Arizona State University NanoFab

CAMBRIDGE ALD STANDARD OPERATION PROCEDURE

Rev D

Table of Contents

1.0 Purpose / Scope

1.1 This document covers the procedure that should be followed for normal operation of the Cambridge NanoTech S100 ALD tool.

1.2 Sample sizes-any size up to one 4" wafer.

2.0 Reference Documents

2.1 19-29-01628_03 Savannah User Manual.

2.2 19-29-01629_01 Savannah Maintenance.

2.3 https://snf.stanford.edu/SNF/equipment/chemical-vapor-deposition/ald/savannah

3.0 Safety

3.1 Follow all safety procedures outlined in the NanoFab Handbook

3.2 Follow safety and handling procedures when working with tool and processing.

3.3 Do not attempt to repair the tool under any circumstances. Submit a service request and contact ASU NanoFab staff.

3.4 Red EMO Button can be pressed at any time an emergency situation arises. Contact NanoFab staff to follow up with any emergency condition.

4.0 Tool Reservation Policies

4.1 Only trained users will be allowed to use this equipment.

4.2 It is recommended to schedule your runs to alert other members of the active use of the tool.

4.3 Our NanoFab 15-Minute rule.

4.3.1 Please start within 15 minutes of your equipment scheduled time or the tool becomes available to anyone. Please place a 'Tool in Use' tag when you arrive to indicate use.

4.3.2 Please have the equipment available for the next user within 15 minutes after your scheduled time.

4.4 Cancellations.

4.4.1 If you cannot meet the equipment schedule, please cancel your iLabs schedule to allow other users to utilize the equipment.

4.4.2 Scheduling on iLabs allows cancellation within 24 hours of your scheduled time. Please email staff if cancellation within 24 hours.

4.4.3 We discourage last second cancellations.

4.4.4 We discourage scheduled equipment no-shows.

4.5 Scheduling ALD Overnight runs.

4.5.1 You may schedule overnight ALD runs up to 18 hours in length on iLabs.

4.5.2 Please place sign signifying Tool in Use or Overnight run on the tool computer.

4.5.3 Please complete the run by the following morning by 9am.

5.0 Cambridge ALD Operational Policies.

5.1 Allowable cycles per run is limitations.

5.1.1 Service Requests for SOP Variance submitted for process runs exceeding 500 cycles.

5.1.2 Excessive cycles over 500 cycles will be charged at a rate of $0.05 a cycle.

5.2 Our tool policy will leave the tool with the Heaters ON and the STBY recipe running during tool idle time.

5.2.1 Heaters will be kept ON with recipe temps to prevent condensation of precursor material in delivery and vacuum components.

5.2.2 STBY recipe will pump down the chamber, stop valve is open with 5sccm N2 flow.

5.2.3 The tool will be left with the program user interface logged on during tool idle time.

5.3 Our NanoFab utilities recipes are stored in ALD Maintenance folder.

5.4 Three ALD processes are available.

5.4.1 HFO_2- Precursor TDAHF Growth rate @1.0Å per 1 cycle.

5.4.2 AL_2O_3- Precursor TMA Growth rate @1.0Å per 1 cycle.

5.4.3 TiO_2- Precursor TDMAT Growth rate @0.50Å per 1 cycle.

5.5 Chamber loading allowable up to one 4” wafer.

5.6 Materials not allowed in our heated ALD chamber.

5.6.1 No photo resist or polymers allowed into tool chamber.

1.1.1 No plastic including Kapton tape.

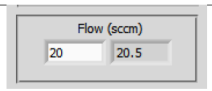
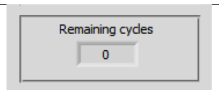
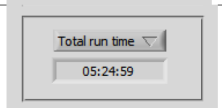
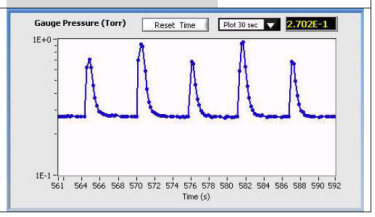
5.6.2 Gold already on wafer is allowed to be processed.

