Cambridge Nanotech ALD SOP

Introduction:
The Cambridge Nanotech Atomic Layer Deposition (ALD) tool allows deposition of various materials at the atomic scale by sequential pulsing of special precursor vapors.

Safety:
Due to the toxic nature of the process gases, the supply cylinders are turned on and off by qualified technical staff only. All tank changes are performed by qualified technical staff only. Do not try to defeat any interlock on the system. Keep your hands away from all moving parts and be sure that all covers are in place when you are processing. If you encounter any equipment problems while operating the system, contact the technical staff in charge of the system. Do not try repairs on your own.

The chamber lid is always hot and will melt gloves and burn fingers – use the handle to open the chamber and avoid touching the lid.

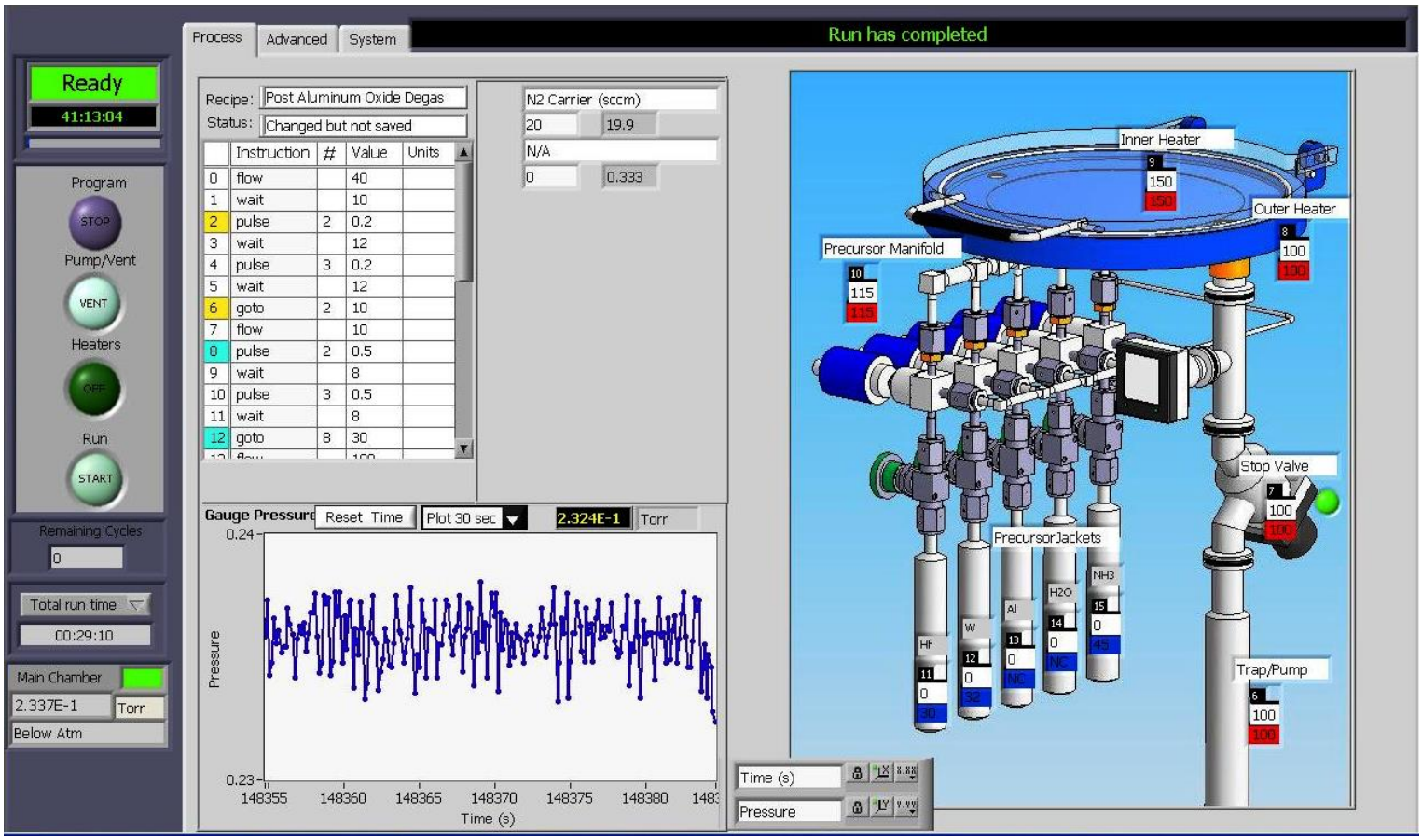
Procedure:
It is mandatory to reserve the system prior to use and to ENGAGE MACHINE prior to starting your process in CORAL.

