

## Veeco FIJI G2 PEALD system

The Fiji is a modular high-vacuum ALD system that accommodates a wide range of deposition modes using a flexible system architecture and multiple configurations of precursors and plasma gases. The result is a new generation ALD platform capable of doing thermal as well as plasma-enhanced deposition of metals and nitrides.

The system contains a load lock chamber for loading substrates external to the process chamber. Both the load lock and the chamber are equipped with turbo pumps to ensure minimal oxygen contamination. The process chamber has a gate-valve to maintain optimum process conditions and minimizing temperature cycling times. EMO buttons are provided on the front and rear of the system.

### **Prerequisites for operating the Veeco ALD system:**

- a) Obtain a NRF ID (if you do not already have one) by completing the [NRF Lab Use Request Form](#) and safety training.
- b) Receive “one on one” training and certification from NRF Staff. Discuss your process with a staff member.

### **Safety**

**DANGER!** Do not remove the covers of the instrument. Do not modify the instrument.

- **HOT Components** - The User must observe caution when loading/unloading samples from the sample stage and when loading/unloading the sample stage from the process chamber. Samples are very hot (>200°C) when immediately removed from the process chamber. Sufficient cooling time must be allowed prior to sample handling.

## **1.0 Pre-Operation**

**1.1** Tool Reservations may be made via the NRF Reservation Page.

<http://nimet.ufl.edu/servicecenter/resources/default.asp>

**1.2** Change gloves. WARNING No solvents or liquids are allowed near the machine, change your gloves before operation!!

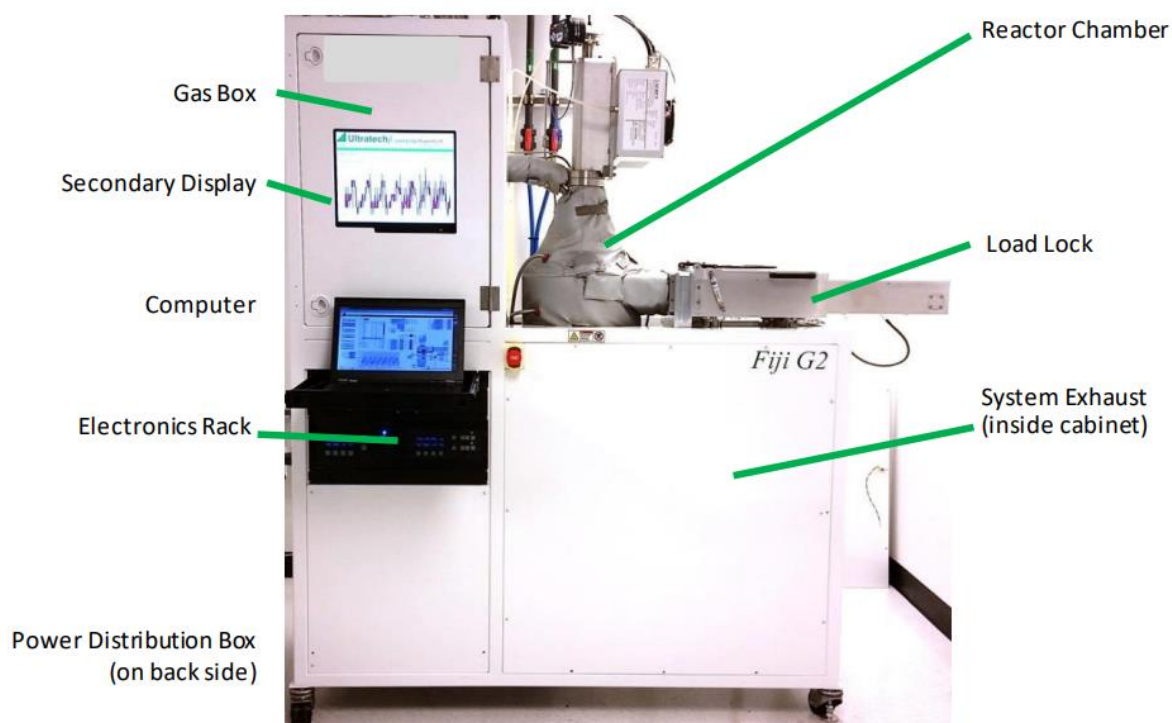
**1.3 ABSOLUTELY no ZnO sample is to be into the ALD system.**

