University of Minnesota Nano Fabrication Center
Standard Operating Procedure

Equipment Name:	ALD		
Coral Name:	ald	**Revision Number:**	8
Model:	Savannah	**Revisionist**:	T. Whipple
Location:	Bay 1	**Date:**	3/14/2017

1 Description

The ALD, Atomic Layer Deposition system is a Savannah series from Cambridge Nano Tech Inc. of Cambridge, MA now owned by UltraTech deposits thin films a layer at a time. This is done by cycling a single precursor for a short amount of time. Then another precursor is cycled. These precursors are alternated to form a film. The more alternating cycles, or loops that are ran the thicker the resulting film will be. The steps to run the ALD system running are very few. It is an easy machine to run.

2 Safety

a The system uses several gases, and of the gases are a pyrophoric gas. Pyrophoric means it will burn if exposed to air.

b The system uses electrical power and is under vacuum, so be aware these items. There should be **no** odor whatsoever. If you smell an odor, put system in STANDBY, and leave the area and Contact staff

c The system is heated, the top plate and other parts of the chamber are very hot so be careful while loading and unloading wafers, do not burn your sleeve.

d Do not edit the recipe beyond the number of cycles. Do not save the recipe.

3 Restrictions/Requirements

a Must be a qualified user on the ALD.

4 Required Facilities

a Compressed air
b Nitrogen
c House Exhaust

5 Definitions

a Precursor – A gas or liquid that is one of the building compounds to make a layer.
b Stop Valve – This is the main valve that opens the chamber to the pump.