Starting an ALD Process:
Before beginning a process, the chamber and precursor temperatures should be set to the temperatures required for deposition and the system allowed to come up to temperature. Typical chamber temperatures are as follow:

Film	Inner Heater (#9)	Outer Heater (#8)
Hafnium Oxide	200°C	150°C
Aluminum (TMA)	200°C	150°C
Tungsten Nitride	350°C	200°C

The precursor temperatures are:

Precursor	TC #	Setpoint
Hafnium	11	95°C
Tungsten	12	90°C
Aluminum (TMA)	13	does not need to be heated
Water	14	does not need to be heated
Ammonia	15	does not need to be heated



The process chamber is kept under vacuum and must be vented to load your sample.

- Make sure all ALD valves on the precursor cylinders are shut.
- Press the Vent button.

When the chamber pressure reaches atmosphere (7.60E+02)

- Set the Nitrogen flow to 20 sccm.
- Open the process chamber lid.

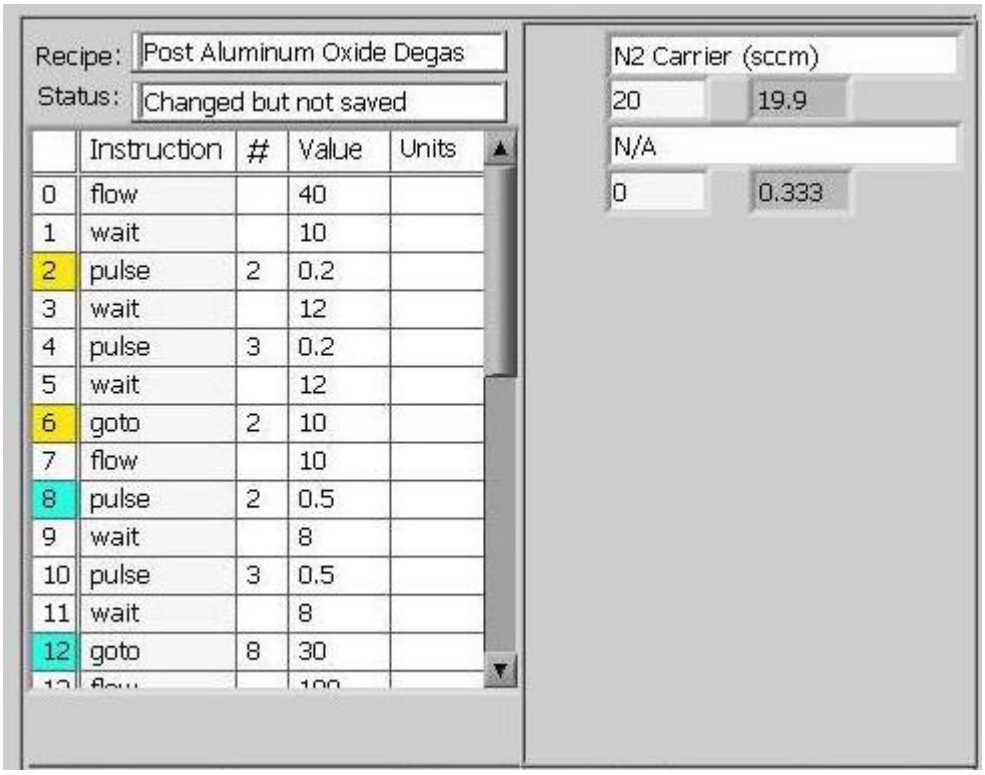
Pieces and wafers should be processed on top of the dummy wafer that should always be present in the chamber.

