



# Cambridge NanoTech Fiji F200 ALD System Operating Procedure

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## Revision History

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1	Philip Kubik	Oct. 26, 2011	Initial Release
1.1	Philip Kubik	Nov. 9, 2011	Sections 6.1, 6.2.3, & 6.2.4.
1.1.6	Philip Kubik	Mar. 15, 2012	Changes throughout.
1.2	Philip Kubik	Jan. 20, 2015	Added ALD manifold purge if manual valve closed. Revised heater temperature ranges.

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## 1 Purpose

This document describes how to operate the Cambridge NanoTech Fiji F200 Atomic Layer Deposition System in the 4D Labs Nanofabrication Facility. It is intended to be a reference for trained users of the system and is not a substitute for training by Nanofabrication Facility staff. In addition, readers of this document are expected to have a basic familiarity with atomic layer deposition (ALD).

## 2 Definitions

**ALD:** Atomic Layer Deposition

**CVD:** Chemical Vapour Deposition

**DEZ:** Diethylzinc

**LL:** Load Lock chamber

**(MeCp)PtMe<sub>3</sub>:** trimethyl(methylcyclopentadienyl)platinum(IV)

**PC:** Process Chamber (aka Main Chamber)

**PVD:** Physical Vapour Deposition

**TBTDEN:** tert(butylimino)tris(diethylamido)niobium

**TDMAHf:** tetrakis(dimethylamido)hafnium(IV)

**TDMASi:** tris(dimethylamino)silane

**TMA:** trimethylaluminum(III)

**TMP:** TurboMolecular Pump

## 3 References

- Cambridge NanoTech, *Fiji Series Product Data*. (Fiji\_Data\_Sheet.pdf)
- Cambridge NanoTech, *Fiji F200 Atomic Layer Deposition System Installation and Use Manual*, Rev. 2.2. Sept. 30, 2010. Preliminary.
- E. Deguns, *Atomic Layer Deposition ...an Introduction and Applications* (Sept. 20, 2011). (ALD\_Introduction-CNT.pdf)

- E. Deguns, *Fiji Training, Part 1* (Sept. 20, 2011). (Fiji\_Training-CNT.pdf)
- E. Deguns, *Fiji Training, Part 2* (Sept. 20, 2011). (Processes and PEALD-CNT.pdf)
- E. Deguns, *ALD Precursors* (Sept. 20, 2011). (Precursors-CNT.pdf)

## 4 Contact

Questions or comments concerning this document or operation of the ALD system should be directed to the tool owner at 4D Labs Nanofabrication Facility, Simon Fraser University, Burnaby, BC, Canada.

## 5 Overview of the ALD System

The ALD system in the 4D Labs Nanofabrication Facility is a Cambridge NanoTech Fiji F200 equipped with a heated process chamber, remote plasma source, turbomolecular pump, and automated load lock for transferring substrates. The system is primarily intended for the deposition of a variety of thin films, including metals, oxides, and nitrides. Typical film thicknesses are monolayers up to tens of nanometers. Thicker films may be deposited but, since typical deposition rates are  $\sim 3\text{--}20$  nm/h, deposition times will be several hours or more.

Some key features are as follows.

- Highly conformable, well controlled layer by layer film deposition.
- Multi-layer film stacks possible.
- Aspect ratios of up to 450:1 in thermal mode or 20:1 in plasma mode are possible.
- Substrates up to 200 mm in diameter and 6 mm thick can be accommodated.
- Substrates may be heated up to 400°C.<sup>1</sup>
- Process chamber turbomolecular pump provides a base pressure  $\sim 2 \times 10^{-5}$  Torr.
- Load lock chamber with automated transfer of substrates into the process chamber.

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<sup>1</sup> The chuck heater will go to 500°C but, currently, there are no processes developed for the Fiji F200 which require more than 400°C.

- All depositions are performed from recipes.
- Available plasma gases are as follows. Any combination of gases may be used simultaneously, with the exception that O<sub>2</sub> & H<sub>2</sub> cannot flow simultaneously.
  - Ar
  - O<sub>2</sub>
  - N<sub>2</sub>
  - H<sub>2</sub>

A wide variety of precursors may be used in the ALD system. Contact the tool owner for a current list of films which can be deposited and precursors available. If the film and precursors you require are not on the current list, discuss your needs with the tool owner well in advance of your need. New precursors must be approved and, typically, 6 weeks should be allowed for purchasing a precursor. **Prohibited** precursors include halides, sulphides, and selenides. Precursors for use in the ALD system must be contained in approved ALD cylinders.

