CORAL Name: ALD-Plasma-Arradiance-XTDP

Location: 8U
Category: Deposition
Contacts: Donal Jamieson, Cody Corey, Elham Borujeny

Table of Contents

Introduction 2
Safety 2
User Qualifications and Responsibilities 3
Operating Procedures 3
Degassing Precursor ALD Valves (minimum of 15 cycles followed by single-pulse check): 3
Venting the Loadlock: 4
Pumping the Loadlock: 5
Transferring Sample to Chamber: 5
Running a Recipe: 6
Transferring Sample to Loadlock: 6
Before You Go, Remember To 6
Degas Precursors (15 cycles followed by single-pulse check): 6
Degas Precursors (150 cycles): 7
Record Process Data: 7
Appendices 7
Appendix A. 7
MIT.nano Recommended Process Parameters for ALD-Arradiance Tools 7
Appendix B. 7
A Quick Guide to MIT.nano ALD Tools: Overview, Tool Selection and Cleanliness 7

Introduction



The Arradiance Atomic Layer Deposition (ALD) tool is capable of depositing nitride and oxide thin films by sequential and repeated pulsing of organometallic precursor vapors and oxidizing/plasma reactants. These precursors/reactants must be thoroughly purged and evacuated from the system after each pulse to avoid parasitic or unwanted reactions. Maintaining extreme cleanliness (including cleanliness of the tool and the substrates) is an essential factor in producing reproducible and high-quality films by ALD. Arradiance-XTDP benefits from a loadlock loading system which maintains better process chamber cleanliness. The plasma ALD availability of the tool allows for lower temperature deposition processes to be performed. The tool is intended for the deposition of high-quality thin films for Electron Devices research. The chamber accepts all size substrates up to 8 inches. Please consult the MIT.nano staff for the allowed substrates and process conditions as well as the films currently available.

Safety

- Do not try and defeat any interlocks on the system – high temperatures, RF power, and dangerous chemicals are contained within the system.
- When loading and unloading samples, make sure not to touch the surface of the wafer holder to prevent contaminating the process chamber. Furthermore, the holder will be hot when removed from the chamber and user burns would result. Allow the hot holder to cool at atmosphere.
- Ensure loadlock lid seal is clear when closing lid. Be careful not to allow the lid to fall shut, improper sealing or user injury could result.
- All organometallic precursors used in this system are extremely dangerous and may react violently if exposed to air.
- Precursors must not be overheated (this includes heating the precursor to higher than the allowed temperatures or leaving any precursor heated for extended periods exceeding a day even at allowed temperatures) as they will degrade and may also become liable to over-pressurization.
- Refer to Appendices for a list of precursors and possible process parameters on this tool. Note that the use of any of the precursors or process parameters is subject to written approval of process review committees.

User Qualifications and Responsibilities

- All users must have a PTC approved process to prevent cross-contamination between systems and processes.
- The user should submit any problems with the system via CORAL and/or NEMO immediately.
- Report to staff when precursor levels drop below 20% via CORAL and/or NEMO.

