



Atomic Layer Deposition Process Development

A 10-step workflow for systematically developing, optimizing, and characterizing ALD processes ¹

Purpose of this guide

Atomic layer deposition (ALD) is a surface-controlled thin-film deposition method based on sequential, self-limiting surface reactions. It offers precise control of film thickness and can provide excellent uniformity over large substrates and conformality on complex three-dimensional structures. A typical ALD cycle consists of a precursor dose and a co-reactant exposure, separated by purge or pump steps. Repeating the cycle builds the film to the desired thickness.

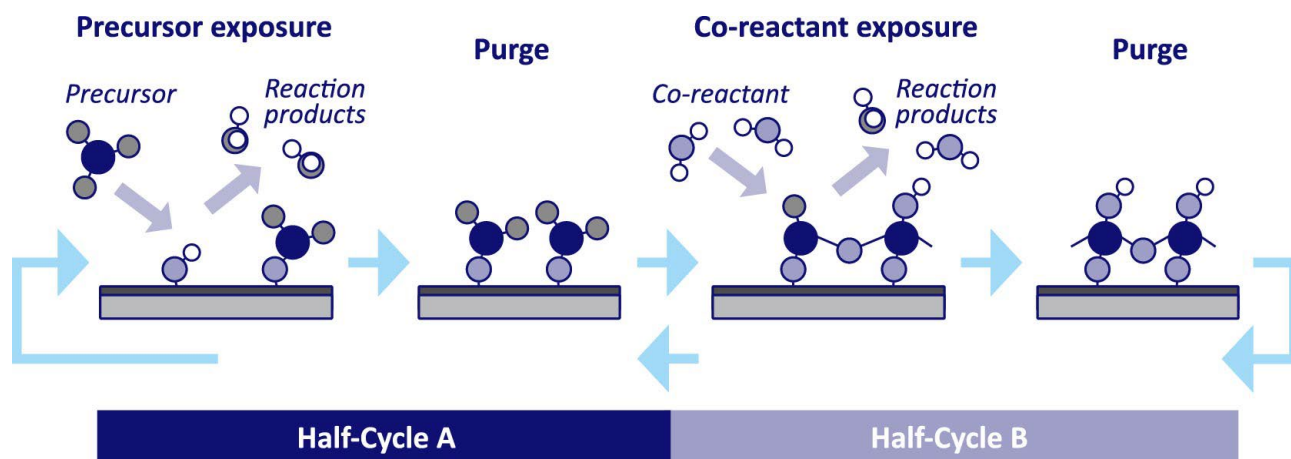


Figure 1: Schematic illustration of a typical ALD cycle consisting of two half-cycles. Sequential precursor and co-reactant doses are separated by purge steps, while self-limiting surface reactions control the amount of material deposited in each cycle.

The basic principle is straightforward, but developing a reliable ALD process is not. Reactant chemistry, dosing, purging, temperature, substrate condition, reactor design and film characterization are closely connected. This guide presents a practical 10-step workflow for developing, optimizing and verifying an ALD process. It is intended especially for researchers who are new to ALD, while also providing a concise checklist for more experienced users.

For a comprehensive introduction to ALD, see: Erwin Kessels, Anjana Devi, Jin-Seong Park, Mikko Ritala, Angel Yanguas-Gil, and Claudia Wiemer, "Atomic layer deposition," [Nature Reviews Methods Primers 5, 66 \(2025\)](#). This guide complements the Primer by focusing specifically on the practical sequence of film growth experiments used to establish conditions for depositing a desired film in a repeatable manner.

The workflow also encourages comprehensive and consistent reporting of experimental conditions, characterization methods, and results. In this way, it can serve as a first step toward a community protocol for generating the structured, high-quality data needed to populate a future AI-ready ALD database.

¹ 2026 revision by Cas Visser, Eleni Poupaki, Adrie Mackus, and Erwin Kessels; based on the 2019 version by Martijn Vos, Adrie Mackus, and Erwin Kessels



The 10-step workflow

The steps below (see also Figure 2) are roughly chronological, but the order is a suggestion rather than a universal guideline. The appropriate sequence depends on the material, process, reactor, availability of tools and intended applications. Also, iteration is often required. Results obtained in later steps may therefore require an earlier step to be revisited.

1. **Reactant selection:** Which precursor and co-reactant will be used?
2. **Initial composition check:** Did the first experiments produce approximately the intended film composition?
3. **Thickness control:** Does the deposited amount of material increase linearly with the number of cycles?
4. **Saturation:** Are the surface reactions during precursor dosing and co-reactant exposure self-limiting, and are the purge times sufficient?
5. **Temperature dependence:** Over which substrate-temperature range is self-limiting ALD behavior obtained?
6. **Detailed composition and material properties:** What are the detailed film composition and material properties, and do they meet the requirements?
7. **Uniformity:** Are the film thickness and relevant properties uniform across the substrate?
8. **Nucleation behavior:** How does growth during the initial cycles differ from steady-state growth?
9. **Conformality:** How uniformly are the film thickness and relevant properties distributed throughout three-dimensional structures?
10. **Practical implementation and validation:** Is the process safe, stable, reproducible, scalable, and compatible with its intended use?

