

Cambridge NanoTech Fiji F200 ALD SOP



1 Scope

- 1.1 This document provides standard operating procedures and instructions to deposit thin films with the Cambridge NanoTech Fiji F200 Atomic Layer Deposition (ALD) system.
 - 1.1.1 This system uses reactive chemistry to generate highly conformal films one atomic layer at a time.
 - 1.1.2 The system supports multiple precursors and can be used to generate a variety of films.

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3 Reference Documents

3.1 Referenced within this Document

3.1.1 None

3.2 External Documents

3.2.1 None

4 Equipment and/or Materials

- 4.1 Cambridge Fiji F200 ALD system
- 4.2 Monitors (bare silicon)
- 4.3 Precursors, as needed
- 4.4 Quartz Plate
- 4.5 Research Grade Argon
- 4.6 Research Grade Nitrogen
- 4.7 Research Grade Oxygen
- 4.8 Substrate Platen
- 4.9 Substrate Platen Wand
- 4.10 Wafer/Sample

5 Safety

- 5.1 Follow all Nanofab safety procedures.
- 5.2 During operation, parts of the ALD become VERY warm – up to approximately 300°C.
 - 5.2.1 Never place flammable materials, such as paper, on or near the reactor chamber.
- 5.3 Do NOT touch the substrate carrier with your hands as it WILL be HOT.
- 5.4 After the chuck has been removed from the chamber, allow sufficient time (at least 10 minutes) for the chuck and sample to cool before handling the sample.
- 5.5 Some precursors are pyrophoric and will combust and/or explode on contact with room air.
 - 5.5.1 Only Nanofab Staff is authorized to handle any precursor material, including water.
- 5.6 Precursors are changed only by Nanofab Staff.

6 System Description

6.1 System Overview

- 6.1.1 See **Figure 1, Cambridge Fiji F200 ALD System Configuration** for a view of the system and major components.