## 6 Procedure

### 6.1 Reserving the ALD System

Prior to use, the ALD system **must** be reserved through the Nanofab Booking web site (<https://nanofabrication.4dlabs.ca>), even if you wish to use the system immediately. When you make your reservation,

- 1) On the reservation form, record the film that you wish to deposit, its thickness, the chuck temperature, and the precursors (excluding gasses) that you will require, e.g., 10 nm Al<sub>2</sub>O<sub>3</sub> at 250°C from TMA.
- 2) Check the reservation immediately preceding your reservation. If the chuck temperature in the previous reservation is higher than what you require, you may need to allocate several hours for temperature stabilization, e.g., about 4 h to cool from 250°C to 200°C and 9 h to cool from 250°C to 100°C.

3) Check the reservation immediately following your reservation. It is your responsibility to ensure that there will be sufficient time for the chuck to reach the temperature required in the following reservation. Although heating of the chuck is rapid (typically < 30 min), cooling takes a long time.

4) **Methylated metal precursors, e.g., TMA and (MeCp)PtMe<sub>3</sub>**

If you wish to use a precursor which has a metal-methyl bond, check the precursors used prior to your booking. Ensure that the process chamber **and substrate carrier surfaces** have been passivated since the last Zn compound deposition. Otherwise, your film may be contaminated. Any films in the process chamber and on the substrate carrier, which contain Zn, should be covered with at least 25 nm of a passivating film or films, not containing Zn. Passivating films should **not** be deposited from a methylated metal precursor, e.g., TMA or (MeCp)PtMe<sub>3</sub>. If passivation is insufficient, the passivation recipe in the C:\Cambridge Nanotech\Recipes\4D folder should be run with the empty substrate carrier in the process chamber. There is no charge for running a required passivation recipe. You may:

- a) run the passivation recipe yourself,
- b) ask a preceding user to run the passivation recipe after they have finished their deposition, or
- c) ask the tool owner to run the passivation recipe.

5) **Zn precursors, e.g., DEZ:**

If you wish to use a precursor containing Zn, check the precursors to be used following your booking. If any booking requires a methylated precursor, e.g., TMA or (MeCp)PtMe<sub>3</sub>, you **must** passivate the process chamber and substrate carrier surfaces after your run by running a passivation recipe, which is located in the C:\Cambridge Nanotech\Recipes\4D folder. There is no charge for running a required passivation recipe. It is your responsibility to ensure that passivation will be completed prior to the following booking.

## 6) Heated Precursors:

If you will be using a precursor which requires heating, allow at least 2 h to preheat the precursor before starting your deposition.<sup>2</sup>

Users are advised to check with tool owner 1-2 days in advance of their booking to ensure that the precursor they require will be installed on the ALD system. In addition, any concerns about process chamber passivation, chuck temperature, or precursor heating may be mentioned.

## 6.2 Depositing a Film

### 6.2.1 Preliminary Checks

Before loading your substrates, do the following.

- 1) Look at the ALD Run Log Sheet to see which films were deposited previously, in case a conditioning run is required before your deposition (cf. Section 6.1: Reserving the ALD System).
- 2) Sign in on the ALD Run Log Sheet, using a black pen.<sup>3</sup>
- 3) Check recent entries in the ALD Log Book for any relevant information.
- 4) Observe the heater temperatures.
  - a) Are they all within the ranges specified in Appendix 1? If not, contact the tool owner.
  - b) If you require different temperatures for your process, bear in mind that you will need to allow additional time for temperature stabilization, especially if you require a lower chuck temperature.
  - c) If any precursor you wish to use requires heating, it should be preheated for at least 2 h prior to commencing deposition.

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<sup>2</sup> Keeping a precursor at an elevated temperature will reduce its storage life. Consequently, after a deposition with a heated precursor, the precursor heater should be turned off unless the precursor will be used again within the next 2 days.

<sup>3</sup> Normally, it is unnecessary to sign into the *ALD Log Book*; however, you should make an entry into the log book if you encounter errors, unexpected behaviour, the system malfunctions, or you wish to make comments for the benefit of the tool owner, yourself, or other users. If there is an error, please record a brief description of the error and the time at which it occurred. You are also encouraged to capture a screen shot (*Fn-Print Screen*) and paste it into a *Paint* file.

- 5) Check that the precursors that you require are installed in the Gas Box and correctly labelled on the *System* screen.<sup>4</sup> If not, contact the tool owner.
- 6) Determine whether the manually operated valve directly on top of the precursor cylinder that you wish to use is open or closed.
  - a) If the valve has a lever handle, its state may be observed.
  - b) If the valve has circular handle, very gently attempt to **close** the valve slightly by rotating the handle **clockwise**.
    - i) If it rotates easily, the valve is open. Rotate back to the initial orientation.
    - ii) If the valve does not rotate easily, do not force it; it is already closed.

### CAUTION

Do not open a closed precursor valve without first purging the dead space. Doing so may irrevocably contaminate an expensive precursor.