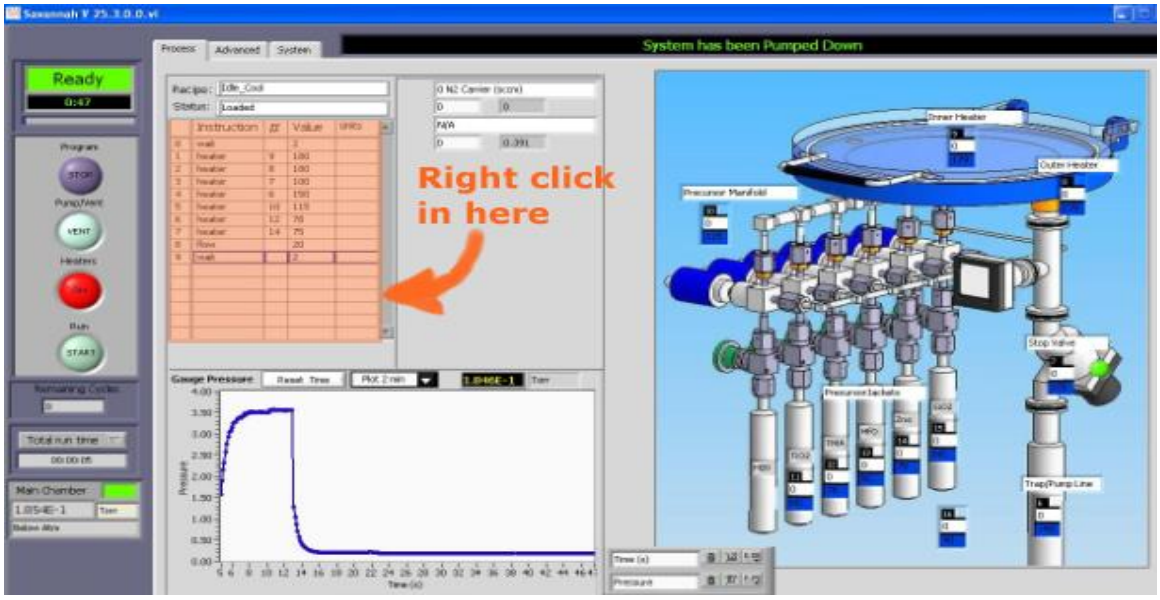
6 Operating Instructions

a LOGGING ON

1 Check Badger for other reservations for the “ald” system first.
2 Enable "ald" on Badger.

b SETUP PROCEDURE *Selecting the operating temperature*

1 The ald system should have been left in Standby with the **Idle_Cool** recipe running. Select the startup recipe to run the temperature to operate at. This is done by the right mouse button while clicking in the recipe display section. This will cause a menu list to be displayed. Click the **Load Recipe** option by pressing the right mouse button and then selecting a **Start_up** recipe. Then press **OK** and then press the **START** button to set temperatures. If you plan to run at lower temperature, vent immediately. See appendix for recipe list.



Select recipe by right clicking mouse in the area displayed above to get recipe menu.

Select the depositing Temperature.

Load a StartUp recipe

Run the recipe to have the temperature set.

Vent the chamber, load sample, pump down.

Select film recipe and the number of cycles.

Start the recipe to deposit film.

c SAMPLE LOADING *Vent Chamber*

1 To vent the system, press the button labeled **Pump/Vent** and change the Flow the value to 100 sccm. After 4.5 minutes the top lid should be lifted open. Place your sample in the chamber. The sample size must be 2cm or larger. Place the surface of the sample to be deposited facing upward. Put sample at the center is best, it might move slightly during the pump down, block small samples with glass slides.

Fig 2. Pump/Vent valve - click to vent chamber 

2 If the film deposition is at a lower temperature - wait with lid open until it cools.
3 Close the cover and align it as you like to.
4 Press the **Pump/Vent** button and it is best to press down on the lid cover while it first pumps down. It help with minimizing the wafer movement.

Fig 3. To Pump/Vent valve to evacuate chamber

5 Adjust the flow to the 20 sccm value and allow the chamber to pump down. It should be pumped down in ~15 Seconds, watch the pressure graph go down.

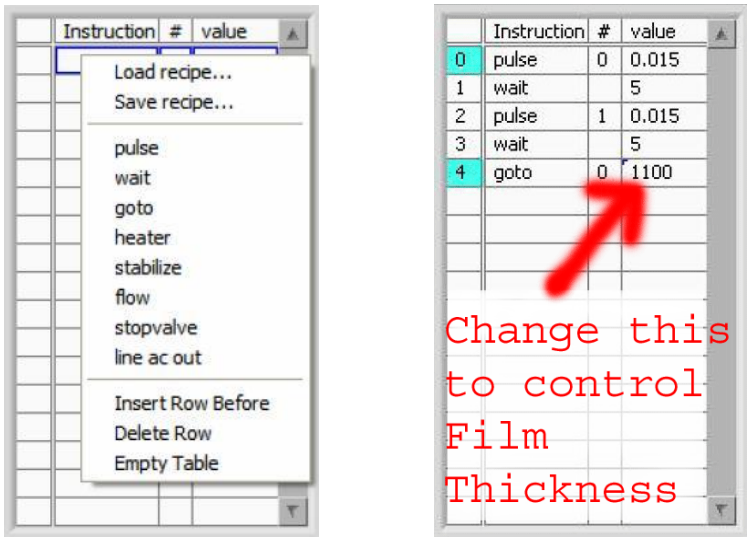


Fig 1. Loading of recipes, only select the Load Recipe option, edit **goto** line.

d STARTING PROCESS *Depositing film*

1. After several minutes the system will be at the correct temperature. When the temperature is within 3 degrees you can start. The outer chuck temperature will be not be at exact setpoint, but this is okay.

2. Load the desired recipe to be ran. This is done the same way as above. For Al2O3 film select the **Al2O3_dep recipe** from the list.

3. Select the number of cycles that you want the process to run by selecting the last line in the recipe, the line with the **GOTO** command and left click the box, and enter the number of cycles in the **Value** column. Notice the time it takes to run.

WARNING:

DO NOT alter the recipe except for the **goto** step value. Making any other adjustment to the recipe could result in an unsafe condition. Seek MNC staff for help if recipe changes are needed to be made.