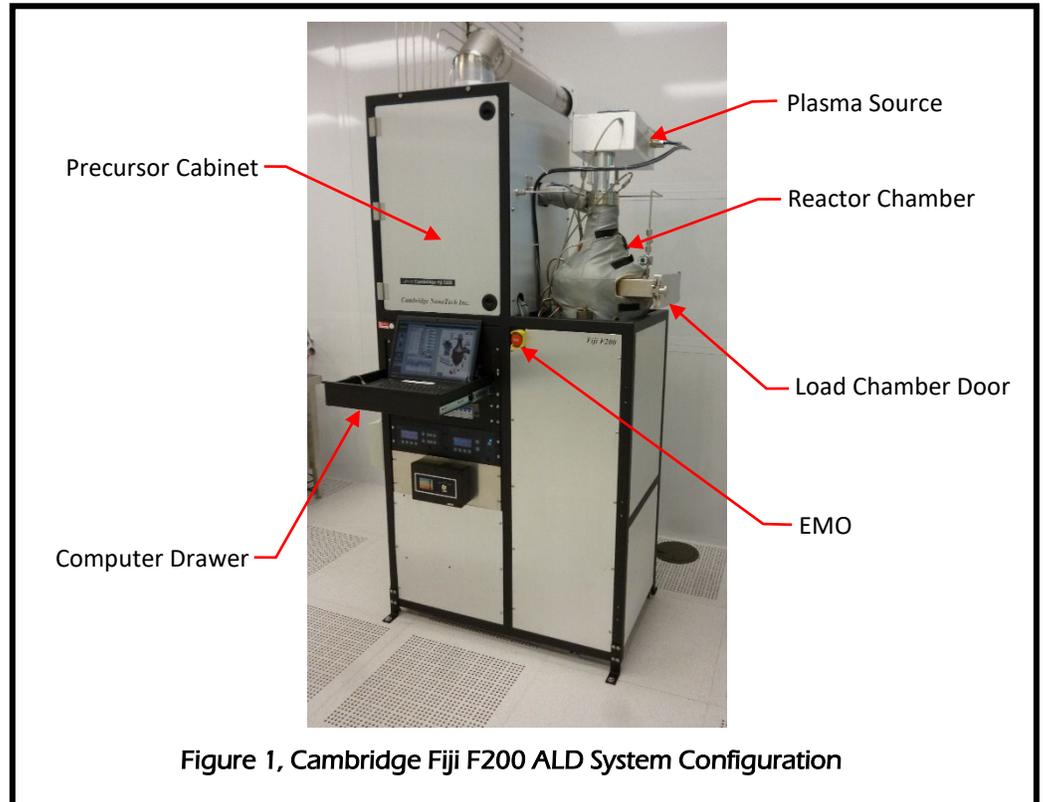
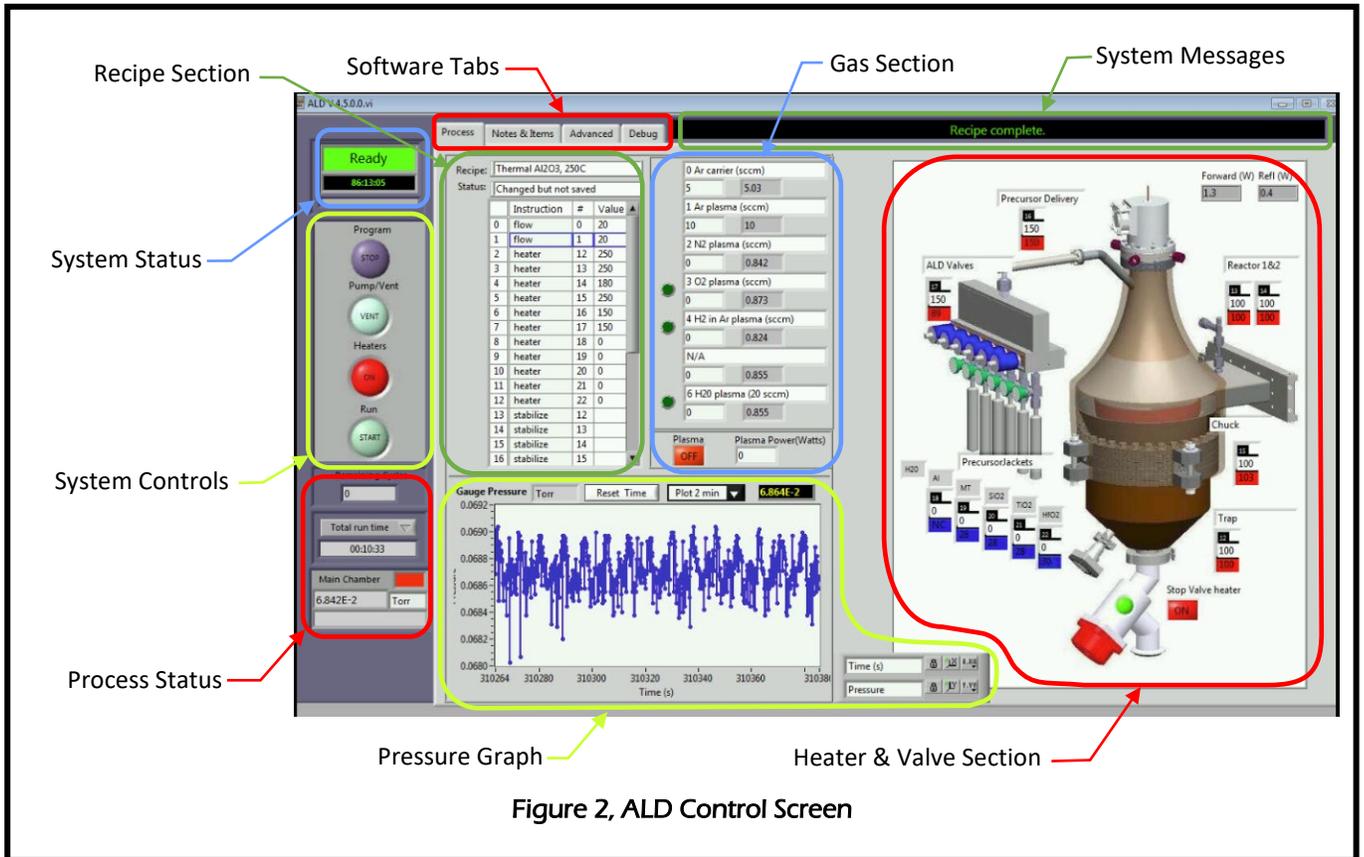


Figure 1, Cambridge Fiji F200 ALD System Configuration

6.2 Software Interface

- 6.2.1 All actions for the system are managed and controlled through a software application processing on a Windows-based laptop computer. (See **Figure 2, ALD Control Screen.**)
- 6.2.2 The application supports 4 different software tabs: Process, Notes & Items, Advanced, and Debug.

