Savannah 100 & 200
Atomic Layer Deposition System

User Manual

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This manual is also available in other languages upon written request.

Notice

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Section 1
Safety

Introduction

Read and follow these safety instructions. Task- and equipment-specific warnings, cautions, and instructions are included in equipment documentation where appropriate.

Make sure all equipment documentation, including these instructions, is accessible to persons operating or servicing equipment.

Qualified Personnel

Equipment owners are responsible for making sure that Cambridge NanoTech Inc. equipment is installed, operated, and serviced by qualified personnel. Qualified personnel are those employees or contractors who are trained to safely perform their assigned tasks. They are familiar with all relevant safety rules and regulations and are physically capable of performing their assigned tasks.

Intended Use

Use of Cambridge NanoTech Inc. equipment in ways other than those described in the documentation supplied with the equipment may result in injury to persons or damage to property.

Some examples of unintended use of equipment include:

- using incompatible materials
- making unauthorized modifications
- removing or bypassing safety guards or interlocks
- using incompatible or damaged parts
- using unapproved auxiliary equipment
- operating equipment in excess of maximum ratings

Regulations and Approvals

Make sure all equipment is rated and approved for the environment in which it is used. Any approvals obtained for Cambridge NanoTech Inc. equipment will be voided if instructions for installation, operation, and service are not followed.

Client Modifications

Modifications to the system including, but not limited to changes to vacuum hardware, electronics, and software, void all warranty and liability.

Personal Safety

To prevent injury follow these instructions.

• Do not operate or service equipment unless you are qualified and have fully read and understood the manual and warning labels on the system. Contact Cambridge NanoTech Inc. with any questions in case of uncertainties.

• Do not operate equipment unless safety guards, doors, or covers are intact and automatic interlocks are operating properly. Do not bypass or disarm any safety devices.

• Before adjusting or servicing equipment, or touching any of the parts, turn off the heaters in the software, wait until all temperature sensors are at room temperature, then shut off the power supply and unplug the main power and wait until all unmonitored parts have cooled down. Lock out power and secure the equipment.

• Relieve (bleed off) pneumatic pressure before adjusting or servicing pressurized systems or components, such as gas cylinders. Never disconnect high pressure gas cylinders without specific knowledge. Refer to your supplier for instructions.

• Obtain and read Material Safety Data Sheets (MSDS) for all materials used. Follow the manufacturer's instructions for safe handling and use of materials, and use recommended personal protection devices.

• To prevent injury, be aware of less-obvious dangers in the workplace that often can not be completely eliminated, such as hot surfaces, sharp edges, energized electrical circuits, and moving parts that can not be enclosed or otherwise guarded for practical reasons.

Fire Safety

To avoid a fire or explosion, follow these instructions.

• Do not place flammable materials underneath, on or near the unit. Do not place paperwork, clothing etc. on or near the unit.

• Do not run the system unattended; do not run the system overnight.

• Do not heat materials to temperatures above those recommended by the manufacturer. Make sure heat monitoring and limiting devices are working properly. Note the maximum temperature settings for different parts. ALD pulse valves are rated to 200 ^{o}C and should not be heated above that temperature. Center heater maximum temperature is 400 ^{o}C, while outer heater should not be set higher then 250 ^{o}C because of the Kalrez™ O-ring. The tee and flexible bellows of the pumping line should not be heated above 150 ^{o}C. Temperature of the precursors should not exceed safety or decomposition temperature of the chemical used. Maximum for the precursor heater jacket is 200 ^{o}C.

• The pump may exhaust small amounts of unreacted precursor. Since Cambridge NanoTech Inc. does not supply the chemicals, responsibility for safe venting and exhausting lies with the customer. General exhaust recommendations include using inert pumping fluid such as Fomblin SV, fireproof metallic exhaust lines to prevent fire. Refer to local codes or your material MSDS for guidance. Minimize precursor use. Do not add vapor traps in the pumping line, upon exposure to air large amounts of trapped precursor may ignite or cause chemical burns.

• Know where emergency stop buttons, shutoff valves, and fire extinguishers are located.

• Clean, maintain, test, and repair equipment according to the instructions in your equipment documentation.

• Use only replacement parts that are designed for use with original equipment. Contact your Cambridge NanoTech Inc. representative for parts information and advice.

Electrical Safety

To avoid electric shocks, follow these instructions.

• At the time of installation the stainless steel system cabinet must be grounded by attaching the supplied ground wire to the facilities grounding loop.

• Turn off and unplug the electronic control unit prior to connecting or disconnecting any sensor, heater, valve or other components.

• Do not disconnect live electrical circuits while working with flammable materials. Shut off main power first to prevent sparking.

Chemical Safety

To avoid chemical hazards, follow these instructions.

• Know the nature of the precursors you are working with (read MSDS).
Some precursors such as trimethylaluminum are pyrophoric, they burn upon exposure to air. Precursors should never be disconnected from the manual valve they were supplied with. Make sure that manual valve is closed before removing the precursor-valve combination from the system.
Pump/purge the space between ALD valve and manual valve before disconnecting any precursor. Always wear proper protection equipment when removing precursors. Precursor replacement should only be conducted by qualified personnel. Read the section on precursor removal before proceeding. Cambridge NanoTech Inc. can be reached for safety assistance with precursor replacement/removal procedure, although the final responsibility lies with the user.

Action in the Event of a Malfunction

If a system or any equipment in a system malfunctions, shut off the system immediately and perform the following steps:

• Disconnect and lock out system electrical power. Close valves and relieve pressures.

• Identify the reason of the malfunction and correct it before restarting the system.

Disposal

Dispose of equipment and materials used in operation and servicing according to local codes.

Section 2
Theory

Atomic Layer Deposition: Principle of Al_2O_3 formation

Atomic Layer Deposition (ALD) is a technique that allows growth of thin films, atomic layer by layer. Deposition of Al_2O_3 from water and trimethylaluminum (TMA) precursors will be used to illustrate the principle of ALD. Recipes for other materials can be found in the literature.
The chemical principle of Al_2O_3 growth from water and TMA is outlined in Figure 1. Five steps can be identified:

1. Put in a sample which is hydroxylated from exposure to air, oxygen or ozone (Figure 1A).

2. Pulse the TMA precursor; TMA will react with the OH groups on the surface. TMA does not react with itself and the monolayer formed passivates the surface (Figure 1B, 1C).

3. Remove unreacted TMA molecules by evacuation and/or purging with nitrogen (Figure 1D).

4. Pulse water (H_2O) into the reactor. This will remove the CH_3 groups, create Al-O-Al bridges, and passivate surface with Al-OH. CH_4 (methane) is formed as a gaseous byproduct (Figure 1E, 1F).

5. Remove unreacted H_2O and CH_4 molecules by evacuation and/or purging with nitrogen (Figure 1G).

Steps (a)-(g) form a ***cycle***. Each cycle produces a maximum of 1.1 Å of Al_2O_3 depending on temperature.Thus, 100 cycles produces 11 nm of Al_2O_3.

Modes of Operation

There are two main modes of operation:

1. Continuously flowing nitrogen carrier gas while pulsing (adding) precursor and pumping continuously
2. Pulsing precursors with stop valve closed and pumping in-between pulses

This research system can operate in either mode.

Pyrophoric Precursors

Trimethylaluminum (TMA) is a liquid at room temperature and is pyrophoric. This means that it burns upon exposure to air. TMA reacts with water vapor in the air. For this reason, the TMA bottle may only be opened in a glove box with inert atmosphere by experienced professionals such as at the chemical supply companies (Strem, Sigma-Aldrich etc).

