ALD-150LE Standard Operating Procedure

Badger Name: Model: Location: P3 ALD KJL ALD-150LE Bay 3, PAN Revision Number:0Revisionist:WanjolDate:January

Wanjohi Kimani January 14, 2021

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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.

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Inactive gas dispersion mechanism

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

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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 $208 \text{ VAC} (+/_ 10\% \text{ line to line and line to neutral}), 3 phase, 60Hz, 60amps$

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.

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Kurt J Lesker Company eKLipse Version: 20200513.3.0.123											– a ×
Kurt J. Lesker	Recipe: Recipe 'PC Recipe: Recipe 'PC System: File 'System System: File 'Recip System: Host Disco System: Host Conn System: RT Contro	Pump' started in threa Pump' completed succ nConfig.db received s e.db' received successi nnected Gracefully ected lier time successfully s	d #1 essfully in thread : uccessfully fully et to 'Fri 06 Nov 2						Ack Hos Msgs	t Comms	Abort System Error or Abort Interlock / Warning
Security Level: Process Engineer	System: File 'System System: File 'Recip	nConfig.db' received s e.db' received success	uccessfully fully						Screenshot		Recipe Running
Logout	Vacuum	Deposition	Process	Heating							
										/	Run Recipe
											Recipe Editor
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					PC Purge MFC 1 Iso						PC Pump
											PC Vent
				ALD Process Active							
				PC Substrate AI2O3 Dep	1 test		Click	"Run Rec	cipe" to launch t	he	
				Capman Press	ure (T)		"Rec	ipe Selec	tor"		
				U.57	g D degC						
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					Closed	VALVES	HEATERS	FLOWS			ſ
											Dashboard
											Chart
											Start Recording
Operation Maintenance UI Configur	ation								Recipe Monitor		Close X

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

(Recipe Selector -	×
Recipe Category v	
ALD Heater Recipe- (100C Deg) ALD Heater Recipe- (130C Deg) ALD Heater Recipe- (180C Deg) ALD Heater Recipe- (200C Deg) ALD Heater Recipe- (250C Deg) ALD Heater Recipe- (50C Deg) ALD Heater Recipe- (50C Deg)	
ALD Heater Recipe. Al203 (271 C Deg) ALD Heater Recipe. Al203 (271 C Deg) ALD Heater Recipe. Al203 (332 C Deg) PR- ALD AB Process. HfO2 (TDMAH + H2O) with RPN PR- ALD AB Process. Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process. TiO2 (TDMAT + H2O) with RPN	
Recipe Thread Any ~	
Close	

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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.

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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.

🚯 RecipeMonitor				- 🗆 🗡
Recipe Name: P(C Pump Recipe Co	mplete 11/10/2020 9):54:53 AM	
Step No: 9			Run T	ime: 00:00:17
Equipment Name	e:			
Operation:	End Recipe			
Step Value:				
Timeout Time (s)):	Time	e Remaining (s): ()
	-			
Skip	Stop	Abort Recipe	Pause	Resume
		Show Progress	Keep On Top	Close
Recipe Thr	read: <mark>1 ~</mark>	Thread Own	ner: Admin	

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 Al₂O₃ film, select:
 PR- ALD AB Process- Src3b_Vapor (TMA) and Src1_Vapor (H₂O) - Al₂O₃ - SR1 For HfO₂ film select:
 PR-ALD AB Process - HfO₂ (TDMAH + H₂O) with RPN For TiO₂ run select:
 PR-ALD AB Process - TiO₂ (TDMAT + H₂O) with RPN