**1.4** Log into the tool by using the TUMI computer in this cleanroom bay.

**1.5** Ensure precursor valves are open prior to using the system. Valve status can be checked on the ALD web page:

<https://nrf.aux.eng.ufl.edu/ccb/resource.asp?id=197> or by contacting staff.

## 2.0 Hardware Overview Images



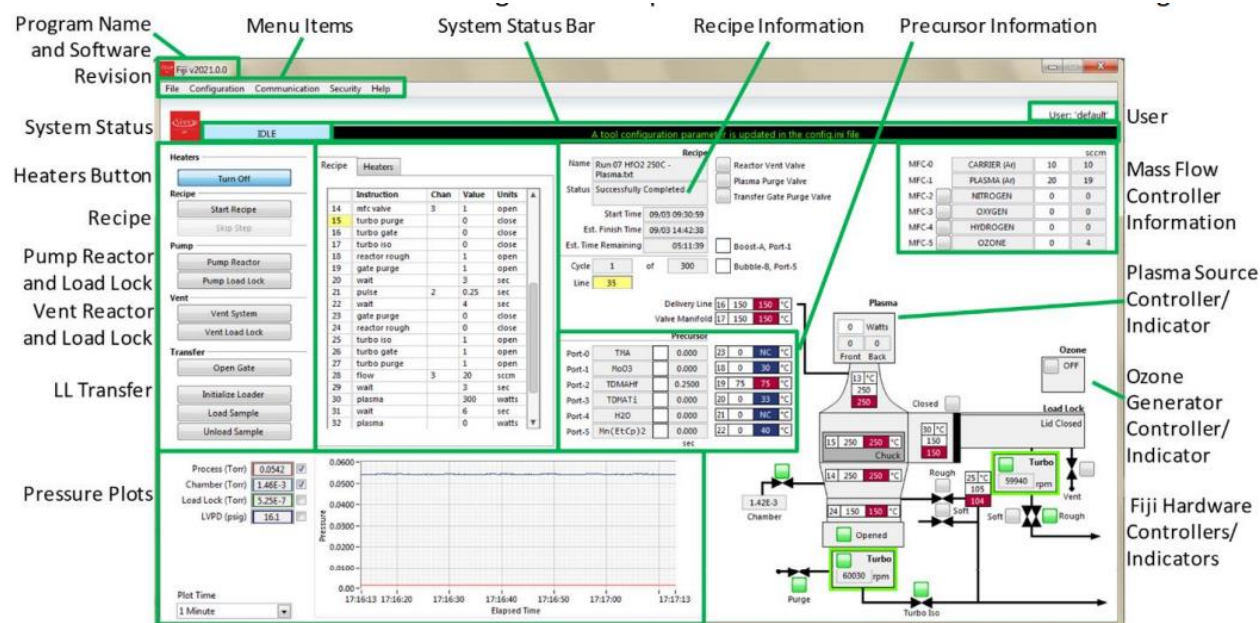
The Veeco FIJI G2 PEALD system is equipped with a sample load lock and fully automated transfer system. These components are visible and accessible in the user side of the cleanroom bay. The remaining portion of the system is behind the bay wall in the cleanroom chase and is not accessible by NRF users.



**Users are not to access components located in the cleanroom chase. This violates NRF policy and the user will be removed from the cleanroom.**

### 3.0 User Interface Overview

The Veeco Fiji G2 PEALD tool is controlled through a software interface. All operation of the system except physically loading/unloading your sample from the substrate holder is performed via this software interface. Figure 1 shows the software screen shot and indicates important system fields. These fields will be referred to in this manual and during processing. This is an image of the main tool interface. Though this screen, recipes will be loaded and run and system status can be verified.



