguishable.

Transmission Electron Microscopy

By the atomic structure, TEM is able to achieve resolution of nanoscale grains, interface, defects and multilayer, which is about several orders of magnitude higher than SEMs can do. SAED, the higher resolution imaging, has the advantage to provide the supplementary structural information. The sample preparation processes might be destructive and also causes artifact.

Atomic Force Microscopy

AFM generates three-dimensional surface topology, together with quantitative measurement of roughness, where R_q is usually calculated as: $R_q = \sqrt{(1/N \sum_{i=1}^N (z_i - \bar{z})^2)}$, N = The total number of data points (pixels) measured within the AFM scan area.

z_i is the measured height value and $\bar{z}$ is the average height, and it has some affect on the scattering, wetting adhesion charge transport and interface quality.

Composition And Chemical State: EDS And XPS

In combination with SEM generally, EDS can identify elements relatively quickly and gives estimated concentrations. XPS is even more surface sensitive and is the only method listed which can differentiate between various chemical states via analysis of binding-energy shifts. As XPS can only sample a very small near-surface area analysis, depth profiling is possible in multilayers but requires ion sputtering, which may alter chemistry and must be carefully monitored and interpreted.

Raman and FTIR Spectroscopy

Vibrational modes can be investigated by Raman spectroscopy and this is a means to observe phase, crystallinity, strain and disorder as well. FTIR

gives additional information, concerning molecular bonding and functional group. Oxides, polymers, carbon compounds and hybrid films can be investigated.

Optical Characterization

It reveals the values of transmittance, absorption. For a direct allowed transition, a Tauc Plot is commonly written as:

$$(\alpha h\nu)^2 = A(h\nu - E_g)$$

Where α is the absorption coefficient; h is the Planck's constant, A is a constant, E_g is the optical band gap. Other exponent must be added to it according to assumption of mechanism. The derived optical gap value via this model depends on the chosen assumption, it cannot be quoted directly, without mentioning transition nature. The measurement of spectroscopic ellipsometry deals with the induced change in polarization on reflection. An application of ellipsometry that results in thickness and the complex optical constants, n and k has been observed with suitable model fit [Soumya, S. S. 2024]. The measurement has also revealed its potential on thin polymer and multi layers on some recent developments [Bhutia, S. Z. *et al.*, 2024].

Photoluminescence

The radiative recombination can be revealed using photoluminescence. Band-edge transitions, defect

states, impurities and recombination routes can be investigated through the position, width and intensity of the emission peak. PL must be measured in conjunction with structural or chemical analysis as peaks occurring at the same position might be associated with different defect sites.

Electrical Characterization

Sheet resistance may be measured by four-point probe whereas carrier type, concentration and mobility can be measured by the Hall effect. In the most straightforward case of a single carrier only it would be written that conductance is expressed as $\sigma = nq\mu$ where n is concentration, q is elementary charge and μ is carrier mobility. The results obtained are sensitive to contacts, Film uniformity, grain boundaries, stoichiometry and the geometry of measurement.

Mechanical Properties and Adhesion

Nanoindentation probes hardness and elastic modulus at low indentation depths; scratch and adhesion tests probe the integrity of coating. Residual stress can be measured by means of the substrate curvature or by means of the diffraction. The role stress in the film is critical as excessive tensile stress or excessive compression stress will cause crack or delaminate, buckle or the warp/bow the wafer.

CHARACTERIZATION MATRIX

Table 2. Complementary roles of common thin-film characterization methods

Technique	Primary output	Typical scale / sensitivity	Strength	Important limitation
XRD	Phase, lattice parameter, texture, crystallite size	Å–nm structural information	Non-destructive, phase specific	Weak for amorphous films; peak overlap
XRR	Thickness, density, roughness	nm-scale layers	Excellent for ultrathin/multilayers	Model-dependent fitting
SEM	Morphology, cross-section	nm–µm	Fast visual assessment	Limited chemical specificity
AFM	Topography, roughness	nm–µm lateral; height sensitive	3D quantitative surface data	Small scan area; tip artifacts
TEM	Interfaces, defects, nanostructure	Å–nm	Highest spatial resolution	Complex sample preparation
EDS	Elemental composition	µm to sub-µm depending on beam/sample	Convenient with SEM	Limited light-element/quantitative accuracy
XPS	Surface composition/chemical state	Top few nm	Chemical-state information	Surface sensitive; possible sputter artifacts
UV–Vis	T, A, optical gap	nm–µm films	Simple optical screening	Model assumptions for α and E_g
Ellipsometry	Thickness, n , k	Å–µm	Non-contact and	Correlated model

			sensitive	parameters
Four-point/Hall	Resistance, carrier density, mobility	Film/device scale	Direct functional information	Requires appropriate contacts/geometry

