University of Minnesota Nano Center

ALD-150LE Standard Operating Procedure

Badger Name: P3 ALD KJL
Model: ALD-150LE
Location: Bay 3, PAN

Revision Number: 0
Revisionist: Wanjohi Kimani
Date: January 14, 2021

Table of contents

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1 Scope

1.1 This document provides detailed instructions on how to operate the thermal KJL Atomic Layer Deposition (ALD-150LE) tool.

Fig 1: ALD 150 LE

2. Tool Description

The KJL ALD-150LE is a thermal Atomic Layer Deposition (ALD) system configured for use with up to 150mm diameter or smaller planar substrates. Substrate thickness is limited is 1.9mm or lower. This ALD tool deposits thin films layers a cycle at a time by dosing a single precursor at a time followed by a purge. Inactive gas (N_2) flows continuously and acts as a carrier gas for the precursor or as a purge gas when no precursor is being pulsed. Film thickness is a function of the number of cycles.

The control software used on this tool is eKLispse. A few features of the tool include substrate heating up to 450C, heated CapMan (150C) for pressure measurements (range 0.001 – 10 Torr), perpendicular flow design, separate chamber inlets for precursor delivery, scalable design that allows for future expansion, etc.

The system has a single-source vapor draw module (Src1) used forH2O delivery and a multi-source vapor draw module (Src 3) with five vapor draw sources (Src 3a to Src 3e) for delivery of other precursors. Src 3a has TDMAT (TiO2), Src 3b has TMA (Al2O3) and Src 3c has TDMAH (HfO2). Src 3d and Src 3e will be filled in the future. There is no Ozone source in the system.

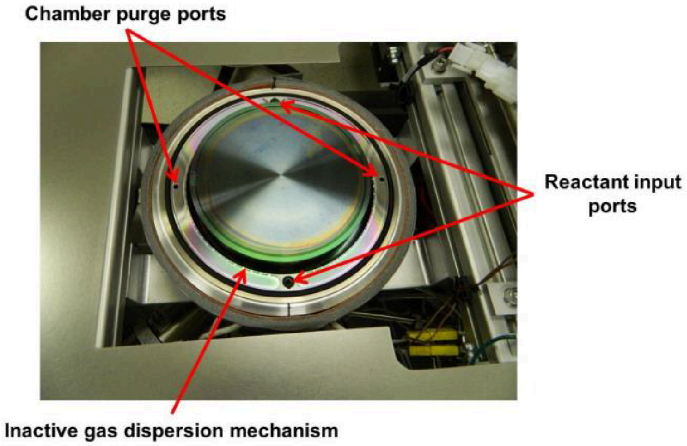


Fig 2: Substrate heater/ lower chamber showing chamber purge ports and reactant input ports