Process Flow

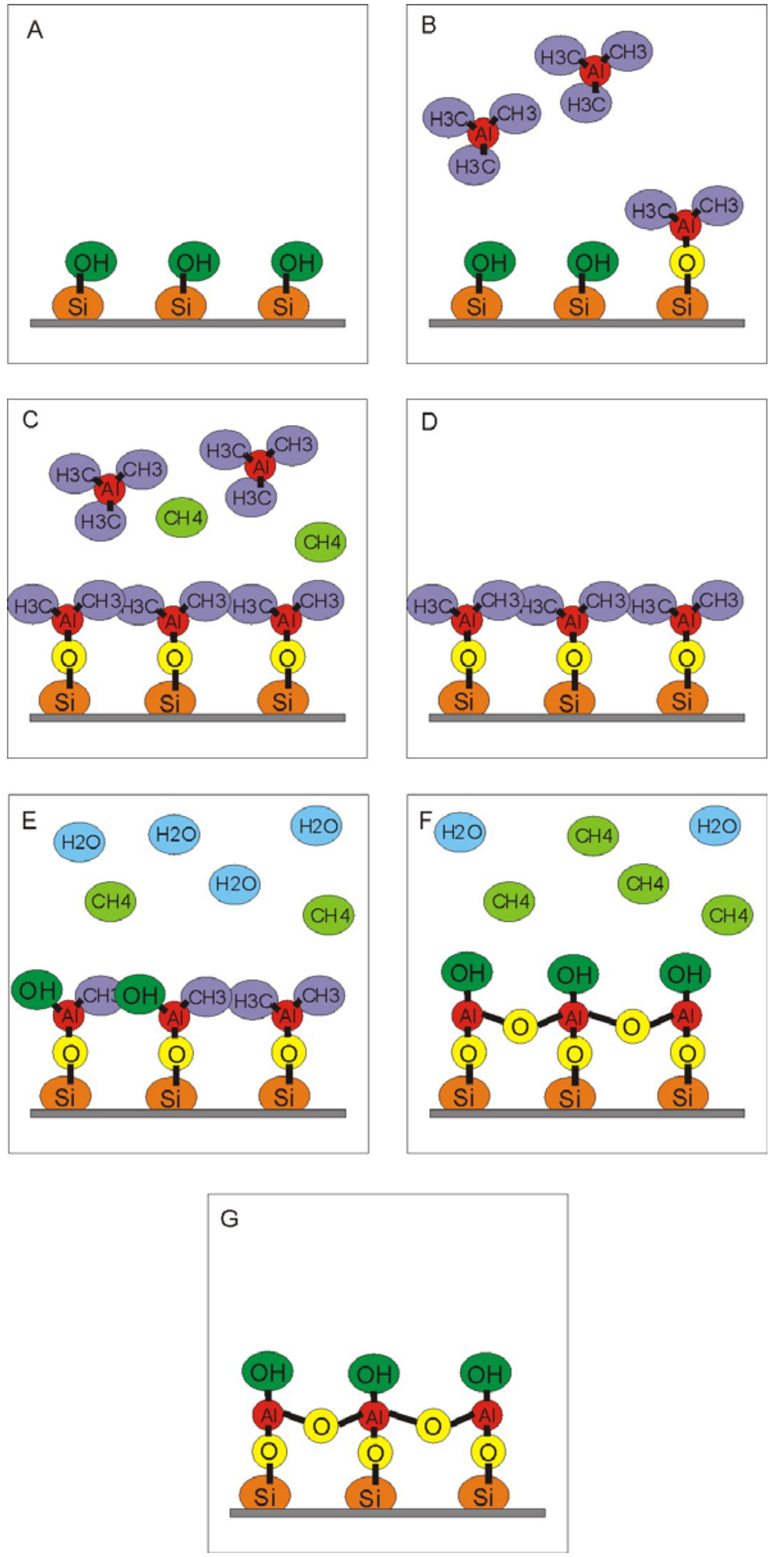


Fig. 1. Chemical reactants in the ALD process are introduced into the deposition chamber as gases, and supplied in pulses delivered to the reactor at different times. Reactants are separated from one another in the flow stream by a purge gas or evacuation. Each reactant pulse chemically reacts with the wafer surface, making ALD a self-limiting process capable of precise monolayer growth.

Section 3 Installation

Introduction

The Savannah 100 & 200 ALD system is shipped disassembled and assembly is required prior to operation. Carefully inspect all supplied parts for damage before proceeding with installation.

Installation must be performed by qualified personnel observing all safety regulations and procedures. Helium leak checking of the entire assembled system is advised.

System Assembly

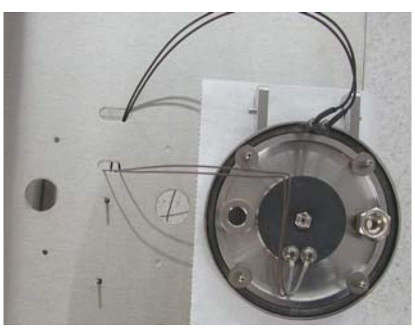
Unpack and identify all parts before the assembly. Start with installing the cabinet at the place of intended use. Make sure that enough space is provided for rear and side access to the system.

Stainless steel cabinet

Reactor Assembly

Tubular (Outer) Heater:

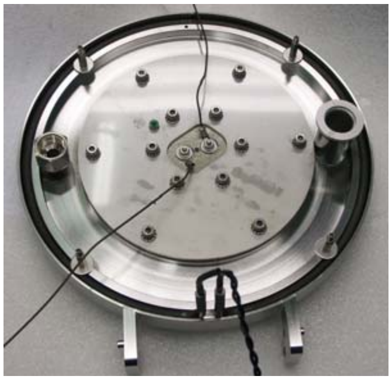
Take the tubular (outer) heater and place it into the skirt of the bottom of reactor aligning the bent areas of the heater in the center between the hinges. Test that the heater is properly aligned by sliding the heaters electrical connections and bent ends through the top elliptical hole in the center of the top of the cabinet. The tubular heater should pass through the center of the hole and should not be touching the sides of the hole. Make sure that the 4 bolts align as well. Once the alignment has been confirmed take 4 washers and slide one over each shoulder bolt and tighten them with 3/4" length 1/4" wide hex nuts.



Inner Disk Heater for Savannah 100:

Take the inner disk heater and place it around the center threading, making sure that the electrical connections are aligned in the center towards the handle, away from the hinges. Tighten the heater down with a 3/8" 5/16" nut using a 5/8" wrench to insure good thermal contact.

Inner Disk Heater for Savannah 200:



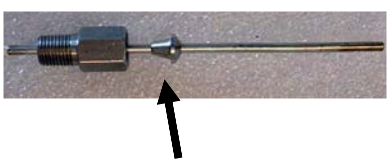
The Savannah 200 substrate heater consists of three disks: a blue glass disk with a printed heating conductor, a center mica insulating disk, and a stainless backplate. The three plates are held together by the ground stud and its nut.

To place the heater, mount it with the center hole centered with the RTD temperature sensor hole in the bottom of the reactor. Following this, place the bolts and tighten these. Make sure these bolts are not too tight as this may damage the glass plate or create non-uniform heating.

When rewiring the cable hookup wires, make sure to place the ring lug over the first mica centering ring. While holding this in place, position the ringlug, large mica washer, metal washer, tooth washer and nut. Note that the welded threaded studs are grounded, so it is important that the ring lugs are not shorted to the threaded studs. It is also important that the ringlug fits over the first small mica washer, otherwise there may be no electrical contact with the printed heating pattern on the blue glass disk.

Thread the outer heater electrical connections for both the Savannah 100 & 200 through the larger center elliptical hole in the top rear of the cabinet. Through the smaller elliptical hole towards the center is where the electrical connection for the inner disk heater is threaded.

RTD's:



Ferrule

Before securing the reactor to the cabinet with 4 nuts, take two RTD's and slip the bottom of the compression fitting plus the ferrule over it. Insert the outer (wall) RTD into a compression fitting welded on the inside part of the top of the cabinet and push it all the way into the hole in the reactor. Be careful not to lift the reactor, but to insure that the tip of the RTD is all the way inside. Start with the RTD closer to the front of the cabinet that goes into the hole on the side of reactor. Repeat this procedure to connect the center RTD, hand tightening the compression fitting nuts (over tightening can break the RTD).

Secure the reactor to the cabinet with 4 nuts and tighten the compression fittings.

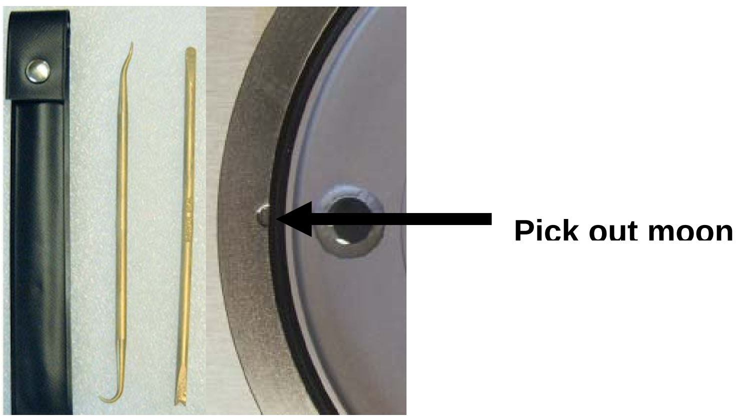
Note: Once the RTD's are in place they do not have to be removed in order to take the reactor off for cleaning or to install it back after the procedure: the reactor can slide over.

Inside of the cabinet with reactor and RTD's installed.

O-ring:

Insert the O-ring into the reactor O-ring groove. Should one need to take out the O-ring, brass O-ring removal tools are provided to pick out the O-ring in the pick out moon. Make sure never to scratch the stainless steel reactor surface or the aluminum lid surface.

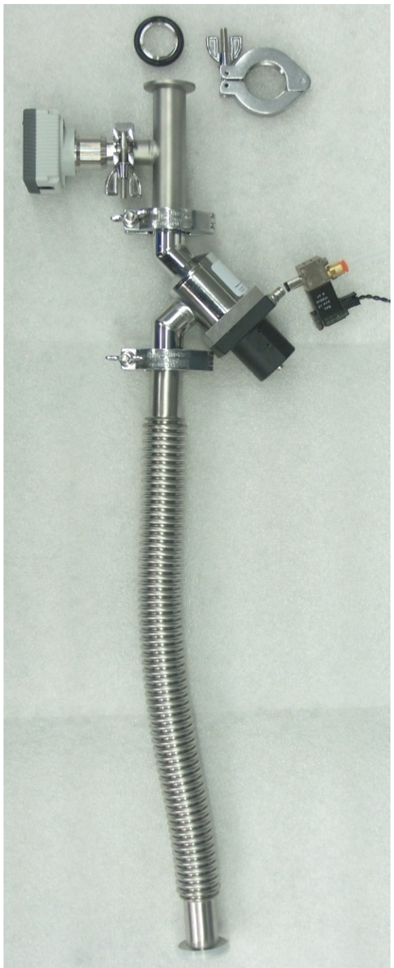
Note: Some deposition can occur on a small area of the o-ring that is exposed to the chamber. Heat cycling can cause this film to flake off. Just wipe the o-ring clean if this occurs.



Lid:

The aluminum lid is placed onto the reactor and hinged on the back.