**Figure 1.** Software screen shot.

Heater idle temperatures are as follows and should remain at these levels for idle.

|                                   |       |
|-----------------------------------|-------|
| ALD Valves (#17)                  | 150°C |
| Precursor delivery line (#16)     | 150°C |
| Tee Heater (#24)                  | 150°C |
| Exhaust Valve Heater (#25)        | 125°C |
| Chuck (#15)                       | 70°C  |
| Upper & Lower Chamber (#13 & #14) | 70°C  |
| Gate Valve (#30)                  | 70°C  |

Due to copper deposition requiring lower temperatures the chuck, upper and lower chamber are kept at a lower temperature than the Cambridge ALD.

**All precursor jackets should be at 0°C for idle.** "NC" indicates no heater jacket is connected for that precursor.

## 4.0 Operation

The system should be running and the software should be displayed on the monitor. Check the system vacuum pressure and the status of the heaters. Chamber vacuum levels are read at the lower left of the program screen. A typical process chamber pressure will be in the 2.5 to 8.5 E-2 Torr range.

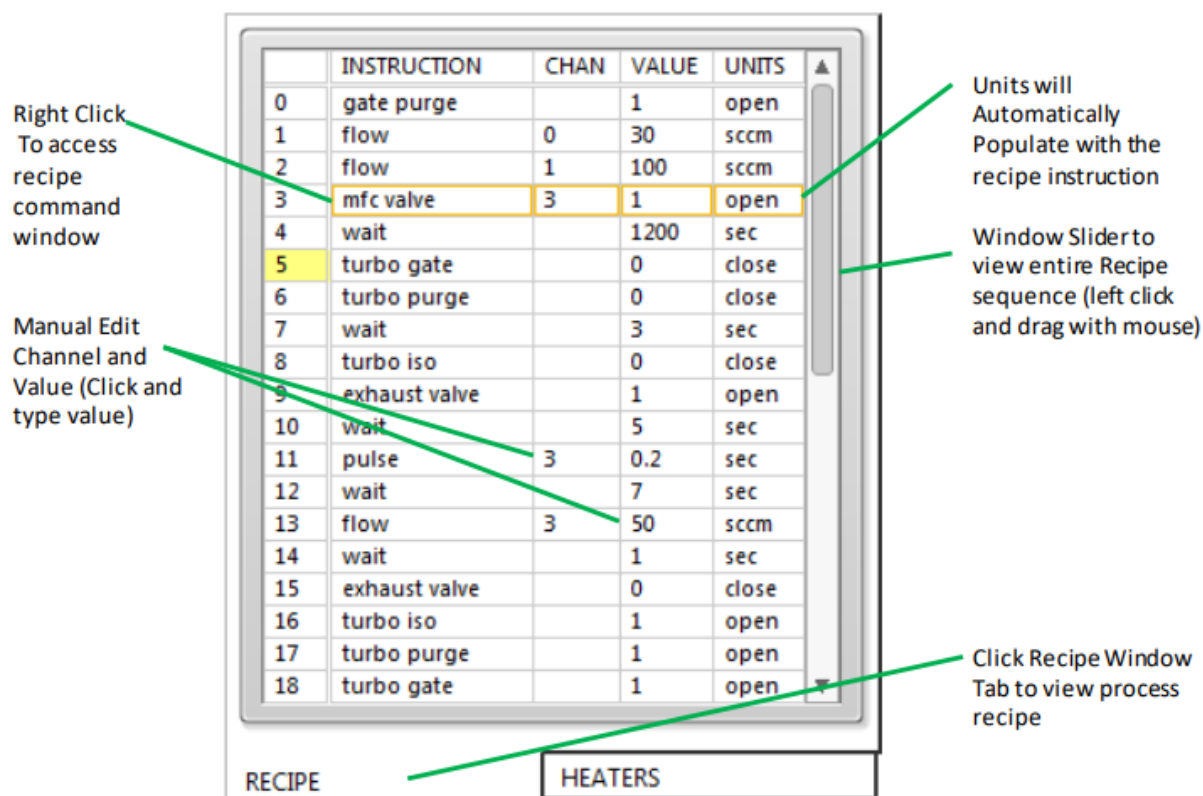
### 4.1 Sample loading

- a. Have sample prepared and ready to load. The bare wafer holder is designed to hold an 8" wafer or small pieces. Use small, supplied washers to pin small substrates down.
- b. Vent the load lock by pressing the "**Vent Load Lock**" button on the left side of the software screen. This will automatically vent the load lock with Argon.
  - i. If the gate valve is open, it will not let you vent. Please consult staff.
- c. Once vented, open the load lock chamber door and load the sample onto the wafer holder. Close load lock door when finished.
- d. Click the "**Pump Load Lock**" button on the left side of the software screen. Wait until the load lock pressure reaches your desired vacuum level.
  - i. For lowest oxygen contamination in nitrides/metals waiting till 2E-6 is recommended. The turbo pump typically achieves this within a few minutes.

### 4.2 Loading recipe and running processes

As an operator, you will only be able to load, modify and run recipes. Modifications to recipes are not saved to the permanent recipe file and are only executed for your run. Any permanent changes to a recipe will have to be performed by NRF staff. Please be sure to provide sufficient time prior to your reservation for staff to create/edit the recipe for you.

- a. Right click anywhere in the recipe table and click "Load recipe...".