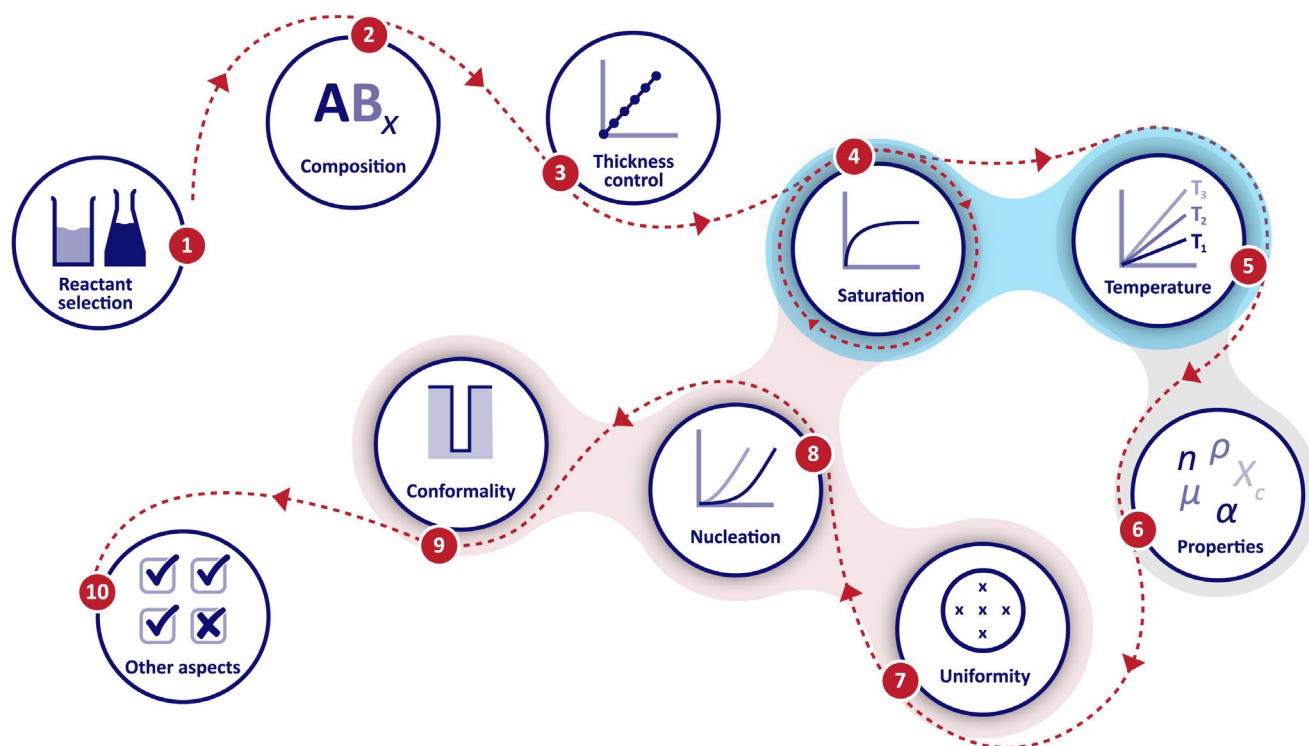


Figure 2: Workflow for the 10-step ALD process-development protocol. The colored circles highlight interdependencies for which it may be necessary to revisit an earlier step. Blue indicates the temperature dependence of saturation behavior, grey the strong influence of temperature on film properties, and red that observations related to uniformity, nucleation, or conformality may warrant reassessing the saturation behavior.



Step 1. Reactant selection

Before setting up an ALD process, select a suitable combination of precursor and co-reactant. The reactants must provide the elements needed for the intended film and must generate a viable sequence of surface reactions: each reactant should react with the surface groups created by the preceding half-cycle and leave the surface in a state that enables the next reaction. Suitable volatility, thermal stability and reactivity of the reactants are essential. The likely reaction products should also be considered because stable or strongly adsorbing by-products can complicate purging, incorporate impurities or inhibit subsequent reactions.

Carry out a literature survey before starting the process development. The [AtomicLimits ALD database](#) is a practical starting point for identifying previously reported precursor and co-reactant combinations, deposition temperatures and delivery approaches, while the [AtomicLimits ReviewBase](#) can help locate relevant review publications. Even when a material has not been deposited previously, information on related chemistries, precursor design, surface reactions and decomposition pathways can guide the selection.