Pumping Line Assembly



All connections of the pumping line are made with Kwik Flange™ (KF) clamps (also called QF or NW by other manufacturers). Make sure that a properly sized (KF16 and KF25), clean Viton™ O-ring with a centering ring is used and clamps are securely tightened by hand.

The pumping line assembly is shown to the right and is attached to the KF exit port of the reactor at the end of the procedure. The wing nut of the horizontal clamps should be facing right.

The pressure gauge is attached to the left side of the tee with the RJ45 connection facing down. The nut of the clamp should be facing down.

Lift the entire assembly (tee, gauge, stop valve, and hose) and attach the top KF16/25 connection of the tee to the KF16/25 exit port of the reactor using a wing nut clamp and a centering ring. Have the nut on the right hand side. The pressure gauge should be facing toward the cabinet door with the stop valve pointing towards the back of the cabinet. Wrap the tee heater around the entire top part of the pumping line, excluding the bellows, after having removed the solenoid from the stop valve actuator (leaving the tubing on the stop valve) and plug it into the electronics box. Reattach the solenoid to the stop valve and plug it in.

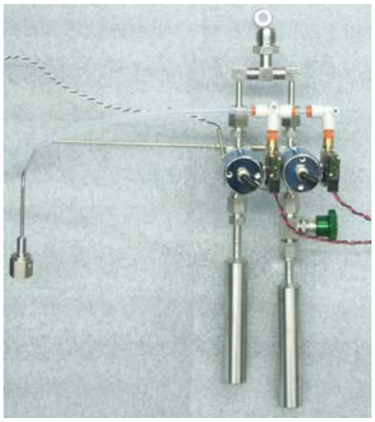
Attach the bellows to the inlet of the pump. The bellows may need to be stretched a little in order to do so. Wrap the bellows hose heater around the bellows part of the pumping line and plug it into the electronics box.

Attach the blue gauge cable and the pneumatic hose of the stop valve. Plug the stop valve in.

Once all of these attachments have been made, the pump can be plugged in. Remember to use Fomblin oil in your b-prepped pump.

Note: Always treat the KF connection flange faces with care and cover them with plastic caps if not in use, scratching can cause leaks. Replace each viton o-ring after use.

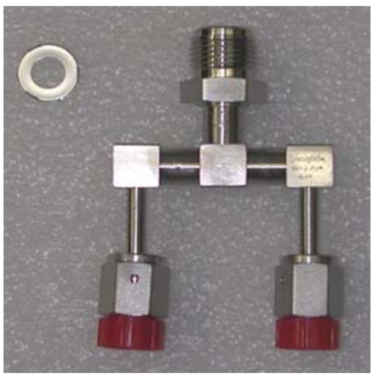
Re: Gauge
The stainless steel tube body of the gauge can be removed for cleaning or replacement. Cleaning of the body should be done very gently as to not break the filament. The gauge comes calibrated, but should calibration at atmosphere and/or vacuum be necessary, please refer to the instruction manual that is included in the Appendix. Note: We recommend not using the copper filter that is shown in the gauge manual. To keep the gauge clean it is important to heat the tee in the pumping line to 150°C, otherwise precursor can condense in the gauge. With the pumping line at 150°C, the gauge tube temperature will be around 60°C. It is also beneficial to keep the dose as low as possible. One can do this by lowering one precursor dose, until uniformity becomes worse, and then double that dose. Same for the other precursor.



Precursor Assembly

Precursor assembly parts are connected by metal face seal VCR™ fittings. A new metal gasket should be used every time a connection is made. Extra care should be taken in order to keep VCR connecting surfaces from scratches. Once disconnected, use plastic covers and plugs to protect polished VCR face surfaces.

Do not over tighten VCR fittings. Once connected, first hand tighten and then tighten by a 45^{o}.

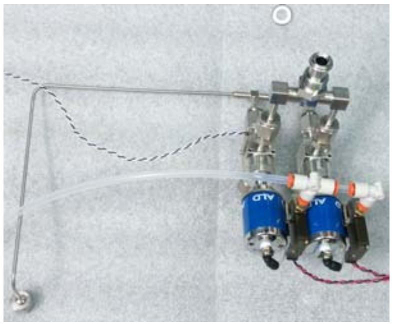


Two port precursor manifold.

Take two high speed ALD pulse valves. Using a new gasket for every VCR-VCR connection attach each valve to the precursor manifold. See picture on the next page for orientation. Hand tighten. Insert the pneumatic hose for each valve into the quick connect fitting. Attach an RTD to one of the valves.

Note: The high speed ALD pulse valves are rated to up to 200 °C.

Two port nitrogen manifold.



Take the nitrogen manifold and insert it into the back of the ALD pulse valves. Hand tighten. The other end of the nitrogen manifold will attach to the MFC after the precursor assembly has been installed into the precursor heater and attached to the reactor. It is best to hold the male nut and only rotate the female nut to prevent torquing.

Precursor manifold with high speed ALD pulse valves and nitrogen manifold attached.

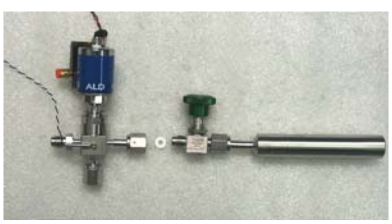
Filling of the Precursor Cylinder

The precursor cylinder should be only filled maximum half way to ensure plenty of vapor space for eg. 20 ml of trimethylaluminum.

Once the manual valve is attached to the ALD valve the space that is formed between these two valves is called the headspace and has to be evacuated prior to the opening of the manual valve.

Note: The arrow on the manual valve attached to the precursor should be pointed toward the cylinder with the chemical.

Never disconnect manual valve from the precursor container, only authorized manufactures can replenish and clean precursor cylinders. Always disconnect cylinder-valve assembly with manual valve closed.



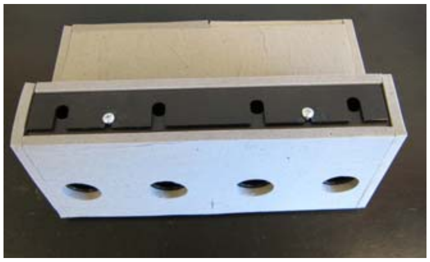
Attach the male VCR fitting on the manual valve on top of the precursor cylinder to the high speed three way valve female nut using a gasket.

The water precursor does not require a manual valve and the ALD valve can be connected directly to the male VCR fitting of the cylinder.

Note: The nitrogen port of the valve is always open and the valve only operates on the precursor inlet.

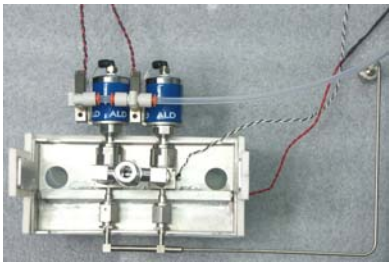
Some precursors require a snap on heating jacket. One is included with each system.

Note: TMA does not need to be heated.

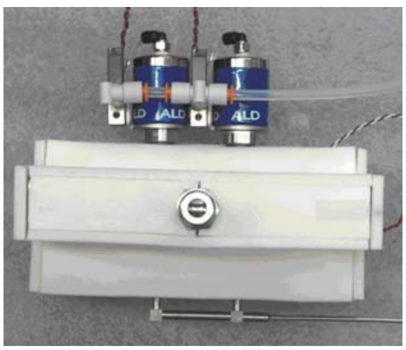


Heater Block for Valves:

Heater block for the ALD pulse valves consists of two sections; top and bottom and is secured by two screws near the top on one side.



Place the precursor assembly into the bottom of the heater block. Thread the wire of the RTD through one of the holes as shown. Attach the top of the heater block, threading the ½" VCR fitting through the top and securing the block with the two screws provided.



Precursor assembly in heater block:

Take the two port precursor assembly in the oven and with a new ½" gasket attach it to the female ½" nut of the reactor. Hand tighten. Follow the same instructions for the larger precursor assemblies. When you are ready to tighten the fitting use a 1 1/16" and the short 15/16" wrench provided. The 1 1/16" wrench holds the ½" female fitting above the cabinet, while the 15/16" wrench tightens the nut inside the cabinet.

Carrier Gas/Pneumatic Gas Connection

Carrier gas (nitrogen, argon or other inert gas) is supplied through the provided mass flow controller (MFC). The supplied MFC is calibrated for nitrogen and a gas correction factor has to be used for other carrier gases. The MFC ¼" male VCR inlet pressure should be around 20 psi (pressure regulator is not provided). The MFC ¼" male VCR outlet is connected to the inert gas input of the 3-port ALD valve on the precursor(s) via a stainless steel nitrogen manifold with VCR connections.

Insert the ¼" PFA tubing for the pneumatic gas into the bottom of the quick-connect tee. 80 psi of air or nitrogen (N_2 preferred) is recommended.

Heat Guard and Hook

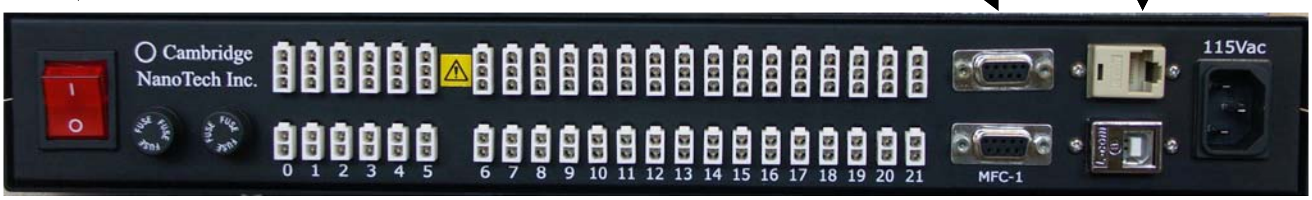
To prevent burning by hot lid and reactor surfaces, a Savannah ALD system comes with a heat guard as a standard safety feature. It is very important to remember to place the heat guard over the reactor each time after loading/unloading the sample.