- Be careful not to come into contact with the chamber surface – the chamber temperature is kept at 150°C and this may melt gloves and result in a serious injury.
- Do not load samples using composite or plastic tweezers as they may melt.

Close the chamber lid and ensure it is centered on the chamber body. Press the Pump button.

Recipes are loaded and edited from the text box in the center of the screen.

- right click anywhere in the recipe area and select 'Load Recipe'
- Choose the desired recipe from the 'Recipes' folder.
- Simple edits may be made to change the number of cycles or the chamber temperature only. Do not change purge times, carrier flow rates or precursor pulse times. Never change the precursor operating temperature, as overheating will result in the premature degradation of the precursor.
- With the sample loaded, and the chamber under vacuum, the process is almost ready to start. The estimated run time is indicated on the screen.



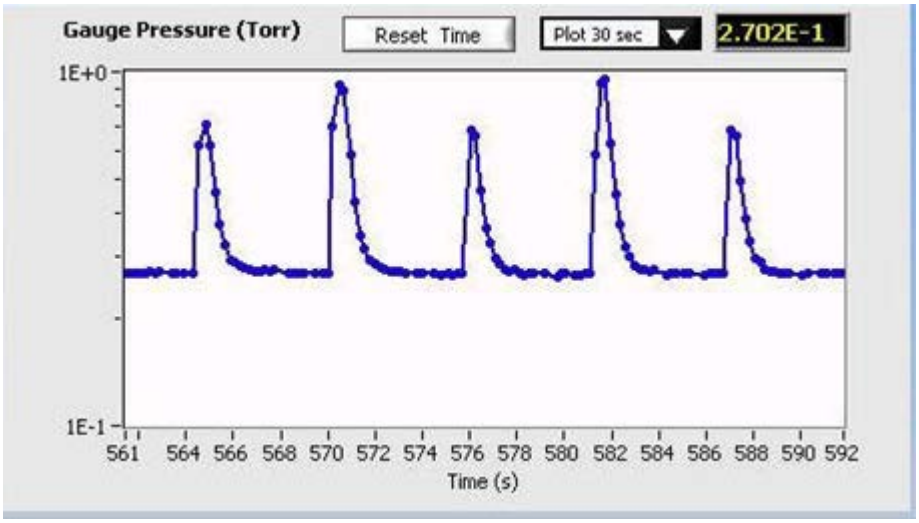
Before starting a recipe, the required precursor/gas valves must be opened on the manifold. Open the door to the ALD cabinet – the precursor manifold is on the left side. The precursors are installed as follows:

Port	**Precursor**	**Chemical Name**
0	Hafnium	Tetrakis(dimethylamido)-Hafnium (IV)
1	Tungsten	Bis(*tert*-butylimino)bis(dimethylamino)-Tungsten
2	Aluminum	Trimethyl Aluminum
3	Water/Ozone	H_2O or O_3
4	Ammonia	NH_3

Each precursor cylinder has a black quarter turn valve installed inline – these valves should remain in the closed position (perpendicular to the cylinder) when not in use.

- Open the valve for the required precursor by turning the valve 90° counter-clockwise.
- When depositing Tungsten Nitride, click on the Advanced tab and set the Overpressure Threshold to 500 torr – when the Ammonia ALD valve is pulsed, the resulting pressure increase can cause an error if this valve is not increased from the default value of 250 torr.

