University of Minnesota Nano Center

ALD Savannah - Standard Operating Procedure

Badger Name:	K1 ALD Savannah Ultratech	**Revision Number:**	9
Model:	Savannah	**Revisionist**:	Wanjohi Kimani
Location:	Bay 1, KH	**Date:**	March 31, 2020

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1 Scope

1.1 This document provides detailed instructions on how to properly operate the thermal Savannah Atomic Layer Deposition (ALD) tool.

2 Tool Description

The thermal Atomic Layer Deposition (ALD) system is a Savannah series tool from Cambridge Nanotech Inc. of Cambridge MA, now owned by UltraTech. The ALD tool deposits thin films a layer at a time. This is done by cycling a single precursor for a short time, and then another precursor is cycled for a short duration. By cycling precursors for a given number of loops, a film of a defined thickness is obtained. More alternating cycles or loops lead to a thicker film. The steps to run the thermal ALD system running are very few. It is an easy machine to run.

3 Safety

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

3.2 The system uses electrical power and runs under vacuum. There should be **no** odor whatsoever. If you smell any odor, put system in STANDBY, leave the area and contact staff

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

3.4 Only edit the number of cycles. Do not edit the recipes beyond that. Do not save recipes.

4 Restrictions/Requirements

4.1 Must be a qualified user on the ALD.

5 Required Facilities

5.1 Compressed air
5.2 Nitrogen
5.3 House Exhaust

6 Definitions

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

7 Operating Instructions

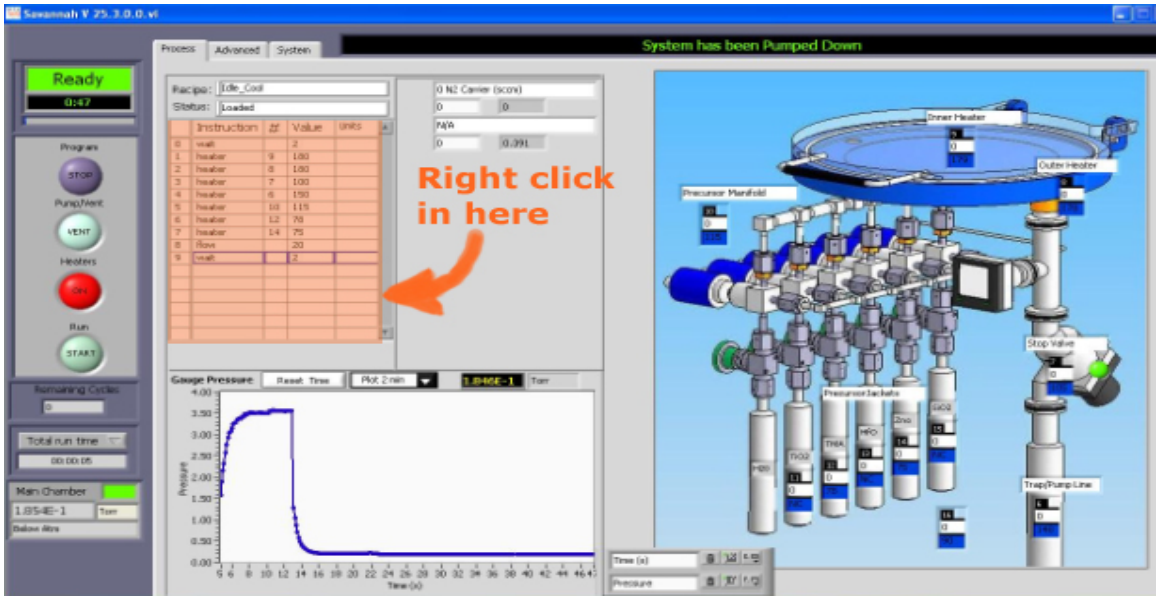
7.1 LOGGING ON

7.1.1 Check Badger for other reservations for the “K1 ALD Savannah Ultratech” system first.

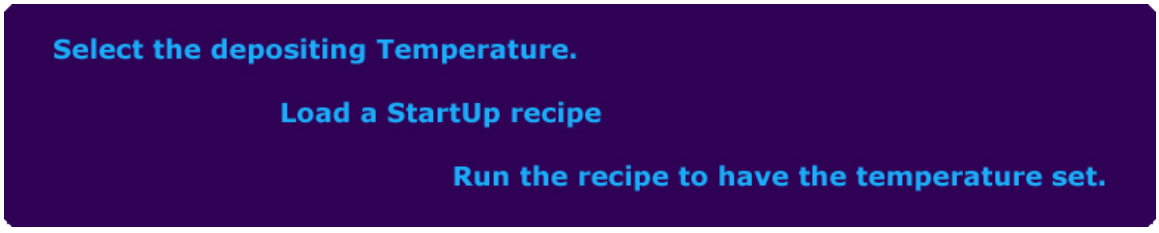
7.1.2 Enable " K1 ALD Savannah Ultratech " on Badger if not reserved or in use.

7.2 SETUP PROCEDURE *Selecting the operating temperature*

7.2.1 The ALD system should be in Standby with the **Idle_Cool** recipe running at idle. Select the startup recipe, which sets the temperature you plan to run at. Do this by right clicking the mouse button 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 **Startup** recipe for your desired temperature. 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.