The best chemical combination is not determined by reactivity alone. Consider precursor and co-reactant availability, purity, cost, safety, storage stability and compatibility with the substrate and intended application. Reactor constraints can limit the possible reactants or delivery methods.

Determine whether the precursor will be delivered by vapor draw, carrier-gas-assisted delivery, bubbling, direct liquid injection or another method, and whether the source, valves, lines and exhaust can be operated safely at the required temperatures and pressures. Consider harmful reaction products, and the safety of the deposited material (see also Step 10). Precursor consumption and precursor stability (especially during prolonged heating) may also influence the precursor choice.

Before the first film-growth experiments, select and document the substrate, an approximate film-thickness range and a suitable fixed starting substrate temperature for Steps 2–4. Select this temperature based on prior literature and database information, reactant volatility and thermal stability, reactor and substrate limitations and the intended application. It is generally advisable to avoid the expected lower and upper limits of the process-temperature range. If little information is available, approximately 250 °C can be a practical starting point for many inorganic ALD processes, provided that this temperature is compatible with the reactants, substrate and reactor. The temperature dependence is subsequently investigated systematically in Step 5. Clearly report whether the stated temperature refers to the programmed setpoint, reactor-wall or substrate-holder temperature or an independently measured or calibrated substrate temperature.



Step 2. Initial composition check

Soon after depositing the first measurable film, perform a rapid check to determine whether the process has produced approximately the intended material. The purpose of this step is straightforward: verify that the expected elements are present and/or that one or more readily measured properties are broadly consistent with the intended material. This is not yet the comprehensive analysis of film composition and properties described in Step 6.

X-ray photoelectron spectroscopy (XPS) can be used for this initial check, but quick proxy measurements may also provide useful warning signals. For example, electrical conductivity may help distinguish a reasonably pure conducting film from an insulating or highly contaminated film, while the refractive index can indicate whether a film is substantially different from the expectations.

If the result is fundamentally different from the target, revisit Step 1 and check reactant delivery, substrate temperature, substrate preparation, reactor conditions and its history and possible cross-contamination before investing in detailed optimization. Less severe deviations may be addressed during subsequent optimization by adjusting the dose, purge or temperature conditions. Repeat the initial check after substantial recipe changes, because film composition and impurity levels may shift during optimization.



Step 3. Thickness control

A central advantage of ALD is predictable control of the deposited amount through the number of cycles. Determine the film thickness, areal density or mass gain for a series of cycle numbers and derive the growth per cycle (GPC) from the slope in the steady-growth regime, where the deposited amount should increase linearly with cycle number. Depending on the measurement, report the GPC as thickness per cycle, atoms per unit area per cycle or mass per unit area per cycle. Avoid using the term “growth rate” when referring to GPC.

Growth can be determined *ex situ* from a series of films deposited using different cycle numbers or followed *in situ* during deposition. Spectroscopic ellipsometry and X-ray reflectometry are commonly used to determine thickness, Rutherford backscattering spectrometry (RBS) can determine areal density and a quartz-crystal microbalance (QCM) can follow mass gain. Select a thickness and cycle-number range over which the measurement is reliable and steady growth dominates. As a practical starting point, films thicker than approximately 10 nm can reduce the relative influence of the initial growth regime. However, the appropriate range depends on the material and measurement technique, as well as the onset of thickness-dependent changes such as crystallization, densification or increasing surface roughness.

A linear increase with cycle number is important for predictable thickness control, but linearity alone does not demonstrate an ALD mechanism. Nucleation delay, substrate-enhanced growth or other thickness-dependent growth behavior may occur during the initial cycles and are investigated in Step 8. Report the individual data points, fitted cycle-number range, slope and uncertainty rather than only a single GPC value.

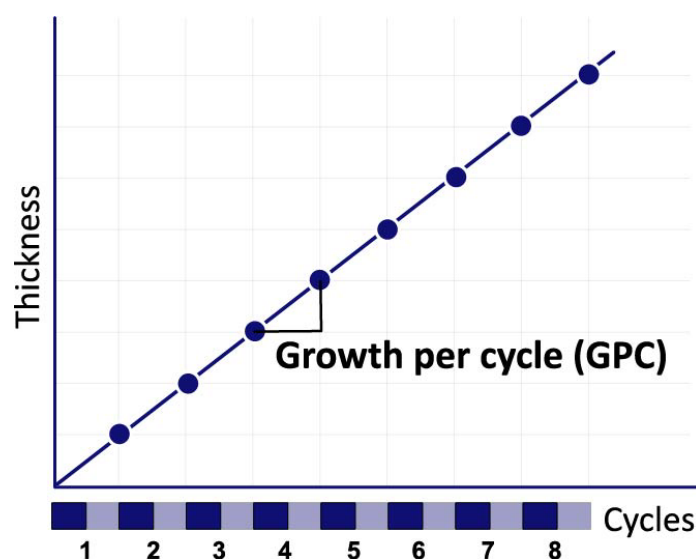


Figure 3: Film thickness as a function of the number of ALD cycles, illustrating linear growth. The growth per cycle (GPC) is determined from the slope of the linear fit.