Press the Start button to being processing. As the recipe progresses you should observe pressure spikes as each ALD valve opens and closes. Absence of a spike during each pulse may indicate a problem with the ALD valve or the exhaustion of the precursor. If you have made an error in the recipe or something does not appear to be functioning correctly, press the Abort button to end the recipe.



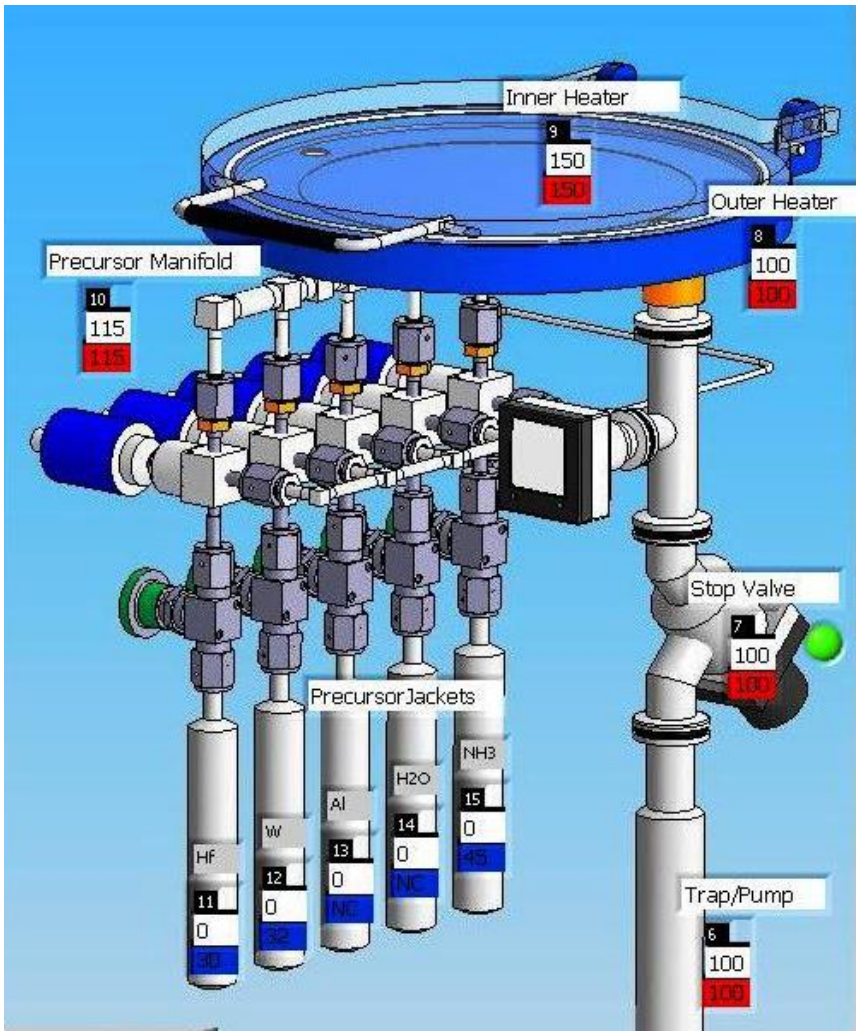
When the recipe has ended, close the precursor valves and Vent the chamber to recover your samples.

If no further samples are being processed, load the recipe "DEGAS" and press Start. This recipe will purge the headspace of excess precursor vapor, helping to maintain purity and increasing the lifetime of the ALD valves.

