

NorthStar™ ALD Operation Manual



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ALD System Operation

Read all of the manuals, including the Safety sections, prior to operating the system.

ALD Operation Principle

Atomic Layer Deposition (ALD) is a vapor phase technique that deposits thin films sequentially, atomic layer by layer. ALD has been described in the literature. It is based on sequential reactions of vapor phase chemicals with the surface of the substrate. The reactions are self-limiting. The surface is first saturated with one chemical precursor, and the excess pumped away. It is then exposed to a second chemical precursor, and the excess is pumped away. The technique provides excellent coverage of topographical features. Relative to other techniques, in its standard mode, the deposition rate is slow.

For more information on ALD, see review articles in the literature, such as; Atomic Layer Deposition: An Overview by Steven M. George, Chem. Rev. 2010, 110, 111–131

As an example of a thermal ALD process, Al_2O_3 can be deposited using precursors of trimethylaluminum (TMA) and water. Alternative recipes and recipes for other materials can be found in the literature.

The substrate is introduced into the deposition chamber, which is evacuated. The surface of the substrate is hydroxylated from exposure to the atmosphere.

One cycle of the deposition sequence can be described as following two half reaction cycles.

1. TMA pulse is admitted into the chamber and allowed to react with hydroxyl groups on the surface. This reaction proceeds until the surface reaction reaches completion
2. The unreacted TMA is pumped away with the help of an inert carrier gas such as Nitrogen or Argon.
3. A water pulse (H_2O) is admitted into the chamber. The H_2O reacts with the surface, removing the CH_3 groups (CH_4 (methane) is formed as a gaseous byproduct), creating Al-O-Al bridges, and passivating the surface with Al-OH once again on the surface.
4. The unreacted H_2O and gaseous byproduct CH_4 are pumped away with the help of the inert carrier gas.

These 4 steps of the cycle can be set up in a recipe as a loop. Each cycle produces up to $\sim 1.1 \text{ \AA}$ (0.11 nm) of Al_2O_3 depending on temperature. For recipe setup, see the SVT Associates **Robo Control User's Manual**.

The ALD process is distinct from Chemical Vapor Deposition (CVD) in that the reactions occur sequentially. CVD mode reactions occur when both reacting precursor chemicals arrive on the surface in the ALD chamber at the same time, and not sequentially. To avoid undesired spurious CVD reactions, it is critical that the purging steps be complete (sufficient time) and all excess precursor be pumped away before pulsing of the second precursor. Only one of the precursors should be exposed to the substrate at a time. CVD reactions in the ALD system will produce non-uniform and excessive deposits.

Depositing a film using ALD

NorthStar ALD system may be operated in either the thermal ALD or the plasma enhanced ALD methods.

There are two modes of operation in the thermal ALD method:

1. Regular mode with continuously flowing carrier gas and pulsing precursor chemicals while continuously pumping.
2. Soak mode for very high aspect ratio structures: Pulsing a precursor with the exhaust valve closed to allow sufficient exposure and saturation of all the surfaces, and then opening the valve to pump/purge. This is then repeated for the other precursor.

In thermal mode, a small internal chamber with lid is preferred.

Plasma enhanced ALD mode (PEALD) – A remote plasma source is designed to enhance the reactivity of many gas species such as N₂, O₂, H₂, NH₃, etc. and lower growth temperatures. A reactive gas, sometimes balanced with Ar in order to boost plasma efficiency is used. PEALD and thermal ALD can be switched easily by removing or putting on an internal lid.

Key growth parameters include precursor pulse time, pulse strength, and purge time for each precursor. Carrier gas and sealing gas flow rates are also important. There are parameter tradeoffs in an ALD process. Longer precursor pulse times can assure that the surface to be coated is saturated with the precursor, but precursor consumption will go up and cycle time is prolonged. Higher carrier gas flow rates help purge the excess precursor faster but may increase base pressure in the chamber thus reducing precursor pulse strength.

In the NorthStar ALD system there is an internal deposition chamber within the vacuum chamber. A flow of inert gas from the top of the chamber into the outer chamber space is used as a “sealing gas” in the thermal mode to help seal the internal lid and suppress precursor from leaking out of the internal chamber.