Vent the chamber, load sample, pump down.

Select film recipe and the number of cycles.

Start the recipe to deposit film.

7.3 SAMPLE LOADING *Vent Chamber*

7.3.1 To vent the system, press the button labeled **Pump/Vent** and change the flow value to 100 sccm. After about 4½ minutes, the top lid should be free to open. *If the lid can be opened, but the software shows it is still venting, click Abort to end the venting message*. Place your sample in the chamber. The sample size must be 2cm or larger. There is a thickness limitation with the default chamber. Place the surface of the sample to be deposited facing upward. Placing 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

7.3.2 If the film deposition is at a lower temperature - wait with lid open until it cools.

7.3.3 Close the lid and align it to the lower chamber.

7.3.4 Press the **Pump/Vent** button and it is best to press down on the lid cover while it first pumps down. It helps with minimizing the wafer movement.

Fig 3. To Pump/Vent valve to evacuate chamber

7.3.5 Adjust the flow to the 20 sccm value and allow the chamber to pump-down. It takes approximately 15 Seconds to pump down; watch the pressure graph go down.

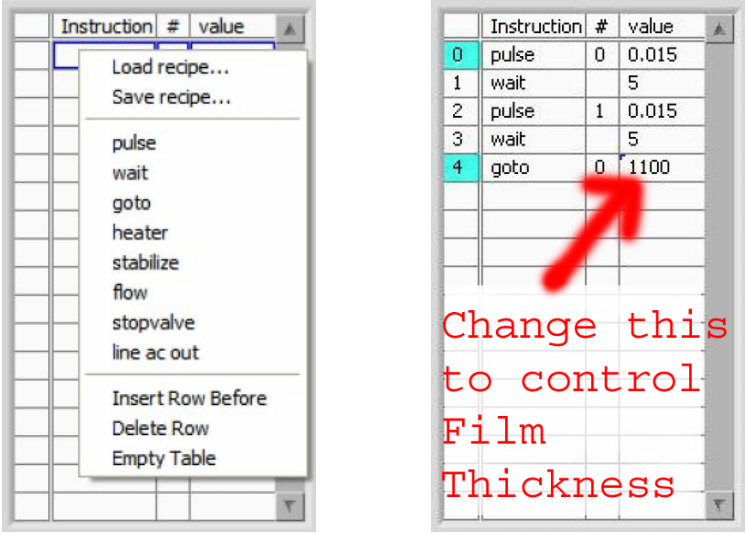


Fig 1. Loading of recipes; only select the Load Recipe option; only edit **goto** line.

7.4 STARTING PROCESS *Depositing film*

7.4.1 You can start a run once the temperature is within 3-degrees of the setpoint. The outer chuck temperature will be not be at exact setpoint, but this is okay.

7.4.2 Load the desired recipe run. Right click in the recipe display area, and then click on **Load recipes**. For Al_2O_3 film, select the **Al2O3_dep recipe** from the list.

7.4.3 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 in the box and enter the number of cycles in the **Value** column. Notice the time it takes to run on the left bottom side of the screen.

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.

7.4.4 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 greyed-out; the START button will change to show ABORT.

