

Standard Operating Procedure: ALD

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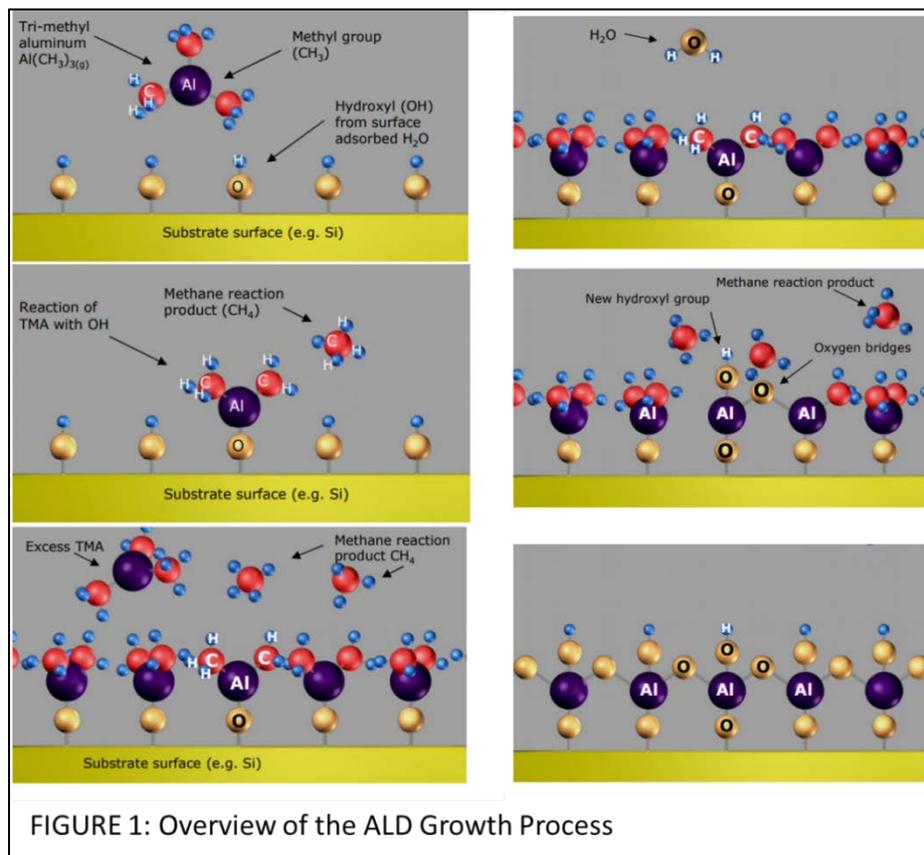
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Hardware Description and Principle of Operation

Ultratech Fiji G2 ALD

Atomic Layer Deposition (ALD) is a technique that allows growth of thin films, atomic layer by layer. The typical ALD reaction is illustrated via the formation of aluminum oxide from trimethylaluminum or TMA, $\text{Al}(\text{CH}_3)_3$, and water. The precursor reacts with hydroxyl groups on the surface of the substrate, liberating methane. The reaction is self-limiting as the precursor does not react with adsorbed aluminum species. Unreacted precursor and the methane (CH_4) liberated from the reaction are



removed by simple evacuation of the sample chamber or by flowing inert gas over the surface. Water reacts with the methyl groups on the deposited aluminum atoms forming both Al-O-Al bridges, as well as new hydroxyl groups. The formation of hydroxyl groups readies the surface for the acceptance of the next layer of aluminum atoms. Methane is liberated as a by-product. The unreacted byproducts are then pumped out of the chamber. Finally, the process begins again with the introduction of precursor A followed by B. Atomic layers are built up one after the other, and shown in Figure 1.