NOTE: Operation of the system is done through the Process tab. Lab Members should not modify information on any other tab.



6.2.3 Process Tab Description

- 6.2.3.1 **Gas Section.** This section displays information (valve #, valve status, Gas Name, current flow rate, and setpoint) for each gas. It also includes information for the plasma source.
- 6.2.3.1.1 In standby mode, MFC setpoints and valve status can be changed manually.
- 6.2.3.2 **Heater & Valve Section.** This section displays information for all the heaters (setpoint and current temperature) and the stop valve status.
- 6.2.3.2.1 In standby mode, heater setpoints and the stop valve status can be changed manually.
- 6.2.3.3 **Pressure Graph.** The graph shows a running history of the reactor/system pressure.
- 6.2.3.4 **Process Status.** This section is for display only and shows the number of cycles (goto loops) remaining in the current recipe, the Total run time of the currently loaded recipe, and the current pressure.
- 6.2.3.5 **Recipe Section.** This section is used to load, save, and modify a recipe. See 9.2 Programming Commands for details of recipe commands.
- 6.2.3.6 **System Controls.** This section contains several buttons that are used to activate/deactivate the system hardware and recipes. The button text shows what action will occur if the button is clicked.
- 6.2.3.6.1 **Program button.** If necessary, use this button to Stop program execution.

- 6.2.3.6.2 **Pump/Vent.** Opens and closes the stop valve (gate valve) to either pump the system down or vent it.
- 6.2.3.6.3 **Heaters.** Turns all the heaters on or off. Turning the heaters on will enable each heater to ramp to the defined setpoint. Turning the heaters off will give all heaters a setpoint of 0.
- 6.2.3.6.4 **Run.** Use this button to Start or Abort the currently loaded recipe.
- 6.2.3.7 **System Messages.** This field will show messages and information about the current recipe.
- 6.2.3.8 **System Status.** This section indicates the current status of the system and the time since the last recipe started or ended.

7 Setup Procedures

NOTE: All samples/substrates must have a thickness less than 6mm.

NOTE: Films thicker than 50 nm (500 Å) require prior approval from Nanofab Staff.

7.1 Verify Chamber Vacuum

- 7.1.1 Ensure the chamber is pumped down (under vacuum).
 - 7.1.1.1 **If the Pump/Vent button in the System Controls section shows "PUMP"**, do the following.
 - 7.1.1.1.1 Close the Chamber Load Door.
 - 7.1.1.1.2 Click "PUMP".
 - 7.1.1.2 **If the Pump/Vent button shows "Vent" and the Main Chamber pressure in the Process Status section is greater than 1E-1 Torr**, do not proceed.
 - 7.1.1.2.1 Notify Staff.

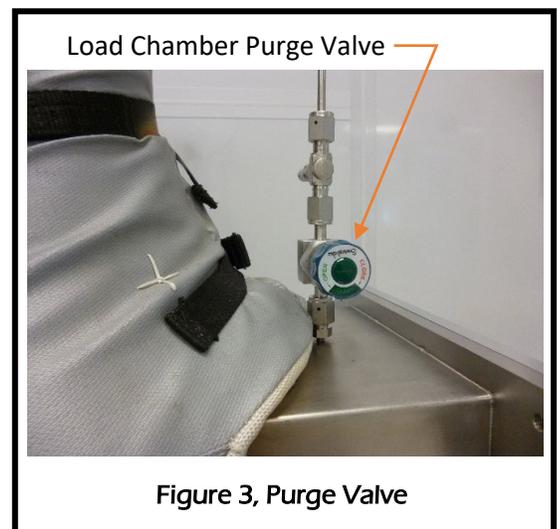
7.2 Verify Heaters

- 7.2.1 **If the Heaters button is red**, click the button.
 - 7.2.1.1 The Heaters button must be green.