4 Press the **START** button and **OK** button, and then watch the recipe. The graph will start changing in the Gauge Pressure display with a repeating pattern. Buttons on the left side will be lighter looking, the START button will change to say ABORT.

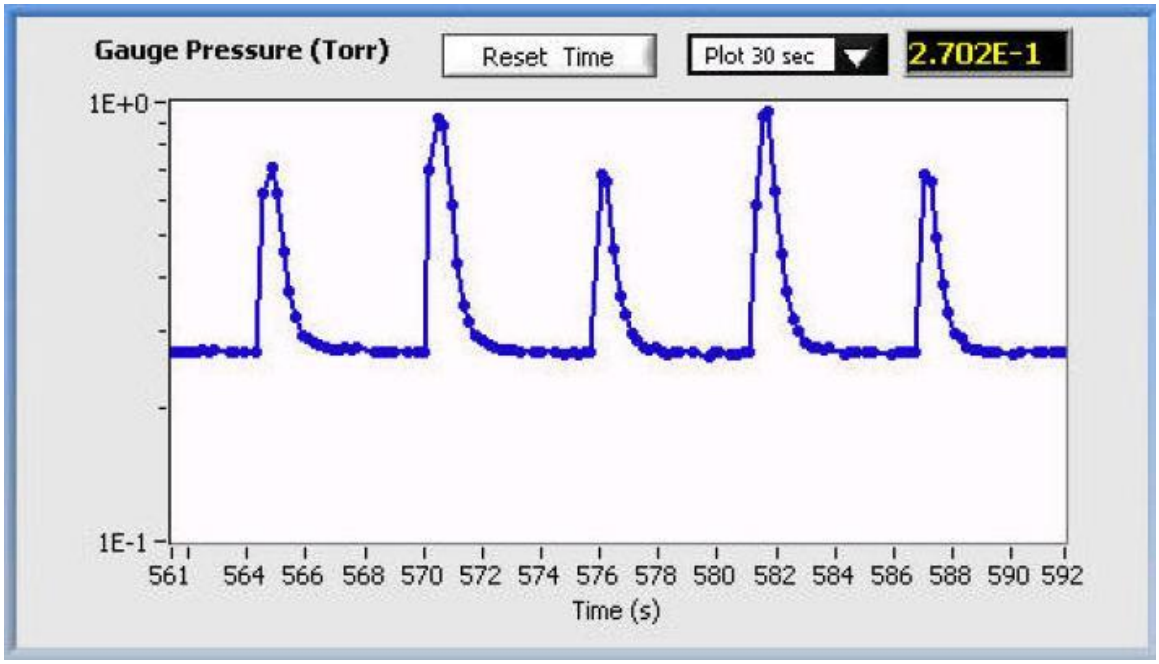


Fig. 2. Notice the repeating wave forms, switching between a higher and lower peak.

5 It best to monitor the process from time to time. Making sure the graph looks correct and the temperature of the heaters are at the correct settings.

6 The amount of time left of the process can be checked by looking at the section labeled **Remaining Cycles** and below that three time options can e selected: **Actual time remaining**, **Total run time** or the **Completion time** the run will be completed.

7 When the run is finished, the process will stop and nothing on the display will be changing. The buttons that were grey looking will now have color, and the Abort button will change back to **START**.

8 Remove the sample by doing the sequence of events that was listed above to vent the chamber. Click on the **Pump/Vent** button and increasing the flow to 100sccm value. It will reach atmosphere in 3 to 4 minutes.

9 Once the chamber is at atmosphere place the lid guard on the holder on the wall and remove the wafer from the system. Also notice the position of the wafer now compared from its loading position. Remember that the wafer and chamber are still hot, so be careful.