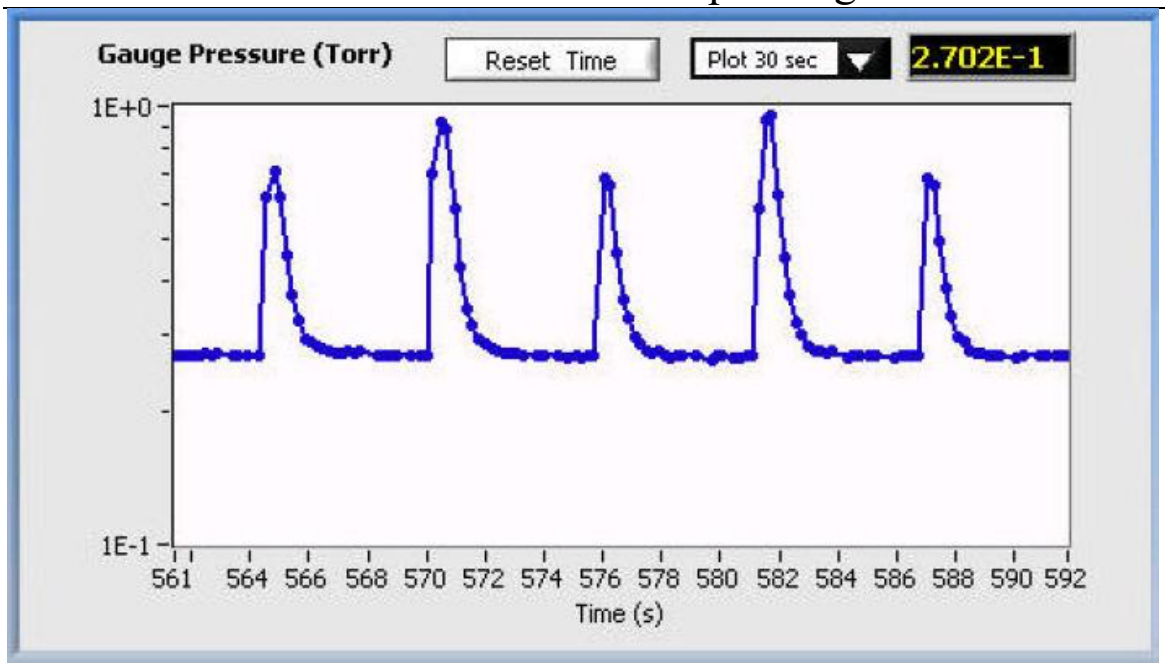


Fig. 2. Notice the repeating waveforms, switching between a higher and lower peak.

7.4.5 It best to monitor the process from time to time. Make sure the graph looks correct and the temperature of the heaters are within range of settings.

7.4.6 The amount of time left of the process is displayed at the section labeled **Remaining Cycles;** below that, three-time options are available: **Actual time remaining**, **total run time** or the **completion time** when the run will be complete. You can switch between the different options as desired.

7.4.7 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**.

7.5 Remove the sample by following the sequence of events above to vent the chamber. Click on the **Pump/Vent** button and increase the flow to a 100sccm value. The chamber will reach atmosphere in about 4½ minutes.

7.5.1 Once the chamber is at atmosphere place the lid guard on the holder on the wall and remove the wafer from the platen. 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.

7.6 Close the lid, pump the system down, and return the lid guard on the system.

7.6.1 Load the **Ilde_Cool** recipe. Run the recipe by pressing START button and OK.

7.6.2 Log out of Badger.

7.7 Warnings

7.7.1 DO NOT EVER **REMOVE** any precursors from the system.

7.7.2 DO NOT PRESS **ABORT** unless there is a problem with the system.

7.7.3 DO NOT EVER **EDIT** a recipe.

7.7.4 DO NOT EVER process ALD films in Bay 1 wet benches.

7.7.5 DO NOT EVER process ALD films in Tylan furnaces.

8.0 Problems and Solutions

8.1 The chamber did not vent.

Check that the normal status of the screen options is correct.
Is the program running, the top button display "Running?"
Does the display say "Waiting for system to vent "? If it is vented, then just press abort button.

8.2 The normal program is not displayed. How do you start the program?
Double click on the icon labeled: **ALD Control**

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

8.4 The precursor is empty, now what?
Contact MNC staff person and remember the number cycles you have run/left to run.

8.5 The deposit rate is much different from 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 that the graph displays to allow viewing of the whole run, any changes observed.