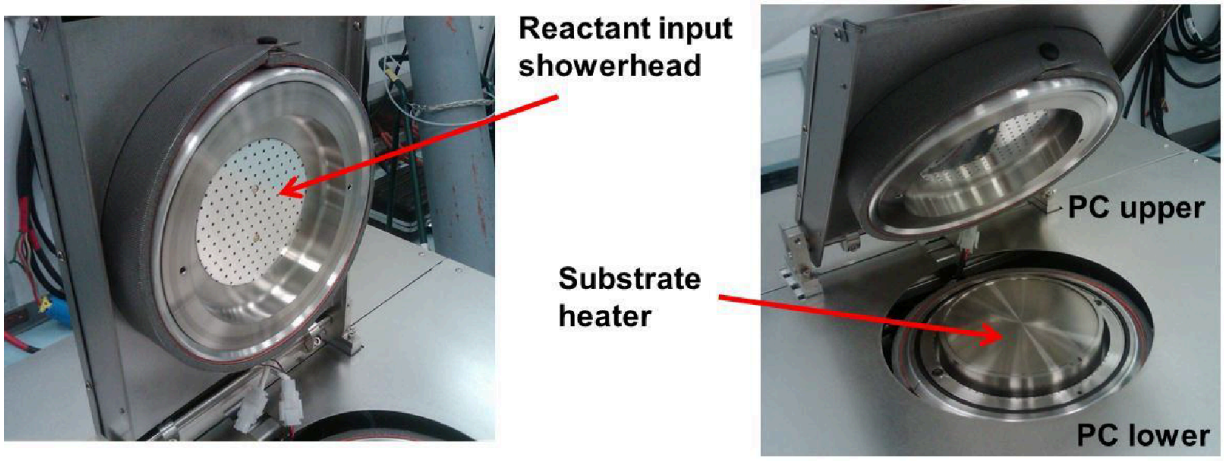


Fig 3: Reactant input showerhead and upper/lower chamber

3 Safety

3.1 Some precursors like TMA 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, or see any potentially hazardous condition during system operation, press the red EMO button on the right side of the tool; leave the area and contact staff.

3.3 The system is heated; the platen, the lid and other parts of the chamber are hot so be careful while loading and unloading wafers.

4 Restrictions/Requirements

4.1 Must be a qualified user on the P3 KJL ALD

4.2 The ALD-150LE is configured for dynamic ALD process, i.e., the isolation valve between the main process chamber and the pump remains open during process. Purge gas (N2) flows continuously through the lines, process chamber and foreline

during process. Static process is not recommended. Static process is confined to the Cambridge ALD in Keller Hall bay 1.

5 Required Facilities

5.1 Compressed air
5.2 Nitrogen
5.3 Pump and Enclosure Exhaust
5.4 208 VAC (+/_ 10% line to line and line to neutral), 3 phase, 60Hz, 60amps

6 Definitions

6.1 Precursor – A solid, liquid or gas that is one of the building compounds to form a film layer.
6.2 PC Roughing valve – This is the main valve that opens the chamber to the pump.
6.3 Vapor draw sources – ideal for liquid/solid phase precursors with enough vapor pressure that do not require any special assistance for effective vapor delivery other than heating.

7 Operating Instructions

7.1 LOGGING ON

7.1.1 Check Badger for other reservations for the “P3 ALD KJL” system first.
7.1.2 Enable "P3 ALD KJL” on Badger if not reserved or in use.
7.1.3 On Windows, click on “**Oper**” and login using password “**1234**”
7.1.4 Launch the eKLipse software by double clicking on the **eKLipse** shortcut on the desktop

7.1.5 On **eKLipse** window on the left top, login with the username “**Oper**” and password “**1234**” and click login

7.2 SETUP PROCEDURE - Selecting the operating temperature

7.2.1 Select the startup recipe, which sets the temperature you plan to run at. Do this by clicking on the “**Run Recipe**” button and selecting the appropriate heater recipe, e.g., **ALD Heater Recipe – (180C Deg)**, in the Recipe Selector. A recipe monitor window will pop up showing the steps associated with running this recipe. If you plan to run at temperatures lower than 130C, you may need to vent right after the heater recipe loading is complete to hasten the cooling process.