Preparation

Make sure the system has been properly outgassed:

- run the in-vacuum heaters at >100 °C to outgas all internal surfaces
- the precursor bubblers should be installed and any headspace between the manual shutoff valves and the ALD valves evacuated.

Check and make sure the cooling water line o-rings for the Growth Chamber and the Plasma Source are sealing. Make sure the carrier gas and gases for the Plasma source are ready.

Power up the system, pumps, and computer. Start the Robo Control software program and turn on the system electronics.

Prior to any use of chemical precursors, the Hot Trap should be on and operating. Leave the Hot Trap on during the use of precursors.

If you are working in the thermal ALD mode, the internal lid cover must be in place and properly sealed; if you are working in plasma enhanced ALD mode, remove the internal lid or insert.

Introduce the Substrate into the ALD Chamber – Vent the deposition chamber to atmosphere and open the outer vacuum chamber lid. Internal surface should be kept >100 °C to suppress moisture adsorption during exposure to atmosphere. **Caution: to avoid injury, avoid touching hot surfaces.** Place the substrate on the pedestal. Close the lid and evacuate the system.

The use of the optional Load-Lock Introduction system allows exchange of substrates while keeping the growth chamber in vacuum, minimizing exposure to moisture and other contaminants.

Establish Good Vacuum and Temperature Stability

Turn the lamp heaters on using the Robo control system. It may take 30 minutes for temperatures to stabilize.

Set up a Recipe

See *Robo Control User's Manual*, "Creating Recipes"

The following are two examples of Al₂O₃ deposition recipes in both thermal and plasma enhanced modes for your reference.

Target Materials	Mode	TMAI Pulse Time(s)	Pulse Strength (Torr)	Purge Time(s)	H ₂ O Pulse Time(s)	Purge Time(s)	Carrier Gas Flow (sccm)	Ar Top/Sealing Gas Flow (sccm)	RF-02	Outer Chamber Gauge Reading (Torr)	Internal Gauge Reading (Torr)	Growth Temp (°C)	Bubbler Temp (°C)
Al ₂ O ₃	Thermal ALD	0.02	0.04-0.1	15	0.02	15	4	2	*	1.1	0.17	120-250	RT
Al ₂ O ₃	PEALD	0.5	0.04-0.1	30	15	5	10	10	2	0.25	0.2	RT or Higher	RT

*Internal lid or insert on for thermal mode, the lid or insert removed for plasma mode

All manifolds including precursor delivery lines and exhaust line should be heated to >100 °C. The outer chamber wall temperature should also be >100 °C. These exposure conditions for Al₂O₃ coating including pulse time, pulse strength (pressure) and purge time for TMAI and O₂ plasma are experimentally determined and meet saturation conditions. You may use them as a reference. Some adjustments of the recipe parameters are necessary depending on growth temperatures and individual systems.

An example of a recipe program

Description	Time Left	TMA	SS1	SS2	H2O	CG	P1	S1	S2	RF	RFV
Set RF Forward 176.000											
Begin Loop for 440 iterations											
Shutters for 0.300 sec.		X				X	X				X
Shutters for 30.000 sec.						X	X				X
Shutters for 25.000 sec.						X	X			X	X
Shutters for 5.000 sec.						X	X				X
End of Loop						X	X				X
Set RF Forward 0.000						X	X				X

Table 1. A sample recipe for PEALD Al₂O₃ in RoboALD program format

TMAI: ALD valve for TMAI precursor source;
 SS1-SS2: ALD valves for spare precursor sources;
 CG: carrier gas on/off valve for MFC control;
 P1: Auto stop valve for exhaust and pump;
 S1-S2: spare function controls;
 RF: plasma power control;
 RFV: On/off valve for top sealing Ar gas and reactive gases for RF plasma.

See *RF Plasma source Manual* for operation of the RF source

Run the Recipe

Start the recipe as described in the Robo Control User's Manual.

Always make certain that the Hot Trap is in operation and at desired temperature before starting a recipe. Make certain that all utilities such as exhaust abatement and cooling water, etc. are in operation before starting a recipe.