  1. This will open the recipe folder. Navigate to the process folder, there the processes are organized by material. Select the desired material folder and then recipe.
  2. Recipes are named by #A/cycle Material Temperature notes.
    - i. Example. 0.73 TiN 250C w 5H2.
    - ii. This would mean this recipe does 0.73A/cycle of TiN at 250C in a hydrogen co-plasma.
    - iii. If unsure what recipe to run, consult staff. Many recipes have additional resistivity or XPS data available upon request.



**Figure 2.** Example recipe screen.

- b. Once the recipe is loaded, as the user, you **must** change the pulse port in the recipe from the element formula to a numerical value as shown in Figure 3. This value **must** correspond accurately to the correct pulse port number otherwise the wrong material will be deposited. On the main Process Screen, the pulse ports are shown and the materials loaded are displayed.

| Precursor |           |  |        |    |           |
|-----------|-----------|--|--------|----|-----------|
| Port-0    | H2O       |  | 0.000  | 23 | 0 NC °C   |
| Port-1    | LVPD-A Co |  | 2.000  | 18 | 0 41 °C   |
| Port-2    | AL TMA    |  | 2.000  | 19 | 0 27 °C   |
| Port-3    |           |  | 1.000  | 20 | 0 28 °C   |
| Port-4    | LVPD-B Cu |  | 2.000  | 21 | 110 75 °C |
| Port-5    | Ti TDMAT  |  | 0.2500 | 22 | 0 29 °C   |
| sec       |           |  |        |    |           |

**Figure 3.** Example precursor port configuration.

- c. If your recipe requires a heated precursor, you **must** also ensure that the correct heater jacket number is listed in the recipe. The heater jacket numbers are listed in the Main Screen and these are in the recipe as "heater" in the recipe

lines. A list of precursors and jacket temperatures are listed below. The jacket heaters are #18-23. A “NC” under a precursor indicates no heater jacket is connected.

**DO NOT change the temperature of heaters #12, #13, #14, #15, #16, #17 in the recipe.**

Titanium (Ti) – 75 to 80°C

Copper (Cu) – 110°C

Cobalt (Co) – 80°C

The idle temperature for all heated precursors is 0°C. The precursor heater jackets should be at 0°C when you log in and when your runs are completed.

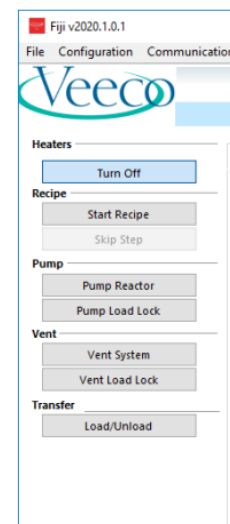
d. The user can change the number of cycles. This change will not be saved to the permanent recipe file. Remember the number of cycles, and report it as “comments” on the TUMI system while logging off. Failure to do so will result in the group being billed inaccurately. The default values are high.