7.3 Purge Chamber

- 7.3.1 Turn the Load Chamber Purge Valve counter-clockwise to the "Open" position. (See **Figure 3, Purge Valve**.)

NOTE: This valve is either open or closed. It does not control the flow rate of the purge gas.



7.4 Select Startup Recipe

- 7.4.1 Right click in the recipe steps table. (See **Figure 4, Recipe Section Detail.**)
- 7.4.2 Click on "Load recipe ..." in the drop down menu.

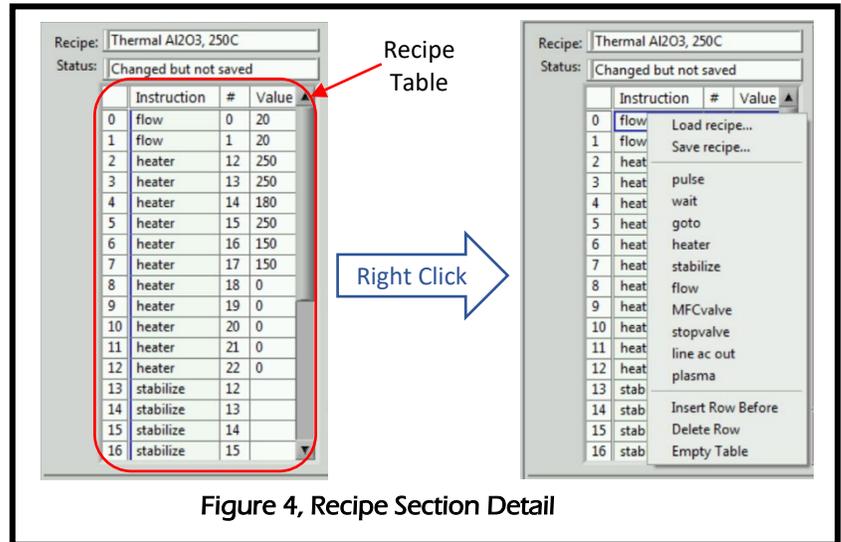


Figure 4, Recipe Section Detail

- 7.4.3 Choose the ALDStartup recipe that matches the desired temperatures. (See **TABLE 1, ALD RECIPE AND PROCESS INFORMATION.**)

TABLE 1, ALD RECIPE AND PROCESS INFORMATION							
Type	Recipe Name	Film Deposited	Chuck Temp (°C)	goto line #	Precursor	Precursor Temp (°C)	Dep Rate (Å/cycle)
Standby	ALDStandby, 100C		100				
Startup	ALDStartup, 150C		150				
	ALDStartup, 200C		200				
	ALDStartup, 250C		250				
	ALDStartup, 270C		270				
Plasma	Plasma Al ₂ O ₃ , 250C	Al ₂ O ₃	250	30	TMA	Room	1.023
	Plasma HfO ₂ , 200C	HfO ₂	200	31	TDMAHf	100	1.017
	Plasma SiO ₂ , 150C	SiO ₂	150	30	3DMAS	Room	0.534
	Plasma SiO ₂ , 200C	SiO ₂	200	30	3DMAS	Room	0.440
Thermal	Thermal Al ₂ O ₃ , 250C	Al ₂ O ₃	250	25	TMA	Room	1.087
	Thermal HfO ₂ , 200C	HfO ₂	200	26	TDMAHf	100	0.976
Precursor Abbreviations		Abbr.	Precursor Material				
		3DMAS	Tris(dimethylamino)silane				
		TDMAHf	Tetrakis(dimethylamino)hafnium				
		TMA	Trimethylaluminum				
Recipes are found in the read-only directory c:\Cambridge Nanotech\Recipes\Masters							

7.5 Run Startup Recipe

- 7.5.1 Press the Start button in the System Controls section. (See **Figure 2, ALD Control Screen.**)
- 7.5.2 At the "Are you sure you want to start a Run?" prompt, click "Yes".
- 7.5.3 Wait for the system temperatures to reach setpoint.

NOTE: Since the system can take several hours to reach desired temperatures (especially when cooling), a lab member could intervene and use the system after another has initiated their process conditions. To prevent this, a member should reserve and enable the tool while the temperatures are stabilizing. Tool use charges may apply.

8 **Film Deposition Procedures**

8.1 Enable in Coral

- 8.1.1 Log in to Coral and Enable the tool.