10 Close the lid and pump the system down and return the lid guard on the system.

11 Load the **Ilde_Cool** recipe. Run the recipe by pressing START button.

12 Log out of Badger.

WARNING:

DO NOT EVER **REMOVE** any precursors from the system.
DO NOT PRESS **ABORT** unless there is a problem with the system.
DO NOT EVER **EDIT** a recipe.
DO NOT EVER process ALD films in Bay 1 wet benches.
DO NOT EVER process ALD films in Tylan furnaces.

7 Problems and Solutions

1. The chamber did not vent.

   Check that the normal status of the screen options are correct.

Is the program running, the top button display " Running " ?
Does the display say " Waiting for system to vent " - if it vents then just press abort button.

2. The normal program is not displayed. How can this be started.
   Double click on the icon labeled: **ALD Control**

3. The system was shutdown, what can be done?
   Contact MNC staff person

4. The precursor is empty, now what?
   Contact MNC staff person and also remember the number cycles you have left.

5. The deposit rate is much different than expected.
   Measure the film on another system, check refractive index of the film too. Confirm that the substrate is normal and have had known results. Set the time the graph displays to allow viewing of the whole run, anything changes seen?

6. The system was shutdown, what can be done?
   Contact MNC staff person

8 Appendix

Images

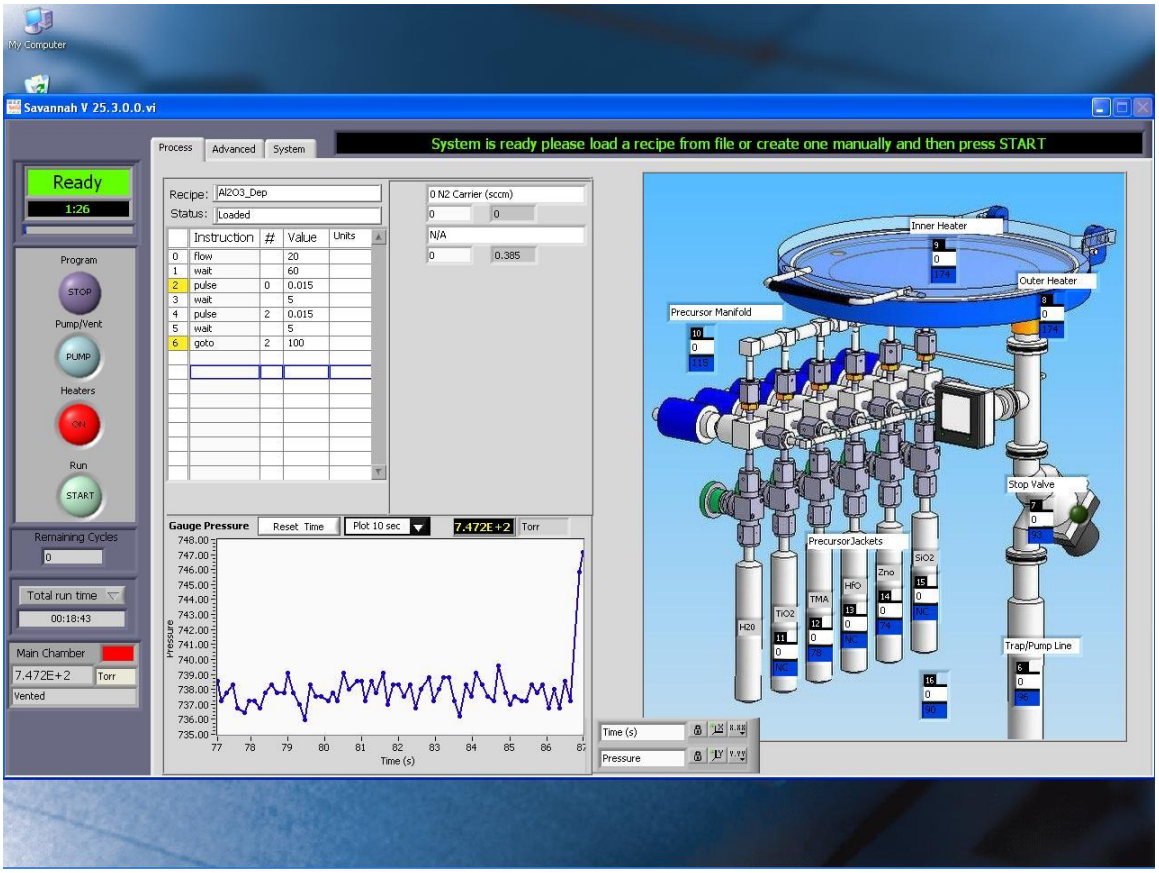


Fig. 3. The main operations screen for the ALD system.

Fig. 4 The two mouse buttons are on the bottom, below the mouse pad area.

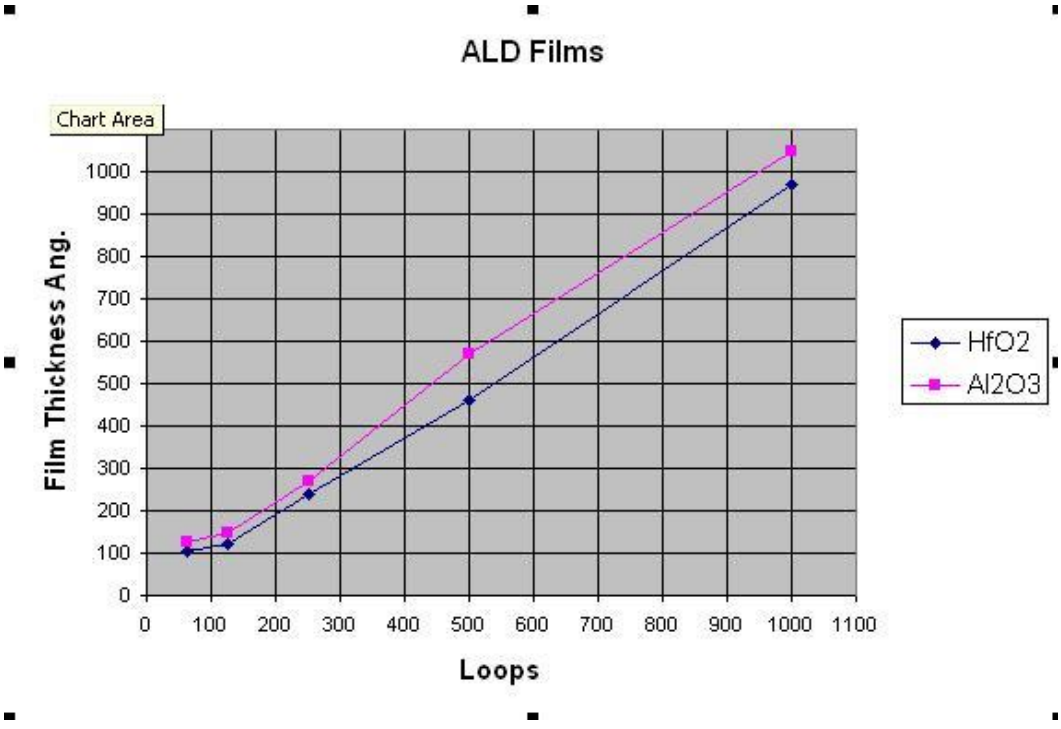


Fig. 5 ALD film thickness for Al2O3 and HfO2 films.

List of Process recipes:

Standard processing recipes:

Al2O3_Dep	Use this for all normal temperature Al2O3 films.
HfO2_Dep	Use this for all normal temperature **HfO2** films.
Idle_Cool	Default recipe to leave the system in while not running.
Start_Up	Same as **Start_Up 250** C recipe
ZnO_Dep	Use this for most **ZnO** films
Oxide---May08	Use this for most **SiO2** files, rate is about ~ 60 Ang / loop
HfO2_130_Degree_Dep	Use this for all lower temperature **HfO2** films.
Al2O3_130_Degree_Dep	Use this for all lower temperature **Al2O3** films.