Please **DO NOT** set the number of cycles to 0 as this will cause the program to run an infinite loop.

All recipes will be modified to reset the process chamber temperatures back to the idle temperatures listed in section 3.0. This is to reduce the occurrence of users forgetting to reset the temperatures at the end of their run.

f. To start the recipe, click on “**Start Recipe**”.  
Click YES in the user response box that appears.

The recipe will auto execute including loading and unloading of your sample from the chamber. There may be a delay if the chuck temperature (#15) or chamber temperatures in the recipe are different from the idle temperature. Once the temperature is stabilized, the recipe will run.



The recipe can be monitored by watching the PROCESS tab, recipe details and the Pressure Status of the system. Each valve operation will result in a pressure change that can be noticed in the pressure graph.

**Do not use the Turn OFF button to abort a recipe, this button will turn off the ALD program and ramp down all heaters. NRF staff must reset the system to enable the software once the program is stopped.**

**NOTE:** Once the recipe starts, keep an eye on the “Remaining Cycles” tab. If it is different than the number entered, ABORT AND CONTACT NRF STAFF IMMEDIATELY.

**NOTE:** Changing the number of cycles after the program has started running will not change the initial settings.

### 4.3 Sample unloading

Once the recipe is finished your samples will already be in the load lock.

- a. Click on “**Vent Load Lock**”, after 2.5 min the chamber will be open.
- b. Be careful about the temperature of the substrate, carefully remove your samples, if they are still hot, you can use the metal table as a heat sink.



**THE SAMPLE AND WAFER HOLDER WILL BE VERY HOT (OVER 100°C).  
USE CAUTION TO NOT TOUCH THE WAFER HOLDER OR SAMPLE RING.  
LET THESE COOL COMPLETELY PRIOR TO REMOVAL.**



- c. After removing your samples press “**Pump Load Lock**” to leave the tool under vacuum.

## 5.0 Stand by Mode

If any changes were made to the Chuck temperature, please reset to the idle temperature of 70°C. You must also reset the precursor jacket temperature(s) to 0°C.

**Load the “blank recipe”** – this will blank out the recipe screen and return any user edited recipe to its default values

Do not close the ALD program this will turn off all the system heaters and will require NRF staff to reset the tool (a hardware reset is required on a non-user accessible component).



**CLOSING THE PROGRAM RESULTS IN  
THE HEATERS LOSING POWER. REACTIVATION OF THE  
SYSTEM REQUIRES NRF STAFF.**

**Log off the ALD system in the TUMI computer and leave the appropriate notations.**

**NOTE:** In the TUMI logoff notes, indicate the type of ALD run (conventional or exposure), the number of ALD cycles, and the source material used. For examples:

Conventional ALD, 278 cycles, Al  
Or  
Plasma ALD, 311 cycles, Hf

**Failure to do so will result in limited access to the ALD system.**

## 6.0 Approximate Heating and cooling times for the ALD

Please be sure to add these heating and cooling times to your reservation to ensure enough time for the system to recover back to the idle temperature for the next user's access.

Idle state 200°C chuck, 195-200°C chamber and cone to

Heating from **200°C** to **300°C** – 1.0 hrs

Heating from **200°C** to **400°C** – 1.0 hrs

Heating from **80°C** to **200°C** – 1.0 hrs

Cooling from **200°C** to **80°C** – 7.5hrs

Cooling from **300°C** to **200°C** – 4.5hrs

Cooling from **400°C** to **200°C** – 5.0hrs

## 7.0 Software and Tool glitches.

Below are some known issues with software and tool you may encounter during operation.

- a. Failing to replace precursor name with valve number on the “go to” part of the recipe will default to pulsing water instead of your precursor.
- b. Failure to set precursor temperature to proper temperature will result on under or no pulses during your run.