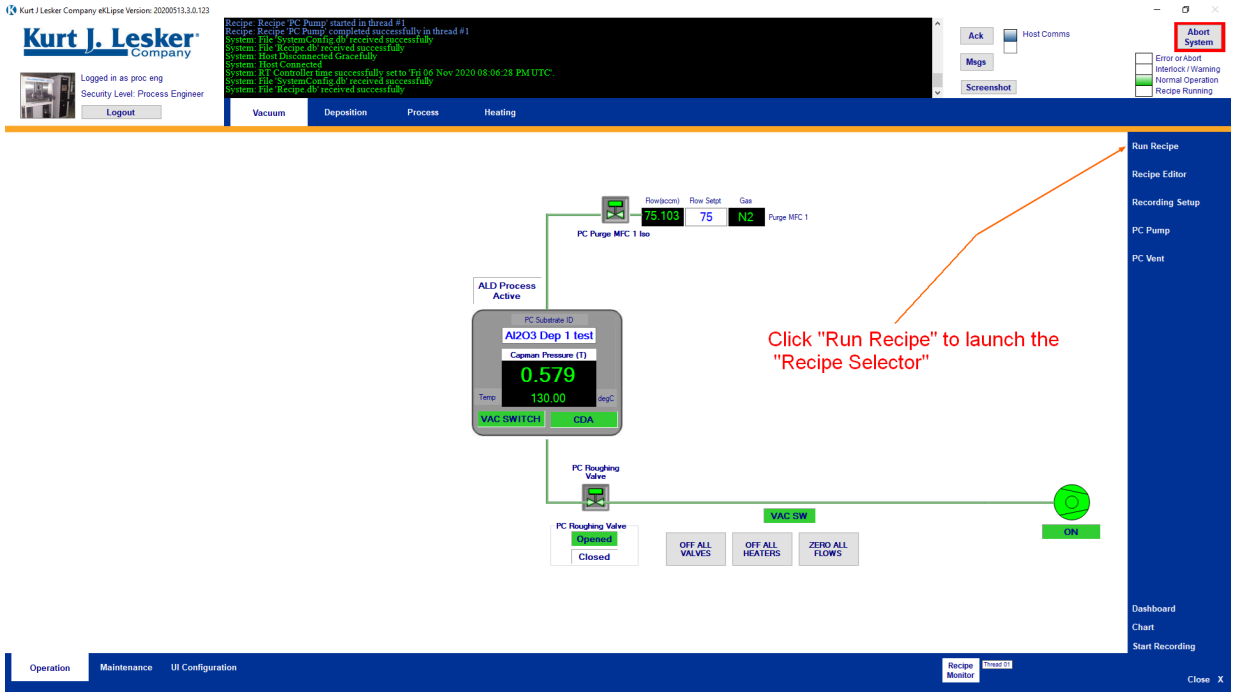


Fig 4: Vacuum screen –Run Recipe on the top right

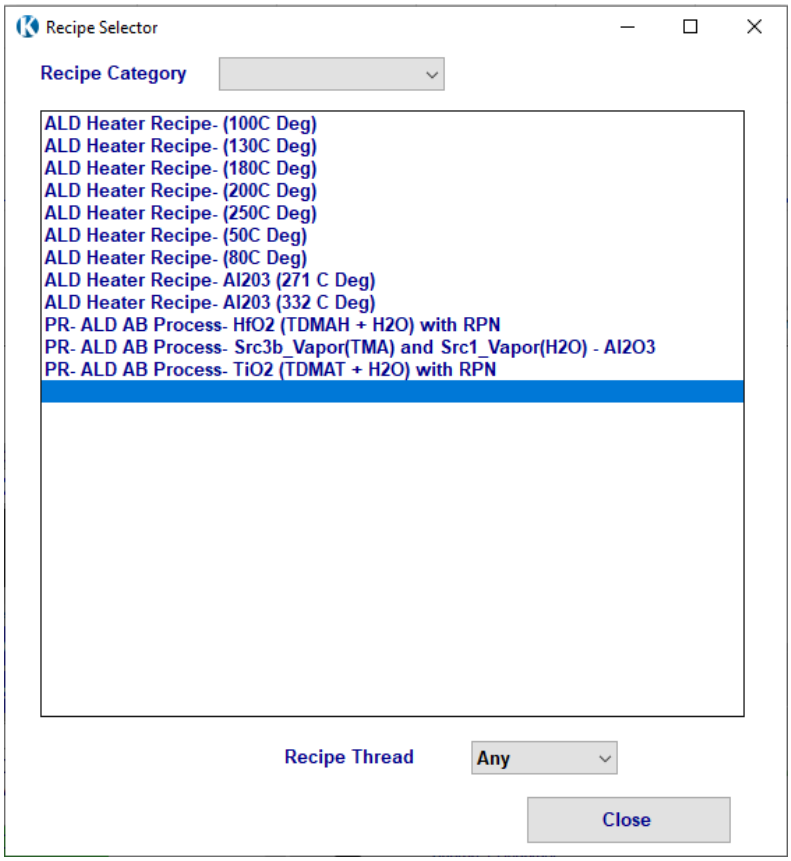


Fig 5: Recipe selector

7.3 SAMPLE LOADING - Vent Chamber and load sample(s)

7.3.1 To vent the system, click on **PC Vent**. After a second or two, the *RecipeMonitor* will show that the PC Vent Recipe has paused. Click **Resume** or **Skip** to acknowledge that you want to vent. Wait for the *RecipeMonitor* to show that venting is complete. It takes about 1 ½ minutes to fully vent the chamber. Lift the lid only when the *RecipeMonitor* shows venting is complete.