When the heat guard is not in use, it can be hung on the hook mounted on the side of the cabinet.

Electrical Connections

Plug all the connections into their appropriate locations on the electronic control unit, which is attached to the inside left wall of the cabinet.

Twenty-two 3-pin connectors in positions 0 through 21 are for 115V AC. Sixteen 2-pin connectors in position 6-21 are for RTDs, and positions 0-5 (2-pin connectors) are for 24V DC valves.



Valve section:

#	Pins	Function
0	• • •	Stop Valve
	• •	ALD Pulse Valve 0 – Plug in the valve (precursor) that you want to pulse first.
1	• • •	
	• •	ALD Pulse Valve 1
2	• • •	
	• •	ALD Pulse Valve 2
3	• • •	
	• •	ALD Pulse Valve 3
4	• • •	
	• •	ALD Pulse Valve 4
5	• • •	
	• •	ALD Pulse Valve 5

(Table is continued on the next page)

Heater and RTD section:

6	• • •	Bellows Heater
	• •	Bellows Heater RTD
7	• • •	Tee/Valve Heater
	• •	Tee/Valve Heater RTD
8	• • •	Outer Tubular Heater
	• •	Outer Tubular Heater RTD
9	• • •	Center Disk Heater
	• •	Center Disk Heater RTD
10	• • •	Valve Heater Block
	• •	Valve Heater Block RTD
11	• • •	Precursor Heater Jacket
	• •	Precursor Heater Jacket RTD
12	• • •	Precursor Heater Jacket
	• •	Precursor Heater Jacket RTD
13	• • •	Precursor Heater Jacket
	• •	Precursor Heater Jacket RTD
14	• • •	Precursor Heater Jacket
	• •	Precursor Heater Jacket RTD
15	• • •	Precursor Heater Jacket
	• •	Precursor Heater Jacket RTD
16	• • •	
	• •	
17	• • •	
	• •	
18	• • •	
	• •	
19	• • •	
	• •	
20	• • •	
	• •	
21	• • •	System Cabinet Cooling Fans
	• •	

Section 4 Operation

READ ALL INSTRUCTIONS IN THEIR ENTIRETY BEFORE OPERATION

System Requirements

Microsoft Windows XP® personal computer.

Minumum requirements are: Pentium 4 processor or higher, Windows XP with SP2, 512MB memory, 500MB free harddisk space, one USB port, 1024x768 (SVGA) graphics resolution or higher.

Software Installation

Software for the Savannah 100 & 200 ALD system can be installed from the supplied CD. See Installation instructions.txt.

Preparation

Make sure that the system is fully assembled and all electrical connections to the control unit are properly made. The control computer is connected to the electronic control unit via a USB cable.

Note: Please ensure that the USB cable is at least a foot away from the pump and other noisy equipment.

The system should be degassed and leak checked (refer to the appropriate section of the manual) prior to use.

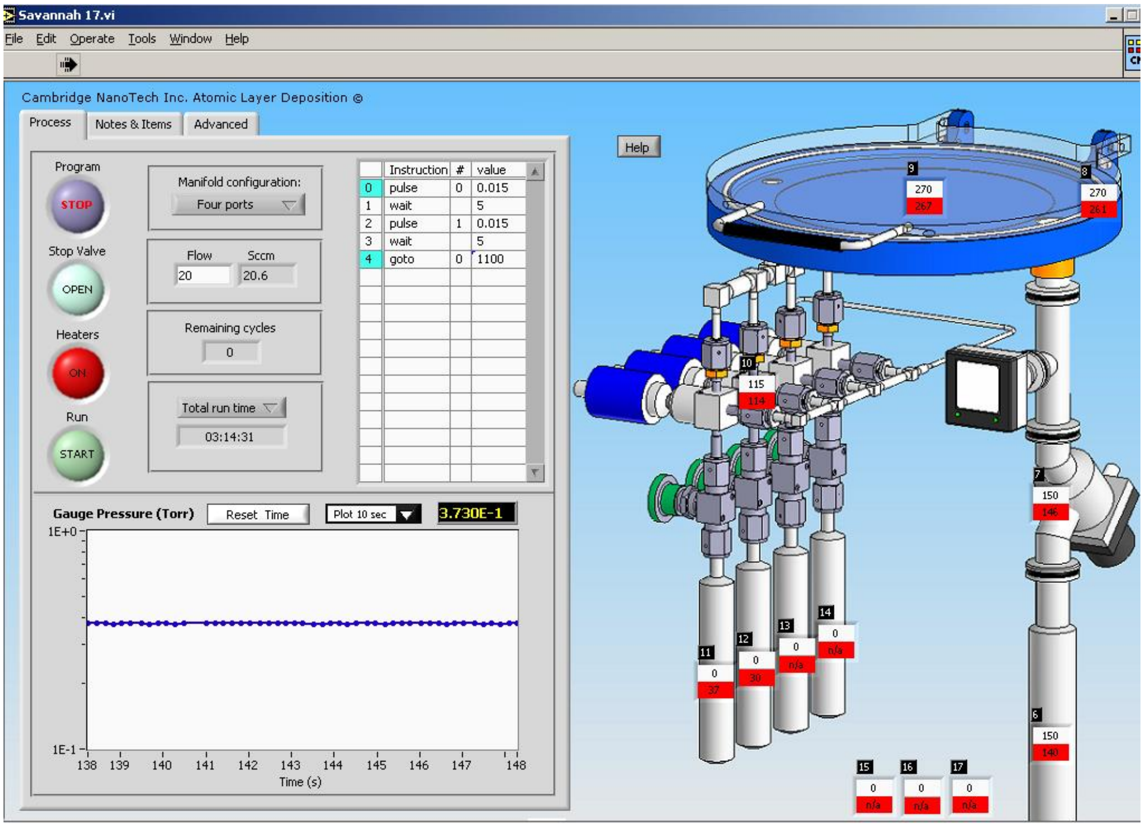
Turn on the electronic control unit and the vacuum pump.

Start the software by double clicking the **“Savannah XX.exe”** program icon. If running the LabView version of the software press the run button (white arrow) in the top left corner. The arrow turns black once the program is running.

Do not run the program with the USB cable disconnected.

Control Software Features

This subsection describes the main features of Savannah control software. The control program allows the operator to control the ALD valves, pumping system, heaters, and to set deposition recipes. The user-interface consists of 3 tab pages: “Process”, “Notes & Items” and “Advanced”, a “Help” button, and temperature setpoint/reading clusters.



Control buttons:

Program – stops the program.

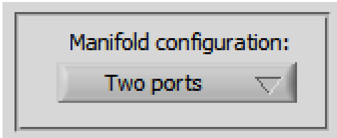
Stop Valve – opens/closes the stop valve to pump down/evacuate the reactor. To prevent the running of a process when the system is vented, when the stop valve is closed, the “Run” button becomes grey, and not clickable.

Heaters – turns ON/OFF all the heaters. When all the heaters are turned, all the temperature setpoints are set to 0^{o}C.

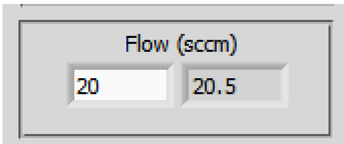
Run – starts/aborts an ALD deposition process. When a process is running, “Program”, “Stop Valve” and “Heaters” buttons are grey and not clickable. To abort a run, simply click on “Run” button once.

Note: Do not close the window of the program while it is running.

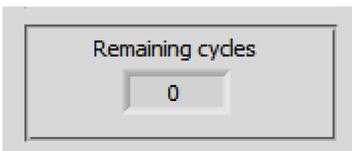
Input and display boxes:



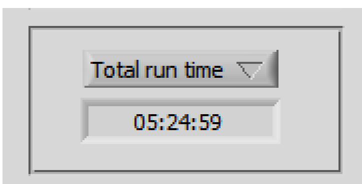
Manifold configuration **–** the dropdown menu allows selecting the correct number of ports on the precursor manifold. The corresponding image of the configuration is displayed in the background image, and each Heater/RTD cluster is automatically arranged to the correct location.



Flow rate control – sets (left) and displays (right) the rate of N_2 flow through the mass flow controller.

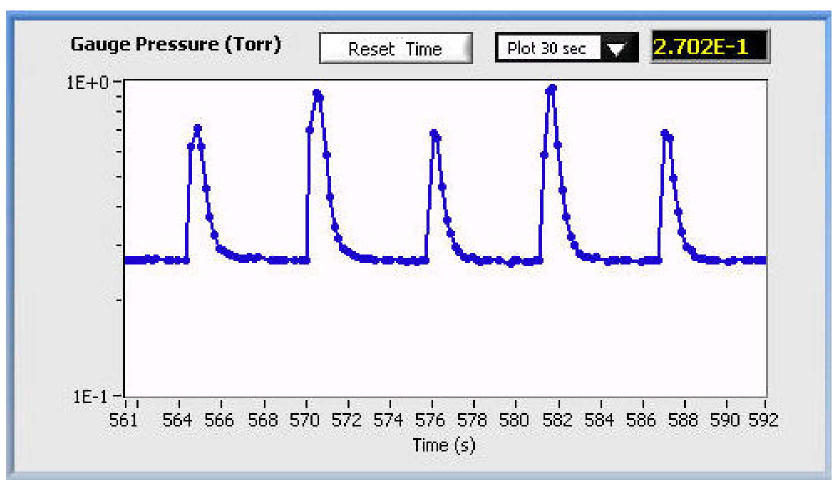


Remaining cycles – displays the number of cycles to be completed in the current loop in a running process. More details about a editing and running a process are given below.