5.7 Operator does not need to be present at the tool when is processing.

5.8 If required, log into computer using the following:

5.8.1 User name: CSSER_ALD

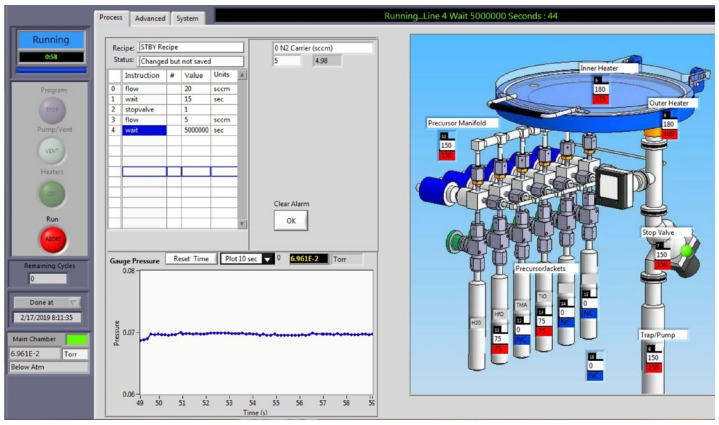
5.8.2 Password: NANFAB_ALD

Control Buttons: **Program –** stops the program. **Pump/Vent** – opens/closes the stop valve to pump down/evacuate the reactor. To prevent the running of a process when the system is vented, when the stop valve is closed, the "Run" button becomes grey, and not clickable. In the 3D drawing, the green LED on the stop valve indicates the open/close status of the stop valve: light green is open and dark green is close. **Heaters** – turns ON/OFF all the heaters. When all the heaters are turned, all the temperature setpoints are set to 0°C. **Run/Abort** – starts/aborts an ALD deposition process. When a process is running, "Program", "Pump/Vent" and "Heaters" buttons are grey and can not be selected. To abort a run, click on the displayed "Abort". **NOTE:** Do not close the window of the program while it is running.	
Flow: **S**ets (left) and displays (right) the rate of N_2 flow through the mass flow controller.	
Remaining cycles: **D**isplays the number of cycles to be completed in the current loop in a running process.	
Run time: **T**he text box shows the total time, the time left or the time a process ended.	
Process Chamber Plot Area: This plot tracks the reactor pressure reading from the pressure gauge installed in the pumping line assembly. Pulses of the precursors show up clearly on the plot. Time scale can be reset by pressing **Reset Time** button.	