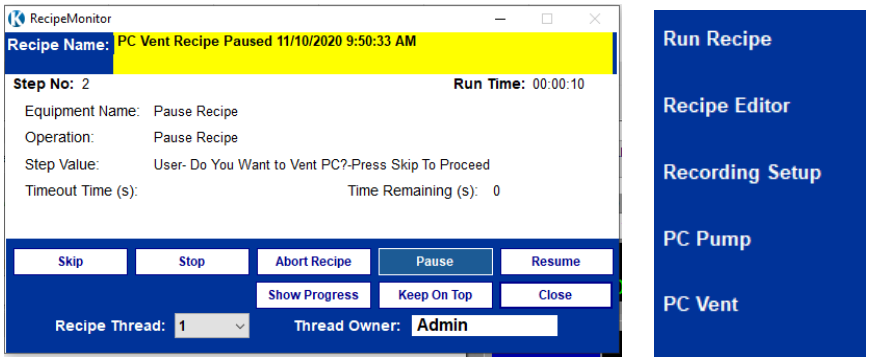
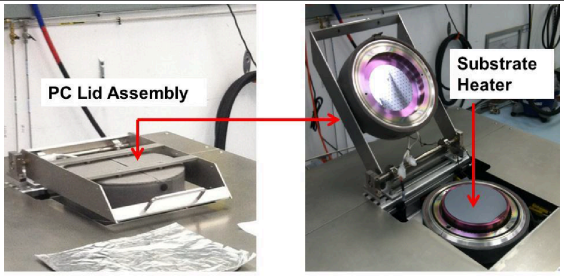


Fig 6: Venting screen on the left and recipe selector on the right

7.3.2 Be careful not to lean on and accidentally push the EMO button when lifting lid or loading/unloading samples.

Fig 8: EMO button

7.3.3 Place your sample in the chamber. The samples can be planar small pieces or up to 6” or 150mm planar substrates. Maximum thickness is 1.9 mm. 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, shield small samples with glass slides.



Mechanical stops support PC lid in open position

7.3.4 If the film deposition is at a lower temperature - wait with lid open until it cools to within five deg of your desired temperature.

7.3.5 Close the lid

7.3.6 Press the **PC Pump** button. It takes about 17 seconds to pump down.

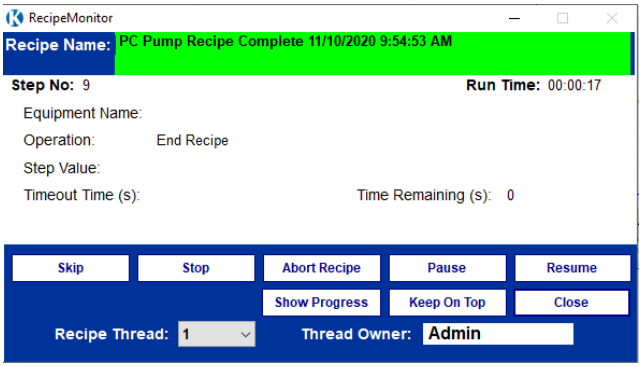


Fig 9: Pump down complete as seen on RecipeMonitor

7.4 STARTING PROCESS - Depositing film

7.4.1 You can start a run once the substrate temperature is within five - degrees of the setpoint.

7.4.2 Load the desired recipe. Click “Run Recipe” on the right side of the screen and select a recipe to run.

For Al_2O_3 film, select:

PR- ALD AB Process- Src3b_Vapor (TMA) and Src1_Vapor (H_2O) - Al_2O_3 - SR1

For HfO_2 film select:

PR-ALD AB Process – HfO_2 (TDMAH + H_2O) with RPN

For TiO_2 run select:

PR-ALD AB Process – TiO_2 (TDMAT + H_2O) with RPN

Zirconia precursor (TDMAZ) and Zinc Oxide precursor (Diethylzinc) are additional precursors in the tool. To find related process recipes, look for the labels PR-ALD-xxx, where the xxx is the name of the relevant precursor.

