

University of Minnesota Nano Fabrication Center

Standard Operating Procedure

Equipment Name: ALD
Coral Name: ald
Model: Savannah
Location: Bay 1
Revision Number: 5
Revisionist: T. Whipple
Date: 9/16/08

1 Description

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

2 Safety

- a The system currently uses several gases, and could have other gases in the future that are a pyrophoric and toxic 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 As the system is being heated the top plate and other parts of the chamber are very hot so be careful while loading and unloading of wafers
- 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.

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6 Operating Instructions

a LOGGING ON

- 1 Check CORAL for other reservations for the ald system.
- 2 Enable "ald" on CORAL.

b SETUP PROCEDURE

- 1 The ald system should have been left in the Standby with the **Idle_Cool** recipe running when it is not in use. Select the startup recipe, and this will set the temperatures to increase to the correct operating value. 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 the **Start_up** recipe. Then press the **OK** button. Select another startup recipe if you plan to running at lower temperature, see appendix for recipe list. **Where does extra "Press OK go?"**

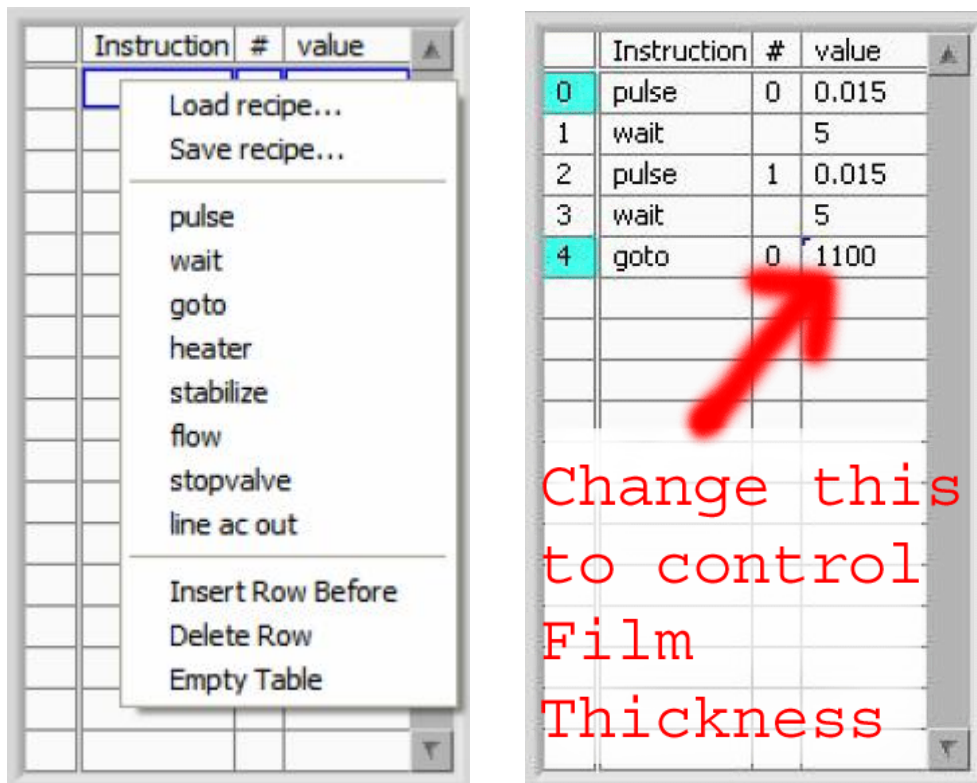


Fig 1. Loading of recipes, only select the Load Recipe option, edit **goto** line.

This will load the recipe to be ran. See the Appendix for keyboard image and labels.

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- 2 After several minutes the system will be at the correct temperature. If the main program window is not displayed see the section labeled “ Problem and solutions” in the Appendix.
- 3 Load the desired recipe to be ran. This is done the same way as above. For Al₂O₃ film select the Al₂O₃_dep recipe and for HfO₂ film select the HfO₂_dep recipe. Also for lower temperature select film_130_Degree_Dep recipe, see appendix for recipe list.
- 3 Load the desired recipe to be ran. This is done the same way as above. For Al₂O₃ film select the Al₂O₃_dep recipe and for HfO₂ film select the HfO₂_dep recipe.
- 3 Select the number of cycles that you want the process to run by selecting the last line in the recipe and select the line with the **GOTO** command and right click the box, and enter the number of cycles in the **Value** column.

c SAMPLE LOADING

- 1 To vent the system, press the button labeled **Stop Valve** and change the Flow the value to 100 sccm. After a few minutes the top lid will be able top 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. Placing the sample at the center is best, but the sample most likely will move slightly during the pump down.



