Chapter 6.42

Picosun Atomic Layer Deposition (ALD)

(picosun) ***(586)***

1.0 Equipment Purpose

1.1 Picosun ALD is a microprocessor controlled system that can atomically deposit a thin layer of metal oxides, pure metals, and with an additional ammonia gas source (not connected at this time) should be able to deposit metal nitrides. Metal oxide films grown by the ALD system are suitable for gate dielectric, electroluminescent display insulators, capacitor dielectrics and MEMS applications. Metal nitride thin films grown by the ALD could be used for diffusion barriers and similar applications.

1.2 **The Load Lock is not functioning as of 11/06/2023. We have moved to load directly into the carrier via the side window. Process is described below.**

2.0 Materials Controls & Compatibility

2.1 Polymer material and other low temperature substrates must undergo process review before use in picosun.

2.2 Metals are allowed in this tool.

3.0 Applicable Documents

3.1 Exam Tool : this tool requires an online exam before qualification.

3.1.1 Timeline (estimated time to completion: 1 week)

3.1.1.1 Get trained by any qualified member.

3.1.1.2 Take the online test in the Nanolab office (open 8A-12P, 1P-5P).

3.1.1.3 Arrange a qualification session with a superuser to show competency on the tool.

3.1.2 Superusers and staff qualify members on this tool. Superusers on this tool may have permission to do advanced techniques

3.2 Picosun Sunale R-Series ALD tool User Manual (Copy in Office).

3.3 Material Safety Data Sheets for TMA and TTIP (Copies in Office and Lobby).

4.0 Definitions & Process Terminology

4.1 Atomic Layer Deposition: A self-limiting chemical process that uses metal precursors to coat an atomically thin layer of metal or metal oxide or metal nitride film on the substrate surface.

4.2 Precursor: Source/s used in the ALD machine contains the desired metal for deposition. This metal is bonded to a hydrophobic alkyl functional group such as Methyl (CH3), which will

ultimately leave a monolayer of metal oxide behind on the substrate (metal is reacted with water vapor to form the metal oxide).

4.3 Source bottle: Up to three sources materials (precursors) can be provided in source bottles installed on this particular ALD machine.

4.4 Injection: The precursor source bottle is pulsed open in specific intervals defined by the process recipe. A carrier N2 gas will then delivers the precursor material to the reaction chamber.

4.5 Typical source materials for this system are:

4.6 TMA: Trimethylaluminum Al(CH3)3 is precursors used to deposit aluminum oxide.

4.7 TTIP: Titanium Tetrakis Isopropoxide, which supplies Ti source for the titanium oxide process.

4.8 Other sources may be available as an alternative to above sources i.e. TiCl4 Titanium Tetrachloride is a precursors used to deposit Titanium oxide. The number of sources that can be installed on this tool are currently limited to three as per available source bottles (hardware limited) on this tool.

4.9 hPa: Hectopascal – (1 hPa = 0.75 Torr)

5.0 Safety

5.1 Follow general safety guidelines for the lab; the safety rules outlined in Chapter 1.01 - Marvell NanoLab Chemical Hygiene Plan and the following:

5.2 Source gas: The system uses highly reactive source gases, great care must be taken when source bottles are changed. Also, the source temperature needs to be lowered to room temperature, before changing the source.

5.3 Chemical Hazard: Do not vent the chamber before purging process is finished. Please note chemicals and their vapor used in this system are highly flammable and toxic. Any suspected leak/s in the system needs to immediately be reported on the WAND.

5.4 Emergency shutdown: In case of an emergency, shut down the whole system by pressing the red emergency button on the machine.

6.0 Process Data

6.1 Process Specification for standard 300°C Al2O3 ~1A/cycle deposition available on the Process Specification section of Nanolab Website

6.1.1 2019/03/19 25pt ellips2 Thickness and Index maps 400cycles Picosun Al2O3 300C