Upon completion of the recipe, turn off the bubbler manual shutoff valves; set the growth parameters in the standby mode either manually or by recipe (not provided). Suggested standby conditions include setting up the substrate temperatures to >100 °C for overnight, running low carrier gas flow constantly.

Extract the Substrate

Reverse the procedure of introducing a sample.

ALD System Safety

Safety Interlocks

The system has 3 safety interlocks to handle possible emergency situations

1. **Growth chamber interlock** – If the growth chamber has any leaks or the pressure rises above a set level (say 5 Torr) in the middle of the growth process, the controller and the pneumatic throttle valve software can automatically shut down and isolate all ALD valves and MFCs so no precursor vapor or by product can leak into the growth chamber or leak outside if the system is vented.
2. **Emergency stop button** – There is a manual stop button. In case of an emergency, push the emergency stop button. All ALD valves, MFCs and the pneumatic throttle valve will shut down and the growth chamber is sealed to prevent any possible precursor vapor leaking outside.
3. **Exhaust for the console** – There is an exhaust port for the console. All precursor bubblers are located in the console. In case of precursor bubbler leakage, the exhaust can prevent the vapor from leaking outside. Make sure both front and back doors of the console are properly closed at all the times to maximize the exhaust flow.

It is strongly recommended that a gas sensor/alarm system working for a specific vapor material be installed near the ALD system. For example, if you work with chloride such SiCl_4 material, an HCl sensor should be installed inside the console or near the growth chamber.

Precursor Bubbler Handling

The source of the precursor chemicals is generically referred to as a “bubbler”. A bubbler passes a carrier gas through a liquid chemical to pick up the chemical. Alternatively, for high vapor pressure materials, it is not necessary to pass the carrier gas through the liquid, but simply have a cylinder of the material, at a controlled temperature, which feeds directly into the ALD valve, called direct vapor drawing.

Know the nature of the precursors with which you are working and read the **Material Safety Data Sheet (MSDS)**, which is available from the chemical supplier. Many ALD precursors are flammable, corrosive or toxic. For example, TMA is pyrophoric; it burns upon exposure to air. It is thus crucial to handle the precursor bubblers safely, which includes knowing how to connect and disconnect a bubbler to the ALD valve and how to store a bubbler if it is not in use.

When connecting a filled bubbler to an ALD valve, make sure to wear a proper mask or respirator, clear the ALD area of irrelevant personnel; double check to make sure the manual on/off valve on the bubbler is closed tightly before loosening the VCR fitting; Loosen the VCR sealing cap in a chemical hood with proper vent system. Ideally there should be a gas sensor around to check if there is a possible leakage from the bubbler. Immediately transfer the bubbler to the ALD console and connect to the ALD fitting, and tighten it. Make sure the line between ALD valve and the bubbler is purged, by opening the ALD valve for a short period of time before you open the manual on/off switch (assuming the system is under vacuum). If the base pressure is not recovered and is higher than the original point, there could be leakage from the bubbler connecting area.

When disconnecting a filled bubbler from an ALD valve, make sure to wear proper mask or respirator, clear the ALD area of irrelevant personnel; double check to make sure the manual on/off valve on the bubbler is closed tightly before loosening the VCR fitting; purge the line between ALD valve and the bubbler to remove any residual precursor in the line; loosen the connecting fitting and cap the bubbler immediately with a VCR cap and gasket.

When a filled precursor bubbler is taken out of use, it is the best to put it back in the original package and store in a safe, properly vented area or chemical hood area. Make sure the on/off valve is tightly sealed and the VCR cap is tightened.

Heating up a Bubbler

Depending on precursor volatility, a bubbler sometime has to be heated to certain temperatures to get sufficient vapor pressure (~1 Torr), for example Hf precursors of TDMAH, TEMAH and TDEAH. Please pay attention to the key properties such as working temperature, decomposition temperatures and maximal ALD growth temperature of the precursor used. This information may be found in the literature. Make sure the thermocouple of a bubbler heater has good thermal contact with the bubbler, and has good measurement stability and accuracy so that the temperature readings are real; ensure the working temperature never goes beyond the decomposition temperature; in addition, when heated, it is recommended that you keep the precursor constantly at the working temperature or slightly below, this minimizes the thermal cycling of a precursor to avoid degradation.