When processing is complete, return the system to idle state temperature using the following values:

Item	**TC #**	**Setpoint**
Foreline	6	100°C
Isolation Valve	7	100°C
Outer Heater	8	100°C
Inner Heater	9	150°C
Manifold	10	115°C

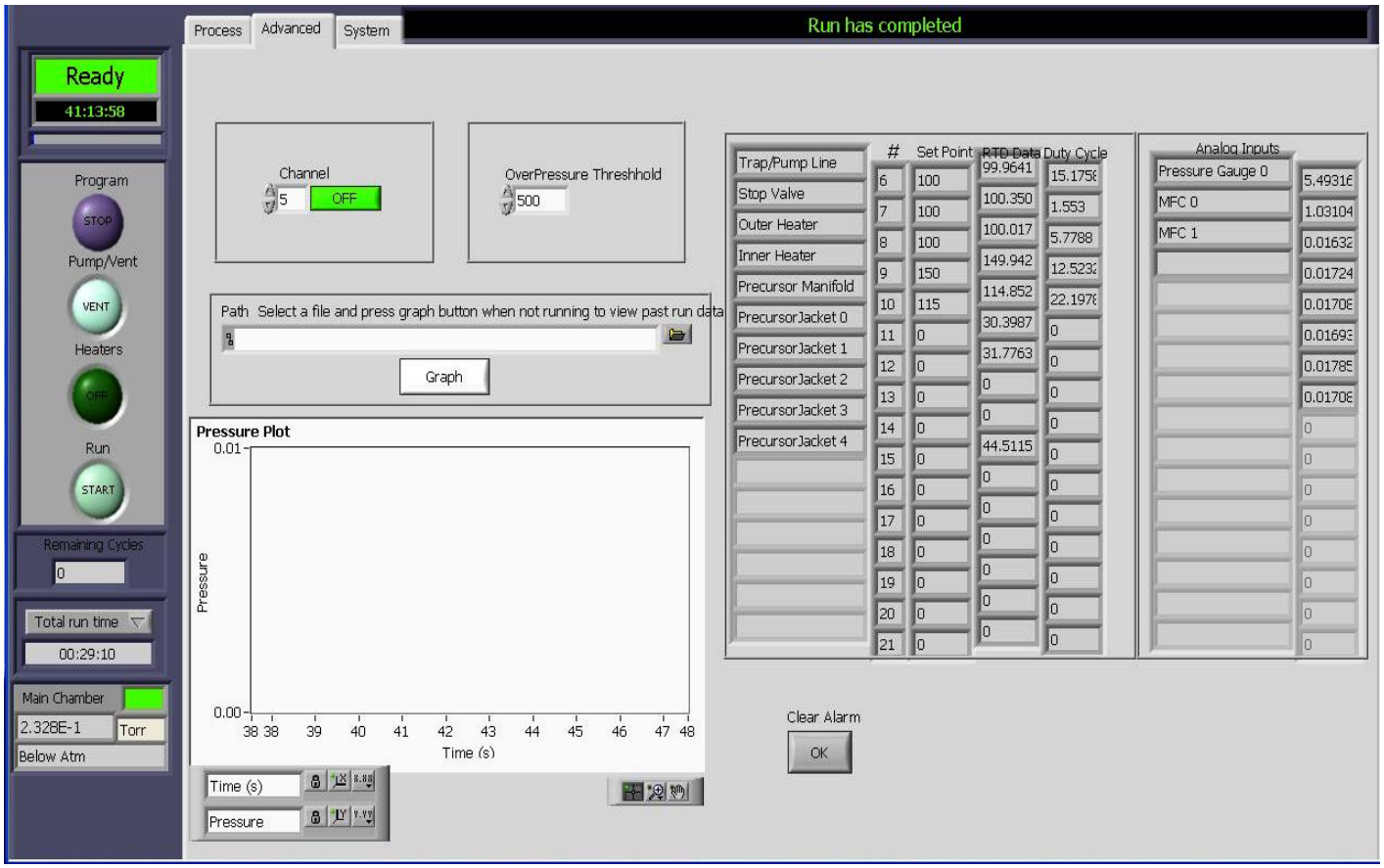
All precursor temperatures should be set to 0°C.