6.2 Low temperature deposition Al2O3 at 200C° with an 8s purge: 500cycles resulted in 44nm film with n=1.66.

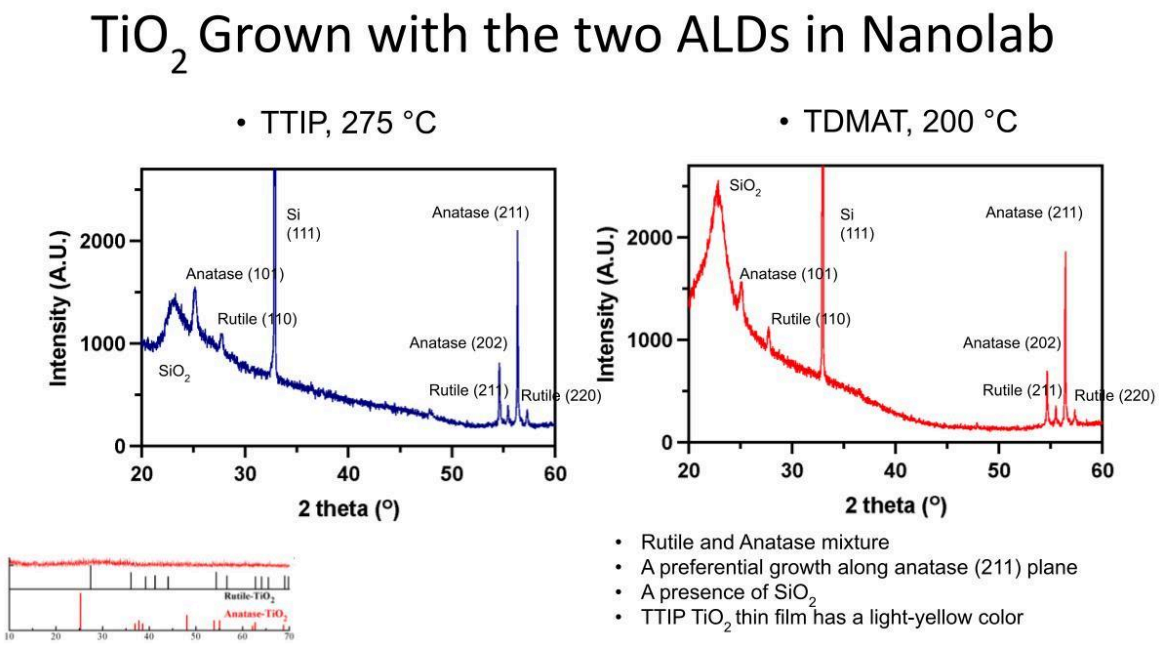
6.3 Process Staff routinely does TiO2 deposition checks and characterization. Please check in with Process Engineer 1 if you have questions.

6.3.1 2020-06-20 1000 cycle TiO2 thickness and index 25pt map from ellips2

6.4 Result of DOUBLE LOAD deposition, in which two wafers are loaded with spacers in between them for a shim effect. Data acquired by XRR method and also compared with cambridge alumina

	Thickness [nm]	Density [g/cm3]	Roughness [nm]	thickness/cycle [nm/cycle]
Cambridge	42.26	3.09	0.415	0.10565
Picosun "bottom"	34.60	3.16	0.434	0.0865
Picosun "top	34.25	3.04	0.458	0.085625

6.5 XRD comparison of thermal TiO2 from picosun and cambridge. (Scan courtesy of Yang Group researchers)



6.6 Al2O3 film etch rate comparisons.

6.6.1 Cambridge film: 500 cycles O2 plasma at 270C

6.6.2 Picosun film: 500 cycles Thermal ALD at 300C

AL2O3 etch rate

Wet Etch Chem	Picosun initial th (A)	Picosun final th (A)	Cambridge initial th (A)	Cambridge final th (A)	time (s)	PIcosun film Etch rate (A/s)	Cambridge film etch rate (A/s)
Pirahna 100C	510	375	473	375	180	0.75	0.54
DI:HF (49%) 100:1	520	379	466	339	30	4.70	4.23
DI:HCl (37%) 100:1	523	520	472	466	180	0.02	0.03

7.0 Available Processes, Gases, Process Notes

7.1 System Description

7.1.1 System Components: Picosun ALD system comprised of the Oxford wafer loader station, deposition chamber, a source cabinet with the electronics and MFCs associated with it, all of which are controlled by the ALD software installed on a user-friendly Select panel controller (PLC).

7.1.2 Precursor Sources: There are currently three types of source bottle available on this system designed to address different precursor materials at specific vapor pressure ranges.

7.1.3 Source A: Liquid source with high vapor pressures above 10 mbar can be used in this source bottle. Several source materials including the TMA (current source) can be used in this source bottle.