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Recipe Category ✓ ALD Heater Recipe. (100C Deg) ALD Heater Recipe. (130C Deg) ALD Heater Recipe. (180C Deg) ALD Heater Recipe. (200C Deg) ALD Heater Recipe. (200C Deg) ALD Heater Recipe. (200C Deg) ALD Heater Recipe. (200C Deg) ALD Heater Recipe. (50C Deg) ALD Heater Recipe. (80C Deg) ALD Heater Recipe. Al203 (271 C Deg) ALD Heater Recipe. Al203 (271 C Deg) ALD Heater Recipe. Al203 (332 C Deg) PR. ALD AB Process. HfO2 (TDMAH + H2O) with RPN PR. ALD AB Process. Sr03 Vapor(TMA) and Src1 Vapor(H2O) - Al2O3 PR. ALD AB Process. TiO2 (TDMAT + H2O) with PRN	Recipe Selector			-	>
ALD Heater Recipe- (100C Deg) ALD Heater Recipe- (130C Deg) ALD Heater Recipe- (180C Deg) ALD Heater Recipe- (200C Deg) ALD Heater Recipe- (200C Deg) ALD Heater Recipe- (50C Deg) ALD Heater Recipe- (50C Deg) ALD Heater Recipe- (80C Deg) ALD Heater Recipe- Al203 (271 C Deg) ALD Heater Recipe- Al203 (332 C Deg) PR- ALD AB Process- Hr02 (TDMAH + H2O) with RPN PR- ALD AB Process- Hr02 (TDMAH + H2O) with RPN PR- ALD AB Process- IfO2 (TDMAT + H2O) with RPN	Recipe Category ~				
ALD Heater Recipe- (130C Deg) ALD Heater Recipe- (180C Deg) ALD Heater Recipe- (200C Deg) ALD Heater Recipe- (250C Deg) ALD Heater Recipe- (50C Deg) ALD Heater Recipe- (80C Deg) ALD Heater Recipe- Al203 (271 C Deg) ALD Heater Recipe- Al203 (322 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB PROCESS- SrC3b_VAPOR(TMA) and SrC1_VAPOR(TMA) and SrC1	ALD Heater Recipe- (100C Deg)				
ALD Heater Recipe (180C Deg) ALD Heater Recipe (200C Deg) ALD Heater Recipe (250C Deg) ALD Heater Recipe (50C Deg) ALD Heater Recipe (80C Deg) ALD Heater Recipe Al203 (271 C Deg) ALD Heater Recipe Al203 (332 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process- TiO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- (130C Deg)				
ALD Heater Recipe (200C Deg) ALD Heater Recipe (250C Deg) ALD Heater Recipe (50C Deg) ALD Heater Recipe (80C Deg) ALD Heater Recipe Al203 (271 C Deg) ALD Heater Recipe - Al203 (332 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process- TiO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- (180C Deg)				
ALD Heater Recipe (250C Deg) ALD Heater Recipe (50C Deg) ALD Heater Recipe (80C Deg) ALD Heater Recipe A1203 (271 C Deg) ALD Heater Recipe A1203 (271 C Deg) ALD Heater Recipe A1203 (332 C Deg) PR- ALD AB Process- HfO2 (TDMAH + H2O) with RPN PR- ALD AB Process- IfO2 (TDMAH + H2O) with RPN PR- ALD AB Process- IfO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- (200C Deg)				
ALD Heater Recipe- (50C Deg) ALD Heater Recipe- (80C Deg) ALD Heater Recipe- Al203 (271 C Deg) ALD Heater Recipe- Al203 (272 C Deg) PR- ALD AB Process- HfO2 (TDMAH + H2O) with RPN PR- ALD AB Process- Sr02 Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process- TiO2 (TDMAT + H2O) with PPN	ALD Heater Recipe- (250C Deg)				
ALD Heater Recipe- (80C Deg) ALD Heater Recipe- AI203 (271 C Deg) ALD Heater Recipe- AI203 (322 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - AI2O3 PR- ALD AB Process TiO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- (50C Deg)				
ALD Heater Recipe- AI203 (271 C Deg) ALD Heater Recipe- AI203 (332 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H20) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H20) - AI203 PR- ALD AB Process- TiO2 (TDMAT + H20) with RPN	ALD Heater Recipe- (80C Deg)				
ALD Heater Recipe- AI203 (332 C Deg) PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - AI2O3 PR- ALD AB Process- TiO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- Al203 (271 C Deg)				
PR- ALD AB Process- Hf02 (TDMAH + H2O) with RPN PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process_ TiO2 (TDMAT + H2O) with RPN	ALD Heater Recipe- Al203 (332 C Deg)				
PR- ALD AB Process- Src3b_Vapor(TMA) and Src1_Vapor(H2O) - Al2O3 PR- ALD AB Process_ TiO2 (TDMAT + H2O) with RPN	PR- ALD AB Process- HfO2 (TDMAH + H2O) with R	PN			
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Recipe Thread Any V	Recipe Thread	Any		~	
Recipe Thread Any ~	Recipe Thread	Апу	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	<	
Recipe Thread Any	Recipe Thread	Any	~		

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.