Step 4. Saturation

Assess whether the surface reactions are self-limiting by determining the GPC and, where relevant, film composition or properties as a function of reactant dose and purge time. For a standard AB-type process, investigate the precursor dose, precursor purge, co-reactant exposure and co-reactant purge. Initially use sufficiently long conditions for three of these four parameters while varying the fourth. Repeat this procedure iteratively, because the four conditions are interdependent and changing one may require the others to be re-evaluated. Include zero-dose experiments in which either the precursor dose or the co-reactant exposure is excluded.

A plateau within experimental uncertainty indicates that increasing the reactant dose or extending the purge time does not materially change the measured result. Where possible, show all individual data points together with replicate measurements or error bars, and explain the criterion used to identify saturation or adequate purging. GPC alone may not reveal all changes; film composition or other relevant properties should also be re-evaluated whether these vary with dose or purge conditions.

Where possible, characterize reactant delivery and exposure at the substrate rather than relying solely on valve-open time. Reactant pressure and flow, reactor volume, pumping speed, delivery geometry and total substrate surface area all affect the amount of reactant reaching the surface. Conditions that saturate a sample near the centre of a reactor may not saturate a large substrate or high-aspect-ratio structures. This should therefore be checked during steps 7 and 9.

Deviations from an ideal plateau may arise from condensation, parasitic CVD (e.g., precursor decomposition), by-product readsorption, insufficient purging or dose- or exposure-dependent changes in film composition or properties. The terms “slow saturation” or “soft saturation” are commonly used to describe this behavior. An insufficient precursor or co-reactant exposure (referred to as under-saturation) may also result in incomplete reactions and increased impurity levels, whereas unnecessarily long doses and purges reduce throughput. Zero-dose experiments can reveal non-ideal growth contributions and may also provide information about etching or substrate modification caused by an individual reactant in the absence of a complete ALD cycle.

Saturation and enough purging should first be established at the selected fixed starting temperature. They should be re-evaluated when the substrate temperature, substrate geometry or other relevant process conditions change substantially.

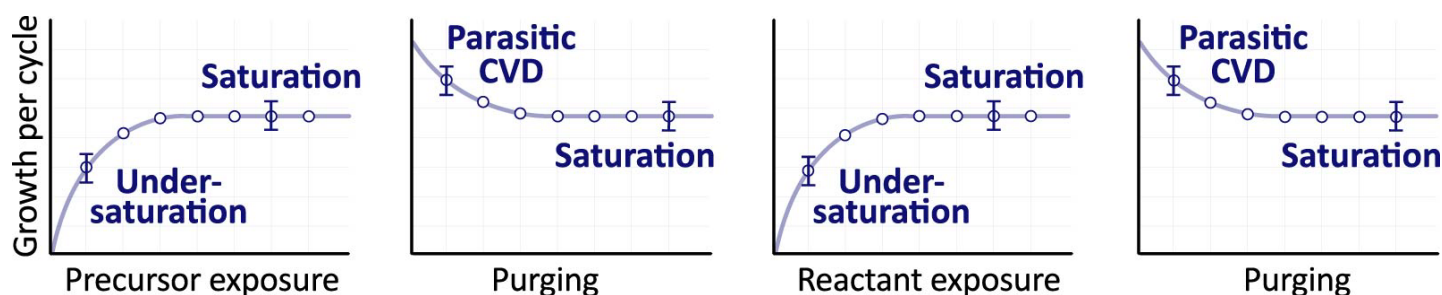
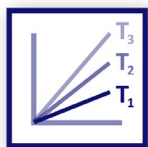


Figure 4: Typical saturation curves for an ALD process showing, from left to right, the effects of precursor exposure, precursor purge time, co-reactant exposure, and co-reactant purge time. It is important to indicate the uncertainties in the data; for simplicity, they are shown here for selected data points only.



Step 5. Temperature dependence

After Steps 2–4 have been established at a fixed starting temperature, investigate its dependence on different substrate temperatures. Deposit films over a relevant temperature range. Consider reactant volatility and thermal stability, substrate and reactor limitations, film requirements, and the intended application. A generic range such as 50–350 °C may be useful, depending on the chemistries, but should not be treated as a requirement.

The ALD window is the substrate-temperature range over which the surface reactions remain self-limiting under the selected process conditions. Contrary to a common belief, the GPC does not need to be constant within this window. Temperature-dependent changes in surface-group density, adsorption, desorption, and reaction pathways can cause the GPC to vary while the surface reactions remain self-limiting, as shown in Figure 5. A GPC-versus-temperature curve alone therefore does not establish the ALD window. Repeat the saturation experiments, or at least the most critical precursor- and co-reactant-dose experiments, at application-relevant temperatures and/or near the lower and upper limits of the investigated range.