8.2 Verify Temperatures

- 8.2.1 From the Heater & Valve Section, ensure temperatures are stable at the desired setpoints.
 - 8.2.1.1 **If they are not**, continue to wait.
 - 8.2.1.2 **If the setpoints are not correct**, repeat the procedures in section 7 Setup Procedures.

8.3 Vent Chamber

- 8.3.1 **If the Pump/Vent button in the System Controls section reads "VENT"**, click the button.

NOTE: The button text will now change to "PUMP".
- 8.3.2 Wait until the chamber pressure is greater than 5.50E+2 Torr.

8.4 Remove Substrate Platen

- 8.4.1 Open the door of the Load Chamber.
- 8.4.2 Insert the loading wand into the Load Chamber.
- 8.4.3 Slide the curved tips of the loading wand under the clips on the substrate platen.
- 8.4.4 Using the wand, slightly lift the substrate platen from the chuck.
- 8.4.5 Slide the substrate platen out of the process chamber.
 - 8.4.5.1 Be careful not to rub against the walls of the load chamber or the chuck.
- 8.4.6 Place the substrate platen on a quartz plate.
- 8.4.7 Do NOT place the platen on any other surface.



CAUTION



The substrate platen may be very hot.

Do NOT touch the platen or place it on any surface other than quartz.



CAUTION



8.5 Load Sample(s)

- 8.5.1 Place the wafer or sample in the center of the substrate platen.

8.6 Load Monitor

8.6.1 Place a monitor on the substrate platen, near the wafer or substrate.

NOTE: The monitor must be bare silicon. It may be a 2" wafer or a piece of a larger wafer.

8.7 Load Substrate Platen

8.7.1 Open the door of the Load Chamber.

8.7.2 Slide the curved tips of the loading wand under the clips on the substrate platen.

8.7.3 Using the wand, lift the substrate platen.

8.7.4 Slide the substrate platen into the process chamber.

8.7.4.1 Be careful not to rub against the walls of the load chamber or the chuck.

8.7.5 When fully inserted, lower the substrate platen so that it rests on the chuck.

8.7.6 Lower the loading wand to release the clips.

8.7.7 Carefully slide the loading wand out the of the Load Chamber.

8.7.8 Place the loading wand it its storage location.

8.8 Pump Down

8.8.1 Close and hold the process chamber door.

8.8.2 Click the Pump/Vent button to pump down the system.

NOTE: The button text will now change to "VENT".

8.8.3 Wait until pressure is less than 1E-1 Torr (100mTorr).

8.9 Load Recipe

8.9.1 Right click in the recipe steps table. (See **Figure 4, Recipe Section Detail**.)

8.9.2 Click on "Load recipe ..." in the drop down menu.

8.9.3 Select the recipe for the desired deposition. (See **Figure 2, ALD Control Screen**.)

8.9.4 Click OK.

8.10 Adjust Recipe

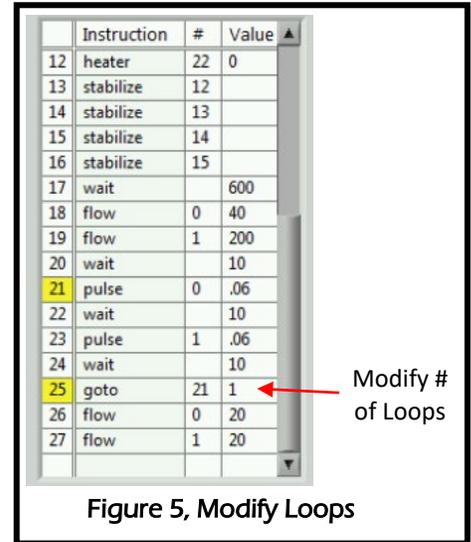
NOTE: Master recipes reside in a write-protected directory. They can be modified, but cannot be saved to the master directory. However, recipes can be saved to user-defined subdirectories.

- 8.10.1 Modify the value of the goto instruction listed in **TABLE 1, ALD RECIPE AND PROCESS INFORMATION** to the necessary number of loops for the target thickness. (See **Figure 5, Modify Loops**.)

NOTE: By default, all recipes will not repeat the loop. Failure to change this value will result in a single cycle for deposition.