- 7) If the manual precursor valve is open, proceed to the next section. If is closed, continue with the remainder of this section.
- 8) On the Vacuum System screen, check that the capacitance manometer *Isolation Valve* is closed.
- 9) On the Process screen, ensure that the Ar gas idle flows are set, i.e.,
  - a) *MFC0 Ar Carrier = 10 sccm*
  - b) *MFC1 Ar Plasma = 30 sccm*
- 10) Turn on all 3 purges.
  - a) *Door Purge*
  - b) *Ellipsometer Purge*
  - c) *Main Turbo Purge*
- 11) Load the pulse recipe for ALD valve #n.

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<sup>4</sup> Note that, when looking into the Gas Box from the left side of the ALD system, precursor cylinders are labelled 0 – 5 from left to right.

- 12) Compare the pulse duration in the recipe to the value appropriate for the current precursor (see Appendix 2: Precursor Pulse Durations). If they are different, change the value in the recipe to the value for the current precursor.
- 13) Press the *Run* button on the left panel.
- 14) At the prompt, confirm that you wish to run the recipe. The pulse recipe should begin to run.
- 15) On the Pressure vs. time graph at the bottom of the Process screen, observe the pressure spikes that result from opening the ALD valve. Initially, there should be a large spike but subsequent spikes should diminish rapidly.
- 16) If, at the end of the pulse run, pressure spikes are no longer observed, proceed to Step 17). Otherwise, if this is the first pulse run, return to Step 13). If pressure spikes persist at the end of the second pulse run, there is probably a leak. **Do not proceed**; instead, contact the tool owner.
- 17) Load the purge headspace recipe for ALD valve #n.
- 18) Press the *Run* button on the left panel.
- 19) At the prompt, confirm that you wish to run the purge headspace recipe. The recipe should begin to run.
- 20) On the pressure vs. time graph at the bottom of the Process screen, no pressure spikes should be observed when the ALD valve opens. If pressure spikes are observed, there is probably a leak. **Do not proceed**; instead, contact the tool owner.
- 21) Open the manual valve on precursor cylinder #n almost fully (1 turn).<sup>5</sup>
- 22) Press the *Run* button on the left panel.
- 23) At the prompt, confirm that you wish to run the purge headspace recipe. The recipe should begin to run.
- 24) On the Pressure vs. time graph, pressure spikes should be observed when the ALD valve opens, unless the precursor requires heating. The amplitudes will depend on the precursor but should stabilize after a few pulses. If the precursor requires heating during deposition, the spikes may be small or non-existent.

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<sup>5</sup> The valve is fully open when its resistance to turning decreases. If the valve is not fully opened, deposits may form on the valve stem, preventing it from closing properly in the future.

### 6.2.2 Substrate Requirements

- Substrates up to 200 mm in diameter and 6 mm thick may be loaded. Multiple smaller substrates may be loaded for the same run.
- Ensure that your substrate is compatible with the process conditions in your recipe.
- Substrates need not be clamped to the substrate carrier; however, you may wish to place dummy substrates around your substrate(s) to keep them in place. Otherwise, substrates may shift in position slightly during pumpdown.

### 6.2.3 Substrate Loading

#### WARNING

The interior of the load lock and the substrate carrier may be hot.  
Proceed cautiously when loading or unloading substrates.

Load your substrates as follows.

- 1) Observe whether the substrate carrier is in the load lock. If not, it is probably still in the process chamber. In that case, contact the previous user or the tool owner before proceeding.
- 2) If the substrate carrier is in the load lock and has substrates on it, contact the previous user or the tool owner before proceeding. The substrates may be cooling and exposing them to atmospheric pressure while hot may damage the film.
- 3) If the load lock is **not** at atmospheric pressure, vent it as follows.
  - a) Open the *Vacuum System* screen.
  - b) Press LL Vent.
  - c) Wait 2-3 minutes for the LL to vent. During this interval load lock pressure should rise to 760 Torr and a dialogue box should open requesting acknowledgement that the LL is at atmospheric pressure. Do **not** acknowledge yet.
  - d) Gently, try to lift the LL door. If it will not lift easily, wait another minute and try again. Repeat until it lifts easily.

- e) Once the LL door can be opened easily, press OK in the dialogue box to acknowledge that the LL is at atmospheric pressure.
- 4) Fully open the LL door.
- 5) Approach the substrate carrier carefully in case it is hot. If it is, allow it to cool to near room temperature.
- 6) Place your substrate(s) onto the substrate carrier. If desired, place additional dummy chips on the carrier to prevent your substrates from shifting position during pumpdown.
- 7) Close the LL door.
- 8) On the *Vacuum System* screen, press Transfer Sample. The substrate carrier should be automatically transferred onto the substrate chuck in the process chamber. Then, a dialogue box should open requesting that you acknowledge that the gate valve is clear. Do **not** acknowledge yet.
- 9) Check that the transfer arm has fully retracted into the load lock and that the LL-PC gate valve opening is clear. If so, acknowledge by clicking Gate Clear in the dialogue box.