Mechanical Pump Safety/Maintenance

1. In principle the ALD process should work in a slightly over-saturated exposure condition, and almost 100% of the precursors are consumed and very little excessive vapor going into the exhaust and the pump. The saturation condition for an ALD system has to be experimentally determined. However, if precursor vapor pulses are oversaturated too much or too excessively, or there is not sufficient purge, a severe CVD reaction can happen which generates a lot of powders or particles inside the growth chamber, down the exhaust and even into the pump. It is thus strongly recommended that you use recipes approved by SVT Associates or minimize precursor use to avoid exhaust problems. This is the fundamental, the most important, root protection for the pump;
2. In practice, it is hard to identify exact saturation conditions or it takes many test runs. Condensation of excessive precursors, particles or other by-products could still be built up gradually along the exhaust line or even precursor delivery lines. If the exhaust line is heated and there is not any filter or trap installed down the exhaust line, most are dumped in the pump. A hot trap or filter is needed which becomes the secondary defense line. A hot trap is capable of decomposing or neutralizing excessive metal organic precursor vapor into less volatile products and hydrocarbon gas, which do not harm the pump and reduce hazard.
3. A chemical corrosive resistant pump/use of Fomblin oil are recommended. Fomblin is ideal but expensive. However it is a trade off of the oil price and pump repair cost. If ordinary mechanical oil is used, frequent replacement is necessary (once a month or even more frequent if in heavy use or very aggressive chemicals are used). Some other types of pump oil which are resistant to O_2/H_2O and metal organics such as AJ Elite Z synthetic vacuum pump fluid from A&J vacuum can last longer than the ordinary one.

Emergency Shutdown Procedure

In an emergency situation such as system malfunction, power shut down, system vent failure:

- Stop the program if you are in the middle of the growth;
- Manually shut down shutoff valves of all bubblers in use;
- When the RF plasma unit is on, turn off, zero all MFCs and switch off all master pneumatic valves to prevent any gas in; and shut down the gas cylinder;
- Shut down the lamp heater, manifold heater, exhaust heater and chamber wall heater;
- Close the automatic throttle valve to isolate from the pump and shut down the pump itself manually.
- Identify the reason of the failure or malfunction and correct it before restarting the system

ALD System Shutdown for Extended Period of Time

Certain ALD precursors can degrade with repeated heat and cool cycles. If precursors must be cooled, or are accidentally cooled as a result of a power failure, we recommend the precursors remain at room temperature until needed to run process.

Complete System Shutdown

- Confirm that the manual precursor isolation valves are closed.
- Close manual gate valve between chamber and loadlock
- Set all MFC flows to 0 sccm
- Close Process gas isolation valves
- Shut off all heaters (Lamp heater, chamber wall heater, manifold heaters, exhaust heater, hot trap, and precursor heaters)
- Close load-lock roughing valve
- Close chamber pumping gate valve
- Turn off the turbo pump, leave the Turbo backing pump run until the turbo stops spinning
- Turn off the load-lock rough pump
- Power down Eurotherms, RF power supply, Ozone generator
- Turn off water
- Close air supply to system
- Close Robo program
- Shut off computer
- Remove main power to the system

Precautions

Do not heat the ALD system to temperatures above those recommended by the manufacturer. Note the maximum temperature settings for different parts. ALD pulse valves are rated to 200 °C and should not be heated above that temperature. The lamp heater maximum temperature is 500 °C, while outer chamber wall heaters should not be set > 150 – 200 °C because of the o-ring. The precursor delivery lines and exhaust lines should not be heated above 200 °C. Temperature of the precursors should not exceed safety or decomposition temperature of the chemical used. The maximum temperature for the precursor heater jacket is 200 °C.

Chamber Cleaning

After many depositions are performed in the ALD system, deposits build up on the internal surfaces. Depending on the materials deposited, thermal cycling of the system can eventually cause cracking and flaking of the deposit. If flakes are visible, clean the chamber with a lint free cloth using the appropriate solvents.