**Please record below other issues you encounter with the tool.**

## 8.0 FAQs and Tips for ALD

### ***What valve temperature should I use?***

The valves on the system (Swagelok pneumatic ALD valves) we recommend the following: Swagelok valves can be heated up to 200 °C. We recommend the Swagelok valves to be heated to 150°C for non-heated precursors (water, TMA etc). Please ask Veeco or staff engineers for settings for your conditions before trying anything at random.

### ***What is the dose that I should use for the precursors?***

We recommend asking Veeco or staff engineers for a recipe before any run since there are many parameters interconnected (substrate material, precursor, temperature, purge time etc.), that one general recipe cannot be mentioned. One general guideline is that the precursor pulse height should be about 1 Torr or less. For TMA and other high vapor pressure materials, one should not pulse for more than about 0.02 seconds, unless Veeco advises otherwise. This is to prevent premature contamination of the system. Also, high vapor pressure materials should not be heated and the heating jacket should be removed to prevent the cylinder from getting hot from the valve heating block above it.

### ***Why is there nitrogen flow and what is the recommended setting?***

Nitrogen (or argon) flow is used for various reasons. The first reason is to quickly purge the system between each pulse. It is important that between the precursor pulses, there is no residue (except for the monolayer chemically bonded to the substrate) of precursor in the process chamber. The presence of two precursors at the same time would cause immediate reaction in vapor phase, which can lead to CVD mode deposition (non-uniformity, thick coating, powder formation). So the combination of temperature, nitrogen flow and pump time between pulses prevents two precursors from seeing each other in vapor phase. At low process chamber temperatures (e.g. 100 °C), the pump time needs to be higher (e.g. 30 sec) than at high temperatures (>150 °C, pump time e.g. 8 sec, for TMA). The higher the temperature, thus the faster the cycles. Another reason for nitrogen flow is to prevent flow of precursor from one pulse valve into the other. It is important that the pulse valves only see their own precursor, thus each valve has a constant nitrogen flow. The constant nitrogen flow out of each valve prevents other precursors to enter, and thus prevents deposition in each valve. A recommended nitrogen flow value is 20 sccm. During venting this is increased to 100 sccm, and also while the process chamber is open, this should be the maximum flow value (100 sccm) to prevent air from entering the valve regions. One should immediately close the lid door inserting/removing a substrate and not leave it open.