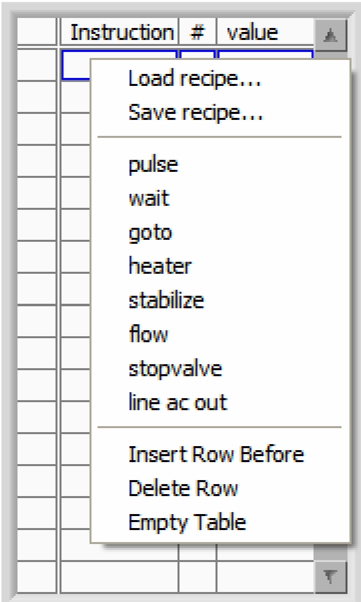
Run time – the text box shows the total time, the time left or the done time of a process depending on item selected in the menu above.

Reactor Pressure 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.

Process Sequence Table:



This table allows programming, loading and saving of the sequence of a process (recipe). Editing of the table is accomplished by selecting commands in a context menu (right-click menu) and typing in numerical values. After right click, the row that the mouse cursor is pointing to becomes selected and is highlighted with a blue frame.

Loading/Saving a recipe – clicking on menu item "Load recipe…" or "Save recipe…" brings up a load/save file dialog allowing access to recipe files saved in computer data storage.

Each command line in the table consists of an automatically assigned line number, an instruction, and two numerical parameters: "#" and "value".

Inserting/deleting a row – clicking on menu item "Insert Row Before" adds a new row before the selected row. Clicking on "Delete Row" deletes the selected row.

Adding/Changing an instruction – clicking on a menu item from "pulse" to "line ac out" first clears the selected row, and then puts the text of the menu item into the instruction cell. Detailed usage of each command is listed below in alphabetical order:

Flow (, value) – sets the flow rate to "**value**", which can be any non-negative number. However, the actual flow rate is limited by the specifications of the mass flow controller in use.

Goto (#, value) – jump to line number "**#**" for "**value**" times. "**#**" must be a non-negative integer and no greater than the largest line number in the recipe. "**value**" must be a positive integer.

Heater (#, value) – sets number of heater (6-17) and temperature.

Line ac out (#, value) – turns the #-th 120V output on (**value**=1) or off (**value**=0).

Pulse (#, value) – opens the "**#**"-th ALD valve for "**value**" seconds and then closes the valve, which completes a precursor pulsing. The valid value of "**#**" is 0,1,2,3,4, and 5, corresponding to the 2-pin socket that the ALD valve is connected to on the electronic box. The range of "**value**" is 0.010 to 10.

Stabilize (#) – waits until heater # has reached its setpoint within 2 degree.

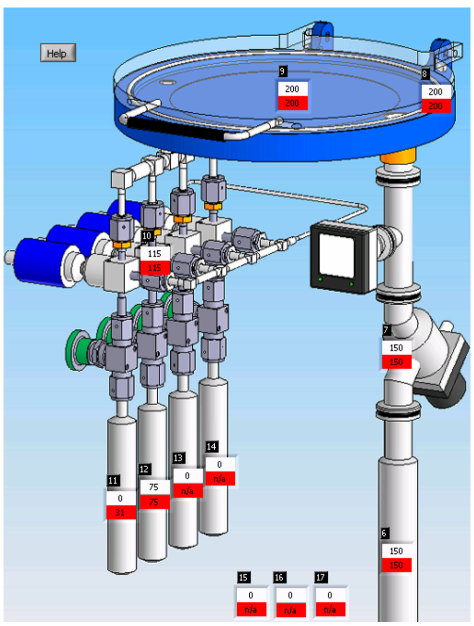
Stopvalve (, value) – closes (**value**=0) or opens (**value**=1) the stop valve.

Wait (, value) – lets the program to wait "**value**" seconds before executing the next command. This command is usually used right after a "pulse" command for purging the pulsed precursor. "**value**" can be any non-negative number.

	Instruction	#	value
0	pulse	0	0.015
1	wait		5
2	pulse	1	0.015
3	wait		5
4	goto	0	800
5	pulse	0	0.015
6	wait		5
7	pulse	4	0.015
8	wait		5
9	goto	5	700

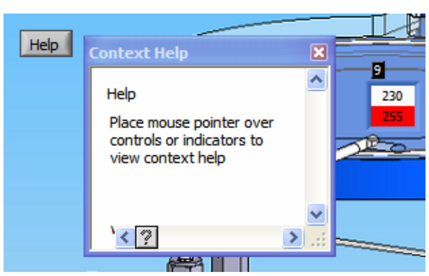
To the left is an example of growing multiple layers of different materials. Line 0 to 4 corresponds to the growth of 800 cycles of the first material using precursors 0 and 1. Line 5 to 9 corresponds to the growth of 700 cycles of the second material using precursors 0 and 4. Line numbers of both **goto** instruction and the line that **goto** jumps to are colored to help identify the range of a **goto** loop.

It is advised to double-check all the parameters in a process sequence before starting a run.



Heater Control Area:

This section of the program window controls the temperature of the heaters. There are 12 input/display clusters numbered 6-17 corresponding to numbers of ports on an electronic box. For each cluster, the temperature setpoint is typed into the white area and the current temperature reading is shown in the red area. If an RTD is not connected to the corresponding port, the temperature reading displays “n/a”

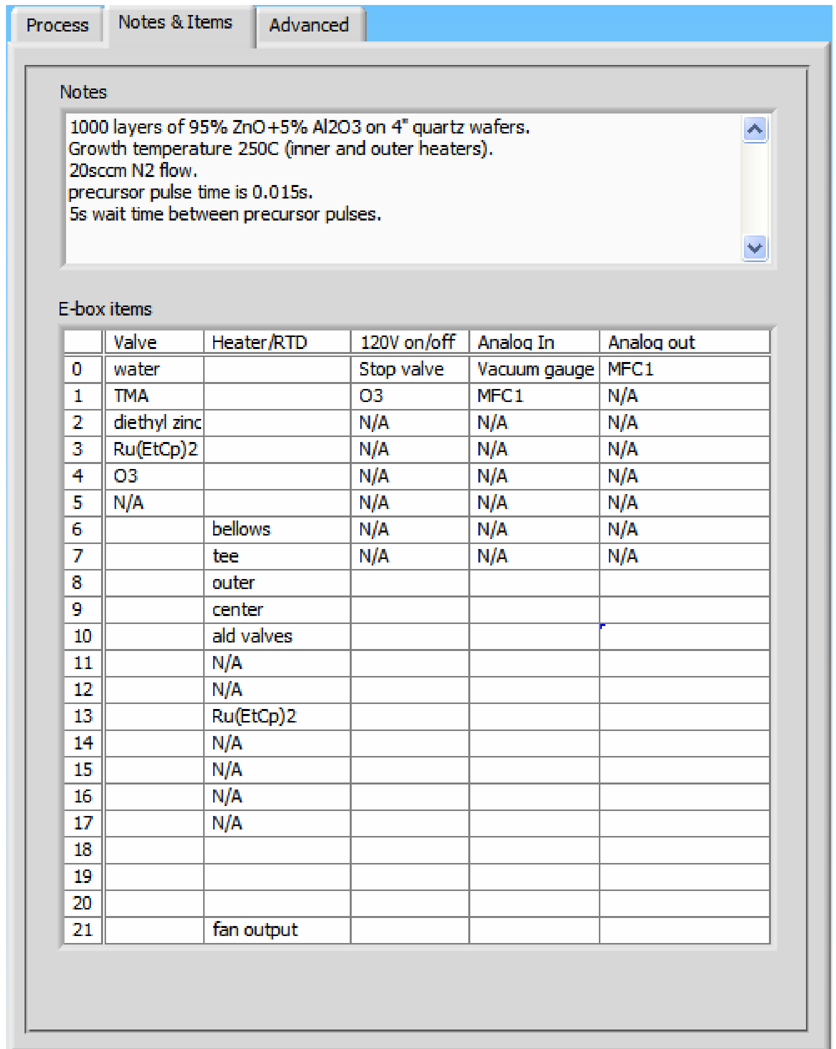


Help Button:

Help button activates context help. Hover over a control to read detailed description.

<u>Notes & Items Page:</u>

This page is used to log information about experimental conditions and the configurations of the ALD system. Comments and notes can be added to the “Notes” text box. “E-box items” provides a customizable list of specifics for each electric port on the electronic box.



	Valve	Heater/RTD	120V on/off	Analog In	Analog out
0	water		Stop valve	Vacuum gauge	MFC1
1	TMA		O3	MFC1	N/A
2	diethyl zinc		N/A	N/A	N/A
3	Ru(EtCp)2		N/A	N/A	N/A
4	O3		N/A	N/A	N/A
5	N/A		N/A	N/A	N/A
6		bellows	N/A	N/A	N/A
7		tee	N/A	N/A	N/A
8		outer			
9		center			
10		ald valves			
11		N/A			
12		N/A			
13		Ru(EtCp)2			
14		N/A			
15		N/A			
16		N/A			
17		N/A			
18					
19					
20					
21		fan output			

Advanced Page:

This page contains controls that users can perform some advanced operations on the electronic box.