6.0 Cambridge ALD setup

6.1 Please record your process parameter entries on iPad ALD run log.

6.2 Please ensure vacuum pump oil level is above minimal line. Shake pump slightly to note level.



6.3 Abort the STBY recipe. Depress Abort on Run button. Depress the Yes button.

6.4 Ensure all Heater remains ON and actual temperatures meet target temps.

6.4.1 Insure Precursor Manifold at 150°C.

6.4.2 Insure Inner Heater at 180°C.

6.4.3 Insure Outer Heater at 180°C.

6.4.4 Insure HfO_2 and TiO_2 Precursor Jackets at 75°C.

6.4.5 Insure Stop Valve at 150°C.

6.4.6 Insure Trap/Pump at 150°C.

6.5 Test chamber pressures.

6.5.1 If required, enter 5 sccm N_2 carrier flow.

6.5.2 Record 5 sccm pressure in mTorr. (Pressure unit on Y scale is Torr)

6.5.3 Enter 0 sccm N_2 carrier flow.

6.5.4 Record base pressure at 0 sccm N2 flow in mTorr.

6.6 Select Precursor valve pertaining to your targeted material to Open (Turn CW).

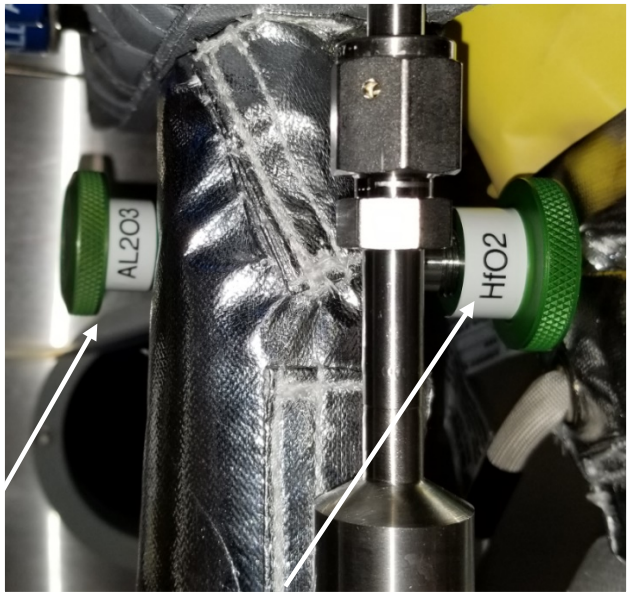
6.6.1 Valve 0- H_2O

6.6.2 Valve 1- HfO_2 (HDAHF) precursor (Green Valve on Rt). Turn CCW 1 turn to Open

6.6.3 Valve 2- AL_2O_3 (TMA) precursor (Green valve on Lt). Turn CCW 1 turn to Open.

6.6.4 Valve 3- TiO_2 (TDMAT) precursor (Red valve on Lt). Turn CCW ¼ turn to Open.

TiO_2 valve (Closed)



Al_2O_3 valve *HfO_2 valve*

7.0 Cambridge ALD Conditioning run.

7.1 Performing a Chamber Conditioning run is recommended. Perform a 10 to 20 cycle conditioning recipe using the same precursor. No substrates are loaded during run.