PROCESS-STRUCTURE-PROPERTY RELATIONSHIPS

The core science problem of thin film research is the linking of processing variables to structure and thus to device performance. A good schematic is Process parameters nucleation & growth microstructure intrinsic properties device performance. For instance, the temperature in the deposition influences the mobility of the surface and so the grain size and orientation; grain boundaries scatter carriers; the scattering changes the resistivity; and the resistivity affects the transparency, conductive or semiconductor device. Similar sequences can be found for optical absorption, catalytic properties and mechanical wearing of surfaces. This relation is generally nonlinear. For instance, a film may have greater crystallinity with greater roughness. It may be more conductive yet less optically transparent, or harder yet more brittle.

APPLICATIONS

Thin films serve multiple purposes as active, passive or protective layers. In the field of electronics, they can be conductor’s semiconductors dielectrics, barriers and magnetic films. For optoelectronics, thin films are mostly used to alter such characteristics as reflection absorption emissions and refractive index. To be efficient at photovoltaics thin absorber buffer transport and transparent-conducting layers need to be connected through well-controlled interfaces. Transparent conductive films such as ITO and AZO show that it is necessary to combine optical transparency and low electrical resistance together; a comparison of deposition methods and materials for these applications is made in a number of recent reviews [Patel, J. *et al.*, 2024; Sharme, R. K. *et al.*, 2024].

Thin films, in energy storage, are used as electrodes, solid electrolytes, protective layers and model interfaces. Plasma-assisted PVD and CVD techniques are promising for energy-conversion and storage materials since plasma chemical reactions can offer the possibility of low temperature processing and the control of surface morphology and the presence of defects. For biomedical and protective applications, sputtered coatings can be tailored to show a high level of

corrosion resistance, wear resistance and biocompatibility [Garg, R. *et al.*, 2024].

EMERGING TRENDS

In-Situ and Operando Metrology

The future for thin film processing involves a greater use of measurement at the deposition tool level. Real time feedback is provided by such in-situ measurements as optical emission spectroscopy, quartz-crystal microbalance, in-situ ellipsometry and residual gas analysis. With less reliance on characterization after process is finished the process reproducibility is likely to be enhanced.

Machine-Learning-Assisted Optimization

Machine learning offers itself as another way to relate deposition parameters to thickness, composition, and functional properties. In just the last few years, there has been successful use of machine-learning prediction of parameters for thin-film dielectrics and active learning on composition mapping in automated magnetron-sputtering systems. A crucial methodological constraint, however, is that models based on machine learning must rely on a well-designed, traceable data set, not upon supplanting physical insight.

Sustainable Processing

Environmental influences are also increasingly prevalent and the design aims for lower consumption for deposition in the future, lower solvent usage, less toxic precursors, more efficient material utilization and longer coating life. The life-cycle economic performance per unit of functional quality, rather than mere deposition cost, is the benchmark.

Large-Area and Flexible Substrates

The challenges of large area scaling include uniformity, temperature gradients, gas flow profiles, target erosion, and precursor delivery and mechanical handling. Furthermore, polymer substrates place restrictions due to operating temperatures, mechanical fragility, and residual stress. Efforts to produce films of sufficient size and homogeneity for industrial implementation have recently been made using PLD, sputtering, and solution methods.

Hybrid and Multilayer Architectures

Designs of properties at the interface offer routes through the manipulation of heterostructures and multilayers. While ALD offers suitable conforming ultrathin layers, sputtering, CVD achieve throughput and thus faster film deposition for desired properties. These techniques can be combined so that strengths of one can overcome limitations of another- e.g. High throughput bulk layer coupled with an ultrathin ALD interface or barrier.