At low temperatures, non-ideal behavior may arise from incomplete or slow surface reactions, condensation or slow desorption of reaction by-products. At high temperatures, deviations may result from precursor decomposition or desorption of reactive surface species. Film composition, structure and other relevant properties may also change substantially with temperature and should therefore be investigated in more detail in Step 6.

Also keep in mind to distinguish the set temperature programmed in the software from the actual substrate temperature. The two may differ substantially, particularly in warm-wall reactors, because of factors such as thermal contact with the substrate holder, process pressure, substrate thickness, reactor configuration and wall temperature. Allow sufficient time for thermal equilibration. Report how the temperature was controlled and measured or calibrated.

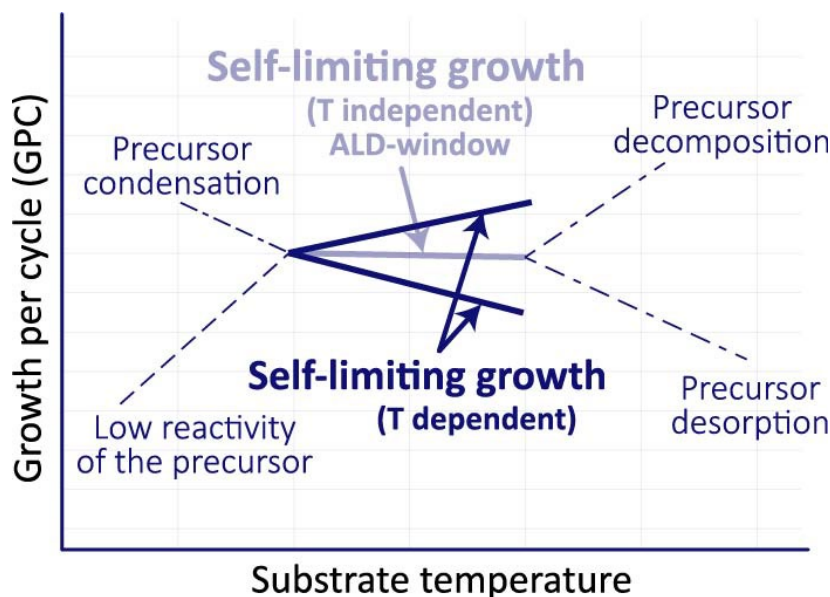


Figure 5: Growth per cycle (GPC) as a function of deposition temperature, illustrating both an idealized temperature-independent GPC and temperature-dependent GPC trends within an ALD window. In either case, the surface reactions must remain self-limiting.



Step 6. Detailed composition and material properties

Once suitable growth conditions and a relevant temperature range have been identified, characterize what material was produced and whether it meets the intended requirements. Revisit the composition in much more detail compared to in Step 2. Depending on the material, investigate the stoichiometry, impurities, chemical bonding, depth distribution and interface composition. Evaluate the film properties in relation to its composition and structure. Relatively small changes in impurity levels, stoichiometry, density or crystalline phase can lead to substantial changes in electrical, optical or mechanical properties.

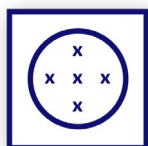
The appropriate characterization depends on the material and application. The table below lists representative properties and techniques. Keep in mind that individual techniques have different depth sensitivity, spatial resolution, assumptions and detection limits. Whenever possible, combine complementary methods rather than relying on a single measurement.



Property or characteristic	Representative technique(s)
Elemental composition, stoichiometry and impurities	XPS, XRF, RBS, ERD, NRA, SIMS
Chemical bonding and oxidation state	XPS, FTIR, Raman spectroscopy
Thickness and areal density	Spectroscopic ellipsometry, XRR, profilometry, TEM, RBS
Mass density and porosity	XRR, RBS combined with thickness, porosimetry
Surface morphology and roughness	AFM, SEM, TEM, XRR
Crystallinity, phase, texture and microstructure	XRD/GIXRD, TEM/SAED, Raman spectroscopy, EBSD
Optical properties	Spectroscopic ellipsometry, UV-visible spectroscopy, photoluminescence
Electrical and dielectric properties	Four-point probe, Hall measurements, capacitance-voltage and current-voltage measurements
Mechanical properties and residual stress	Wafer-curvature measurements, nanoindentation

XPS = X-ray photoelectron spectroscopy; XRF = X-ray fluorescence spectroscopy; RBS = Rutherford backscattering spectrometry; ERD = elastic recoil detection; NRA = nuclear reaction analysis; SIMS = secondary ion mass spectrometry; FTIR = Fourier-transform infrared spectroscopy; XRR = X-ray reflectometry; TEM = transmission electron microscopy; AFM = atomic force microscopy; SEM = scanning electron microscopy; XRD = X-ray diffraction; GIXRD = grazing-incidence X-ray diffraction; SAED = selected-area electron diffraction; EBSD = electron backscatter diffraction.