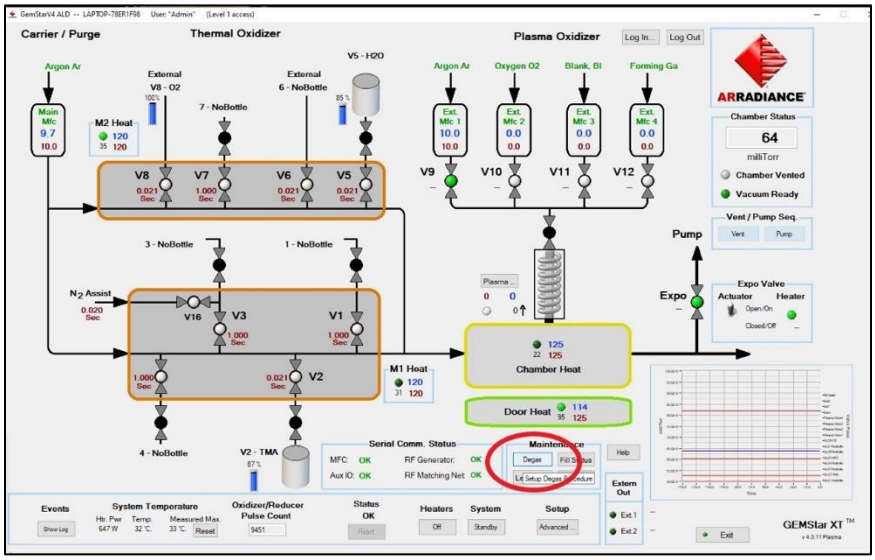
Operating Procedures

Reserve and engage the system on CORAL and/or NEMO. Report any issues on CORAL/NEMO.

Degassing Precursor ALD Valves (minimum of 15 cycles followed by single-pulse check):

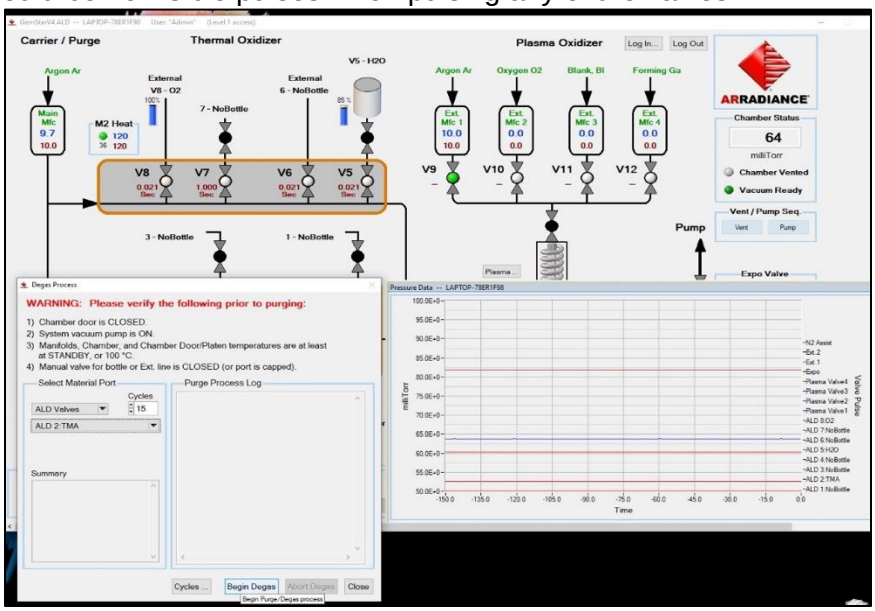
Users must Degas ALL the ALD valves for a minimum of 15 cycles BOTH before AND after their reserved time regardless of which valves they are using and regardless of whether the ALD valves have a bottle installed for them or not. The degas process ensures the system is in a clean state at the start and completion of all runs which helps maintain film quality over time. The 15 cycles of degas along with single-pulse checks also provide a means to detect anomalies in pulse pressure due to tool and/or user errors earlier and prevent tool damage. Follow the procedure below to perform this mandatory 15 cycles of degassing.

1. Verify that all precursor bottles are shut (the manual valve on each precursor bottle must become closed “manually”). Click on the “Degas” button on the main chamber screen.



2. From the drop-down menu at the bottom, select the option that allows you to degas all ALD valves (that is, “All ALD (and N2) Valves”).

3. Run the Degas program by pressing “Begin Degas” (default is 15 cycles for each valve) and monitor the live pressure plot to ensure there are no visible pulses. Wait until the Degas program is completed, then perform single-pulse checks by pulsing the ALD valves (that have just been Degassed) one time, one by one – there should be no visible pulses when pulsing any of the valves.



4. In case that pressure pulses are still present after degassing during the single-pulse checks, go back and repeat the Degas process one-more time, pulse all ALD valves once again one-by-one after the Degas process is completed to confirm that there are no visible pressure pulses, and make sure to report that you had to degas more than one time via Coral/NEMO. Repeat the entire process until there are no pressure pulses visible for any of the ALD valves during single-pulse checks.