7.1.4 Source B: Pico Solid source bottle at this station can be used for liquid or solid sources with vapor pressures above 1mbar at temperatures less than 200°C. Several source materials including the TTIP (current source) can be used in this source bottle.

7.1.5 Source C: This source has the same design as source A, however, it is currently dedicated to DI water as an oxidizing agent for metal oxide processes.

7.1.6 MFCs: There are 4 MFCs available on the system, three of which are used for the sources and one for the intermediate space N2.

7.2 System Control Description

7.2.1 The main control of ALD is a PLC based system with Select screen user interface. The operations are divided into seven main pages which can be accessed by Selecting graphic buttons on the bottom portion of the display screen. Some of the pages contain sub-pages. The functions of these main pages are described below:

7.2.2 PI CHART – Displays the system conditions, including temperatures, pressures, gas flows and etc.

7.2.3 RECIPE – Used to load, edit, and save process recipes.

7.2.4 DEPOSIT – Used to start, stop, and abort a process run. It also displays the schematics of process steps.

7.2.5 The DEPOSIT page contains a schematic drawing of the process flow. Each circle stands for a process step. The highlighted one stands for the current process step being executed.

7.2.6 There are two circles with capital “M” in their center, each representing a step that needs lab member’s attention: First one to open the source valves before deposition. Second one to close the source valves after the deposition.

7.2.7 In the center of the page, there are several colored rectangular boxes that stand for process conditions. Red means a process condition is not satisfied. Green means the process is ok to run.

7.2.8 ACTIONS – Contains some operation sequences used for routine system maintenance, e.g. source line purge.

7.2.9 MANUAL – Used to operate some system components manually, e.g. open/close valves, set chamber/source temperatures, set purge gas flows.

7.2.10 TRENDS – Used to view the charts of all the process parameters.

7.2.11 SETTINGS – Used to set system settings. Also used to log out the system.

7.2.12 BACK – Used to go back to previous control page.

7.2.12.1 Note: In this manual, all the above main control buttons, displayed on the bottom of the Select screen, are in bold and capital case. All the other control buttons on each control page are bold, with only the first letter capitalized.

7.2.12.2 The wafer elevator and load lock gate valve are controlled via another small Select screen control located on the lower shelf under the main Select screen. It is used, along with two other manual valves, for wafer loading/unloading operations. There are four Select screen buttons:

7.2.12.3 <VALVE> - Used to open or close the gate valve between the process chamber and the load lock.

7.2.12.4 <HOME> - Used to home the wafer elevator and lift wafer up from process chamber for unloading.

7.2.12.5 <Z1> - Used to move the elevator to intermediate position when pushing/pulling the load lock rod.

7.2.12.6 <Z2> - Used to lower the elevator to the process position. It also closes the process chamber.

7.2.12.7 Note: In this manual, all the buttons for load lock/elevator control are bounded by <> to avoid confusion with the main process control buttons.

7.3 Available Sources

7.3.1 TMA: Trimethylaluminum Al(CH3)3, which supplies aluminum source for the aluminum oxide process. (Source A)

7.3.2 TTIP: Titanium Tetrakis Isopropoxide, which supplies Ti source for the titanium oxide process. (Source B, a heated source, process temperature 80°C)

7.3.3 DI Water: DI water vapor acts as oxidation agent. (Source C)

7.4 Available Recipes

OXIDE	Precursor	Nanolab Inventoried and replenished	Deposition Rate
Al2O3	TMA	Standard Process – Supported and Replenished	~1 Å/Cycle
TiO2	TTIP	Supported Process – Supported and Replenished	~0.2-0.25 Å/Cycle

7.5 Members are allowed to change the number of cycles, temperature, precursor purge time in their own recipes in their own folders. NEVER CHANGE A STANDARD RECIPE IN THE STANDARD FOLDER

7.6 Process Notes

7.6.1 Samples with photoresist are not allowed in the Picosun ALD system.

7.6.2 The process chamber can accommodate one 6” or one 4” wafer at a time. Many small samples can be loaded at a time on a 6” pocket wafer, which can be checked out from the office. Please note the ALD deposition only goes on the topside of the sample.

7.6.3 Take extra care not to crash the elevator or the gate valve in the system, as it does not have all the necessary sensors for a fully automated operation.