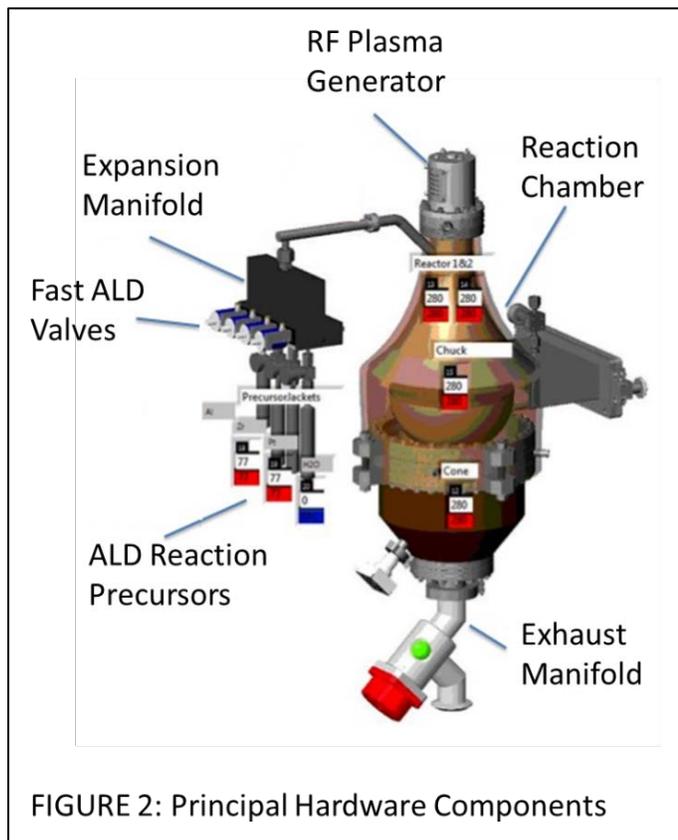
The ALD system consists of a heated reaction chamber, to which the reaction precursors are plumbed using fast ALD valves. As shown in figure 2 below. The system accommodates two growth modes: thermal and plasma. In the thermal growth mode, water is used as a source of oxygen, as described above. In the plasma mode, an RF generator positioned at the top of the

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chamber is used to dissociate oxygen (or nitrogen) gas and supply radicals for the formation of the dielectric film.

Material Requirements

The ALD growth process involves the use of volatile chemical reactants. Therefore the materials on which ALD growth takes place should be inert. Typically silicon, glass, or metal substrates are ideal. Many ALD precursors are known to react with organic materials, such as photo or electron beam resists. Furthermore, most ALD processes are calibrated in the 100 – 300 C temperature range. The sample should be able to withstand that heat for a prolonged period of time. Please consult staff (and literature) prior to growing on uncommon substrates.



Procedure

Estimated Time: Typical processes go from 1 to 4 hours. Screen will display process time when recipe is loaded.

Loading and unloading samples into the Fiji system can initially appear tricky if you are not familiar with all the hardware and all of its functions. The instructions for loading the sample (described below) assume that when you come to the tool both the load lock and the process chamber are under vacuum. If that is not the case, some of the steps described below might have to be carried out in a different order. Only users who are very familiar with the hardware are allowed to deviate from this procedure, other users should contact staff for help. Figure 3 shows the software screen that you should encounter when coming to the tool. Note that both the load lock and process chamber are under vacuum, the Argon flow rate for MFC-0 is set to 5 sccm, the Argon flow rate for MFC 1 is set to 10 sccm, and heaters 13, 14, 15 are set to 150 C. At this point

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you are ready to vent the load lock, load the sample, and run a recipe. After you are done with the growth it is imperative that you return the tool to the state described above.