Side Note (i): Valve 16 (V16, labelled N2 Assist) allows Ar to flow in, which then gets trapped behind valve 3 (V3) when V3 is closed. While this is not an issue (since Ar is the inert carrier gas for this tool), manually pulsing V3 after degassing V16 should evacuate the trapped Ar.

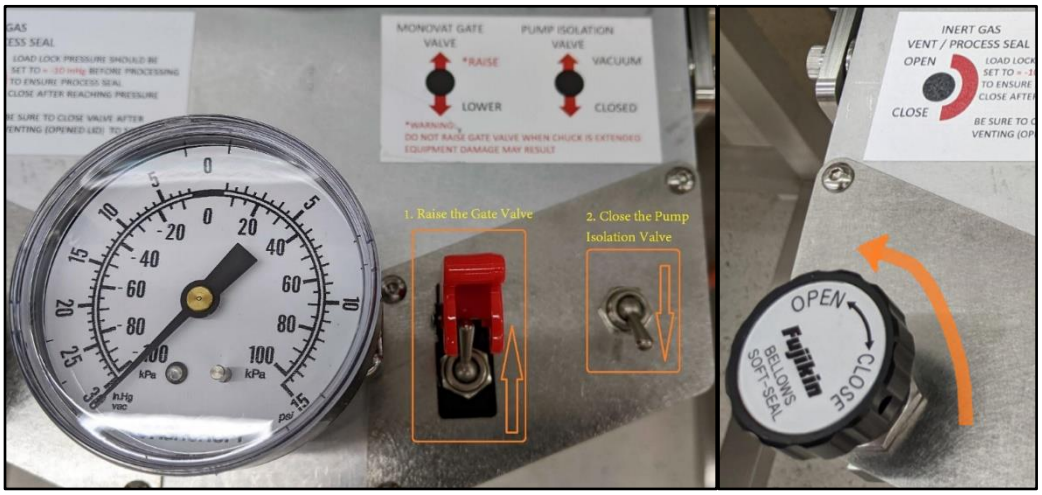
Side Note (ii): When starting a deposition (see detailed steps below), open the manual valves on the bottles for the required precursors only.

Side Note (iii): All precursor bottles must be shut (that is, the manual valve on each precursor bottle must be closed) at all times, except during the deposition when only the required precursors can be open.

Venting the Loadlock:

1. Verify the Monovat Gate Valve is raised (closed).
2. Close the Pump Isolation Valve.

3. Open the Inert Gas Vent/Process Seal valve (Inert Gas Valve).



4. When the loadlock pressure gauge reads atmosphere (0kPa), the loadlock lid clamps may be unscrewed and the lid be opened.
5. Close the Inert Gas Valve.
6. Load sample onto the wafer holder in the loadlock.

Pumping the Loadlock:

1. Close the loadlock lid and tighten the lid clamps.
2. Open the Pump Isolation Valve by switching it to Vacuum; pressure will drop to -100kPa.



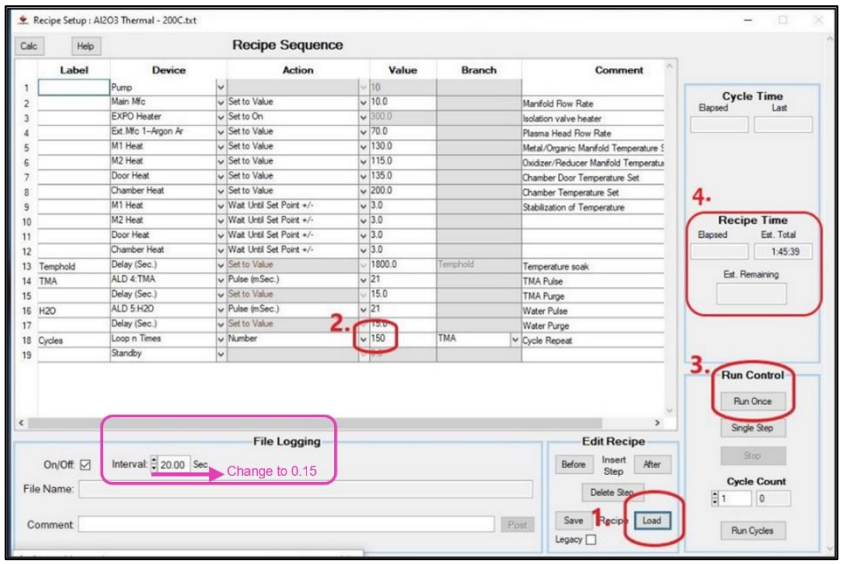
Transferring Sample to Chamber:

1. Verify the loadlock pressure is at -100kPa.
2. Open Monovat Gate Valve.
3. Close Pump Isolation Valve.
4. Turn the load arm wheel until the wafer holder is fully inserted in the process chamber.
5. Keep a slight pressure on the wheel to ensure a good seal and open the Inert Gas Valve.
6. Allow the loadlock pressure to reach -40kPa, then close the Inert Gas Valve.

Running a Recipe:

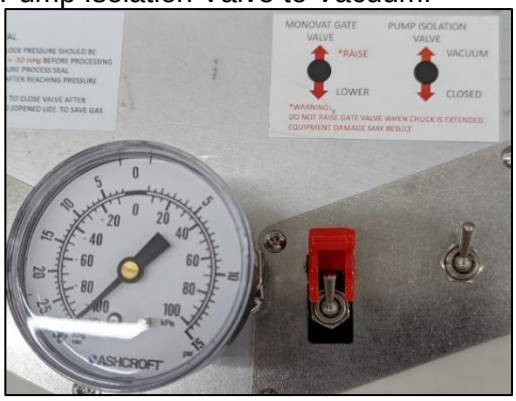
0. Make sure that the “Interval” under “File Logging” is set to 0.15 seconds.

1. Open the desired recipe using the “Load” button on the Recipe Setup window.
2. Set the number of cycles on the Cycles step of the recipe.
3. Click “Run Once” under the “Run Control” section of the window.
4. The estimated process run time will populate in the “Est. Total” box under the “Recipe Time” section.
   Note: Do not change any other process parameters or variables. Unapproved parameter modification could result in precursor degradation, runaway reactions, and damage to the tool or personnel. Custom recipes can be written with support and permission from the staff.



Transferring Sample to Loadlock:

1. Verify the process has completed fully.
2. Pump the loadlock to base pressure (-100kPa) by switching the Pump Isolation Valve to Vacuum.
3. Turn the load arm wheel to remove the wafer holder from the process chamber.
4. Isolate the loadlock from the process chamber by switching the Monovat Gate Valve to the Raised (closed) position.
   Note: If the Monovat Gate Valve doesn’t close, verify the load arm is all the way back in the loadloack and making contact with the sensor. The sensor located at the back end of the arm wheel’s pathway senses when the arm is safely all the way out of the reactor and allows the Monovat Gate Valve to close. If the Monovat Gate Valve still doesn’t close, don’t take further actions and immediately report the issue via Coral/NEMO.
5. Vent the loadlock to retrieve the sample as outlined above.
6. Remove sample from the loadlock and pump the loadlock to base pressure (-100kPa).



Before You Go, Remember To …

Degas Precursors (15 cycles followed by single-pulse check):

Close all the precursor manual valves. Degas ALL the precursor ALD valves for 15 cycles as outlined above and confirm that there are no visible pressure pulses for any of them when you perform single-pulse checks.

Degas Precursors (150 cycles):
If you are the last user of the day (based on Coral/NEMO reservations at the time that you finish), you must do one more step after all the above is done: You must run the degas process overnight - doing this is similar to running the Degas process as outlined above with the exception that you must increase the number of degas cycles from 15 (which is the default value) to 150.