### 6.2.4 *Running a Recipe*

This section describes how to make minor modifications to and run a deposition recipe. For information about writing recipes, refer to Section 6.3.

#### 6.2.4.1 *Loading, Checking, & Modifying a Recipe*

Film deposition in the ALD system is accomplished exclusively by running recipes; manual processing is normally impractical. Users have the option of running a standard 4D Labs recipe or their own recipe. 4D Labs recipes are located in the C:\\Cambridge Nanotech\\Recipes\\4D folder. Create your own subfolder within the C:\\Cambridge Nanotech\\Recipes folder to store your custom recipes.

You may make temporary changes to a 4D recipe but do **not** save any changes to a 4D recipe file. If you wish to make permanent changes, save the recipe to your own folder with a different filename, e.g., with your initials in place of "4D". Normally, it is inadvisable to change the duration of a pulse command; consult with the tool owner before doing so.

To run a 4D recipe do the following. The procedure for running your own recipe will be similar.

- 1) Right click on the recipe area of the *Process* screen and select Load recipe.
- 2) In the navigation window, open the 4D folder, select the desired recipe, and click the Open button.
- 3) Check whether the ALD valve number in the #cell of each *pulse* command corresponds to the actual ALD valve number of the precursor you desire by looking inside the gas box and at the *System* screen.<sup>6</sup> If not, select the #cell and type in the correct ALD valve number.
- 4) Change the film thickness to the desired value by changing the number of cycles in the *Value* column of the *goto* command(s) in the recipe.
- 5) Do **not** save the changes unless you save to a different filename in your own recipe folder.

#### 6.2.4.2 Running a Recipe

Before running a deposition recipe, do the following.

- 1) Confirm that your substrate is loaded into the process chamber.
- 2) Turn on all 3 purges (on the *Vacuum System* screen).
  - a) Door Purge
  - b) Ellips Port Purge
  - c) Main Turbo Purge
- 3) On the *Vacuum System* screen, check that the capacitance manometer Isolation Valve is closed. **The Isolation Valve must be closed during deposition.**<sup>7</sup>

To run your recipe, do the following.

- 1) Press the Run button on the left panel.
- 2) At the prompt, confirm that you wish to run the recipe. The recipe should begin to run. If it does not, look at the message in the *Status* bar at the top of the Fiji window; it may inform you of the reason.
- 3) Set the P vs. t graph time scale so that it will display a few deposition cycles.

<sup>6</sup> There are more precursors available than ALD valves so precursors may migrate to different positions from time to time.

<sup>7</sup> If the capacitance manometer Isolation Valve were left open during deposition, the (expensive) manometer would become coated with the film and be damaged. If the Isolation Valve were left open when a recipe is run, an interlock will close it; however, it is not good practice to rely on this feature.

- 4) Scroll through the recipe to display any important parameters that you may have changed, e.g., the number of cycles. At the end of the recipe, a screen shot will automatically be captured and saved to the C:\Cambridge Nanotech\Logfiles\Reports folder, providing you with a record of recipe parameters which may not have been saved.

It is good practice to observe the first few cycles of your deposition to ensure the following.

- Temperatures are correct.
- Background pressure is correct, typically 0.1–0.2 Torr.<sup>8</sup>
- Precursor pulses have reasonable amplitudes, typically in the range 5 – 20 mTorr.
- Plasma gases are flowing (if required).
- The plasma turns on for the desired time.

If possible, check the above from time to time during the run. In addition, you could check the corresponding log file after the run has completed.

Running other applications during a recipe is not recommended because communication between the Fiji software and hardware may be interrupted, resulting in a communication error and abortion of the recipe.

#### 6.2.4.3 Cleanup

After your run is completed, do the following.

- 1) Turn off all 3 purges (on the *Vacuum System* screen).
  - a) Door Purge
  - b) Ellips Port Purge
  - c) Main Turbo Purge
- 2) Ensure the following.

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<sup>8</sup> This is the true pressure, e.g., as measured by the capacitance manometer. The Pirani gauge pressure is valid for nitrogen but not necessarily for other gasses. In particular, for argon the Pirani gauge pressure will be somewhat lower than the true pressure. Since the Isolation Valve must be closed while running a recipe, your process pressure must be calibrated when no recipe is running. See Section 6.3.2.4.