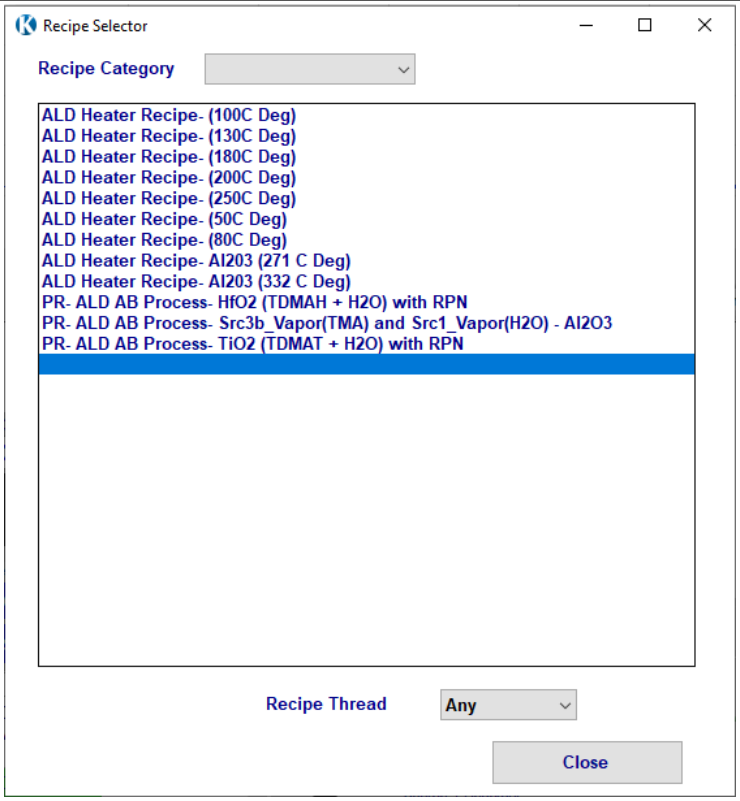


Fig 10: Recipe Selector after clicking on "Run Recipe"

7.4.3 In the Recipe User Set Values, select the number of cycles that you want the process to run by typing a value for the ALD cycle setpoint.

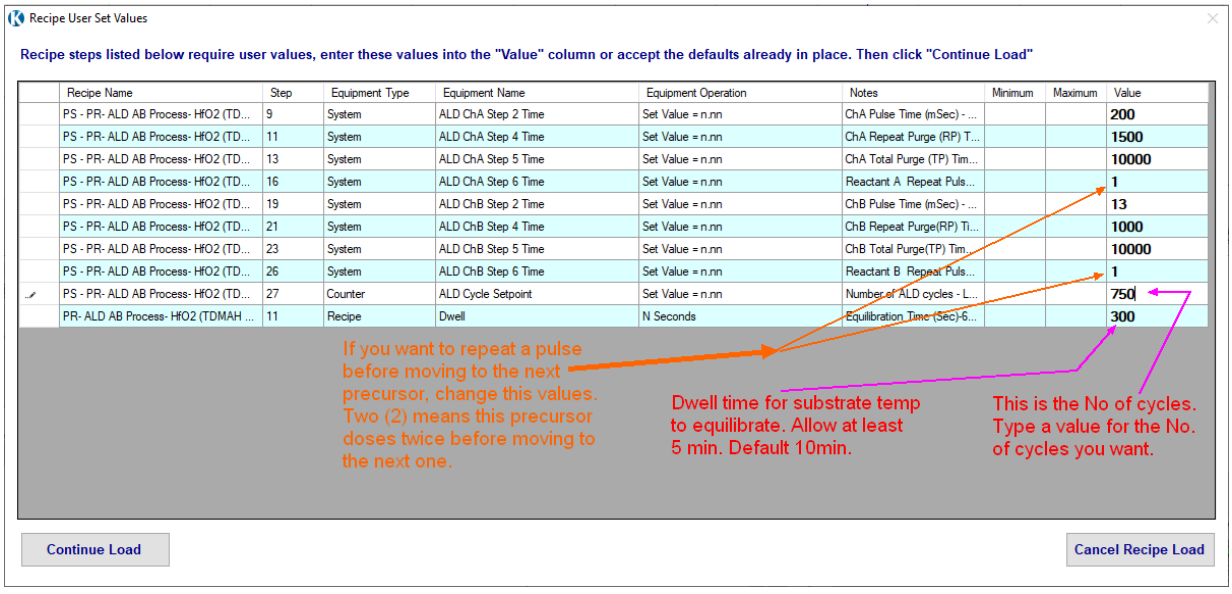


Fig 11: User set values for No of cycles, equilibrate time and dose repeat

7.4.4 Press the **Continue Load** button and then watch the recipe. The graph will start changing in the Gauge Pressure display with a repeating pattern on the deposition or process screen.