Record Process Data:
Fill out the logbook of the tool with Date, MIT ID, Process run, and process parameters – Pulse and Purge times as well as number of cycles. If film thickness is measured at this point, add to the Results section.

Appendices

Appendix A.
MIT.nano Recommended Process Parameters for ALD-Arradiance Tools

Appendix B.
A Quick Guide to MIT.nano ALD Tools: Overview, Tool Selection and Cleanliness

MIT.nano Recommended Process Parameters for ALD-Arradiance Tools

Location: 8U

Contact: "mitnano-ald-advisors@mit.edu"

Current Tool Limits:

- Maximum allowed deposition temperature (i.e., chamber temperature) on Arradiance tools is **250°C**.
- Maximum allowed film thickness on Arradiance tools is **20nm**.
- Chamber pressure at thermal equilibrium right before starting any depositions (that is, while **110sccm of argon** is flowing through the chamber) **must NOT exceed 168mTorr**.
- Plasma **must NOT remain ON for more than 45 second** at a time.
- Any abnormal observations during the deposition and characterization steps **must be immediately reported** -- examples include (but are not limited to): Abnormal growth per cycle (GPC) values, presence of carbon or fluorine in the film in XPS compositional studies, unusual plasma operation (e.g., plasma color or color intensity), presence of ghost precursor pressure pulses upon manual pulsing of any precursor bottles after degassing with the bottle's manual valve closed.
- Precursors that are allowed to be heated **must not be left** at their allowed elevated temperature when not needed.

List of key components' temperature (unless stated differently in the recommended process parameters):

Door Temperature: Determined based on Chamber Temperature as per vendor's recommendations listed on the right
M1-Heat: 120°C (except when using TMI†)
M2-Heat: 120°C (except when using TMI†)
†When using TMI: M1-Heat = M2-Heat = 100°C

Chamber Temperature (°C)	Door Temperature (°C)
100	80
150	100
175	120
200	135
250	150

MIT-nano Recommended Process Parameters:

ALL processes must be submitted to PTC for approval; ALD processes that propose to use parameters different from the table below must include the exact proposed run conditions and why they were chosen.

Material, Deposition Method	Precursor(s), Precursor Temperature [°C]	Deposition Temperature (i.e., Chamber Temperature) [°C]	Pulse Duration [milliseconds] / Purge Duration [seconds]
Al2O3, TALD	TMA and H2O, both at room temperature (i.e., not heated)	250°C only	60ms TMA/45s purge/ 30ms H2O/45s purge
Al2O3, PEALD	TMA, at room temperature (i.e., not heated)	150°C, 175°C, 200°C, 250°C	60ms TMA/45s purge/ 10s O2-plasma/15s purge
AlN, PEALD	TMA, at room temperature (i.e., not heated)	250°C	60ms TMA/45s purge/ 20s FG-plasma/15s purge
HfO2, PEALD	TDMAHf, at 75°C for 1-3 hours (the 1-3 hours of wait is to ensure stable vapor pressure)	200°C, 250°C	100ms TDMAHf/45s purge/ 10s O2-plasma/15s purge
ZrO2, PEALD	TDMAZr, at 75°C for 1-3 hours (the 1-3 hours of wait is to ensure stable vapor pressure)	200°C, 250°C	100ms TDMAZr/45s purge/ 10s O2-plasma/15s purge
HZO, PEALD	TDMAHf and TDMAZr, both heated to 75°C for 1-3 hours (see the two rows above)	200°C, 250°C	Supercycles consisting of (x cycles HfO2 / y cycles ZrO2)
GaN, PEALD	TEG, at room temperature (i.e., not heated)	250°C	20ms TEG/45s purge/ 40s FG-plasma/15s purge
Ga2O3, PEALD	TEG, at room temperature (i.e., not heated)	200°C, 250°C	20ms TEG/45s purge/ 10s O2-plasma/15s purge
ZnO2, PEALD	DEZ, at room temperature (i.e., not heated)	200°C, 250°C	60ms DEZ/45s purge/ 10s O2-plasma/15s purge
TiN, PEALD‡ **[GPC = 0.7 Ang/cycle]**	TDMATi, heated to 60°C for 3 hours (3 hours of wait is to ensure stable vapor pressure)	160°C only ((Door @110°C))	2000ms TDMATi/45s purge/ 40s FG-plasma/15s purge
In2O3, PEALD **[GPC = 0.85 Ang/cycles]**	TMI, room temperature (i.e., not heated)	150°C only ((Door @100°C)) **M1-Heat = M2-Heat = 100°C**	1000ms TMI/45s purge/ 10s O2-plasma/15s purge