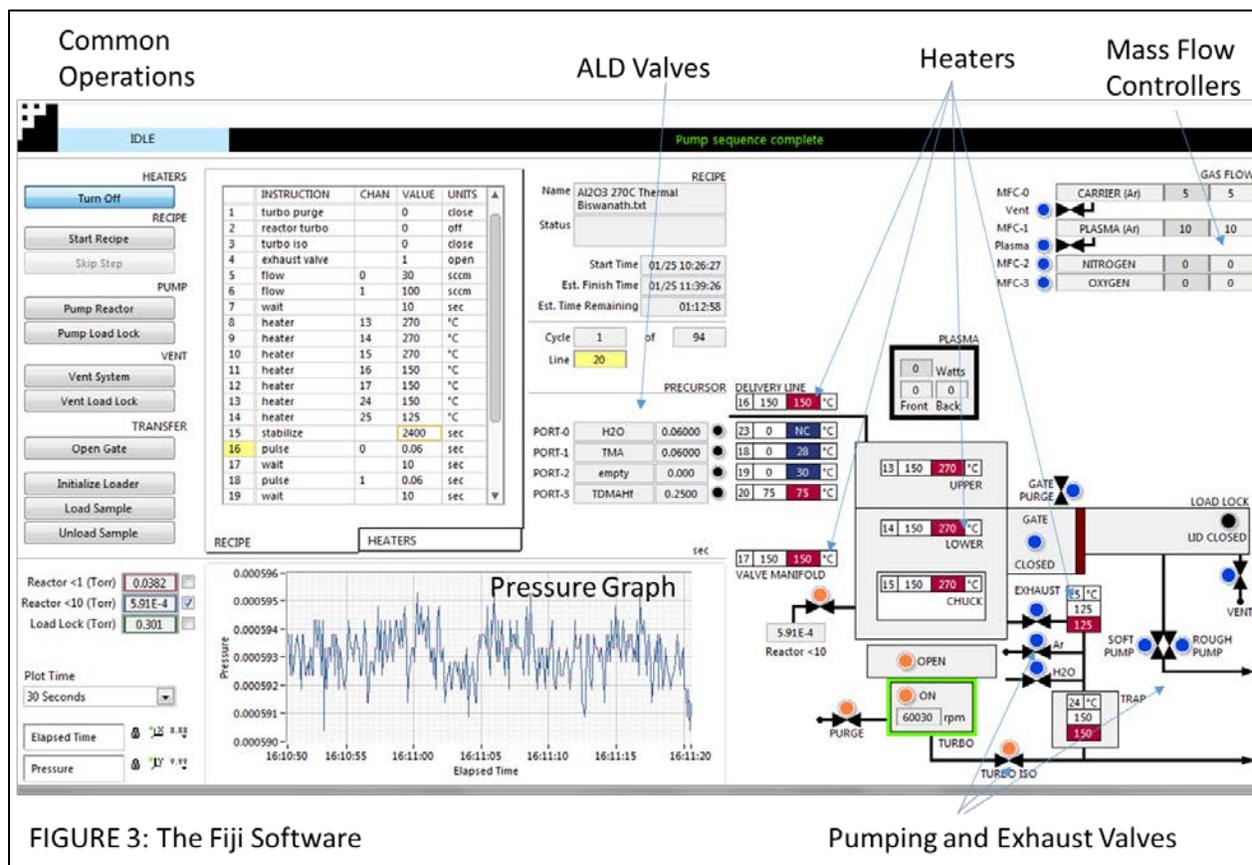


FIGURE 3: The Fiji Software

Pumping and Exhaust Valves

Load Sample

In order to load the sample please follow the following steps (also see Figure 4 for a graphical reference):

1. Verify that both the load lock and the reactor are under vacuum.
2. Vent the load lock.
 - a. Load your sample onto the sample holder. When loading the sample, make sure that there is no gap between the fork that holds the sample holder and the sample holder. See figure 4 for the correct position of the sample holder and the lifting fork.