7.1.1 Load your ALD growth recipe.

7.1.2 Rt. Click on program area and Open your intended recipe from your recipe folder.

7.1.3 Update number the recipe cycles you intend to run.

7.1.4 Depress Start on the Run button. Depress Yes. Conditioning recipe will start.

7.1.5 Monitor Precursor and your H_2O pulses on the pressure readout display.

7.1.6 The precursor valve used will correspond to Pulse number.

7.1.6.1 Pulse 0 is H_2O

7.1.6.2 Pulse 1 is HfO_2

7.1.6.3 Pulse 2 is Al_2O_3

1.1.1.1 Pulse 3 is TiO_2.

1.1.2 When conditioning run is completed, you may now vent chamber and load your samples.

7.2 Venting the chamber.

7.2.1 Depress Vent on the Pump/Vent button. Pressure displayed >760 Torr.

1.2 Please remove the chamber safety barrier on top of tool.

7.3 Load your substrates.

7.3.1 Remove the chamber barrier.

7.3.2 Load your substrates using tweezers. Recommended to load small pieces on a silicon carrier wafer.

7.3.3 Recommended to add a silicon witness pieces to measure thickness.

1.2.1 Recommended to load samples starting in the center of the platen if possible.

7.3.4 Close chamber lid.

1.2.2 Place the chamber safety barrier back on top of tool.

7.4 Pump down chamber.

7.4.1 Depress Pump on the Pump/Vent button.

7.4.2 Allow your wafer to acclimate to chamber temps for 5 minutes.

8.0 Cambridge ALD Growth run.

8.1 Load your growth recipe.

8.1.1 Rt. Click on program area and Open your intended recipe from your recipe folder.

8.1.2 Update number the recipe cycles you intend to run.

8.2 Depress Start on the Run button. Depress Yes. Growth recipe will start.

8.2.1 Monitor Precursor and your H_2O pulses on the pressure readout display.

8.2.2 The precursor valve used will correspond to Pulse number.

8.2.2.1 Pulse 0 is H_2O

8.2.2.2 Pulse 1 is HfO_2

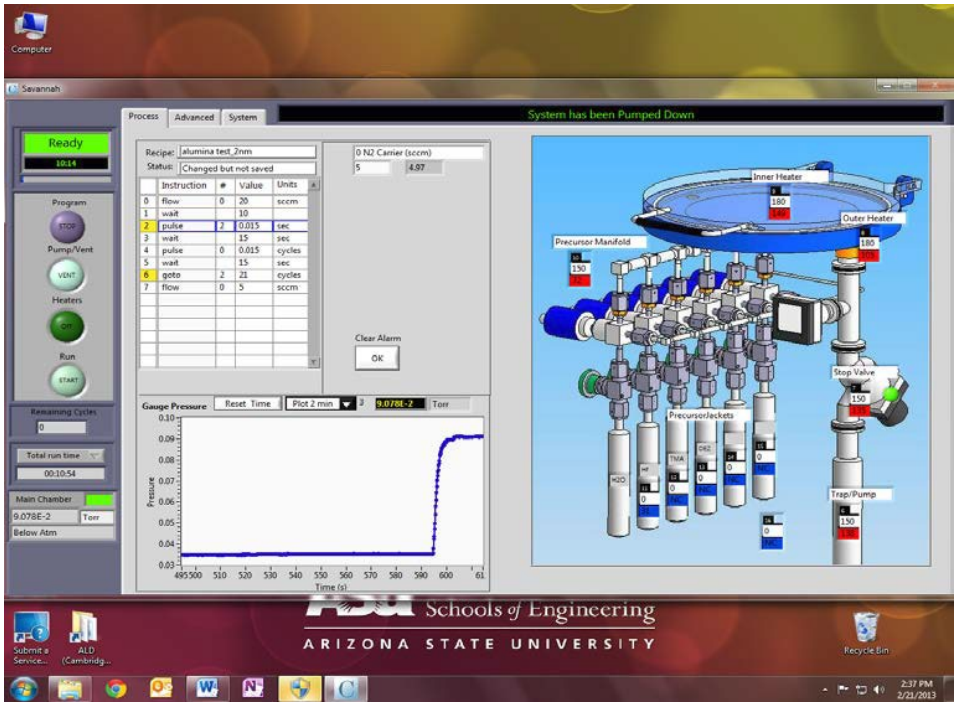
8.2.2.3 Pulse 2 is Al_2O_3

1.2.2.1 Pulse 3 is TiO_2.

8.3 Record both the active Precursor pulse and the H_2O pressures on the log sheet.

8.4 When the recipe is completed, the top progress line will indicate 'Run has Completed'.

8.5 Please notify ASU NanoFab staff of any run abnormalities. We do depend on your judgement during your activities to spot potential tool issues early.



9.0 Cambridge ALD run completion.

9.1 Vent chamber.

9.1.1 Depress Pump/Vent button to Vent.

9.2 Unload substrates.

9.2.1 Remove the chamber barrier and hang on the right side of chamber hook. Pressure reflects on screen and graph.

9.2.2 Unload your substrates using tweezers. Please ensure they are cooled down before placing on cassette, compact or plastic surfaces.

9.2.3 Close chamber lid.

9.2.4 Place the chamber safety barrier back on top of tool.

9.3 Pumpdown chamber.

9.3.1 Depress Pump/Vent button to Pump.

9.4 Close active Precursor valve. This is Important*.

9.4.1 Valve 1- HfO_2 precursor (Green Valve on Rt).

9.4.2 Valve 2- Al_2O_3 precursor (Green valve on Lt).

9.4.3 Valve 3- TiO_2 precursor (Red valve on Lt).

9.5 Run Purge recipe from the ALD Maintenance folder to clear precursor in hardware.

9.5.1 Rt. Click on program area and select and Open the active precursor purge recipe.

9.5.1.1 Purge1 HfO_2.

9.5.1.2 Purge2 $Al2O_3$.

9.5.1.3 Purge3 TiO_2.

9.5.2 Depress Run button to Start. Depress Yes. Recipe will start.

9.5.3 Monitor pressure display. One pulse should be generated and then a flat line. If more than one pulse seen, then the precursor valve may have been left open.

9.5.4 When the recipe is completed, the top progress line will indicate 'Run has Completed'.

9.6 Start the STBY recipe from the ALD Maintenance folder.

9.6.1 Rt. Click on program area and select and Open STBY recipe.

9.6.2 Depress Run button to Start. Depress Yes. Recipe will start. Ensure recipe is running.

9.6.3 Please leave tool with STBY recipe running.

9.6.3.1 The heater, chamber pumped down and N2 flow will remain ON indefinitely.

10.0 Cambridge ALD Process Data

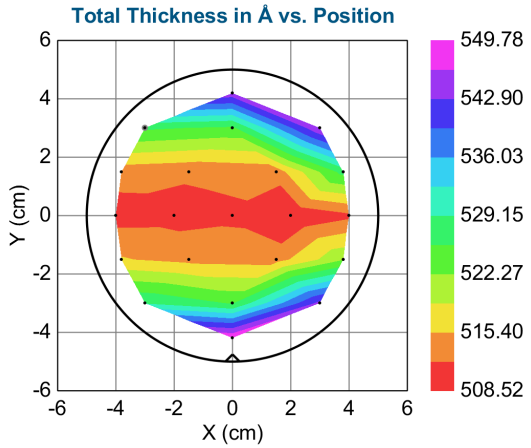
10.1 HfO_2 ALD film 500 cycle run (01/24/19) on silicon data.