‡ 300 cycles of “degas” must be performed after TiN deposition (that is, two times the usual 150 cycles of “degas”).

APPENDIX:

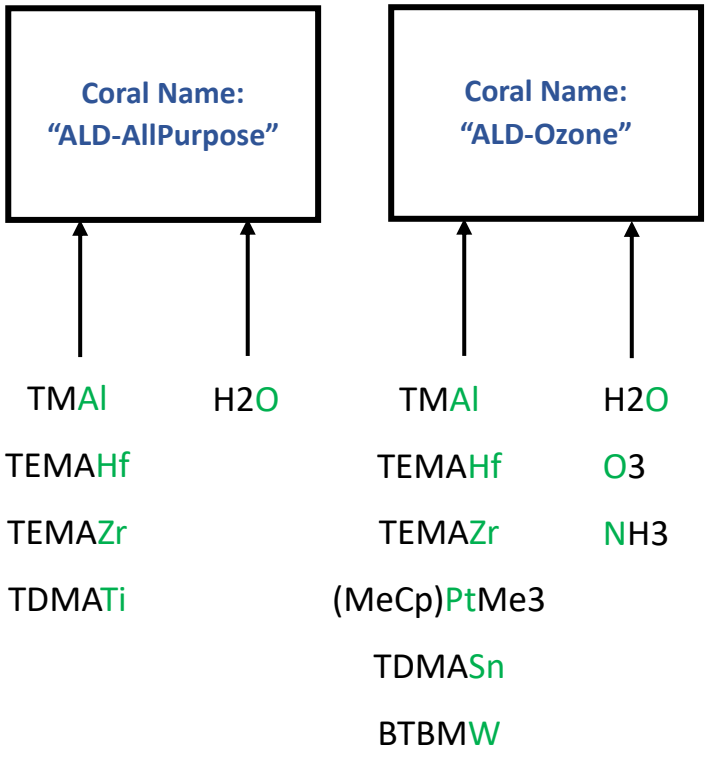
List of abbreviations:

TALD: Thermal ALD	TEG: Triethylgallium
PEALD: Plasma Enhanced ALD	DEZ: Diethylzinc
TMA: Trimethylaluminum	TDMATi: Tetrakis(dimethylamido)titanium
TDMAHf: Tetrakis(dimethylamido)hafnium	TMI: Trimethylindium
TDMAZr: Tetrakis(dimethylamido)zirconium	FG: Forming Gas (a 95%-5% mixture of N2-H2)

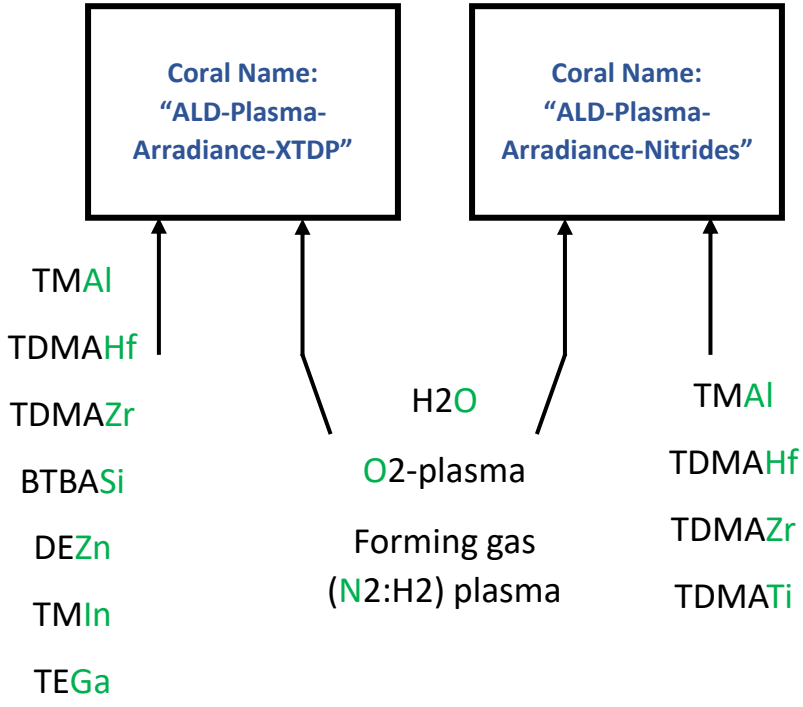
A Quick Guide to MIT.nano ALD Tools: Overview, Tool Selection and Cleanliness