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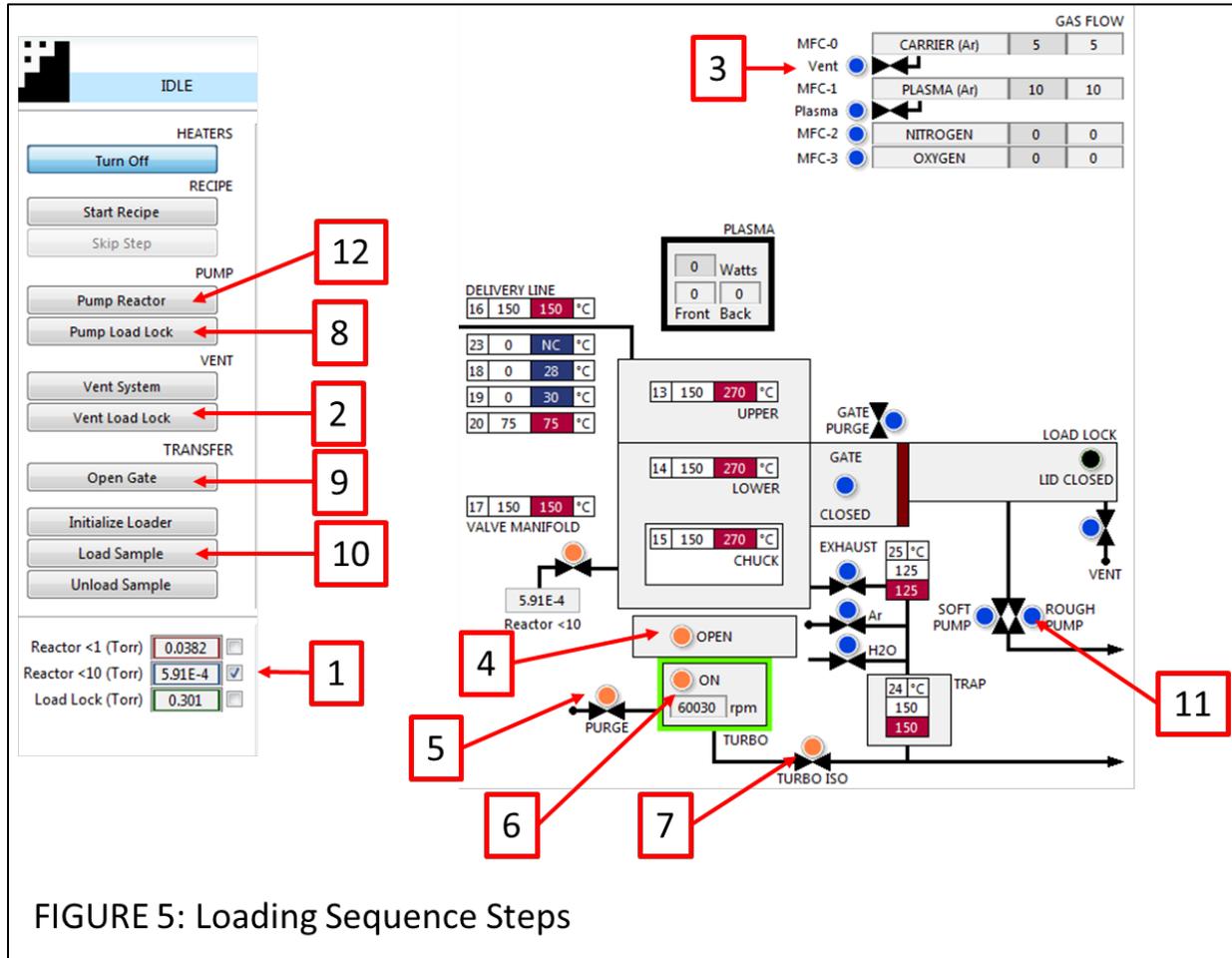
3. Turn off the Argon flow by setting the values of the MFC-0 and the MFC-1 controllers to zero.
4. Isolate the chamber by closing the isolation valve (press the orange button).
5. Turn off the turbo purge.
6. Turn off the turbo.
7. Close the turbo isolation valve.
8. Now you are ready to pump the load lock, you can do so by pressing the **Pump Load Lock** button.
9. Once the load lock has been pumped, you are ready to load the sample. To do so, open the gate valve by pressing the **Open Gate** button.
10. Now load the sample by pressing **Load Sample**. CAUTION: do not press **Unload Sample**, as that will damage the transfer arm.
 - a. Once the sample is successfully loaded, you should close the gate valve, by pressing the **Close Gate** button. It is the same button you just used to open the gate valve.
11. At this point you can pump the reactor again. To do so you must isolate the roughing pump from the load lock, by pressing the **Rough Pump** button, which should now be orange.
12. After isolating the roughing pump, press **Pump Reactor** and wait for the pumping sequence to complete.