- a) MFC0 Ar Carrier = 10 sccm
  - b) MFC1 Ar Plasma = 30 sccm
  - c) All other gas flows are set to 0 and the MFC valves are closed
  - d) Plasma power is *OFF*
  - e) Turbo Speed is  $\cong 48,000$  rpm
  - f) Heater temperatures are correct (cf. Appendix 1)
  - g) Process chamber pressure is in the range  $2 - 10 \times 10^{-3}$  Torr<sup>9</sup>
  - h) APC valve = 100%
- 3) Turn off any precursor heaters, unless the heated precursors will be used again within the next 2 days.
  - 4) Complete your entry on the *ALD Run Log Sheet*, including final process chamber pressure. Final process chamber pressure should be measured on the process chamber Pirani gauge under the conditions listed in items 1) and 2) above.<sup>10</sup>
  - 5) If the final process chamber pressure exceeds  $1 \times 10^{-2}$  Torr, inform the tool owner at your earliest convenience.
  - 6) If requested by the next user, set the process chamber or precursor heater temperatures as needed.

### 6.2.5 Substrate Unloading

Substrates should be unloaded as follows.

- 1) Press Transfer Sample on the *Vacuum System* screen. The substrate carrier should be automatically transferred to the Load Lock.
- 2) Reset the argon idle flows as follows.
  - a) MFC0 Ar Carrier = 10 sccm
  - b) MFC1 Ar Plasma = 30 sccm
- 3) Turn off all 3 purges (on the *Vacuum System* screen).
  - a) Door Purge
  - b) Ellips Port Purge
  - c) Main Turbo Purge

<sup>9</sup> For a recently calibrated pressure gauge, the main chamber pressure with the idle flows on should be  $3 \times 10^{-3}$  Torr. As the gauge tube becomes coated with films, the indicated pressure will rise.

<sup>10</sup> Normally, it is unnecessary to sign into the *ALD Log Book*; however, you should make an entry into the log book if you encounter errors, unexpected behaviour, the system malfunctions, or you wish to make comments for the benefit of the tool owner, yourself, or other users. If there is an error, please record a brief description of the error and the time at which it occurred. You are also encouraged to capture a screen shot (*Fn-Print Screen*) and paste it into a *Paint* file.

- 4) Wait for your substrate to cool to near room temperature, e.g., for substrates which were heated to 250°C, allow at least 1 h.
- 5) When the substrate carrier has cooled sufficiently, go to the *Vacuum System* screen and press LL Vent.
- 6) Wait 2-3 minutes for the LL to vent. During this interval, load lock pressure should rise to 760 Torr and a dialogue box should open requesting acknowledgement that the LL is at atmospheric pressure. Do **not** acknowledge yet.
- 7) Gently try to lift the LL door. If it will not lift easily, wait another minute and try again. Repeat until it lifts easily.
- 8) Once the LL door can be opened easily, press OK in the dialogue box to acknowledge that the LL is at atmospheric pressure.
- 9) Fully open the LL door.
- 10) Approach the substrate carrier carefully in case it is hot. If it is, allow it to cool.
- 11) Remove your substrate(s) from the substrate carrier.
- 12) Close the LL door.<sup>11</sup>
- 13) If requested by the next user, run the passivation recipe in the C:\\Cambridge Nanotech\\Recipes\\4D folder.

Leave the Fiji software running.

### 6.2.6 Data Logs

Information about your run, which may be useful to you, is recorded in various data logs. Data logs are tab delimited text files. Specifically, the following log files are available in the C:\\Cambridge Nanotech\\Logfiles folder.

- Event Files: Continuously logs events such as run start & end times, MFC resets, errors. Recipe names are not logged.
- Heater Data: Logs heater temperatures during runs.
- Pressure Data: Logs process chamber pressure during runs.
- RF Data: Logs forward & reflected RF power supplied to the plasma during runs.

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<sup>11</sup> The load lock may be left at atmospheric pressure.

Graphs of the logged parameters versus time may be viewed from within the Fiji software on the *Advanced* screen as follows.

- 1) Click on the open folder icon next to the Path window.
- 2) In the Path window, browse to the appropriate log file.
- 3) Click Graph.
- 4) Adjust the scales as required by using the icons under the graph.

Alternatively, log files may be downloaded to another computer and viewed by opening with a spreadsheet programme such as Microsoft Excel.

## 6.3 Recipes

### 6.3.1 *Introduction*

The purpose of this section on deposition recipes is to outline good practices and 4D Labs conventions for ALD recipe writing. For the mechanics of writing recipes, refer to Section 4 of the Fiji operator's manual. A quick reference to recipe commands and their associated parameters can be found in Section 6.3.3: Recipe Quick Reference.

Recipe files are located in the C:\Cambridge Nanotech\Recipes folder. Each user should create his own subfolder identified by his name. In addition, each user should identify his recipes by including his name or initials in the filename. Users are responsible for backing up their recipes.

Users are welcome to use and even modify the standard recipes in the C:\Cambridge Nanotech\Recipes\4D folder but if you wish to save any changes, the recipe must be saved to the user's own folder and the filename changed, e.g., by replacing "4D" in the filename with the user's name or initials.