7.7 Standard TiO2 recipe changes 04/01/2023

7.7.1 Precursor temp 75C

7.7.2 Pulse TTiP = 0.1s

7.7.3 Purge = 8s at 200sccm

7.8 Equipment Acceptable Parameter Window

7.8.1 Chamber Temperature: 150 – 350 C

7.8.2 Number of cycles: Up to 2000

7.8.3 Exceeding these limits **requires staff approval**

8.0 Equipment Operation

8.1 Login Procedure

8.1.1 You should not have to log in to the system because we have stopped logging out of the system. However if you find the system logged out, please contact staff

8.1.2 Select the Login button, and enter the password using the graphic keyboard. The password, which is case sensitive, is given only to qualified lab members. Select the PI button at the bottom of the page to go to PI Chart screen shown in Figure 1.

8.2 System Check

8.2.1 Go to PI CHART page.

8.2.2 Set the source purge nitrogen flows to 150 sccm

8.2.3 Set the chamber purge flow to 350 sccm.

8.2.4 Set chamber temperature to 100°C and the source to room temperature.

8.2.5 Check chamber pressure: Should be ~7-10 hPa.

8.2.6 Move elevator to <Z2>.

8.2.7 Check parameters on mercury to determine last deposition material.

8.2.8 If material deposited immediately prior differs from material being deposited in current run, run a dummy run for 100A of deposition to ensure there is no flaking.

8.3 Loading Recipe

8.3.1 Go to the RECIPE screen.

8.3.2 Select Open Recipe button.

8.3.3 Select either the Al2O3 or TiO2 recipe.

8.3.3.1 Note: If you do not see these two recipe options, Select Change Directory button, and change the active directory to “STANDARD”, then repeat recipe loading.

8.3.3.2 The source type is defined in the recipe. Without loading the recipe, the source temperature cannot be set.

8.3.3.3 YOU MAY ADJUST 1) the Number of cycles on page 4/4

8.3.3.4 YOU MAY ADJUST 2) The Purge time after each pulse - talk to staff first

8.3.3.5 YOU MAY ADJUST 3) The temperature of deposition - talk to staff first

8.3.3.6 DO NOT SAVE OVER THE STANDARD recipes without changing the name first!!

8.3.3.7 Make sure you keep enough FLUSH times in the recipe - 5 recommended - since we are now opening the chamber to atmosphere every single time!

8.4 You can change stabilization time and number of flushing cycles in the recipe. If you want the Flushing step in your recipe you must allow for at least 1 minute of Stabilization time afterwards

8.5 DIRECT LOAD INTO THE CHAMBER - FOLLOW THIS PROCESS EVERY TIME

8.5.1 Check hand valves are closed on sources (TMA and H2O)

8.5.2 Check that the temperature in the chamber is LESS than 100C. It's not a good idea to open a hot chamber to atmosphere. Better wait!

8.5.3 Go to SETTINGS page

8.5.4 Change OPEN LID limit to 1000

8.5.5 Go to ACTIONS page

8.5.6 VENT the reactor

8.5.7 Watch PT1 increasing. It should hit 1000 in a few minutes

8.5.8 Move the z-lift to the <Z1> location by pressing <Z1>

8.5.9 Open the large window on the right of the tool with the large black knob

8.5.10 Load wafer using tweezers. Keep in mind it's still pretty hot inside.

8.5.11 Double wafer load is possible using alumina tabs as spacers. Talk to process staff or superuser about the pros/cons of this method.

8.5.12 Close the window. Turn the knob firmly closed but don't kill it.

8.5.13 SETTINGS page - change Open Lid limit to 80hpa

8.5.14 ACTIONS page - cancel VENT

8.5.15 ACTIONS page - execute EVACUATE

8.5.16 Immediately open the cabinet underneath, locate and press up the overflow blank flange. Watch the PT1 decrease.

8.5.17 Lower the lift by pressing <Z2> on the controller.

8.5.18 Proceed when you've verified V1 opened and the chamber pressure is ~10hpa or below.

8.6 Set Chamber/Source Temperature

8.6.1 Select MANUAL button

8.6.2 Select 3/3 button on the top right of the MANUAL page (Figure. 2).

8.6.3 Set the chamber/source temperature as the following table:

Recipe	Chamber Heater TE1 Max	Chamber Temp TE2	Source Heater TE21 Max	Source Temp TE20

Al2O3	450°C	300°C	NA	NA
TiO2	450°C	275°C	100°C	80°C

It takes about an hour for the chamber temperature to stabilize at set point. After the chamber temperature stabilizes, set TE1 Max to 400°C for better temperature control.