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

9. Appendix

Images

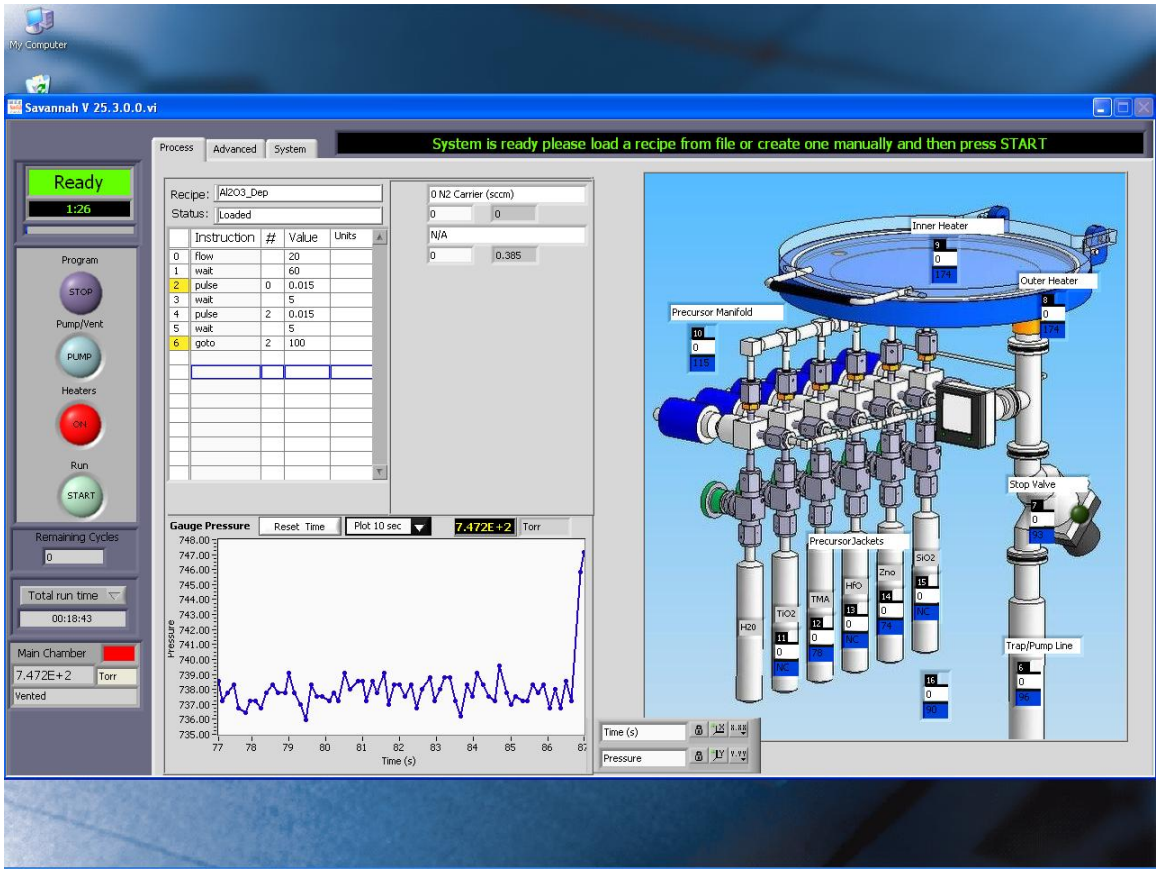


Fig. 3: The main operations screen for the ALD system.