### ***What is the expo for, should I use it?***

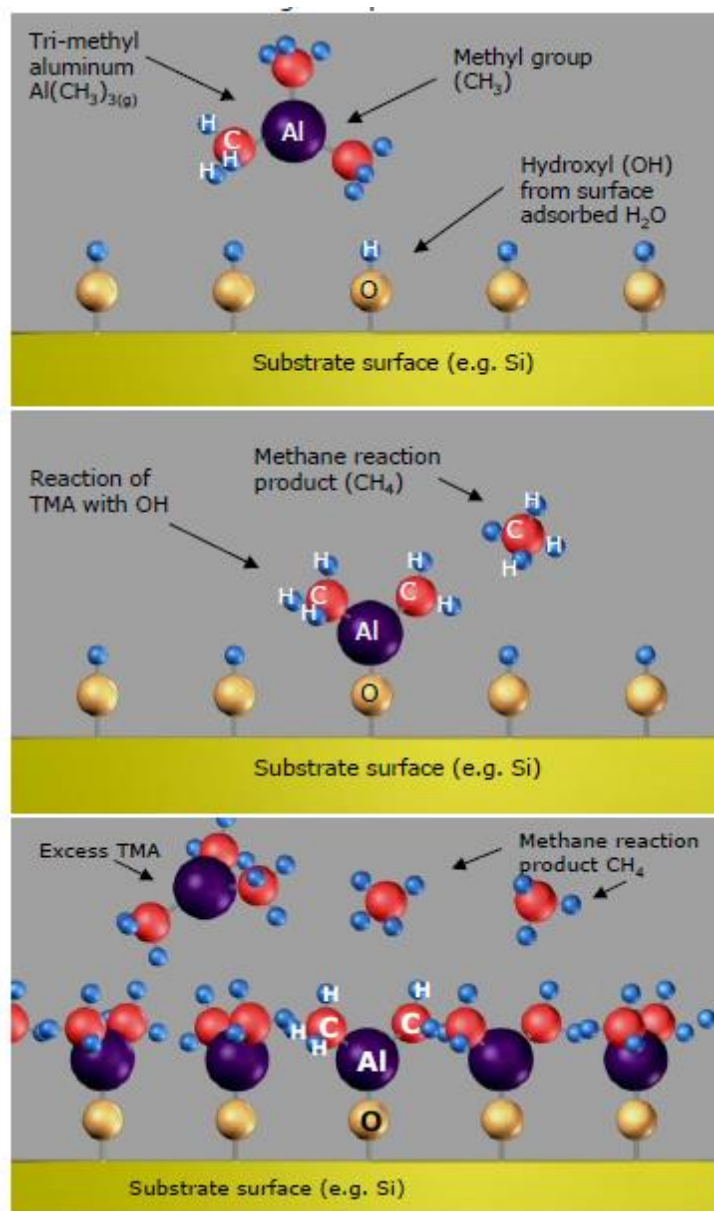
Expo deposition means that prior to pulsing a precursor, the main vacuum valve (valve between process chamber and pump) is closed, then one precursor is injected, then the substrate gets exposed during the expo time (for example if expo = 20 seconds, the main vacuum valve opens after 20 seconds). This mode is only used when one needs to coat very high aspect ratio structures (>1:10). The disadvantage of using expo runs is that since the precursor does not simply flow very fast from cylinder through valve through manifold over substrate into pump, instead, it sits in the process chamber for expo seconds, and some of the precursor can migrate to all valves. This can cause some deposition in the valves, especially if the expo time is long. To reduce this effect, one could pulse two times in expo mode (for very high aspect ratio structures) to prevent the process chamber pressure from getting too high during the expo time. If one wants to get better nucleation on hydrophobic substrates, we don't recommend expo mode deposition. Instead, one could pulse two times the same precursor in multi dosing deposition mode (expo=0 seconds), and this would only be needed for say the first few cycles. This may be especially useful for thin gate dielectrics.

## Atomic Layer Deposition: Principle of $\text{Al}_2\text{O}_3$ formation

Atomic Layer Deposition (ALD) is a technique that allows growth of thin films, atomic layer by layer. The typical ALD reaction is illustrated via the formation of  $\text{Al}_2\text{O}_3$  from trimethylaluminum or TMA,  $\text{Al}(\text{CH}_3)_3$ , and water,  $\text{H}_2\text{O}$ . Recipes for other materials can be found in the literature.

### Step 1: Introduction and adsorption of precursor A to the surface.

The precursor, trimethylaluminum reacts with hydroxyl groups on the surface of the substrate, liberating methane. The reaction is self-limiting as the precursor does not react with adsorbed aluminum species.