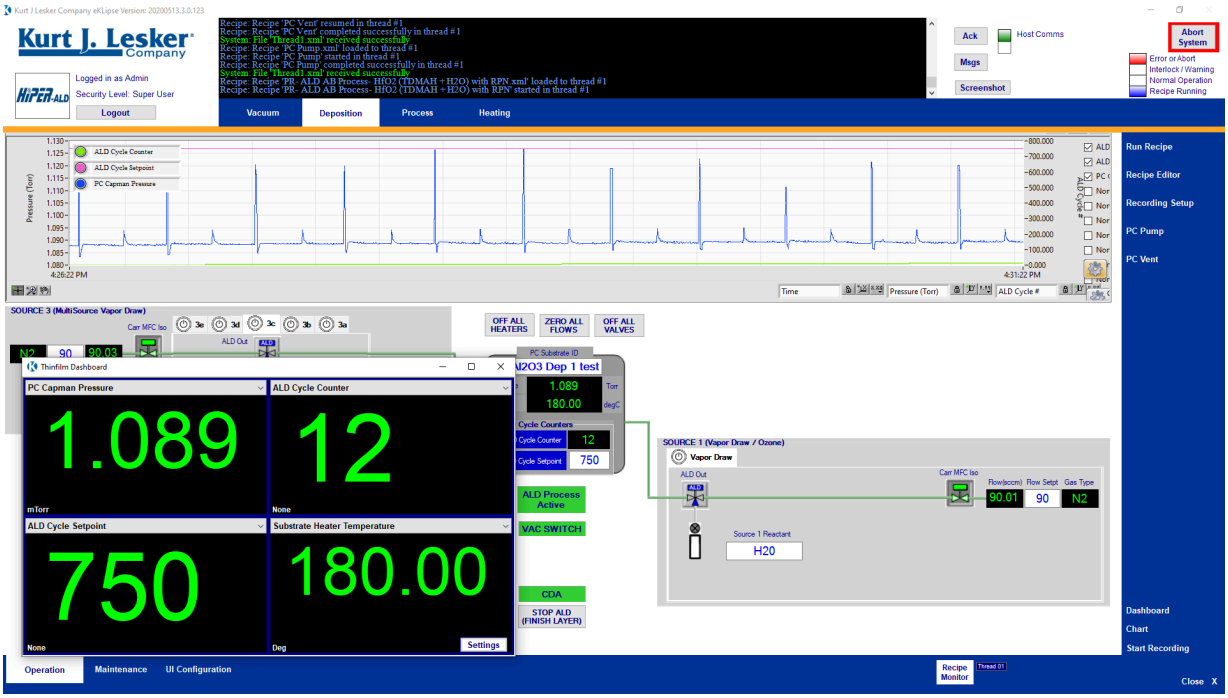


Fig 12: Deposition screen with thinfilm dashboard embedded

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.