Other Information:

In the upper left of the main screen are tabs to access further systems options:

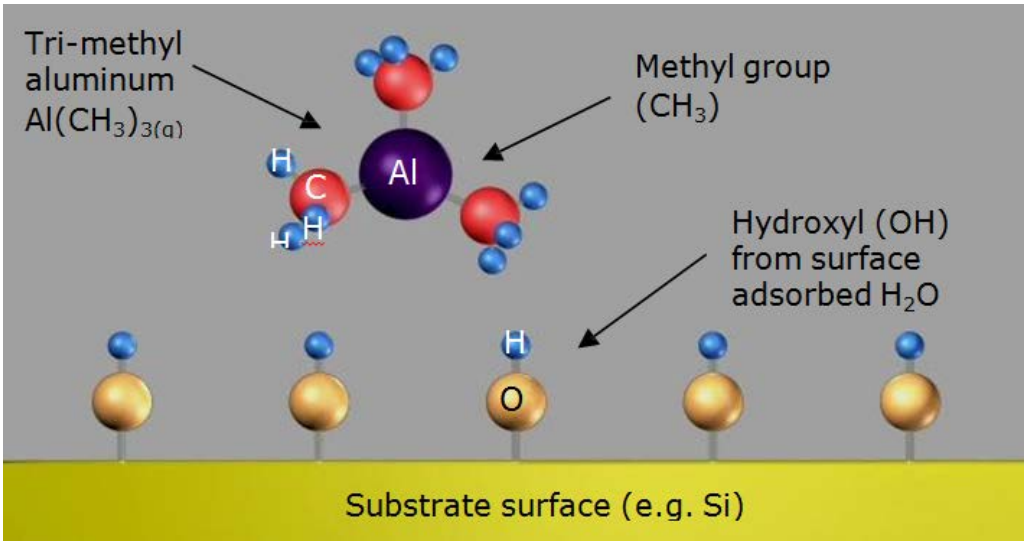
Clicking on the Advanced tab will bring you to a process run review screen.