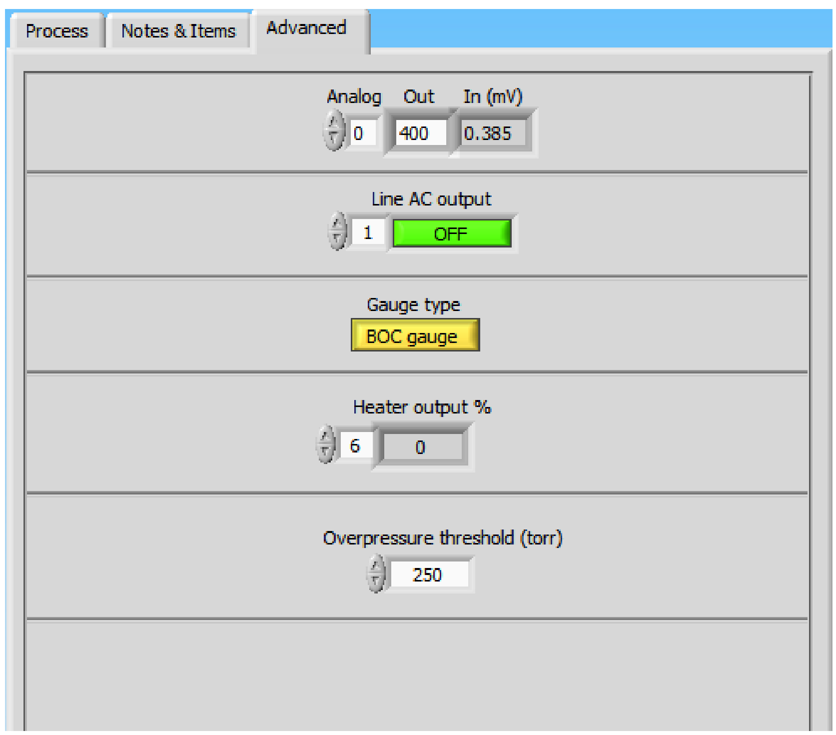
Analog in/out – Analog in/out monitors and sets the voltage levels for the vacuum gauge and the mass flow controller. Contact Cambridge NanoTech Inc. before using this function.

Line AC output – allows switching on/off the 120V (220V in Europe) power output from 3-pin ports 1-5 on the electronic box.

Gauge type – allows choosing between a BOC gauge and an Inficon gauge. All the ALD systems come standard with a BOC gauge. Therefore, it's usually not needed to modify the default value, which is "BOC gauge".

Heater output% – monitors the percentage of heater power outputs from 3-pin ports 6-17 on the electronic box.

Overpressure threshold (torr) – prevents running a process when the reactor pressure is above certain level. By default, the threshold is set to 250 torr. This value rarely needs to be changed. If during an exposure mode run, the pressure rises above the threshold and interrupts the run, it is helpful to increase the threshold value.



Venting the System

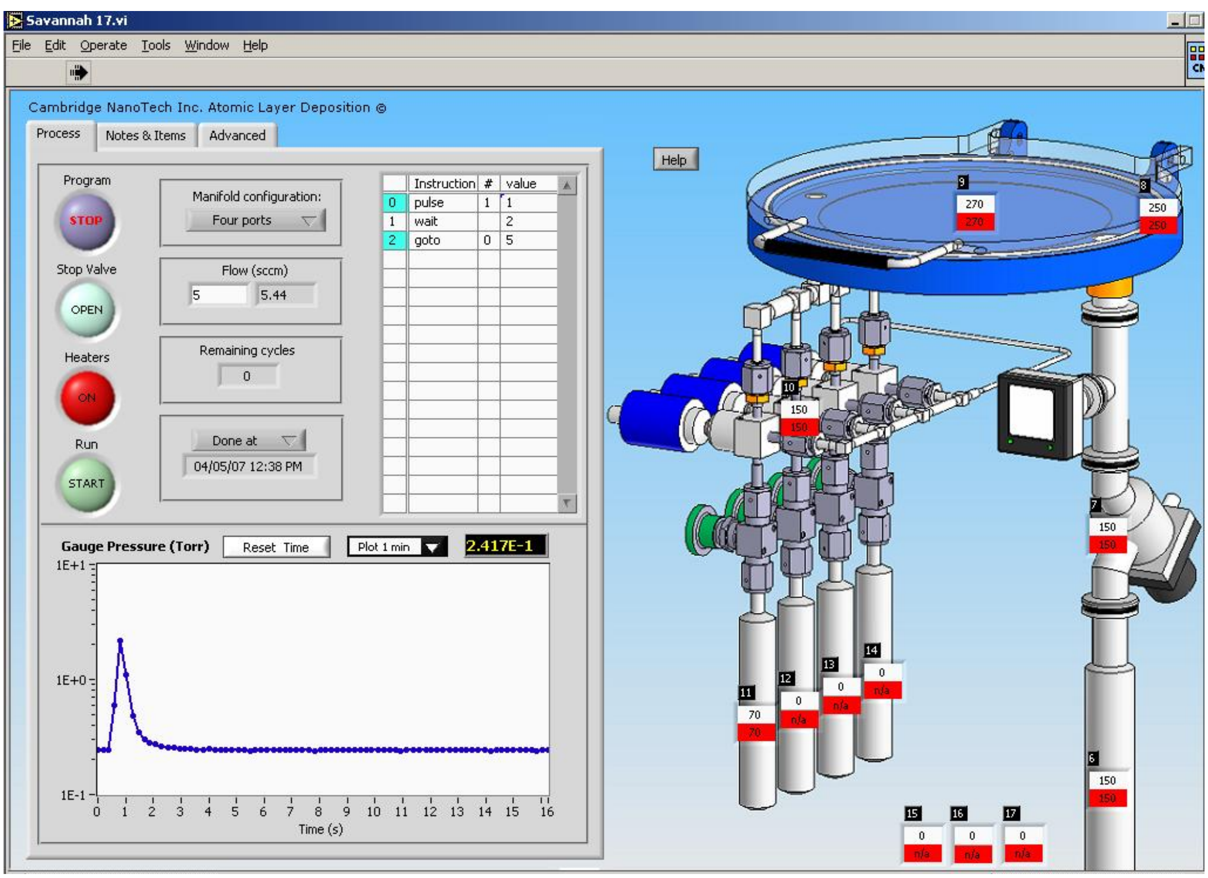
The system is vented by closing the **Stop Valve** (reactor is isolated from the pump) and filling the reactor with carrier gas. For faster venting, a higher flow can be set via the MFC (100 sccm). The system should be at least 80 ^{o}C upon venting. Never vent at room temperature.

Degassing the System

Before using the system for the first time after installation or when new connections or precursors have been added or replaced, the system has to be degassed and checked for leaks.
Power up the system and evacuate the reactor (Stop valve is open). Set the ALD valve heater to 115-150^{o}C, tee and bellows heaters to 150^{o}C, and the inner and outer reactor heaters to 200-270^{o}C respectively. Wait until the temperature reaches the set point for each heater. Note the base pressure of the system.
Degas the water cylinder by doing a run with about 3-5 cycles, “Valve 0” **Pulse** time of 1 second, and the **Pump** time set around 2-3 seconds. Recommended setting for **Flow** is 0-5 sccm.

After the water source degassing run is done and the temperatures have stabilized note the base pressure of the system.

Degas the headspace between the ALD and manual valve of the second precursor by doing the following. Keeping the manual valve on the precursor (trimethylaluminum (TMA) in this example) still closed do a run with valve 1 opening for a 1 seconds **Pulse** time. The **Pump** time is again around 2-3 seconds and the **Flow** is 5 sccm.



Loading/Unloading the Sample

When the base pressure during this run is the same as the base pressure of the system then this area has been degassed.

Note: Degas the headspace of atmospheric gases before opening the manual valve every time the space between manual and ALD valves has been exposed to air. Also degas this area after closing the manual valve if the ALD valve is to be removed.