- 2 Close the cover.
- 3 Press the Stop Valve button and it is best to press down on the lid cover while it first pumps down. This seems to help with minimizing the wafer movement. Adjust the flow to the 20 sccm value and allow the chamber to pump down. It should be pumped down in 1 to 2 minutes. Watch the pressure graph and see it stabilize.

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 NFC staff help if recipe changes are needed to be made.

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

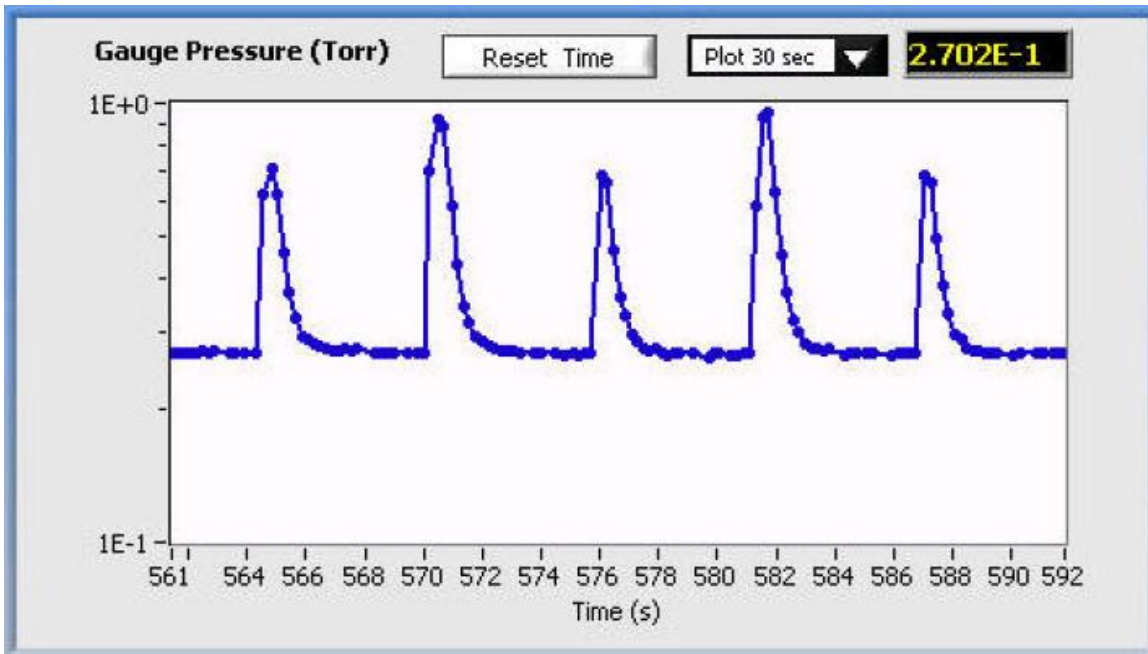


Fig. 2. Notice the repeating wave forms, switching between a higher and lower peak.

- 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 be 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 **STOP Valve** 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.

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- 10 Close the lid and pump the system down and return the lid guard on the system.
- 11 Load the **Idle_Cool** recipe. Run the recipe by pressing START button.
- 12 Log out of CORAL.

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.

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 “ ?
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 NFC staff person
4. The precursor is empty, now what?
Contact NFC staff person
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 NFC staff person