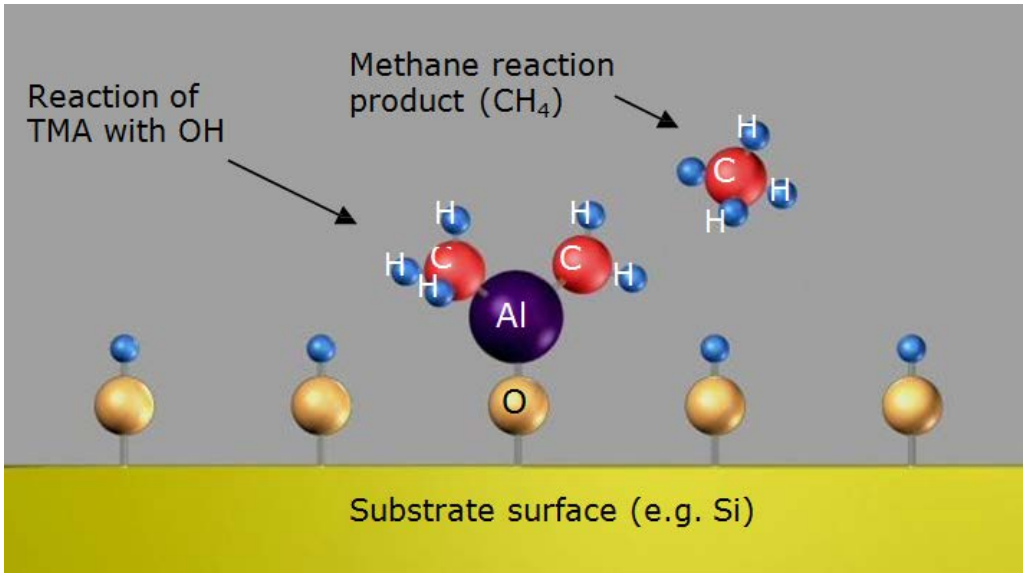


Atomic Layer Deposition: Principle of Al_2O_3 formation

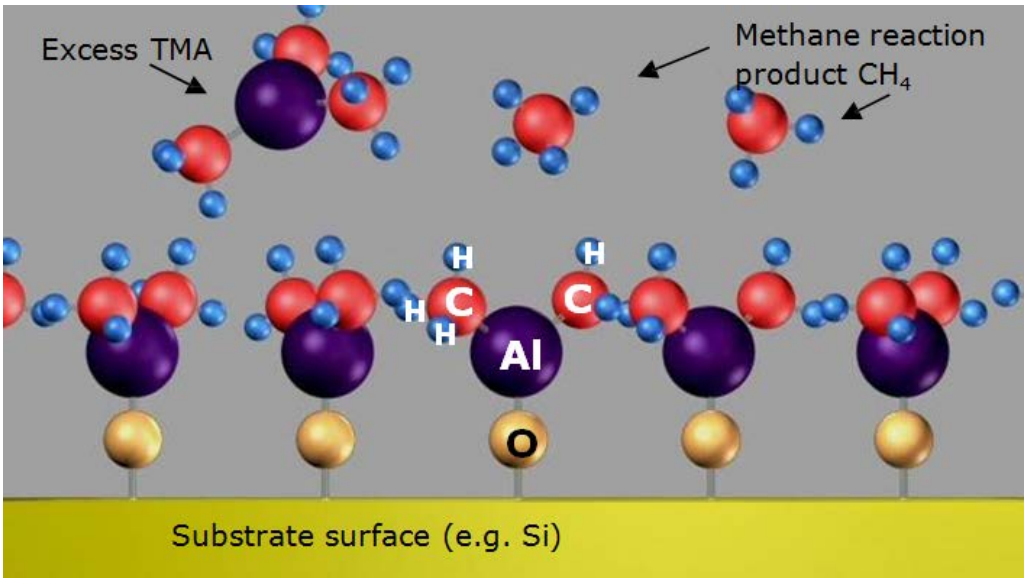
Atomic Layer Deposition (ALD) is a technique that allows growth of thin films, atomic layer by atomic layer. The typical ALD reaction is illustrated via the formation of Al_2O_3 from trimethylaluminum (TMA) - $Al(CH_3)_3$, and water - H_2O. Recipes for other materials can be found in the literature.

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.