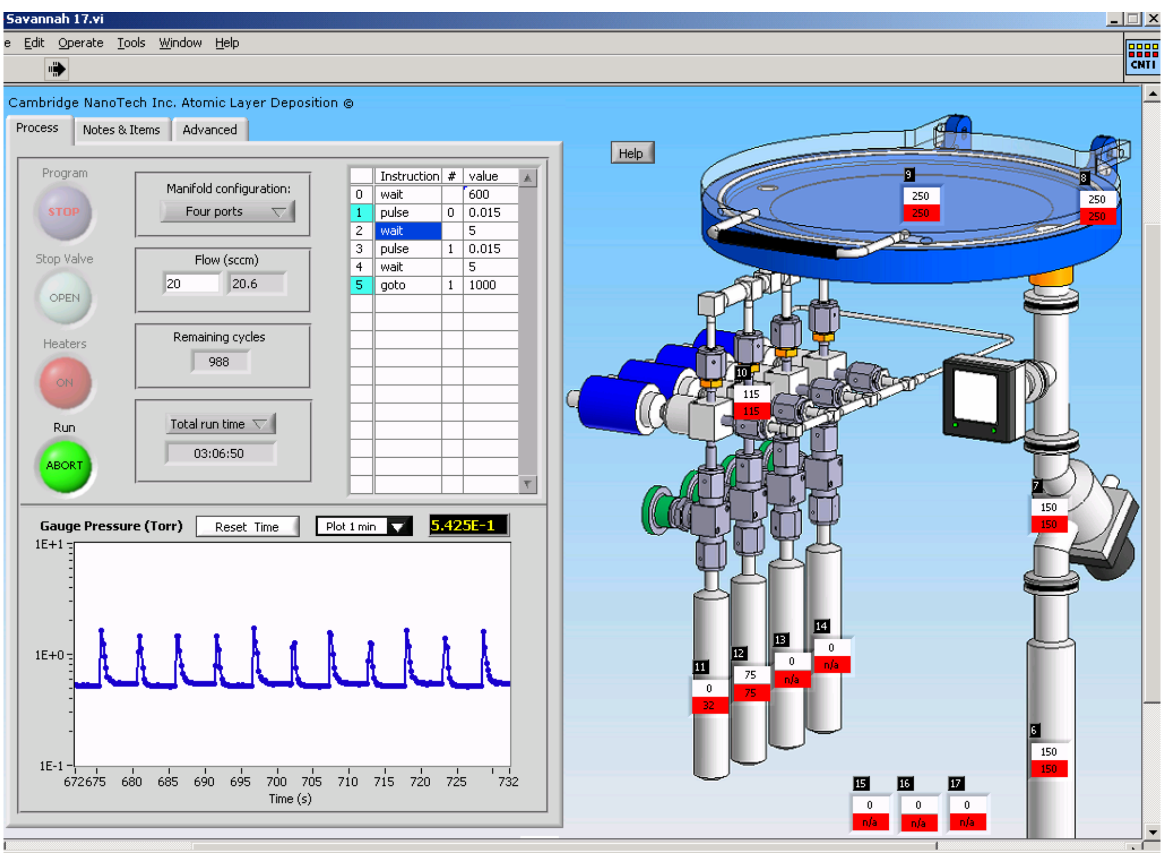
Performing a Growth Run

To load/unload the sample vent the reactor (refer to Venting section of the manual). Once the reactor is at atmospheric pressure, open the lid and place the sample into the recessed area in the center of the reactor. Close the lid and evacuate the system.

ALD growth of Al_2O_3 is used to demonstrate a deposition procedure.

After system has been degassed and vented:

⇒ Load the sample
⇒ Pump down the reactor
⇒ Input the recipe into the process sequence table.
⇒ For valves 0 and 1 set **Pulse** time to 0.015sec, **Wait** time – 5 sec.
⇒ Set number of cycles to 1000, carrier gas **Flow** to 20 sccm
⇒ Delay time can be used to provide enough time for pre-run purge and temperature stabilization.
⇒ Run data is automatically saved in c:\ALD data directory.
⇒ Press **Start Run** button.

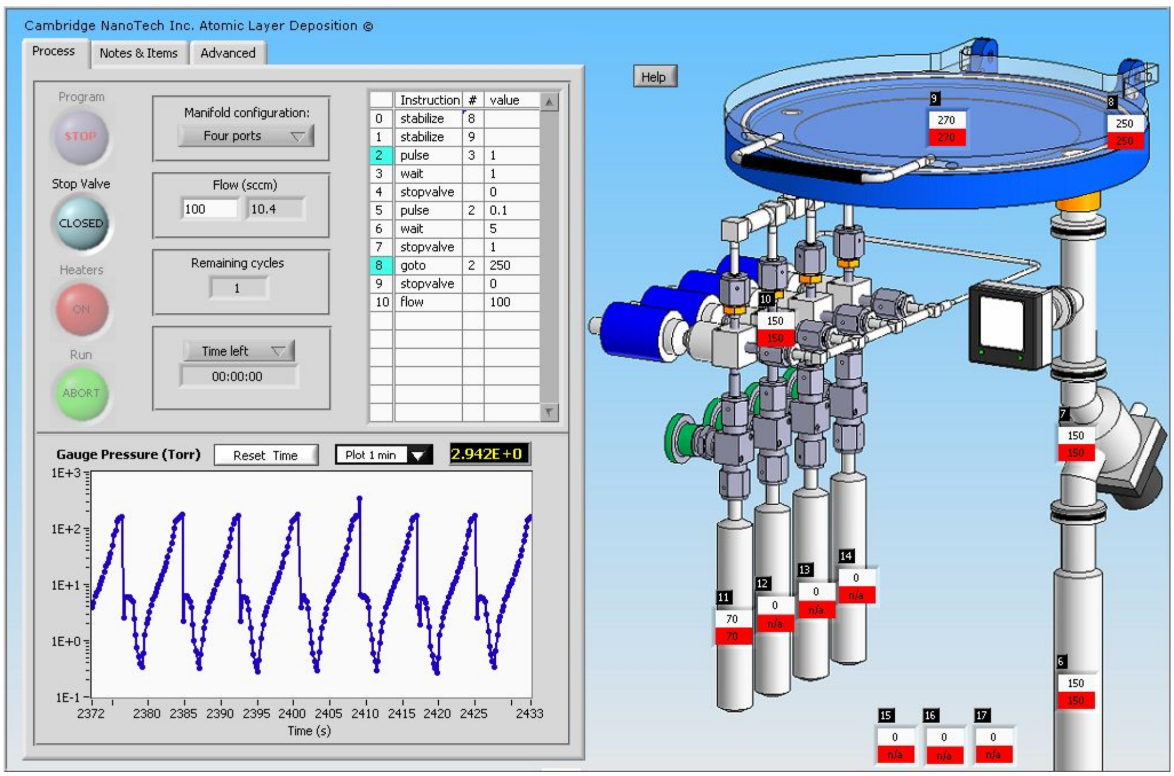


Use of Exposure Mode

In this example about 100 nm of aluminum oxide is deposited. This thickness leads to a beautiful blue color film, and if the wafer is turned over an “ALD frame” can be seen on the backside of the wafer.

Exposure mode can be used for the coating of ultra high aspect ratio structures. This is achieved by dosing a precursor when the stop valve is closed. A wait time follows the dose to allow enough time for the precursor to fully react with the sample surface. In the following screen shot, before pulsing valve 2 for 0.1s, the stop valve is first closed. A wait time of 5s follows the pulse. After the wait, the stop valve opens to allow purging unreacted precursor.

This example is only used to illustrate the principle of using the stop valve during a run.



Section 5 Technical Specifications

Technical Information

Electrical Specifications

Electrical Supply 110V, 10A
Main supply voltage fluctuation not to exceed ±10% of the nominal supply voltage.
Fuses .... ¼"x1¼" fast acting 250V/10A

Facilities Recommendations

It is recommended to use a corrosion resistant (filled with PTFE oil, e.g. Fomblin™, preferably SV oil) rotary vane pump with a recommended pumping speed of 3.5 cfm.

Use of an uninterruptible power supply for the control computer is strongly recommended. Alternatively one can use a notebook computer with a good battery as a back up power.

A supply of high purity nitrogen through stainless steel tubing with a ¼" VCR female end connection and minimum pressure of 20 psi (see Mass Flow Controller specifications for maximum pressure ratings) is required.

80 psi of air or nitrogen (N_2 preferred) for pneumatic actuation in ¼" PFA tubing.

It is recommended to use a PC compatible computer with a fresh installation of Windows 2000 or XP Operating System and an available USB port.

Environmental Specifications

Operating Temperature ....15-40 ^oC(60-100 ^oF)
Humidity .... Max 80% at 30 ^oC (50% at 40 ^oC)
Storage Humidity .... 0-95% relative humidity, non-condensing
Indoor use only.
Pollution degree 2
Installation category... II
Operation Altitude .... Max 2000m

Note: This equipment has been tested and found to **comply** with the limits for a **Class A** digital device, pursuant to **part 15** of the **FCC Rules**. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at user's own expense.

Section 6 Appendix

OEM manuals for the pressure gauge, mass flow controller, and optional equipment are listed in PDF format on the install CD.

Frequently Asked Questions

Please find some of the most frequently asked questions for the Savannah ALD systems below. For an updated list please check our website.

I just received the system, what oil should I put in the pump?

If you received a pump from Cambridge NanoTech, then the pump is “B-prepped” also known as Fomblin prepped. The pump must be filled with Fomblin which has to be purchased by the customer for example from Inland vacuum. We recommend Fomblin SV for its low vapor pressure and inert nature. Ordinary oils are flammable and should not be used. Inland will also clean your oil for you.

Should Cambridge NanoTech Inc. come over and install the system?

In our experience, our customers can very well hook up the Savannah ALD system themselves, after reading the manual and consulting with Cambridge NanoTech Inc. We recommend purchasing the precursor material at least 4 weeks before expected delivery of the system, since the lead time of the precursor material can vary. The required customer supplied items can be found on the quote, or in a detailed specifications document (inquire). It is very important to read the manual; this can not be said enough. It is also useful to contact Cambridge NanoTech Inc. support if things are not clear. We also provide instant messenger support, which is especially useful for international customers (MSN messenger and Yahoo messenger, Please inquire!).

I can’t open the lid, it seems to be stuck!

It is often difficult to open the lid when the reactor is cold. It is best to keep the system warm at all times. We find that the lid opens very well at 80 °C and above. We also provide optional Kalrez O-rings with a proprietary coating which has anti-stick features. Please inquire if needed. It is always good to have a spare O-ring, in case something happens with the old O-ring although we find that the Kalrez O-ring performs extremely well for more than a year.

Frequently Asked Questions continued

The base pressure sometimes goes up, is there something wrong with my gauge?