## 6.3.2 Recipe Writing

### 6.3.2.1 General Guidelines

- In general, you are less likely to have errors in your recipe if you modify an existing recipe, instead of writing a new recipe from scratch.
- Two or more recipes may be combined most easily by using a text editor or a spreadsheet application.
- Remember that if you insert lines above or within a *goto* loop, you must change the value in the #column of the *goto* command.
- For safety reasons, O<sub>2</sub> & H<sub>2</sub> cannot be run simultaneously. Consequently, it is good practice to close H<sub>2</sub> valve before opening the O<sub>2</sub> valve and vice versa.
- At the end of your recipe,
  - reset the Ar flows to the standard idle flows,
  - turn off all other gasses used in the recipe and set their flows to zero,
  - reset the APC to 100%, and
  - turn off precursor heaters,
- If requested by the next user and compatible with your substrates and films, you may wish to reset process chamber and precursor temperatures at the end of your recipe. In general, for process chamber temperatures, this should only be done if the next user requires lower temperatures.

### 6.3.2.2 Temperature

ALD processes occur in specific temperature windows so considerable caution must be exercised before changing process chamber temperatures<sup>12</sup> in an established recipe; doing so may have unexpected and undesirable ramifications. Proposed process chamber temperature changes should be discussed with the tool owner.

The following heaters must **always** be set to 150°C.

- Precursor Delivery Line
- ALD Valves

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<sup>12</sup> Reactor 1, Reactor 2, Chuck, & Cone.

- APC Valve.

Since ALD is a chemical process, changing process chamber temperatures affects not only the film morphology, as in PVD, but also the deposition rate and even the nature of the chemical reactions that occur. Outside the ALD window, undesirable processes such as CVD and precursor decomposition, condensation, and desorption occur. Such reactions may result in poor film quality and contamination of delivery lines. In addition, temperature cycling of the process chamber promotes flaking of deposits on the process chamber walls.

Even if the process chamber temperature remains within the ALD window, one must bear in mind that as the process chamber temperatures are reduced,

- Purge times must be increased or undesirable CVD processes may occur, e.g., see Table 6-1.
- Plasma exposure times must be increased or reactions will be incomplete, e.g., see Table 6-2.

Table 6-1: Purge times and deposition rates as functions of process chamber temperature for ZnO thermal deposition from DEZ and H<sub>2</sub>O.<sup>13</sup>

Chuck and Trap heaters (°C)	Reactor 1 & 2 heaters (°C)	DEZ purge time (s)	H <sub>2</sub> O purge time (s)	Dep. Rate (nm/cycle)
100	100	45	60	~0.15
150	150	30	40	~0.15
200	200	10	20	~0.135
250	250	5	10	~0.12
300	250	5	5	~0.12

Table 6-2: O<sub>2</sub> plasma exposure time as a function of process chamber temperature for deposition of Pt from (MeCp)PtMe<sub>3</sub> and O<sub>2</sub> plasma.

Chuck Temperature (°C)	O <sub>2</sub> plasma exposure time (s)
90	60
150	30
200	20
270	10

<sup>13</sup> Cambridge NanoTech Corp., *Zinc Oxide Fiji Thermal ALD Recipe*, Report R08-12-2011.

### 6.3.2.3 Precursor Pulses

Consult with the tool owner before changing the duration of a precursor pulse in a recipe. In all 4D recipes, the precursor pulse duration has been optimized. Shortening the pulse will reduce your deposition rate. Lengthening the pulse will not increase your deposition rate significantly, instead it will simply waste the precursor, all of which (apart from H<sub>2</sub>O) are expensive.

### 6.3.2.4 Process Chamber Pressure

During deposition, the process chamber background pressure<sup>14</sup> should be ~ 0.1–0.2 Torr (recipe dependent). **Before running your new recipe**, you should check that the Ar and reactive gas flows and APC valve setting produce the appropriate pressure by using the following manual procedure.

- 1) Turn on all 3 purge gas flows.
- 2) Set the APC valve opening, typically 12%-15%.
- 3) Open the capacitance manometer Isolation Valve.
- 4) Set the desired Ar flows (carrier & plasma).
- 5) Open the MFC valves for the reactive gases you wish to flow.
- 6) Set the desired reactive gas flows.
- 7) Adjust the gas flows and APC valve to get the desired process chamber background pressure.
- 8) Observe the TMP speed for a few minutes to ensure that it does not drop below 48,000 rpm. If it does drop, you must reduce the gas flows until it remains above 48,000 rpm. If the resulting pressure is too close, close the APC valve more.
- 9) The resulting Ar gas flows and the APC setting can be used in your recipe.
- 10) Reset the Ar gas flows to the idle settings.
- 11) Turn off the reactive gas flows and close the MFC valves.
- 12) Open the APC valve to 100%.