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

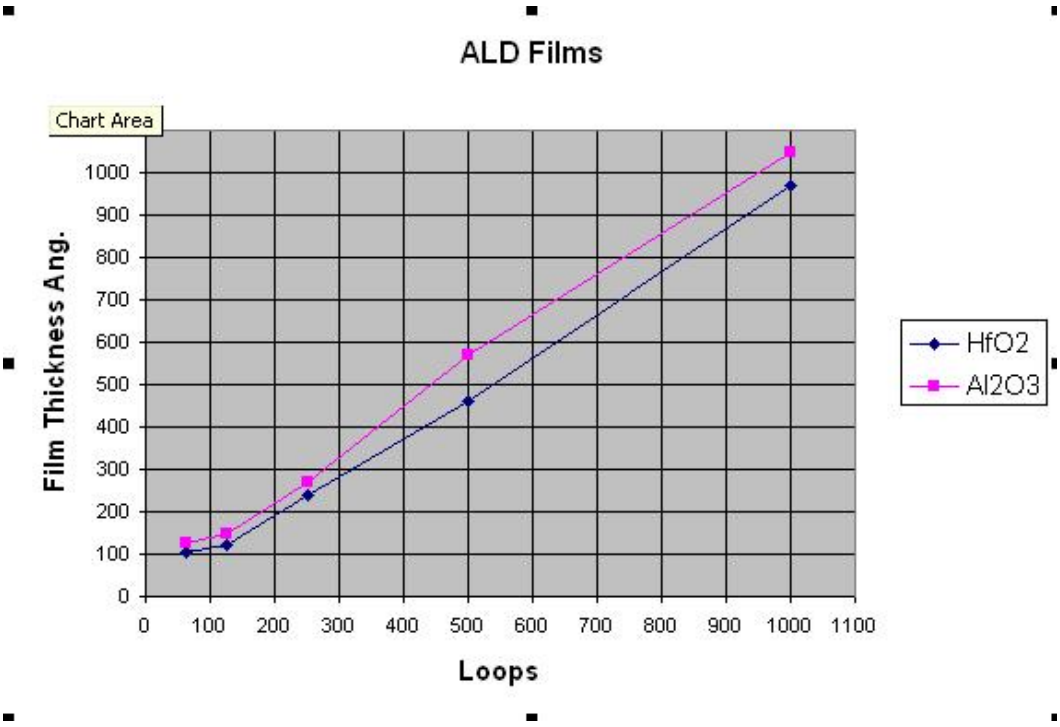


Fig. 5: ALD film thickness for Al_2O_3 and HfO_2 films.

List of Process recipes:

Standard processing recipes:

Al2O3_Dep	Use this for all normal temperature Al_2O_3 films.
HfO2_Dep	Use this for all normal temperature **HfO_2** films.
ZrO2_250	Use this for all normal temperature **ZrO_2** 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
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

Al_2O_3: Trimethylaluminum [TMA] and water vapor.
Sigma-Aldrich Part number: 663301-25G
precursor at room temperature

HfO_2: Tetrakis(dimethylamido)hafnium(IV) [TDMAH] and water vapor.
Sigma-Aldrich Part number: 666610-25G
precursor at 75°C

SiO_2: Tris(dimethylamino)silane **[**TDMAS**]** and ozone**.**
Sigma-Aldrich Part number: 759562-25G precursor at room temperature

TiO_2: 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

ZrO2: Tetrakis(dimethylamine)zirconium [TDMAZr] and water vapor
Cas# 19756-04-08
Precursor at 75°C

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 LSE Gaertner ellipsometer (Thinoxide; set expected thickness to 30Å and expected RI to 1.46). Normal thickness is 15Å – 30 Å.

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.

Al_2O_3 can be wet etched using BOE ~ 350 Å/min. **Do not use in Bay 1**.
Dry etching of Al2O3 can be done but at a slow rate.
Al_2O_3 can be used as an etch mask for DRIE, has great etch selectivity > 1000:1

See application note for more details on this.

HfO_2 can be etched by RIE. Can withstand some BOE etching. **Do not use in Bay 1**.

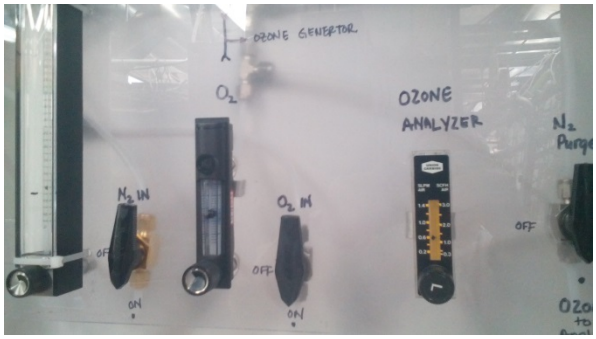
Lower temperatures process will have a higher deposition rate, see chart on wall. The film quality and step coverage is reduced; the exact amount has not been measured.

10.0 OZONE operation.

The ozone process is only 4 easy steps

10.1 Step1:

Make sure that the new SiO_2 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

10.2 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 clockwise until it is reads 80% Then go in front 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

10.3 Step3:

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

10.4 Step4:

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