Location: 8U
Category: Deposition
Contact: "mitnano-ald-advisors@mit.edu"

Cold wall (substrate heater available up to 300°C, typically ≤ 250°C)
Thermal ALD Only



Hot wall (substrate heater available up to 300°C, typically ≤ 250°C)
Both Thermal ALD and PEALD



A guide for choosing the right tool:

Increasing tool cleanliness requirements

ALD Tool Name	Use Requirements	Example Applications	Allowed Samples
Coral Name: ALD-AllPurpose	Need medium quality thin and thick (<~30nm) films of Al_2O_3, HfO_2. Or need to deposit on samples with organic materials.	Capping layer deposited on top of polyimide substrate	Most samples are allowed, including approved organic materials.
Coral Name: ALD-Ozone	Need high quality thin and thick (<~30nm) films of Al_2O_3 and HfO_2. Or need metal precursors such as Pt, Sn, W. Or need ozone-assisted deposition.	Thick gate dielectric or passivation layer. For example, insulation layer for capping high aspect ratio silicon vias	no organics or tool contaminants; samples must be clean.
Coral Name: ALD-Plasma-Arradiance-XTDP	Need very high quality (high breakdown field, low leakage) thin (<20nm) dielectric films of AlN, Al_2O_3, HZO, TiN, GaN, SiO_2, and IGZO. Or Need plasma-assisted ALD deposition.	Passivation film to cap sample. For example, 10nm of ALD AlN as surface capping layer as an etch stop layer	No organics or tool contaminants; samples must be extremely clean.
Coral Name: ALD-Plasma-Arradiance-Nitrides	Need very high quality (high breakdown field, low leakage) thin (<20nm) dielectric films of AlN, Al_2O_3, HZO, and TiN.	Gate dielectric of very high-performance transistors. For example, 15nm of ALD Al_2O_3 for GaN FinFETs	No organics or tool contaminants; samples must be extremely clean.

APPENDIX:

>> Recommended Sample Cleaning Procedures:

Maintaining extreme cleanliness (including cleanliness of the tool and the substrates) is an essential factor in depositing reproducible and high-quality films by ALD. Thus, it is critical for samples to be extremely clean before entering ALD chambers since unclean samples may cause unwanted contamination to the ALD chambers and processes.

When possible, **it is required to do a 10-minute piranha clean** (3:1 sulfuric acid to hydrogen peroxide) on samples immediately before placing them in the ALD chambers. This can be done for nearly all dielectric and semiconductor samples that do not contain metals.

If a user cannot perform a piranha clean on their samples, they must submit a request to PTC to get an exception from the reviewing committees in writing.

ONLY IF a piranha clean is not possible, **it is required to do a 1-hour 70°C hot NMP clean**, followed by solvent rinsing, to remove organic residues on the samples immediately before placing them in the ALD chambers. This is especially important when previous steps have involved use of photoresist (AZ3312, nLOF, AZ10xT, etc...). In the particular case of PMMA only, 1 hour in acetone is also a sufficient cleaning step.