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<sup>14</sup> Background pressure is the pressure produced by the Ar carrier and plasma gases in the absence of precursor pulses. Note that the Pirani gauge only measures the true pressure for certain gasses, such as nitrogen. In particular, for argon it indicates a pressure lower than the true pressure. The capacitance manometer measures the true pressure, independent of the gas type; however, it cannot be used during deposition.

- 13) Turn off all 3 purge gas flows.
- 14) Close the capacitance manometer Isolation Valve.

**Before running your plasma mode recipe**, you should check that Ar & reactive gas flows and the APC valve setting allow a plasma to be struck by using the following manual procedure.

- 1) Turn on all 3 purge gas flows.
- 2) Set the APC valve opening, typically 12%-15%.
- 3) Open the capacitance manometer Isolation Valve.
- 4) Set the desired Ar flows (carrier & plasma).
- 5) Open the MFC valves for the reactive gases you wish to flow.
- 6) Set the desired reactive gas flows.
- 7) Type 300 into the Plasma Power (W) box at the top right of the *Process* screen.
- 8) Press the red Plasma OFF button to turn the plasma on. The plasma should strike. If not, adjust the gas flows and APC valve as needed, bearing in mind that plasmas strike more easily in Ar than in reactive gases so you may need to increase the fraction of Ar.
- 9) Once the plasma has ignited, watch it for a minute or two to ensure that it is stable.
- 10) Observe the TMP speed for a few minutes to ensure that it does not drop below 48,000 rpm. If it does drop, you must reduce the gas flows until the TMP speed remains above 48,000 rpm. If the resulting pressure is too close, close the APC valve more.
- 11) The resulting Ar gas flows and the APC setting can be used in your recipe.
- 12) Set all reactive gas flows to zero.
- 13) Close the MFC valves for all reactive gases.
- 14) Reset the Ar gas flows to the idle settings.
- 15) Open the APC valve to 100%.
- 16) Turn off all 3 purge gas flows.
- 17) Close the capacitance manometer Isolation Valve.



### 6.3.3 Recipe Quick Reference

A list of recipe instructions and valid parameters may be found in Table 6-3 . For details regarding recipe writing, refer to Section 4 of the Fiji operator's manual.<sup>15</sup>

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<sup>15</sup> Cambridge NanoTech, *Fiji F200 Atomic Layer Deposition System Installation and Use Manual*, Rev. 2.2. Sept. 30, 2010.

Table 6-3: Recipe Instructions and allowed parameters for the # & Value columns.

Instruction	#	Value	Units	Component
goto	Any line no.	Any integer		Recipe
wait		> 0.00 in 0.05 s increments	s	Recipe
stopvalve <sup>1</sup>		0, 1		Stop Valve
bypassvalve <sup>2</sup>		0, 1		Bypass Valve
roughpurge <sup>3</sup>		0, 1		Not in use - RoughPurge Valve
plasma		Any value in range 0 – 300	W	Plasma Power
APC		Any value in range 0 - 100	%	APC percentage open
stabilize <sup>4</sup>	12 – 23 <sup>5</sup>			Specified heater
heater	12	0 - 325	°C	Cone / Trap
	13	0 – 300	°C	Reactor 1
	14	0 - 300	°C	Reactor 2
	15	0 - 400	°C	Chuck
	16	150	°C	Precursor Delivery Line
	17	150	°C	ALD Valves
	18	0 – 200	°C	Precursor Jacket 0
	19	<b>WARNING</b>		Precursor Jacket 1
	20	Not to exceed safety or decomposition temperature of the specific precursors in use.		Precursor Jacket 2
	21			Precursor Jacket 3
22		°C	Precursor Jacket 4	
23	150	°C	APC Valve	
pulse	0 – 5 <sup>6</sup>	0.015 s increments up to 30 s	s	ALD Valve
MFCvalve <sup>7</sup>	0	0, 1		Argon Carrier
	1	0, 1		Argon Plasma
	2	0, 1		N <sub>2</sub> Plasma Gas
	3	0, 1		O <sub>2</sub> Plasma Gas
	4	0, 1		H <sub>2</sub> Plasma Gas
	5	N/A		Not in use - NH <sub>3</sub> gas
	6	N/A		Not in use - CF <sub>4</sub> gas
7	N/A		Not in use - Ozone	
flow	0	0 - 200	sccm	Argon Carrier
	1	0 – 1000	sccm	Argon Plasma
	2	0 - 200	sccm	N <sub>2</sub> Plasma Gas
	3	0 - 200	sccm	O <sub>2</sub> Plasma Gas
	4	0 - 200	sccm	H <sub>2</sub> Plasma Gas
	5	0 - 200	sccm	Not in use - NH <sub>3</sub> gas
	6	0 - 200	sccm	Not in use - CF <sub>4</sub> gas
7	0 – 1000	sccm	Not in use - Ozone	
line ac out <sup>7</sup>	1	0, 1		Not in use - Ozone Generator
	2	0, 1		Not in use - Rough Pump Purge
	3	0, 1		Not in use - Ellipsometer Purge

1) Valve between back of turbomolecular pump and roughing pump. 0 = Closed, 1 = Open.