8.6.3.1 Do not set TE1 above 500°C or you may damage the chamber.

8.7 ALD Deposition

8.7.1 Select RECIPE button.

8.7.2 Select 4/4 button on the top right corner of the RECIPE page (Figure 7).

8.7.3 Enter the desired deposition cycles in the field on the right side of the screen.

8.7.4 Select DEPOSIT button.

8.7.5 Select the Start button. The ALD deposition process will start. It will bypass “Soft Pump Down”, “Main Pump Down” steps because the tool contains a load lock.

8.7.6 When the process is at the “Flushing” step, open the lower cabinet doors in front of the tool and open the desired source bottle. There are three source bottles in the cabinet: Source A (currently TMA for Al2O3) on the left, Source B (currently TTIP, for TiO2) in the center, and Source C (currently DI water).

8.7.7 Hold the bottle with one hand securely, and with the other hand open the source bottle/s valves, as per follows:

8.7.8 For Al2O3 deposition: open Source A and C.

8.7.9 For TiO2 deposition: open C. (Source B will be controlled by the system automatically)

8.7.10 After “Flushing: step is completed, the process bypasses the “Heating” step, because the process chamber temperature needs to be at set point. After “Stabl. Time” step (put in 5 minutes, if at all), the process starts “Pulsing”, which is the ALD deposition. The screen will display the estimated process finish time.

8.7.11 To monitor the source pulsing pressure, Select ACTIONS button.

8.7.12 Select 3/3 button on the top right corner of the ACTIONS page. (The PI page does not refresh fast enough to see the pulsing.)

8.7.13 Select Pt10 button to monitor source A,

8.7.14 Select Pt20 button to monitor source B,

8.7.15 Select Pt30 button to monitor source C.

8.7.16 Pressure spikes are showed continuously on a data logging chart when the source valve pulses. For source A and C, the pressure spikes upwards. For source B, heated source, the pressure spikes downwards due to gas flow setup.

8.7.16.1 Note: You can only monitor the press spike of the source one at a time. Wait till the data logging finishes, then start monitor the other source. It you Select other Pt button during data logging, you will get an “access denied” displayed warning.

8.7.17 Select DEPOSIT button again to display the DEPOSITION page. After the deposition step finishes, the process skips “Cooling” step, because in the standard recipe the end temperature is set the same as the deposition temperature.

8.7.17.1 Note: Do not set the end temperature in the recipe to lower temperature. Doing so will make the process hold at “Cooling” step for a long time because the process chamber cools very slowly.

8.7.18 Open the lower cabinet doors in the front of the tool.

8.7.19 Close sources valves that have been opened

8.7.20 Select Unload button. The process will go to “Vent” step.

8.7.21 Select Open Lid button.

8.7.22 Select Shut Down button. The last two buttons have no effect on the process flow because the tool has a load lock. The processed wafer now is ready for unloading.

8.8 Post ALD Source Purge

8.8.1 Open the lower cabinet doors, and double check that all the source valves are closed.

8.8.2 ******WARNING******

If you run the Source Purge with the source valve open, all the precursor in the bottle will be purged out. It needs to be refilled again. MAKE SURE THIS DOES NOT HAPPEN !!!

8.8.3 Select ACTIONS button. In the center of ACTIONS page, there is a Purge Source box with A, B, C, D buttons.

8.8.4 Select A button if you ran Al2O3 recipe. Do not run a purge on source B (TiO2 recipe). There is no shut-off valve and you will use up a great deal of precursor by running a purge on source B.