10.1.1 Woollam Ellipsometer film avg Index (*n*) is 2.1092 (@632.8nm.

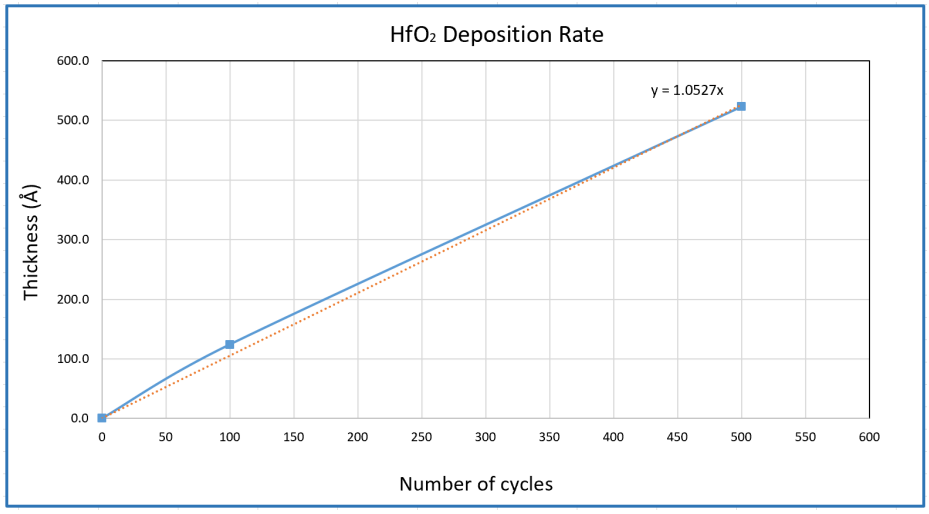
10.1.2 Woollam ellipsometer film avg Thickness is 522.62Å.

10.1.3 Woollam Ellipsometer 21pt. non-uniformity is 7.890%.

10.1.3.1 The 4" wafer flat (^) is facing the front of tool.



10.1.4 HfO_2 Thickness(Å) vs. Cycles dep rate.



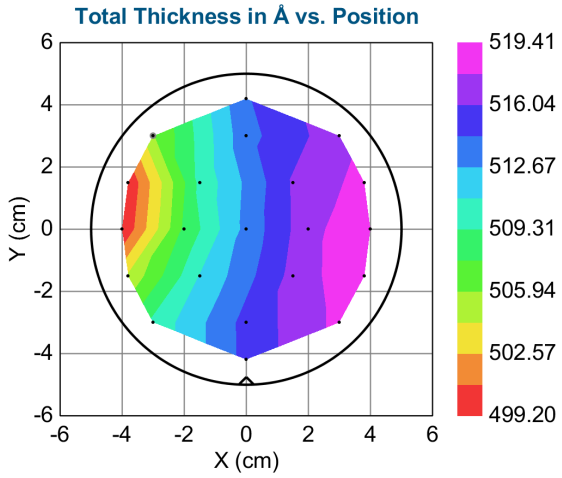
10.2 Al_2O_3 ALD film 500 cycle run (01/24/19) on silicon data.