Characterize films at relevant thicknesses and deposition conditions. Some film properties depend on thickness. In addition, air exposure or sample preparation may alter the composition, structure or properties of films. Report the measurement method and settings, calibration, analysis model, number of samples measured, uncertainty, error bars and detection limits. Where values are inferred through a model, state the assumptions and avoid reporting more precision than the measurement supports.



Step 7. Uniformity

Uniformity over large substrates or across a substrate batch when doing a run with multiple wafers is an important merit of ALD and a sensitive process diagnostic. Saturation measured on a small sample at the center of the substrate table does not demonstrate that all positions in the reaction chamber receive sufficient precursor and co-reactant dosing or purging. Non-uniformity can indicate insufficient dosing, precursor depletion, temperature gradients, by-product effects, parasitic CVD or limitations of the reactor and delivery geometry.

Deposit on the largest representative substrate or batch that the reactor can handle and map thickness at a sufficient number of clearly defined positions. When relevant, also map composition, resistivity,



refractive index, crystallinity or other properties when applicable. Moreover, good thickness uniformity does not guarantee uniform material properties. Define the uniformity metric explicitly and report the mean, range or standard deviation together with measurement uncertainty.

Uniformity is listed as Step 7, but it is often useful to check uniformity at an earlier stage and again after major changes in the recipe or temperature. Poor uniformity may require returning to Steps 4 and 5 or reviewing reactor operation and conditioning. Distinguish within-substrate uniformity from run-to-run repeatability and system-to-system repeatability, which is addressed in Step 10.



Step 8. Nucleation behavior

Growth during the initial ALD cycles can differ from steady growth because the precursor reacts with the starting substrate and its surface groups rather than with the deposited film. Depending on the substrate and chemistry, growth can initially be approximately linear, accelerated or delayed, as shown in Figure 6. Determine this behavior using a series of low-cycle samples or an *in situ* method with sufficient sensitivity and cycle resolution, such as spectroscopic ellipsometry.

Nucleation affects film closure, crystallinity, resistivity and other properties of ultrathin films. Island or Volmer-Weber-type growth can precede formation of a closed film, whereas other systems may approach layer-by-layer growth. A change in structure at a critical thickness, such as crystallization, can also change the apparent GPC after nucleation. Differences between starting surfaces may be undesirable for conventional deposition or rather beneficial for area-selective ALD applications.

Report the substrate used, pre-treatment, chamber conditioning and storage of the sample, as nucleation is especially sensitive to surface history. State the thickness or areal-density method, its detection limit and the fitting method used to distinguish initial growth from steady growth. Do not interpret the nucleation curve without considering measurement sensitivity, surface roughness and the possibility of island formation, which are not well represented by a continuous-film optical model.

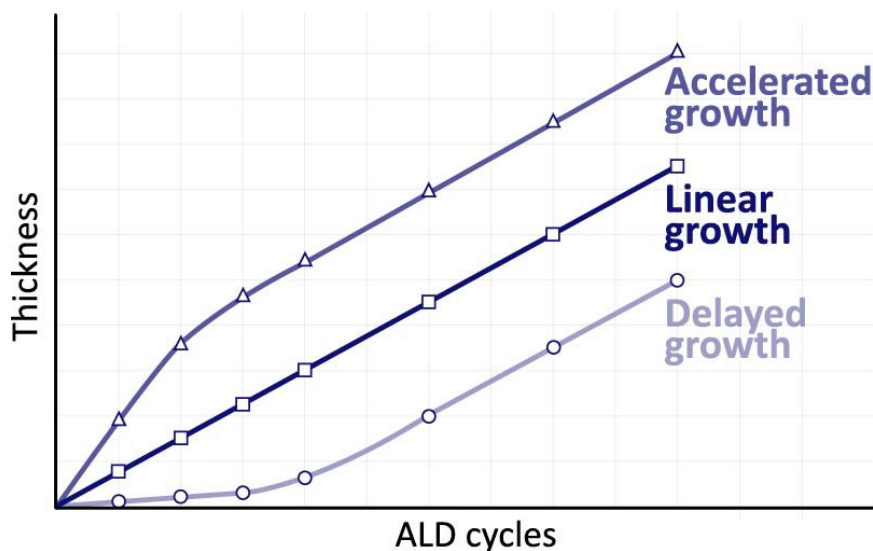


Figure 6: Thickness as a function of number of ALD cycles, illustrating accelerated, linear, and delayed growth during the initial cycles.



Step 9. Conformality

Conformality is the ability to deposit a film with uniform thickness, ideally with uniform composition and properties, throughout a three-dimensional structure shown in Figure 7(a). It is a defining practical advantage of ALD and should be experimentally assessed at a level appropriate to the intended application. A process intended for planar substrates may require only basic verification, whereas applications involving high-aspect-ratio structures require more tests. Claims about conformality should be limited to the structures and experimental conditions investigated. If suitable test structures are not available, no general claim of conformal growth can be made.