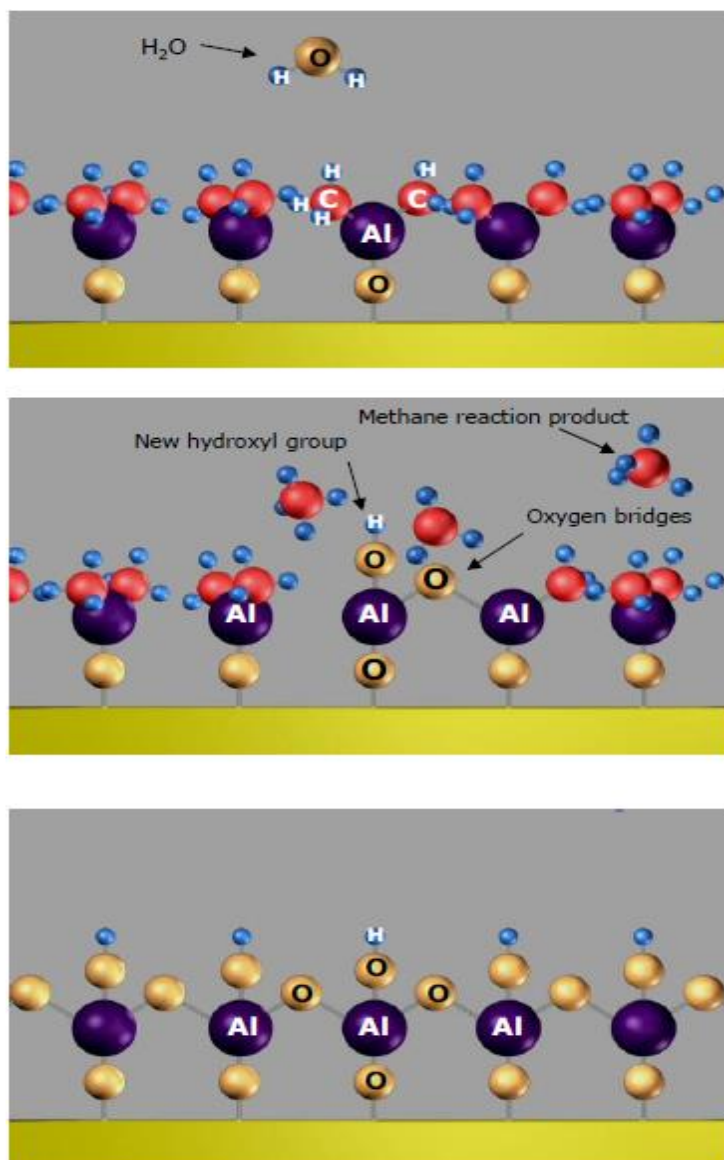


**Step 2: Removal of the unreacted precursor and reaction products.**

Unreacted precursor and the methane ( $\text{CH}_4$ ) liberated from the reaction are removed by simple evacuation of the sample chamber or by flowing inert gas over the surface.

**Step 3: Introduction and adsorption of precursor B to the surface.**

Water reacts with the methyl groups on the deposited aluminum atoms forming both Al-O-Al bridges, as well as new hydroxyl groups. The formation of hydroxyl groups readies the surface for the acceptance of the next layer of aluminum atoms. Methane is liberated as a by-product



**Step 4: Removal of the unreacted precursor and reaction products via evacuation and/or inert gas flow.**

**Step 5: Repeat to create layers**

The process begins again with the introduction of precursor A followed by B. Atomic layers are built up one after the other.

| <b>Veeco ALD Precursor surcharge starting 7/1/25</b> |                                                         |  |  |                             |
|------------------------------------------------------|---------------------------------------------------------|--|--|-----------------------------|
| <b>Part</b>                                          | <b>item</b>                                             |  |  | <b>Price/pulse</b>          |
|                                                      |                                                         |  |  | <b>Assume<br/>4mg/pulse</b> |
| <b>Al</b>                                            |                                                         |  |  |                             |
| 98-4003                                              | TriMethylAluminum (TMA)                                 |  |  | <b>\$0.16</b>               |
|                                                      |                                                         |  |  |                             |
| <b>Ti</b>                                            |                                                         |  |  |                             |
| 98-4015                                              | Tetrakis(dimethylamido)titanium(IV)                     |  |  | <b>\$0.24</b>               |
|                                                      |                                                         |  |  |                             |
| <b>Cu</b>                                            |                                                         |  |  |                             |
| 29-7100                                              | Bis(N,N'-di-sec-butylacetamidinato)dicopper(I),99%      |  |  | <b>\$0.70</b>               |
|                                                      |                                                         |  |  |                             |
| <b>Co</b>                                            |                                                         |  |  |                             |
| 27-0475                                              | Bis(cyclopentadienyl)cobalt(II), min. 98% (Cobaltocene) |  |  | <b>\$0.35</b>               |
|                                                      |                                                         |  |  |                             |
| <b>Ni</b>                                            |                                                         |  |  |                             |
| 28-1301                                              | Bis(cyclopentadienyl)nickel, 99% (Nickelocene)          |  |  | <b>\$0.15</b>               |