You are now ready to run a recipe. You can do so by right clicking anywhere in the CURRENT RECIPE window (see figure 3), loading the desired recipe, and pressing the **Start Recipe** button on the left side of the screen.

NOTE: At the end of your recipe make sure to set the temperature of heaters 13, 14, and 15 back to 150 C. This will allow the tool to cool down for the next user.

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Unload Sample

When unloading the sample you must follow the above steps in reverse order. Once your recipe finishes, both the load lock and the reaction chamber should both be under vacuum. In that case you should:

1. Open the gate valve.
2. Click unload sample.
3. Wait for the sample to unload.
4. Once the sample unloads, close the gate valve.
5. Then vent the load lock and remove your sample. CAUTION: the sample holder will be hot. Be careful not to burn yourself.

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6. Once the sample is unloaded, follow the steps in the “loading the sample” section in order to first pump the load lock, and then pump the main chamber.
7. Prior to leaving the tool you **MUST** verify that:
 - a. The load lock is under vacuum.
 - b. The process chamber is under vacuum, that is, the turbo isolation valve is open, the turbo purge is on, the turbo is on, and the turbo isolation valve is open.
 - c. MFC-0 is set to 5 sccm.
 - d. MFC-1 is set to 10 sccm.
 - e. Heaters 13, 14, and 15 are set to 150 C.

Figure 3 shows a screen shot of the software when it is in the state described above.

Emergency Stop

Critical

- If the tool is smoking or a gas leak has occurred, press the EMO button if possible and leave the cleanroom.

Non-Critical

- Press **Skip Step** until you reach the end of the process.

Allowed Activities

- Load and unload samples.
- Run standard recipes.
- Modify recipes, after discussing the changes with the clean room staff.

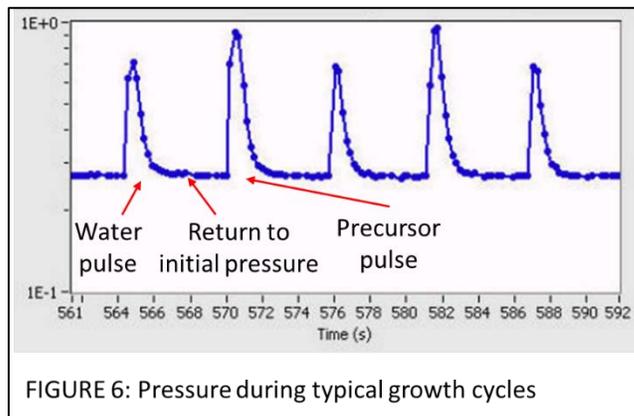
Disallowed Activities

- Changing any software / hardware configuration of the tool.
- Change the ALD precursors.

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What to watch out for during operation

Figure 6 shows a typical pressure graph for a thermal ALD process. The process begins with a water pulse, then the unreacted water is pumped out of the chamber, then a precursor pulse. If the pressure graph during your thermal process does not look like this, then something is wrong. All the water and precursor pulses should look identical, that is, the areas under those curves should be the same. You can change the “plot time” option in the ALD software to monitor the long term progress of the growth.



Common Troubleshooting Tips

- If the heaters are off, or if there are communication issues between the software and the hardware, restart the Fiji software.

Badger Criteria

Report Problem

- Pumping issues.
- Loading issues.

Shutdown

- Tool can't reach vacuum.

When to call staff?

- If either the main chamber or the load lock are not pumping down as expected.

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- If the pressure graph during your recipe does not look like the graph in Figure 6.
- If the sample holder falls off of the loading arm (or the loading fork) when you are either loading or unloading the sample.

Revision History:

1. 1/25/2018 – MB created the original document