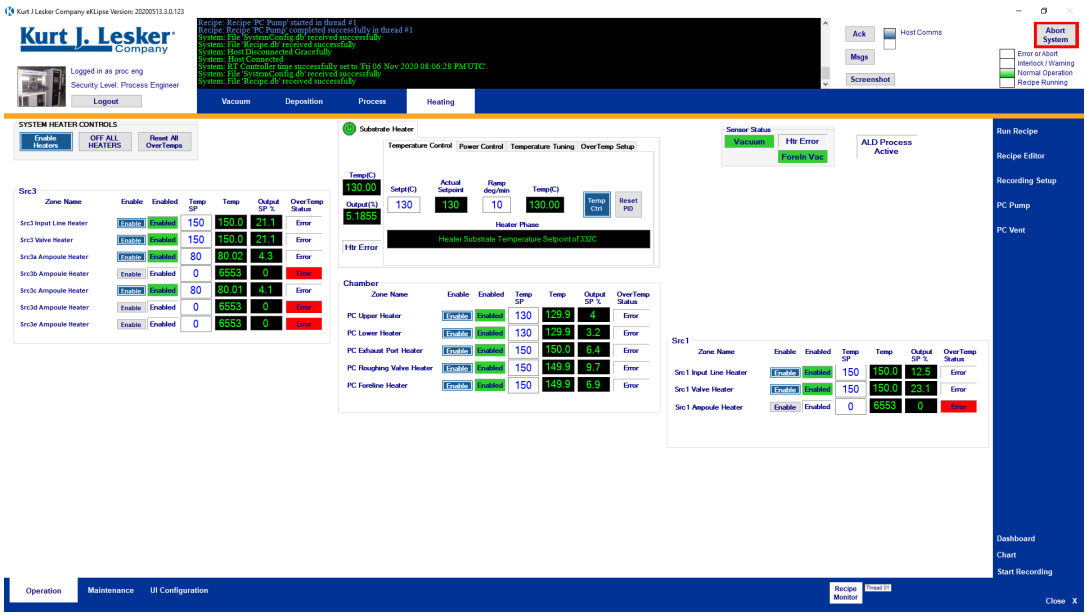


Fig 13: Heating screen. Note the different heating zones, e.g., Chamber, Src 3, etc.

7.4.6 Use DASHBOARD to see the present status of system parameter at a glance; like the total number of cycles, completed cycles, chamber pressure, substrate heater, etc. Use setting button on the Dashboard to customize the appearance and content of the display. Select up to 20 signals at a time (fewer looks better). Expand the dashboard so you can view the deposition progress from the exterior hallway without having to go into the cleanroom.