On systems that are equipped with the plasma source, an Oxygen plasma can be used to clean (by combustion) excessive organic deposits in the chamber. Similarly, a Hydrogen plasma can be used to prepare an ALD chamber for nitride deposition to suppress oxygen inclusion into the film.

Warranty and Limitations of Remedies

SVT Associates warrants that all equipment manufactured by it shall be free from defects in materials and workmanship under normal use and service for a period of twelve (12) months from the date of shipment from SVT Associates manufacturing facility. This warranty is subject to SVT Associates equipment being installed, maintained, and operated in accordance with the operating and maintenance instructions accompanying each item manufactured by SVT Associates. Warranty shall be void if SVT Associates equipment is modified by the CUSTOMER or used in other than the recommended manner or applications. Purchased equipment incorporated into any item supplied by SVT Associates will be covered by said manufacturer's warranty.

SVT Associates warrants that, at the time of delivery, any other products processed or manufactured and sold by it hereunder are free of defects in material and workmanship and conform to COMPANY specifications. No warranty is provided by SVT Associates for products sold hereunder which are not manufactured or processed by SVT Associates, but the manufacturer's warranty for such products, if any, shall be assigned to the CUSTOMER without recourse to SVT Associates. The foregoing warranties are in lieu of and exclude all other warranties not expressly set forth herein, whether expressed or implied by law or otherwise, including without limitation any warranty of merchantability or fitness for a particular purpose. In no event will SVT Associates be liable for any consequential damages.

IN THE EVENT OF SVT ASSOCIATES LIABILITY, WHETHER BASED ON CONTRACT, TORT (INCLUDING BUT NOT LIMITED TO NEGLIGENCE AND STRICT LIABILITY) OR OTHERWISE, THE CUSTOMER'S SOLE AND EXCLUSIVE REMEDY WILL BE LIMITED; SVT ASSOCIATES HAS THE FOLLOWING OPTIONS; TO REPAIR OR REPLACEMENT (F.O.B. SVT ASSOCIATES MANUFACTURING PLANT) BY THE COMPANY OF ANY NON-CONFORMING ITEM FOR WHICH CLAIM IS MADE BY THE CUSTOMER OR TO REPAYMENT OF THE PORTION OF THE PURCHASE PRICE PAID BY THE CUSTOMER ATTRIBUTABLE TO THE NON-CONFORMING ITEM. SVT ASSOCIATES WILL NOT BE LIABLE FOR ANY OTHER DAMAGES, WHETHER DIRECT, INCIDENTAL, CONSEQUENTIAL OR OTHERWISE.

Return Policy

Any request by the CUSTOMER for return of standard products other than for warranty claims under warranty hereof, for all or any part of purchase order accepted by SVT Associates, shall be subject to the following conditions:

- A. Notification must be made to SVT Associates by the CUSTOMER within thirty (30) days of original shipping date.
- B. A "RETURN GOODS AUTHORIZATION" number must be assigned to and accompany all goods or materials being returned by the CUSTOMER to SVT Associates. Said number must be assigned by SVT Associates prior to any and all returns. Goods not accompanied by a "RETURN GOODS AUTHORIZATION" number will be refused by SVT Associates and returned at the CUSTOMER'S expense.
- C. CUSTOMER shall prepay shipping charges for products being returned to SVT Associates.
- D. Products being returned to SVT Associates should be properly crated for shipment, and the CUSTOMER shall bear the risk of loss until delivered to SVT Associates.
- E. Products being returned to COMPANY must be returned in the condition originally received by the CUSTOMER and free from damage, use or modification which would render the product unusable for resale by SVT Associates.
- F. All applicable taxes, duties, insurance, and shipping charges shall be the sole responsibility of the CUSTOMER.

- G. Goods being returned for other than warranty repair shall be subject to a restocking charge of twenty (20) percent of the original sales price of the returned item.

Returning Equipment for Repair or Servicing

Before shipping equipment for repair or servicing, obtain a Return Authorization Number assigned by SVT Associates, Inc.

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