8.9 PUMP and PURGE before VENTING

8.9.1 Select MANUAL button.

8.9.2 Select 3/3 button in the top right corner of the MANUAL page.

8.9.3 Change TE1 Max to 160°C, TE2 to 100°C. If you ran TiO2 recipe, Change TE20 and TE21 to 0°C (no heating).

8.9.4 Select SETTINGS button.

8.9.5 Set Open Lid pressure to 300hpa

8.9.6 Go to Actions and VENT

8.9.7 When PT1 reaches 300hpa it will idle and stop venting

8.9.8 ACTIONS page press CANCEL on VENT

8.9.9 ACTIONS page - start EVACUATE wait till it pumps to base ~10hpa then hit CANCEL

8.9.10 REPEAT 8.9.6-8.9.10 at least 3 times

8.9.11 Go to SETTINGS page and change Open Lid to 1000hpa

8.9.12 Move the lift to <Z1>

8.10 VENT for real this time, remove wafer and PUMP back down as in section 8.5 above

9.0 Troubleshooting Guidelines

9.1 Problem: Cannot log in.

9.1.1 Cause: The password is case sensitive.

9.1.2 Solution: “m” - Level 2 , “o” - Level 1

9.2 Problem:PT2 pressure does not change when the load lock 3-way valve is turned to pump.

9.2.1 Cause: The PT2 valve, in the back of the tool, should be opened manually.

9.2.2 Solution: Open PT2 valve.

9.3 Problem: No source pressure spike during the pulsing step.

9.3.1 Cause: The source valve not open or the precursor in the source is running low.

9.3.2 Solution: Check the source valve. If it is opened fully, report the problem. The precursor needs refill.

9.4 Problem: Process completed without problem, but no film on the wafer.

9.4.1 Cause: The precursor can not be absorbed on the substrate or precursor is out

9.4.2 Solution: contact process staff

9.5 Problem: The deposit film is not uniform.

9.5.1 Cause: The wafer surface not properly cleaned/prepared.

9.5.2 Solution: Properly clean/prepare wafer. Dirty wafer not only create this problem, but also contaminate the process chamber.

9.6 Problem: recipe for Al2O3 is stuck at the Heating Step even though chamber temperature is reached

9.6.1 Cause: Ti precursor source from previous deposition is not at room temperature

9.6.2 Solution: on the Manual 3/3 page change the Source B TE20 to match the current temperature so that a green OK square appears. Call staff if you need help with this.

9.7 Problem: Recipe Save Alarm is on and can't save the recipe

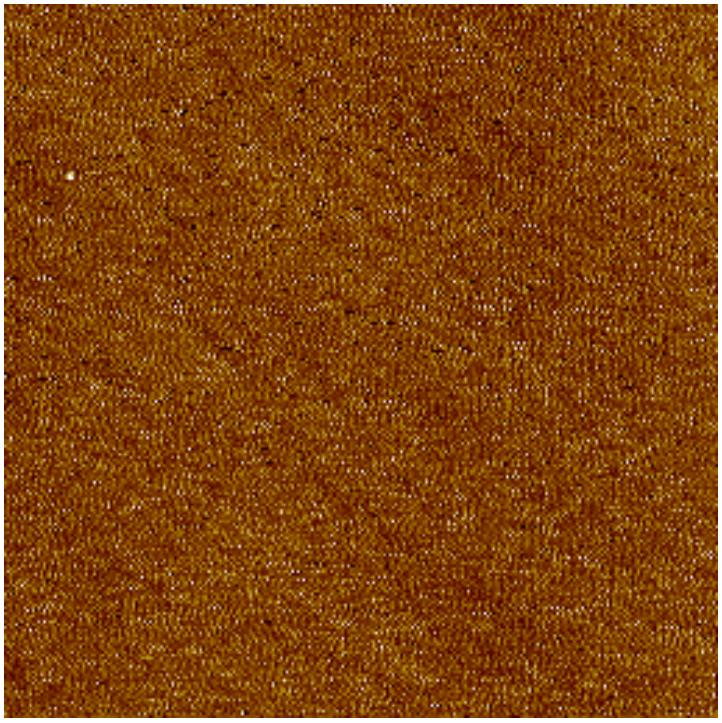
9.7.1 There is a workaround. Talk to Sam (PE1)