Conformality can be evaluated using vertical trenches or vias, followed by cross-sectional analysis using scanning electron microscopy (SEM) or transmission electron microscopy (TEM). Dedicated [lateral high-aspect-ratio test structures](#), such as PillarHall chips from Chipmetrics shown in Figure 7(b), make it possible to measure thickness profiles over long lateral distances after opening the structure, often without preparing conventional cross-sections. Use structures with known geometries and report their relevant dimensions, aspect ratios and surface areas

Report film thickness or other relevant quantities as a function of position within the structure rather than providing only a single top-to-bottom ratio. Define any conformality or penetration metric precisely and report the measurement uncertainty.

Reactant transport, surface-reaction or sticking probabilities, ozone decomposition, recombination of plasma-generated radicals, by-product effects and total exposed surface area are all limitations for the penetration into demanding structures. Increasing the reactant exposure or using stop-flow, multiple-dose or multiple-pulse strategies may improve conformality. Saturation, adequate purging and throughput should then be reassessed under these modified conditions. Poor conformality may require the dose and purge conditions established in Step 4 or the substrate temperature investigated in Step 5 to be revisited.

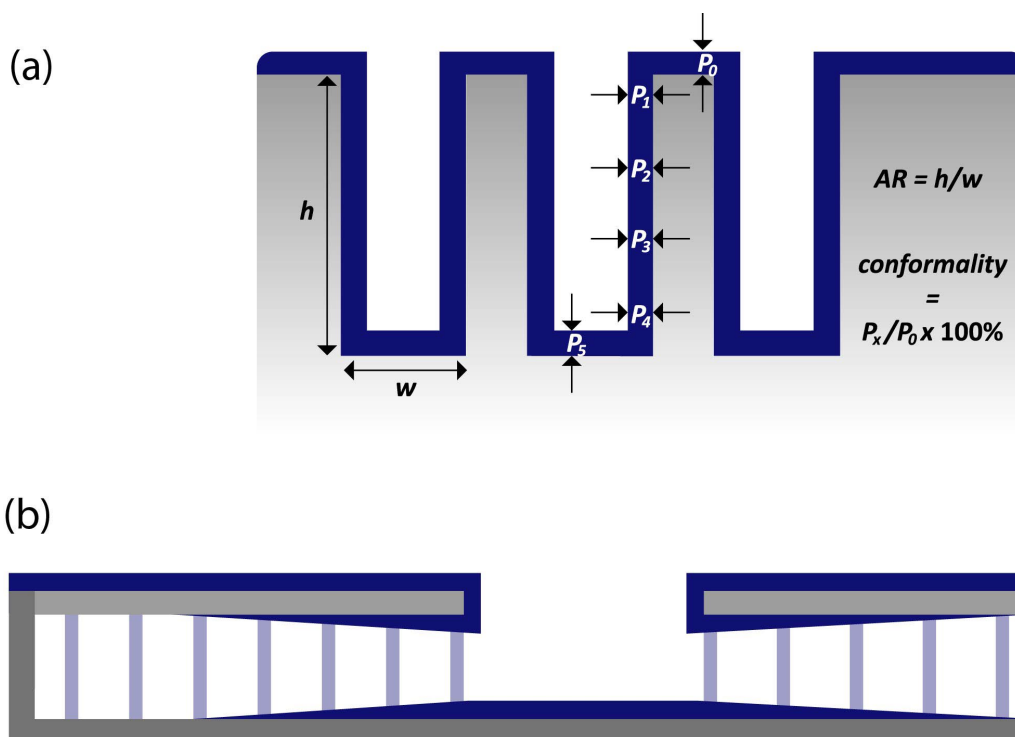


Figure 7: (a) Schematic representation of vertical trenches. Conformity is defined as the local film thickness at different positions within a trench relative to the thickness on the top surface. (b) Schematic representation of a lateral trench, known as a PillarHall structure. After deposition, the top membrane can be removed to measure the thickness profile and film properties along the trench.



Step 10. Practical implementation and validation

A process is not complete when favorable data has been obtained only once on a small wafer or a large substrate. It must also be safe, stable and reproducible, and it should be assessed under conditions that represent its intended use. It is recommended to address the following aspects throughout process development and consolidate the findings during validation of the full process:

- **Safety and by-products:** Confirm safe handling, storage, delivery and disposal of all reactants. Identify possible hazardous reaction and decomposition products associated with the deposited film, cleaning and maintenance.
- **Film and interface stability:** Assess changes during storage, air exposure, humidity, temperature cycling, annealing, chemical exposure or other relevant environments.
- **Run-to-run and batch-to-batch repeatability:** Repeat the recipe on different days or substrate batches. Report variation in thickness, composition and key properties, not only the average result.