Temperature setup Recipes:
Start_Up__50_Degrees
Start_Up__80_Degrees
Start_Up_100_Degrees
Start_Up_130_Degrees
Start_Up_160_Degrees
Start_Up_235_Degrees
Start_Up_250_Degrees
Start_Up_300_Degrees

Precursors used in the ALD system

Al2O3: Trimethylaluminum [TMA] and water vapor.
Sigma-Aldrich Part number: 663301-25G
precursor at room temperature

HfO2: Tetrakis(dimethylamido)hafnium(IV) [TDMAH] and water vapor.
Sigma-Aldrich Part number: 666610-25G
precursor at 75 C

SiO2: Tris(dimethylamino)silane **[** TDMAS**]** and ozone**.**
Sigma-Aldrich Part number: 759562-25G precursor at room temperature

TiO2: Tetrakis (dimethylamido)titanium(IV) [TDMAT] and water vapor.
Sigma-Aldrich Part number: 669008-25G
precursor at 78 C

ZnO: Diethylzinc [DEZ] and water vapor.
Sigma-Aldrich Part number: 668729-25G
precursor at room temperature

Common issues to be aware of with the ALD system..

Make sure to include a bare Si sample to measure added thickness from ALD. Best to premeasure the Bare Si wafer using ellipsometer program' thintest ' normal is 30Ang.

Mouse cursor stuck in the upper right corner? Log out and back in - Badger thing.

The ALD process depends on the surface condition of the substrate.

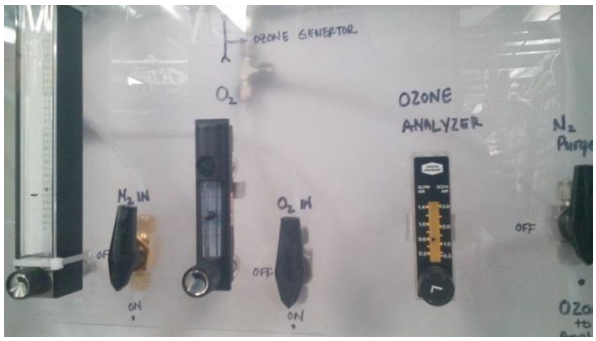
Al2O3 can be wet etched using BOE ~ 350 Ang/min. **Do not use in Bay 1**.
Dry etching of Al2O3 can be done but a slow rate.
Al2O3 can be used as an etch mask for DRIE, has great etch selectivity > 1000:1
See application note for more details on this.

HfO2 can be etched by RIE . Can withstand some BOE etching **Do not use in Bay 1**.

Lower temperatures process will have a higher deposition rate, see chart on wall. The film quality and step coverage will be reduced, the exact amount has not been measured.

OZONE operation.
The ozone process is only 4 easy steps

Step1:
Make sure that the new SiO2 precursor is installed in the system.
Turn on the three valves in back above the ozone – all need to be pointing down – turn them all CCW. Next turn on the main power breaker to the ozone generator.



Gas valves are on – all pointing downward

Main power switch turned ON for ozone generator

Step2:
Go to the ALD computer screen and run the program ' Ozone Prime ' while that is running go in back and turn the Ozone ON & OFF/RESET switch down to OFF/RESET and then back up. This will clear the error E06 that was displayed. Next spin the dial of the POWER knob clock wise until it is reads 80% Then go in front and and confirm the pressure inside the system.

Clear error E06 by switch down and up

Turn the knob many times to get 80% power level

Step3:
Look at the analyzer to make sure there is a ozone value, if it is not shutdown the system.
Write down the values and monitor all the values during the run.

Step4:
Shut down the system by running' Ozone_off' recipe and then turning off the gasses in back and then turning off the main generator power switch.