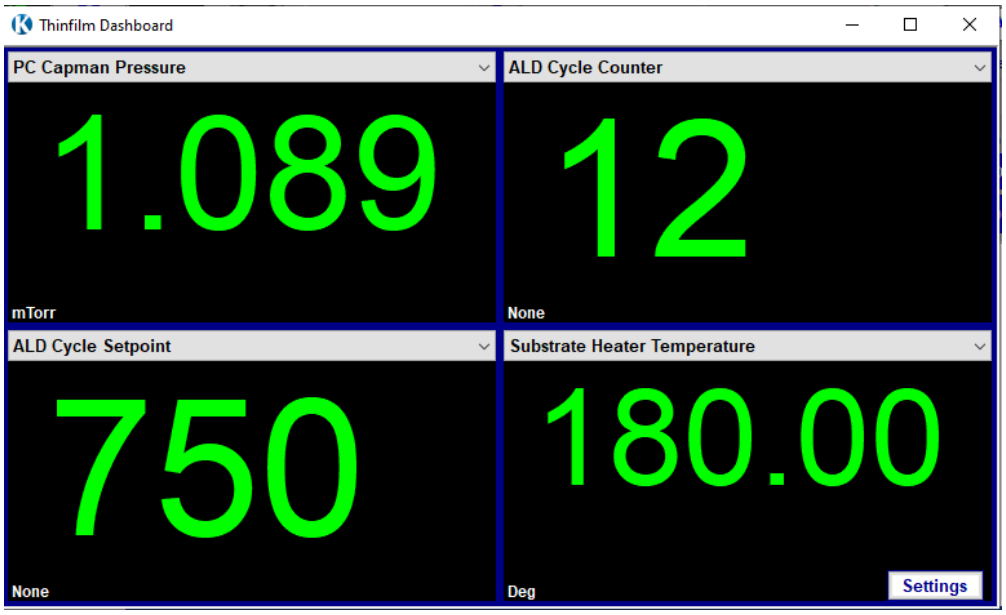


Fig 14: ThinFilm Dashboard

7.4.7 When the run is finished, the process will stop, and the recipe monitor will indicate the same

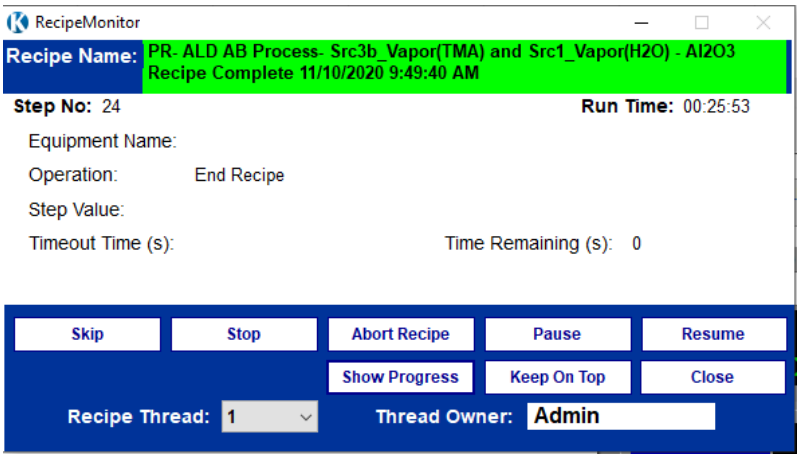


Fig 15: Process complete - RecipeMonitor

7.5 SAMPLE UNLOADING

7.5.1 Remove the sample by following the sequence of events above to vent the chamber. Click on the **PC Vent** button and then click on skip or resume to vent

the chamber. The chamber will reach atmosphere in about one to one and a half minutes.

7.5.2 Once the chamber is at atmosphere lift the lid and remove the wafer from the platen. In addition, notice the position of the wafer now compared from its loading position. Remember that the wafer and chamber are still hot so handle carefully.

7.6 **SYSTEM IDLE**

7.6.1 Close the lid and pump the system down using **PC Pump**

7.6.2 Run the **ALD Heater Recipe – (130C Deg)** recipe.

7.6.3 Log out of the eKLispse software

7.6.4 Log out of Badger.

7.7 WARNINGS

7.7.1 NEVER **REMOVE** any precursors from the system.

7.7.3 NEVER **EDIT** a recipe. Seek MNC help for any recipe changes.

8.0 Problems and Solutions

8.1 The system was shutdown, what to do now?
Contact MNC staff person

8.2 The precursor is empty, now what? Contact MNC staff person and remember the number cycles you have run/left to run. If you had the DASHBOARD running, consult it to determine how many cycles have elapsed.

9.0 Appendix

9.1 List of Process recipes:

9.1.1 PR-ALD AB process – Src3b_Vapor (TMA) + Src1_Vapor (H2O) _ Al2O3:
Use this for all normal temperature Al_2O_3 films.

9.1.2 PR-ALD AB Process-HfO2 (TDMAH + H2O) with RPN Recipe

9.1.3 PR-ALD AB Process-TiO2 (TDMAT + H2O) with RPN Recipe

9.2 Temperature setup Recipes:

9.2.1 ALD Heater recipe – (50C Deg)

9.2.2 ALD Heater recipe – (80C Deg)

9.2.3 ALD Heater recipe – (100C Deg)

9.2.4 ALD Heater recipe – (130C Deg)

9.2.5 ALD Heater recipe – (180C Deg)

9.2.6 ALD Heater recipe – (200C Deg)

9.2.7 ALD Heater recipe – Al2O3 (250C Deg)

9.2.8 ALD Heater recipe – Al2O3 (332C Deg)

More temperature setup recipes have been added. Hop over to the tool to find if what you need is included.

9.3 Precursors used in the ALD system

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

9.3.2 **HfO_2:** Tetrakis(dimethylamido)hafnium(IV) [TDMAH] and water vapor.
Sigma-Aldrich Part number: 666610-25G
precursor at 85°C

9.3.3 **TiO_2:** Tetrakis(dimethylamido)titanium(IV) [TDMAT] and water vapor.
Sigma-Aldrich Part number: 669008-25G
precursor at 82°C

9.3.4 **ZrO2:** tetrakis(dimethylamido)zirconium(IV) [TDMA-Zr] and water vapor.
Sigma-Aldrich Part number:
precursor at 75°C

9.3.5 ZnO: Diethylzinc and water vapor.
Sigma-Aldrich Part number:
precursor at room temperature

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

9.4.1 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.

9.4.2 Al_2O_3 can be wet etched using BOE ~ 350 Å/min. **Do not use in Bay 1 Keller Hall**. 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

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

9.4.4 Lower temperatures process will have a higher deposition rate. The film quality and step coverage are reduced; the exact amount has not been measured.