During deposition one usually overdoses the precursor by a small amount in order to get uniform films and good saturation of each cycle. This means that the residual precursor goes into the pumping port and can deposit all the way in the pump. The gauge consists of a heated filament and temperature sensor. Over time, the coating also deposits onto the filament, and one can recalibrate the sensor to new calibration settings. One needs to calibrate at atmospheric pressure, and at a pressure lower than 1e-5 Torr. This means that once every half a year one could take off the gauge and place it on a turbo pump and recalibrate. Please refer to the gauge manual PDF that was provided on your CD.

My software crashes and shows 1e-7 on the gauge readout and very large numbers on the temperature readout, what is the problem?

This has happened once where the USB cable was mounted in close proximity to a turbo pump. The USB communication was thus not stable. In another instance it can happen if one unplugs one of the cables while the system is running. Unplugging or plugging cables into the electronics box should only be done with the software stopped and the electronics box turned off. It is best that the users call us to report if this problem occurs.

My system is quite dirty, how should I clean it?

Over time a thicker coating develops in the reactor and pumping line. This can flake off, especially if the reactor and other components are heat cycled. Heat cycling can accelerate flaking because the difference of thermal expansion of stainless steel and ceramics like Al_2O_3. It is best to keep the heat cycling to a minimum. The cleaning depends on the material deposited. The lid can be resurfaced with a scotch brite sponge, however to guarantee leak tightness, it would be better to send it back to us for resurfacing. The reactor can be bead blasted clean. The reactor can also be sent back to our company for cleaning.

When I mount the nitrogen manifold, it seems to torque the whole precursor line

When mounting the nitrogen manifold, make sure to NOT turn the male nut on the manifold, and turn the Female nut on the ALD valve 45 degrees (1/8th of a turn) after hand tightening them.

Frequently Asked Questions continued

The NW O-rings in the pumping line are dirty, should I replace them?

The NW O-rings in the pumping line are Viton O-rings. In addition to a coating that can develop during extended use, they can also flatten because of the high temperature of the pumping line (compression set). Because of these reasons, we recommend to always replace the pumping line O-rings with a new O-ring during re-assembly.

The base pressure of the systems seems to be high.

After extended use the pump fluid may have to be replaced/cleaned or the pump may need to be sent out for cleaning. One should take a look in the view port and examine the cleanliness. This often happens after 6 months of extended use without a trap. It may also be an indication that one is using too large of a dose. If too much water mixes with the Fomblin pump oil it starts to look milky and can affect the pumps ability to function.

What spare parts do you recommend purchasing?

We recommend a spare gauge, lid and VCR gaskets. If budget allows we recommend a spare Kalrez O-ring, pump and reactor. The pump will have to be sent out for cleaning and for continued use a spare pump can come in handy.

How do I tighten the VCR fittings?

With VCR fittings in the pumping line, one always has to replace the metal gasket with a new unused gasket. One cannot mount it without a gasket. Hand tighten both nuts and then a total of 45 degrees ($1/8^{th}$ turn) further tightening with two wrenches.

The lid doesn't swing smoothly

Sometimes when the reactor is hot and the lid has been open for more than 5 minutes, the lid will cool down. The thermal expansion of the reactor makes the distance of the reactor hinges wider than the distance of the lid hinges and this can cause some friction. It is recommended to open the lid only momentarily when replacing a substrate. This is good not only for proper rotation of the lid, it also prevents flaking of deposited coating on the lid, and helps air from reaching the valves against the flow of the nitrogen. During venting it is also best to keep 100 sccm nitrogen flowing, to prevent air from reaching the valves. This will ensure a long valve lifetime.

Should I mount all the materials that I want to deposit with a big manifold or should I replace the precursor cylinders every time?

For systems with the blue pneumatic ALD valves, we recommend the following: 1. *If different materials are deposited within the same run:* then one needs to mount them all at the same time. 2. *If different materials are deposited within two weeks but not in one run:* best to have separate ALD systems for different films, then different materials can be run by different users at the same time. If it is not possible to purchase separate systems, then one can leave unused precursor lines mounted with manual valve to the precursor cylinder closed. 3. *If different materials are not used within two weeks:* then it is best to use only the precursor lines for the active materials (no unused lines/valves attached, smallest manifold possible), and replace the cylinders + manual valves and its associated ALD pulse valve. Make sure to close the manual valve and degas the space between manual valve and ALD valve (headspace) extensively with the temperature of the precursor cylinder and ALD valve section that is the same as would be used during deposition. To avoid any cross contamination or precursor residue in the ALD valve, degas for example for an hour with 0.1-1 second pulse time, and until no pulses are seen anymore. (Note1. Due to the internal structure of the Parker solenoid valves, it is better to remove the parker pulse valve along with the cylinder + manual valve when replacing a precursor line). Note 2. NEVER remove precursor cylinder with manual valve open, and NEVER open manual valve in air when there is precursor material in the cylinder.

What orientation should I put the precursor line manual valve?

If your are using a manual valve with a green round handle, then the manual bellows valve should be oriented with the arrow pointing in the direction of the precursor cylinder. This is important because otherwise the large area bellows is pointed towards the ALD valve, which increases the purge out time, when one wants to remove the precursor cylinder + manual valve. A special note should be made to the chemical suppliers, that they mount this valve in this proper orientation after filling the cylinder. (Please see the manual.) If you are using a manual valve with black handle then the orientation is not critical, since it is a symmetric ball valve.

Frequently Asked Questions continued

Should I put a heating jacket around a cylinder with TMA or water?

The vapor pressure of TMA and water is high enough at room temperature, so it doesn't need heating. In fact the heating jackets should not even be mounted around the TMA and water cylinder, because the TMA cylinder would get hot through the temperature of the ALD valves. Any precursors with such high vapor pressure do not need heating jackets.

What is the best plot time on the gauge pressure graph?

It is recommended to plot only several pulses, for example 30 seconds or 1 minute total plot time. Setting this value to 1 hour during a run can reduce delay precision, because with a 1 hour plot time many data points need to be refreshed, which consumes a lot of processing resources. It is possible to set the plot time to 1 hour to get an overview of pulse heights, but plot times > 5 minutes are not recommended during a run.

What valve temperature should I use?

Depending on the valves that are on the system (Parker solenoid valves or Swagelok pneumatic ALD valves) we recommend the following: The Parker valves should be between heated to 80 °C. Do not heat the Parker valves higher or keep them at a lower temperature. The Swagelok valves can be heated up to 200 °C. We recommend the Swagelok valves to be heated to 115 °C for non-heated precursors (water, TMA etc), and up to 175°C for heated precursors. Please ask Cambridge NanoTech Inc. for settings for your conditions before trying anything at random.

What is the dose that I should use for the precursors?

We recommend to ask Cambridge NanoTech Inc. for a recipe before any run since there are many parameters interconnected (substrate material, precursor, temperature, purge time etc.), that one general recipe cannot be mentioned. One general guideline is that the precursor pulse height should be about 1 Torr or less. For TMA and other high vapor pressure materials, one should not pulse for more than about 0.02 seconds, unless Cambridge NanoTech advises otherwise. This is to prevent premature contamination of the system. Also, high vapor pressure materials should not be heated and the heating jacket should be removed to prevent the cylinder from getting hot from the valve heating block above it.

Why is there nitrogen flow and what is the recommended setting?

Nitrogen (or argon) flow is used for various reasons. The first reason is to quickly purge the system between each pulse. It is important that between the precursor pulses, there is no residue (except for the monolayer chemically bonded to the substrate) of precursor in the reactor. The presence of two precursors at the same time would cause immediate reaction in vapor phase, which can lead to CVD mode deposition (non-uniformity, thick coating, powder formation). So the combination of temperature, nitrogen flow and pump time between pulses prevents two precursors from seeing each other in vapor phase. At low reactor temperatures (e.g. 100 °C), the pump time needs to be higher (e.g. 30 sec) than at high temperatures (>150 °C, pump time e.g. 8 sec, for TMA). The higher the temperature, thus the faster the cycles. Another reason for nitrogen flow is to prevent flow of precursor from one pulse valve into the other. It is important that the pulse valves only see their own precursor, thus each valve has a constant nitrogen flow. The constant nitrogen flow out of each valve prevents other precursors to enter, and thus prevents deposition in each valve. A recommended nitrogen flow value is 20 sccm. During venting this is increased to 100 sccm, and also while the reactor is open, this should be the maximum flow value (100 sccm) to prevent air from entering the valve regions. One should immediately close the lid after inserting/removing the substrate and not leave it open.

What is the expo for, should I use it?

Expo deposition means that prior to pulsing a precursor, the stop valve (valve between reactor and pump) is closed, then one precursor is injected, then the substrate gets exposed during the expo time (for example if expo = 20 seconds, the stop valve opens after 20 seconds). This mode is only used when one needs to coat very high aspect ratio structures (>1:10). The disadvantage of using expo runs is that since the precursor does not simply flow very fast from cylinder through valve through manifold over substrate into pump, instead, it sits in the reactor for expo seconds, and some of the precursor can migrate to all valves. This can cause some deposition in the valves, especially if the expo time is long. To reduce this effect, one could pulse two times in expo mode (for very high aspect ratio structures) to prevent the reactor pressure from getting too high during the expo time. If one wants to get better nucleation on hydrophobic substrates, we don't recommend expo mode deposition. Instead, one could pulse two times the same precursor in multi dosing deposition mode (expo=0 seconds), and this would only be needed for say the first few cycles. This may be especially useful for thin gate dielectrics.