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8 Appendix

Screen images

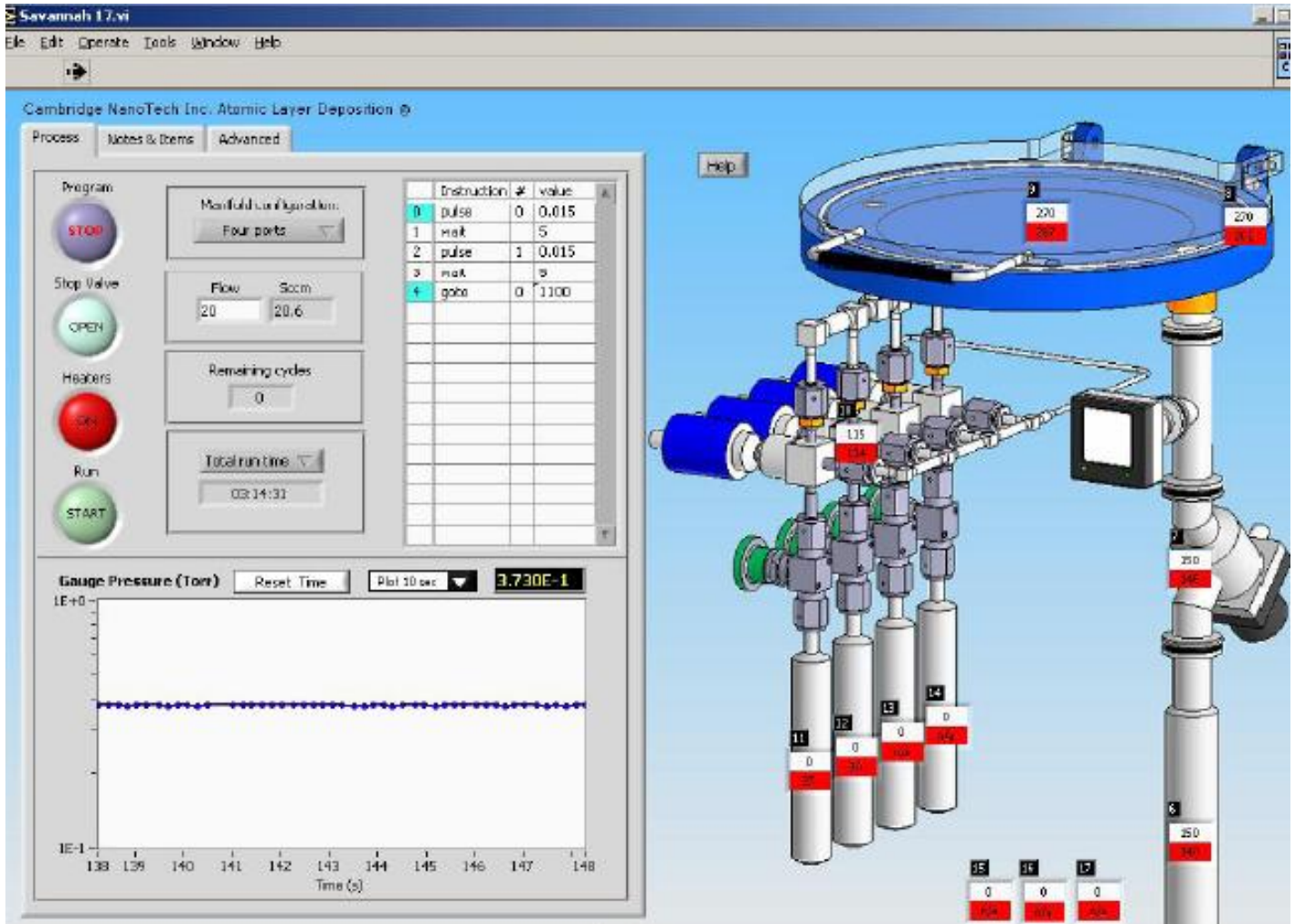


Fig. 3. The main operations screen for the ALD system.