- **Reactor history and process robustness:** Document wall conditioning, possible memory effects, cross-contamination, precursor residues, cleaning and maintenance periods.
- **Process transfer and tool matching:** When transfer matters, compare the process on another nominally identical or different reactor. Expect that dosing and purging conditions depend on reactor geometry, reactant delivery and pumping. Compare actual substrate temperature, GPC, composition and properties rather than just the difference in dosing/purging times.
- **Precursor delivery, stability and use:** Verify stable delivery over time and monitor possible precursor source ageing or supply. Record the supplier and batch where relevant and quantify or estimate the consumption. Avoid unnecessary overdosing while retaining saturation.
- **Throughput and scalability:** Evaluate cycle time, productivity, precursor utilization, and scale-up to larger substrates or batches. Consider the trade-off between throughput and the process margins required to ensure saturation, uniformity, and conformality. In this context, the growth rate expressed as thickness per unit time may be more relevant compared to the GPC alone.
- **Integration:** Test compatibility of the process with substrate preparation, underlying layers, vacuum breaks, post-deposition treatment and other subsequent process steps. Check whether integration changes interfaces, composition, defects or film properties.
- **Application or device validation:** Evaluate the film in the relevant structure, environment or device corresponding to the application. Interface states or localized defects that are hard to measure in other characterization techniques may become visible only in application-level tests.
- **Reporting of the final data:** Archive the complete recipe, metadata, raw and processed data, uncertainty analysis, unsuccessful and unexpected results and the criteria used to select the final process. A concise process summary should link to the underlying experimental records rather than replace them.

Using the workflow

The workflow should be used systematically without being treated as rigid. A well-developed ALD process is supported by corresponding evidence: the intended material is formed - the deposited amount is predictable - the surface reactions are self-limiting under relevant conditions - composition and properties are understood - uniformity, nucleation and conformality have been assessed - and the process is reproducible and suitable for the respective application. This requires that the complete experimental and analytical context should also be recorded and reported.

A shared protocol does not guarantee direct comparability between laboratories, but it is a practical first step towards complete and uniform ALD data. Community-agreed terminology and reporting templates can build on this workflow to support structured, AI-ready ALD databases.



Appendix: Documenting ALD process development

A process-development experiment is only interpretable and reusable when its experimental and analytical context is known. Report not only the result, but also the information needed to understand how it was obtained, assess its uncertainty and reproduce it. Where possible, retain both the raw and processed data. These documentation practices also help lay the groundwork for ALD process-development data that follow the FAIR principles: Findable, Accessible, Interoperable and Reusable.

A first step toward a protocol for populating an ALD database

The 10-step workflow identifies which aspects of an ALD process should be systematically investigated, while the documentation requirements below identify the experimental and analytical context that should be recorded. Used together, they can serve as a first step toward a community-driven protocol for generating complete, consistent and reusable datasets for the inclusion in a future AI-ready ALD database.

The present guide does not yet define a complete standard for database submission. A reporting template and database schema will be needed in the future to specify mandatory and optional fields, controlled terminology, units, identifiers, data formats and submission procedures.

For each process-development experiment, at minimum, document:

- Objective and decision context: the purpose of the experiment, the parameter being tested, the target material and properties, relevant constraints, and the criterion used to decide the next experiment.
- Reactor and run identity: reactor type and configuration, date, run identifier, operator where relevant, chamber history, conditioning or cleaning, maintenance status, substrate loading and sample position.
- Substrate information: material, supplier or batch where relevant, dimensions, orientation, surface termination, pre-treatment, cleaning and storage
- Precursor and reactant information: chemical identity, supplier, grade or purity, batch where relevant, precursor state and source temperature, delivery method, carrier-gas conditions, line temperatures and information about storage or ageing.
- Complete recipe: sequence of all steps, dose or exposure times, purge or pump times, gas flows, pressures, number of cycles and temperature information. For plasma-enhanced ALD also include the plasma source, plasma power, frequency, gas composition, pressure, exposure time, and substrate bias conditions where applicable.
- Measurement information: analytical technique, instrument and relevant settings, calibration and analysis model, sample location where relevant, film thickness at characterization, sample transfer or air exposure, and any data-processing choices.



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- Results and data quality: numerical values with units, replicate measurements, uncertainty or error bars, detection and quantification limits where relevant, treatment of background signals and outliers, and clear distinction between measured and derived quantities.
- Complete outcomes: successful, unsuccessful, negative and unexpected results; deviations from the intended protocol; observations made during the run; and the reasoning behind the next experimental step.

The required details depend on the experiment, but reporting should be sufficiently complete that another researcher can interpret the result without relying on undocumented knowledge of the local reactor or analysis procedure. Consistent terminology, units, sample identifiers and file naming greatly improve the value of the resulting dataset.