- 8.10.2 **If desired**, modify the heater setpoints at the end of the recipe.

NOTE: All deposition recipes are written to return to the standby temperatures at the end of the deposition. There may be occasions when other temperatures are wanted (i.e., back-to-back runs).



	Instruction	#	Value
12	heater	22	0
13	stabilize	12	
14	stabilize	13	
15	stabilize	14	
16	stabilize	15	
17	wait		600
18	flow	0	40
19	flow	1	200
20	wait		10
21	pulse	0	.06
22	wait		10
23	pulse	1	.06
24	wait		10
25	goto	21	1
26	flow	0	20
27	flow	1	20

Modify # of Loops

Figure 5, Modify Loops

8.11 Run the Process

- 8.11.1 Press the Start button in the System Controls section. (See **Figure 2, ALD Control Screen**.)

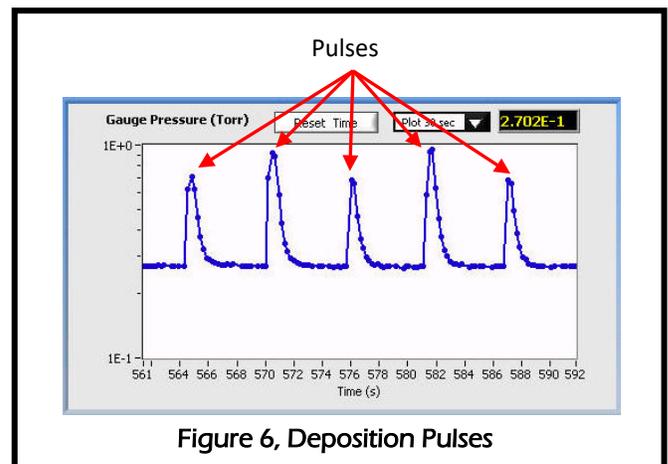
- 8.11.2 At the "Are you sure you want to start a Run?" prompt, click "Yes".

- 8.11.3 During the 600 second wait instruction, note the system pressure.

NOTE: This is the base pressure to be recorded later in Coral.

- 8.11.4 Monitor the first several pulse commands.

- 8.11.4.1 Ensure there is a rise in pressure from each pulse command. (See **Figure 6, Deposition Pulses**.)



- 8.11.5 **If running a plasma recipe**, ensure the plasma ignites.

- 8.11.6 Wait for the end of the recipe.

NOTE: There will be a "Recipe complete." message in the System Messages section.

8.12 Vent Chamber

8.12.1 Follow the procedure in paragraph 8.3 Vent Chamber.

8.13 Remove Substrate Platen

8.13.1 Follow the procedure in paragraph 8.4 Remove Substrate Platen.

8.14 Remove Wafer/Sample

8.14.1 Wait a minimum of 5 minutes for the platen and sample to cool.

8.14.2 Remove the wafer/sample from the platen.

8.14.3 Remove the monitor from the platen.

8.15 Load Substrate Platen

8.15.1 Follow the procedure in paragraph 8.7 Load Substrate Platen.

8.16 Place ALD in Standby

8.16.1 Close and hold the process chamber door.

8.16.2 Click the Pump/Vent button to pump down the system.

NOTE: The button text will now change to "VENT".