	Recipe Name	Step	Equipment Type	Equipment Name	Equipment Operation	Notes	Minimum	Maximum	Value
	PS - PR- ALD AB Process- HfO2 (TD	9	System	ALD ChA Step 2 Time	Set Value = n.nn	ChA Pulse Time (mSec)			200
	PS - PR- ALD AB Process- HfO2 (TD	11	System	ALD ChA Step 4 Time	Set Value = n.nn	ChA Repeat Purge (RP) T			1500
	PS - PR- ALD AB Process- HfO2 (TD	13	System	ALD ChA Step 5 Time	Set Value = n.nn	ChA Total Purge (TP) Tim			10000
	PS - PR- ALD AB Process- HfO2 (TD	16	System	ALD ChA Step 6 Time	Set Value = n.nn	Reactant A Repeat Puls			1
	PS - PR- ALD AB Process- HfO2 (TD	19	System	ALD ChB Step 2 Time	Set Value = n.nn	ChB Pulse Time (mSec)			13
	PS - PR- ALD AB Process- HfO2 (TD	21	System	ALD ChB Step 4 Time	Set Value = n.nn	ChB Repeat Purge(RP) Ti			1000
	PS - PR- ALD AB Process- HfO2 (TD	23	System	ALD ChB Step 5 Time	Set Value = n.nn	ChB Total Purge(TP) Tim			10000
	PS - PR- ALD AB Process- HfO2 (TD	26	System	ALD ChB Step 6 Time	Set Value = n.nn	Reactant B Repeat Puls			1
	PS - PR- ALD AB Process- HfO2 (TD	27	Counter	ALD Cycle Setpoint	Set Value = n.nn	Number of ALD cycles - L			750 🔶
	PR- ALD AB Process- HfO2 (TDMAH	11	Recipe	Dwell	N Seconds	Equilibration Time (Sec)-6			300
If you want to repeat a pulse before moving to the next precursor, change this values. Two (2) means this precursor doses twice before moving to to equilibrate. Allow at least 5 min. Default 10min. This is the No of cycle Type a value for the No of cycles you want.									

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

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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.

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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.

🔇 Thinfilm Dashboard	– 🗆 X
PC Capman Pressure v	ALD Cycle Counter 🗸 🗸 🗸
1.089	12 None
ALD Cycle Setpoint ~	Substrate Heater Temperature V
750	180.00
None	Deg Settin

Fig 14: ThinFilm Dashboard

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

🔇 RecipeMonitor				- 🗆 🗙					
Recipe Name: PF	R- ALD AB Process- ecipe Complete 11/	Src3b_Vapor(TMA) 10/2020 9:49:40 AM) and Src1_Vapor(H	120) - A12O3					
Step No: 24			Run T	ime: 00:25:53					
Equipment Name	e:								
Operation:	End Recipe								
Step Value:									
Timeout Time (s)):	Time	e Remaining (s):)					
			1						
Skip	Stop	Abort Recipe	Pause	Resume					
		Show Progress	Keep On Top	Close					
Recipe Th	read: <mark>1 ~</mark>	Thread Own	ner: Admin						

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.

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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.

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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 Al₂O₃ 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)
- 9.3 Precursors used in the ALD system
 - 9.3.1 Al₂O₃: Trimethylaluminum [TMA] and water vapor. Sigma-Aldrich Part number: 663301-25G precursor at room temperature
 - 9.3.2 HfO₂: Tetrakis(dimethylamido)hafnium(IV) [TDMAH] and water vapor. Sigma-Aldrich Part number: 666610-25G precursor at 75°C
 - 9.3.3 **TiO₂:** Tetrakis(dimethylamido)titanium(IV) [TDMAT] and water vapor. Sigma-Aldrich Part number: 669008-25G precursor at 78°C
- 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 Al₂O₃ 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. Al₂O₃ can be used as an etch mask for DRIE, has great etch selectivity > 1000:1
 - 9.4.3 HfO₂ 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.