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

ALD Films

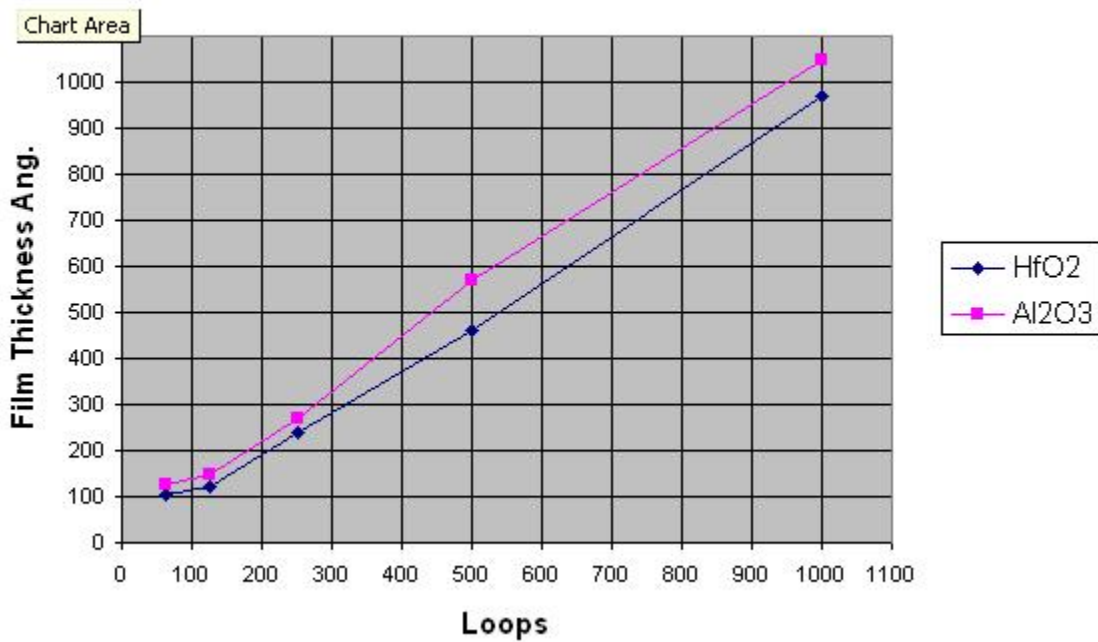


Fig. 5 ALD film thickness for Al₂O₃ and HfO₂ films.

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List of Process recipes:

Standard processing recipes:

Al₂O₃_Dep
HfO₂_Dep
Idle_Cool
Start_Up
ZnO_Dep

Custom Recipes:

Start_Up_100_Degrees
Start_Up_130_Degrees
Start_Up_160_Degrees
Start_Up_235_Degrees
Start_Up_250_Degrees

HfO₂_130_Degree_Dep Use this for all lower temperature HfO₂ films.
Al₂O₃_130_Degree_Dep Use this for all lower temperature Al₂O₃ films.

Precursors used in the ALD system

Al₂O₃:

Trimethylaluminum (TMA) and water vapor.

HfO₂:

Tetrakis(dimethylamido)hafnium(IV) and water vapor.

SiO₂:

TRIS(TERT-BUTOXY)SILANOL and Trimethylaluminum.

ZnO:

Diethylzinc (DEZ) and water vapor.

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

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.

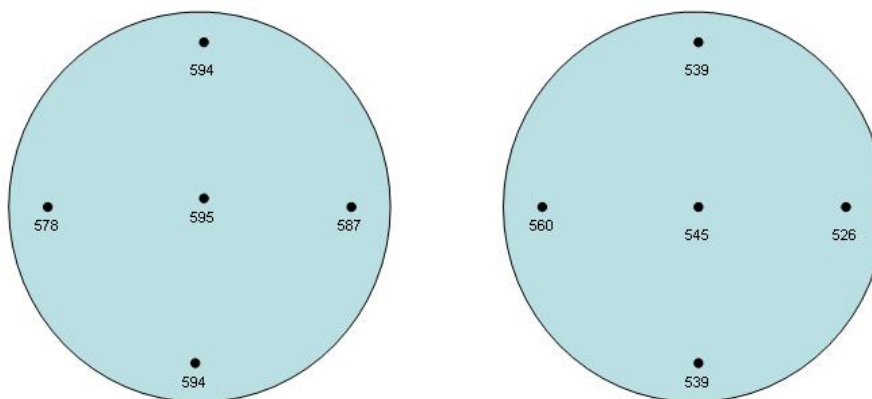
HfO₂ can be etched by RIE . Can withstand some BOE etching.

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

The oxide process is a non normal ALD process. The thickness per cycle is ~110Ang
To adjust the number of cycles for the oxide recipe, you must scroll off screen to get to the step that contains this. To make the recipe to scroll down, just high-light the right column and while holding the left button scroll down and keep going until the last step is displayed. Once it is visible then just edit the time as normal. Look at the pervious page for the source material used to make this film.

Thickness in Å

Gaertner Ellipsometer.



This run had the source at 95 C.



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Fig. 6 Thickness results of 5 loops of the Oxide recipe, wafers positioned as shown.