8.16.3 Wait until pressure is less than 1E-1 Torr (100mTorr).

8.16.4 Right click in the recipe steps table. (See **Figure 4, Recipe Section Detail**.)

8.16.5 Click on "Load recipe ..." in the drop down menu.

8.16.6 Select the ALDStandby, 100C recipe.

8.16.7 Click OK.

8.16.8 Press START to run the program.

8.16.9 Click OK in the recipe start confirmation window.

8.16.10 Turn the Load Chamber Purge Valve clockwise to the "Closed" position. (See **Figure 3, Purge Valve**.)

8.17 Measure Monitor

8.17.1 Using the Nanospec, n&k, or Woollam Ellipsometer, measure the thickness on the monitor wafer.

8.18 Disable in Coral

8.18.1 Log in to Coral and Disable the tool.

8.18.2 Enter all requested information.

9 Process Notes

9.1 Definitions and Process Terminology

- 9.1.1 Plasma. The Inductively-Coupled Plasma (ICP) coil produces a very high density power output that can be used to enhance film deposition.
- 9.1.2 Heaters. The system includes several heating elements to maintain a stable temperature of various components.
- 9.1.3 Gas Delivery. Mass Flow Controllers (MFC's) are used to control the rate process gases flow into the reactor chamber.
- 9.1.4 Precursor. A chemical compound that includes the element intended for the deposition cycle. Precursors are generally highly reactive and must be treated with extreme care.
- 9.1.5 Plasma Deposition. A deposition technique that uses an inert gas with a plasma energy source in addition to the precursor. The chuck may be kept at room temperature, but is frequently controlled to higher temperatures.
- 9.1.6 Thermal Deposition. A deposition technique that does not use a plasma energy source. This technique normally uses pulses of water in addition to the precursor and temperature of the chuck to control the deposition rate.
- 9.1.7 Exposure Mode. Pulsing precursors with the stopvalve closed temporarily between pulses. This technique is normally used for precursors with a low vapor pressure.

9.2 Programming Commands

9.2.1 TABLE 2, RECIPE INSTRUCTIONS describes the programming commands available for a recipe with their parameters and primary function.

Instruction	#	Value	Description																								
flow	0 – 5	0 – 200	Sets the setpoint of MFC “#” to “value” in sccm. The associated MFCValve must be open to ensure flow.																								
goto	0 – recipe max	≥ 1	Sends the recipe to line number “#” for “value” loops. Please note a Value of 0 or less will result in an infinite loop. Goto loops can be nested.																								
heater	12 – 22	0 – max	Sets the setpoint of heater “#” to “value”. <table border="1" style="margin-left: 20px;"> <thead> <tr> <th>#</th> <th>Heater</th> <th>Max</th> </tr> </thead> <tbody> <tr> <td>12</td> <td>Trap</td> <td>250</td> </tr> <tr> <td>13</td> <td>Reactor 1</td> <td>300</td> </tr> <tr> <td>14</td> <td>Reactor 2</td> <td>180</td> </tr> <tr> <td>15</td> <td>Chuck</td> <td>300</td> </tr> <tr> <td>16</td> <td>Precursor Delivery</td> <td>150</td> </tr> <tr> <td>17</td> <td>ALD Valves</td> <td>150</td> </tr> <tr> <td>18-22</td> <td>Precursors</td> <td>150, but varies by precursor</td> </tr> </tbody> </table>	#	Heater	Max	12	Trap	250	13	Reactor 1	300	14	Reactor 2	180	15	Chuck	300	16	Precursor Delivery	150	17	ALD Valves	150	18-22	Precursors	150, but varies by precursor
#	Heater	Max																									
12	Trap	250																									
13	Reactor 1	300																									
14	Reactor 2	180																									
15	Chuck	300																									
16	Precursor Delivery	150																									
17	ALD Valves	150																									
18-22	Precursors	150, but varies by precursor																									
line ac out	n/a	0, 1	Turns “#” ac plug on (1) or off (0). These plugs can be used for accessory equipment. Currently, none are used in this system.																								
MFCValve	1 – 5	0, 1	Opens (1) or closes (0) the downstream pneumatic valve for the specific gas (enables actual gas flow through the “flow” instruction).																								
plasma		0 – 300	Sets the setpoint of the ICP coil to “value” watts. When a recipe uses the plasma, there MUST be a “plasma 0” instruction to turn the plasma off.																								
pulse	0 – 5	0.01 – 10	Opens (pulses) the selected precursor ALD valve, “#”, for “value” seconds.																								
stabilize	12 – 22		Waits until heater “#” is ±2°C of setpoint.																								
stopvalve		0, 1	Opens (1) or closes (0) the stop valve (gate valve). Close the stopvalve to use Exposure mode for materials with very low vapor pressure. A “stopvalve 1” instruction MUST be included to end the Exposure mode step and prevent excess CVD deposition in the chamber.																								
wait		≥ 0	Waits/pauses for “value” seconds before moving to the next instruction in the recipe.																								

10 Revision History

Rev	Date	Originator	Description of Changes
3	05 Jun 2019	T. Olsen	Update document format.
2	02 Apr 2018	T. Olsen	Major rewrite.
1	04 Aug 2010	Sam Bell	Original Release.