COMMON SOURCES OF ERROR AND REPORTING RECOMMENDATIONS

Failure to include process variables critically determines the difficulty of reproducing the study of thin films. To be acceptable by at least the lowest common denominator researchers authors will, report substrate material and cleansing procedure, purity of film precursor or target material, the chamber base pressure, working pressure, gas used in chamber and its pressure, the type and operating regime/power of the target material source or source temperature, substrate temperature, the length or rate of the film deposition, substrate-source distance, the film thickness, post deposition annealing temperature and method (if done) and the geometry of the measurement (if appropriate). Characterisation must provide uncertainty limits where it is achievable.

XRD must clearly mention any instrumental broadening and stated assumptions from peak fitting. SEM/AFM require good scale bars, ideally averaged from several positions. The assumed transition, fitting range used and (if relevant) error estimates when determining the optical bandgap should be quoted. Contact geometry, dimensions of sample and any environmental conditions are needed for electrical measurements. Crucially, avoid over-interpreting a single measurement technique. For example a broad XRD peak is NOT, on its own, indicative of a nano crystalline film. The EDS atomic percent is NOT necessarily representative of the bulk atomic percent. The Tauc intercept is not necessarily the fundamental electronic bandgap and a single arbitrary measurement of AFM roughness is NOT guaranteed to be representative.

CHALLENGES AND FUTURE RESEARCH DIRECTIONS

Reproducibility between various laboratories and between different deposition methods remains one

of the most serious issues since recipes that appear to be identical at first glance can result in quite different plasma settings, gas-phase precursor's deliveries, surface conditions and even temperature profiles. Design of the interface has become increasingly a key thing with advancement towards 3-D device structures and decreasing dimensional scale. As a result, characterization techniques need not only to provide better spatial resolution but also to be capable of identifying hidden interfaces effectively.

The problem of managing the residual stress and controlling defects is still the main issue for film reliability. A film which shows an excellent performance straight after the deposition may be prone to degradation under thermal cycling, exposure to humidity, electrical bias or mechanical strain. It is necessary that the metrology techniques follow the evolution of ultrathin and multi-layered structures. For example, in a scenario of model-based analysis, factors like layer thickness, degree of surface roughness, atomic composition, as well as optical properties may interconnect in a non-linear manner, which should be carefully taken into consideration. Upscaling processes should ensure that the microstructural features found in lab-scale deposition are still preserved in larger systems. To achieve this reactor models, even precursor/plasma flows, statistical process control, as well as more and more automatic feedback devices will be needed. Data driven optimization is becoming a crucial part of research and development, but in order for models to be trusted and valid, data must be collected in a standardized way, metadata uncertainty must be assessed, and models must be based on physical properties that describe meaningful and quantifiable descriptors.

CONCLUSION

Coating the fabrication of the thin film and characterization in total is basically an integrated scientific and engineering endeavor which includes the deposition conditions nucleation microstructure, interfaces and functional properties. PVD methods viz. evaporation, sputtering and PLD allow tight control on material flux and are very good in making dense coatings and films of complex compositions. CVD and PECVD provide scalability and conformity in growth while ALD through self-limiting reactions can be exceptional in controlling ultrathin conformal layers. Solution-based methods viz sol, gel, spin coating, spray pyrolysis and CBD are very nice alternatives as very simple low-cost and

large-area techniques in the production of oxide and semiconductor films.

Only multiple techniques combined together will result in good characterization. XRD/XRR give information on structure while SEM/AFM/TEM display morphology and interfaces ; EDS/XPS are used to check composition and chemical states; Raman/FTIR are very effective tool in detecting bonding and vibrational structure; UV-Vis, PL and ellipsometry measure optical response; and four-point/Hall testing establish electrical performance. It is only when such measurements are jointly interpreted in a context of process-structure-property that the most solid conclusions are derived.

The main trend is atomic deposition, development of plasma-assisted low-temperature processes, large-area fabrication, in-situ measuring techniques, environmentally conscious processing and AI-assisted optimizations. Still, the key is not so much to produce more coating technology, as in fact to design the process so as the structure and performance of the film can be deliberately chosen. This way, one can say that the primary opportunity is not in expanding the number of deposition techniques, but rather, in developing process windows that are reproducible, measurable and scalable in which the structure and performance of the film could be designed deliberately.

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