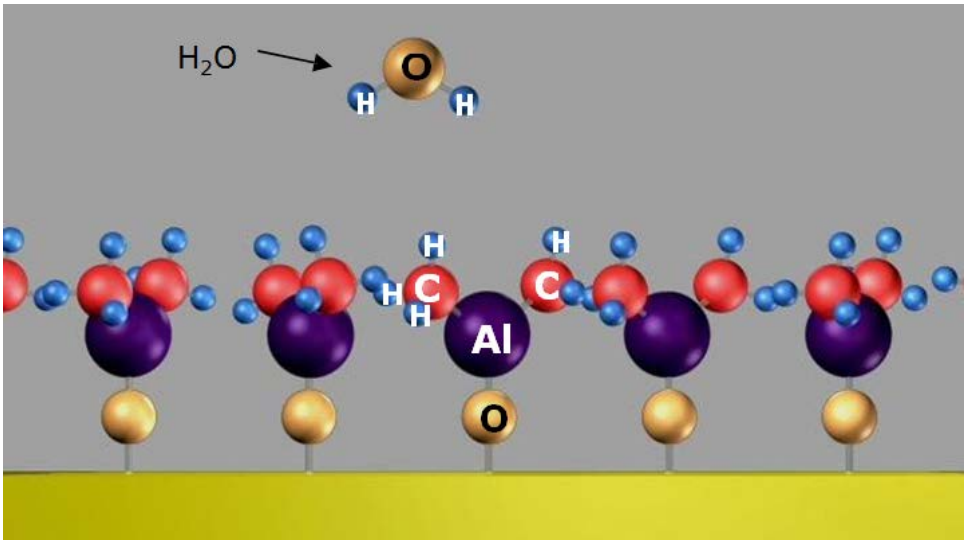


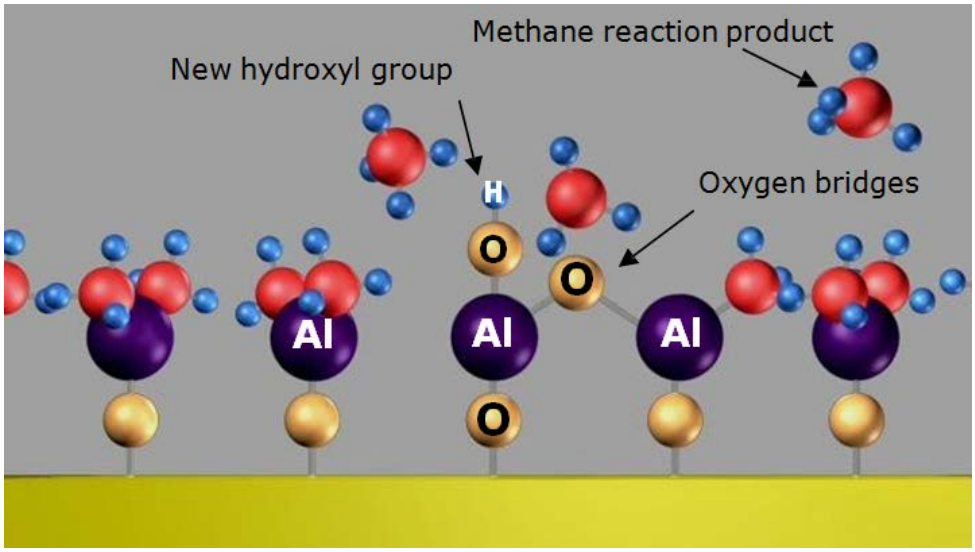


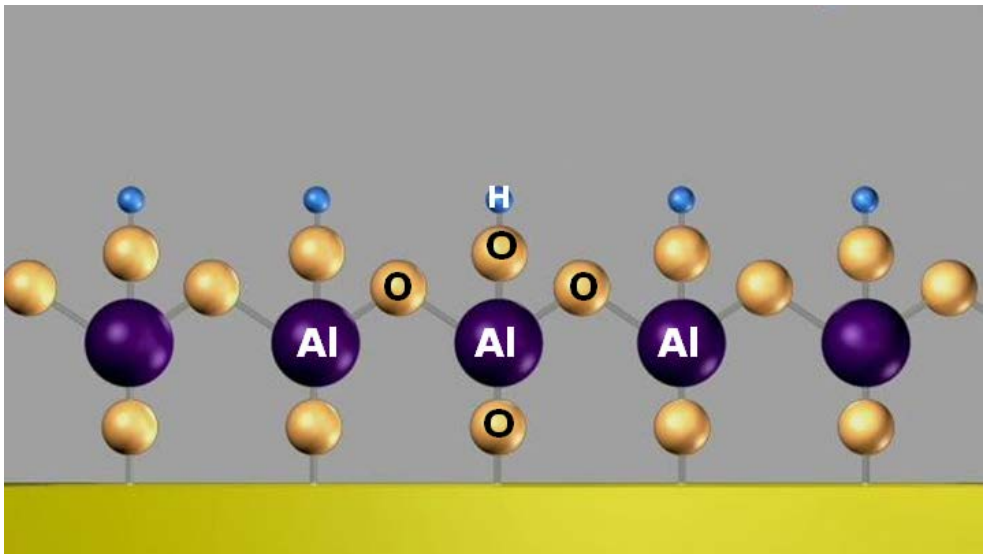
Step 2: Removal of the unreacted precursor and reaction products.
Unreacted precursor and the methane (CH_4) liberated from the reaction are removed by simple evacuation of the sample chamber or by flowing inert gas over the surface.



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 is repeated until a film of the desired thickness is deposited. Each atomic layer is grown on the order of 1Å per cycle.