2) Valve between process chamber and roughing pump. 0 = Closed, 1 = Open.



- 3) Not applicable. No Rough Purge Valve on this system.
- 4) Stabilize causes the recipe to pause until the heater # entered in the 'NUMBER' cell is within  $\pm 1^\circ\text{C}$  of the setpoint.
- 5) Heater reference ID.
- 6) Any ALD Valve reference ID.
- 7) 0 = Closed, 1 = Open.

## 7 Appendix 1: Heaters

A list of the heaters in the ALD system is shown in Table 7-1, along with the allowed temperature ranges and the standard temperatures. Note the following constraints.

- The following heaters must always be kept at 150°C.
  - Precursor Delivery Line
  - ALD Valves
  - APC Valve.
- Reactor 1 and Reactor 2 should always be set to the same temperature.
- At temperatures of 250°C and below, the following heaters should normally be at the same temperature. Above 250°C, this rule is superseded by the temperature limits of the individual heaters.
  - Reactor 1 and Reactor 2. **Do not exceed 300°C.**
  - Chuck. **Do not exceed 400°C.**<sup>16</sup>
  - Cone. **Do not exceed 325°C.**

Table 7-1: Heater locations, temperature ranges, and standard temperatures.

Heater No. <sup>1</sup>	Heater Name	Temperature Range (°C)	Standard Temperature (°C)
12	Cone / Trap	0 - 325	250
13	Reactor 1	0 – 300	250
14	Reactor 2	0 - 300	250
15	Chuck	0 - 400	250
16	Precursor Delivery Line	150	150
17	ALD Valves	150	150
18 <sup>2</sup>	Precursor Jacket 0	0 – 200	0
19	Precursor Jacket 1	<b>WARNING</b> Not to exceed safety or decomposition temperature of the specific precursors in use.	Depends on precursor installed (see recipe).
20	Precursor Jacket 2		
21	Precursor Jacket 3		
22	Precursor Jacket 4		
23	APC Valve	150	150

1) These are the heater numbers which appear on the *Process* screen. To get the numbers on the *Advanced* screen, subtract 12 from these values.

2) For H<sub>2</sub>O. A temperature setpoint equal to zero, means that the heater will be off so the precursor will be at room temperature.

<sup>16</sup> The chuck heater will go to 500°C but, currently, there are no processes developed for the Fiji F200 which require more than 400°C.

## 8 Appendix 2: Precursor Pulse Durations

Typically, the pulse duration for a specific precursor in pulse recipes is the same as that in deposition recipes. In contrast, the pulse duration in headspace purge recipes is 1 s for all precursors. Pulse durations for various precursor pulse recipes are listed in Table 8-1.

Table 8-1: Precursor pulse durations for pulse recipes.

Precursor				Pulse Duration
Name	CAS No.	Formula	Acronym	(s)
diethyl zinc, zinc ethane	557-20-0	ZnEt <sub>2</sub>	DEZ	0.06
tert(butylimino)tris(diethylamido)niobium	210363-27-2	(tBuN=)Nb(NEt <sub>2</sub> ) <sub>3</sub>	TBTDEN	1
tetrakis(dimethylamido)hafnium(IV)	19962-11-9	Hf(NMe <sub>2</sub> ) <sub>4</sub>	TDMAHf	0.25
tetrakis(dimethylamido)titanium	3275-24-9	Ti(NMe <sub>2</sub> ) <sub>4</sub>	TDMAT	0.1
tetrakis(dimethylamido)zirconium	19756-04-8	Zr(NMe <sub>2</sub> ) <sub>4</sub>	TDMAZ	0.25
titanium isopropoxide	546-68-9	Ti(OiPr) <sub>4</sub>		0.25
trimethyl(methylcyclopentadienyl)platinum(IV)	94442-22-5	(MeCp)PtMe <sub>3</sub>		1
trimethylaluminum	75-24-1	AlMe <sub>3</sub>	TMA	0.06
tris(dimethylamido)aluminum(III)	32093-39-3	Al <sub>2</sub> (NMe <sub>2</sub> ) <sub>6</sub>	TDMAA	0.06
tris(dimethylamino)silane	15112-89-7	HSi(NMe <sub>2</sub> ) <sub>3</sub>	TDMA Si	0.1
water		H <sub>2</sub> O		0.06