10.2.1 Woollam ellipsometer film avg Index (*n*) is 1.6490 (@632.8nm.

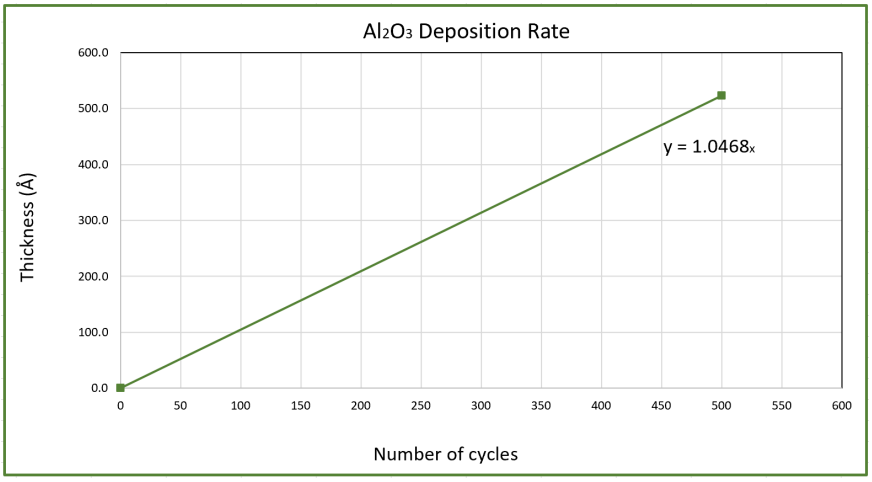
10.2.2 Woollam ellipsometer film avg Thickness is 512.41Å.

10.2.3 Woollam ellipsometer 21pt. non-uniformity is 3.94%.

10.2.3.1 The 4" wafer flat (^) is facing the front of tool.



10.2.4 Al_2O_3 Thickness (Å) vs. Cycles dep rate.



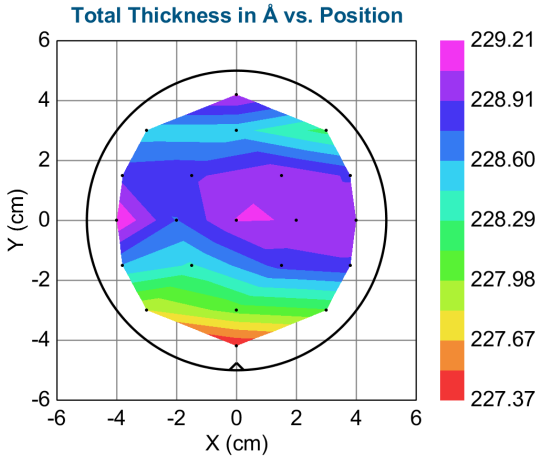
10.3 TiO_2 ALD film 500 cycle run (10/26/18) on silicon data.

10.3.1 Woollam Ellipsometer film avg Index (*n*) is 2.421 (@632.8nm.

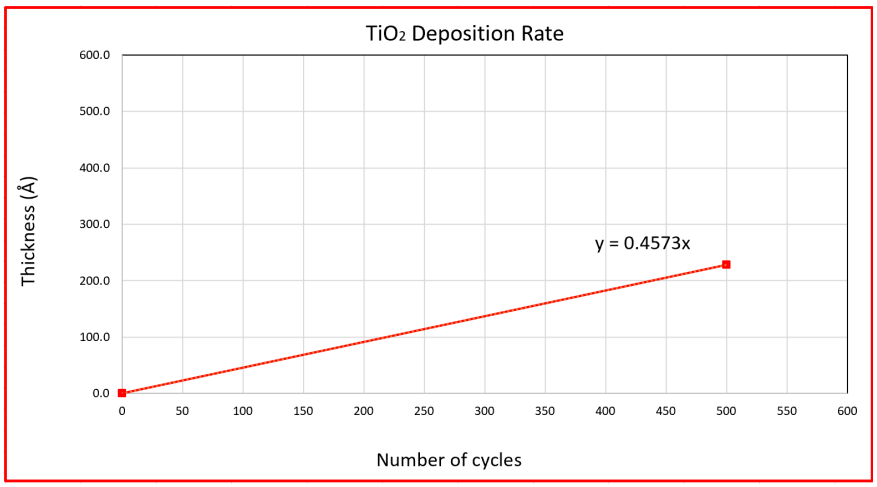
10.3.2 Woollam ellipsometer film avg Thickness is 228.63Å.

10.3.3 Woollam Ellipsometer 21pt. non-uniformity is 0.80%.

10.3.3.1 4" wafer flat (^) is facing the front of tool.



10.3.4 TiO_2 Thickness (Å) vs. Cycles dep rate.



11.0 Revision History

Effective Date	Originator	DESCRIPTION OF REVISION	Issue
02/19/13	Art Handugan	Initial Release	A
05/30/13	Art Handugan	Gas Configuration change	B
01/25/19	Jaime Quintero	Checklist version, shutdown procedures.	C
08/23/19	Jaime Quintero	Reservations/Cancellations and updates	D