10.0 Study Guide

10.1 There is no study guide available at this time

11.0 Appendices, Figures & Schematics

11.1 AFM Roughness Ra =0.299nm Film: AL2O3 300C 400cycles



11.2

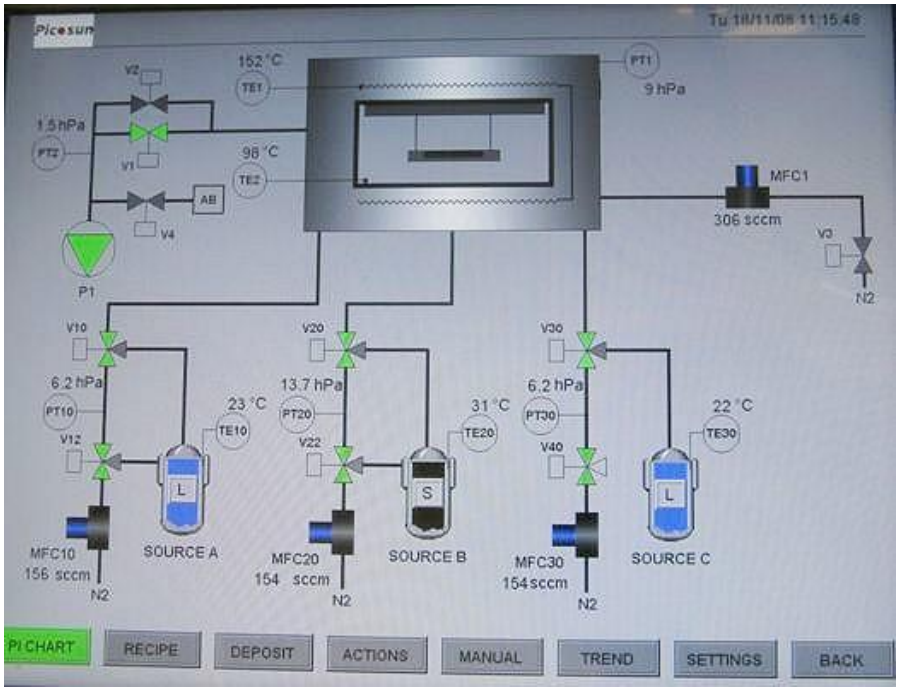


Figure 1 - PI Chart Screen

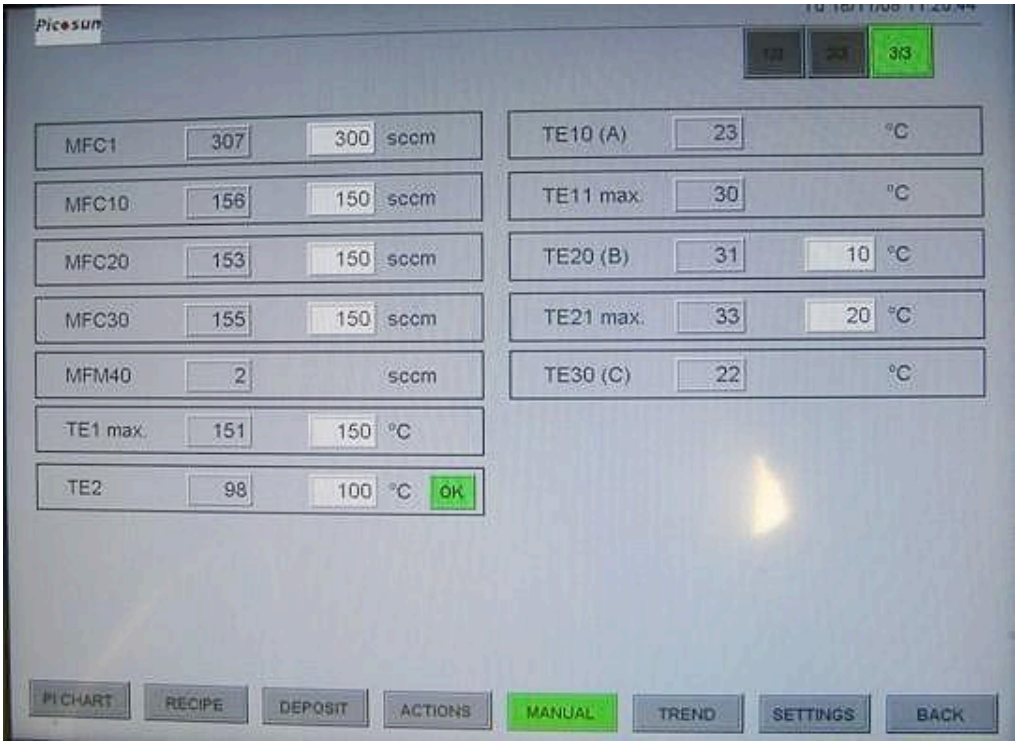


Figure 2- Manual Screen No. 3

Figure 3 - Manual Screen No.1

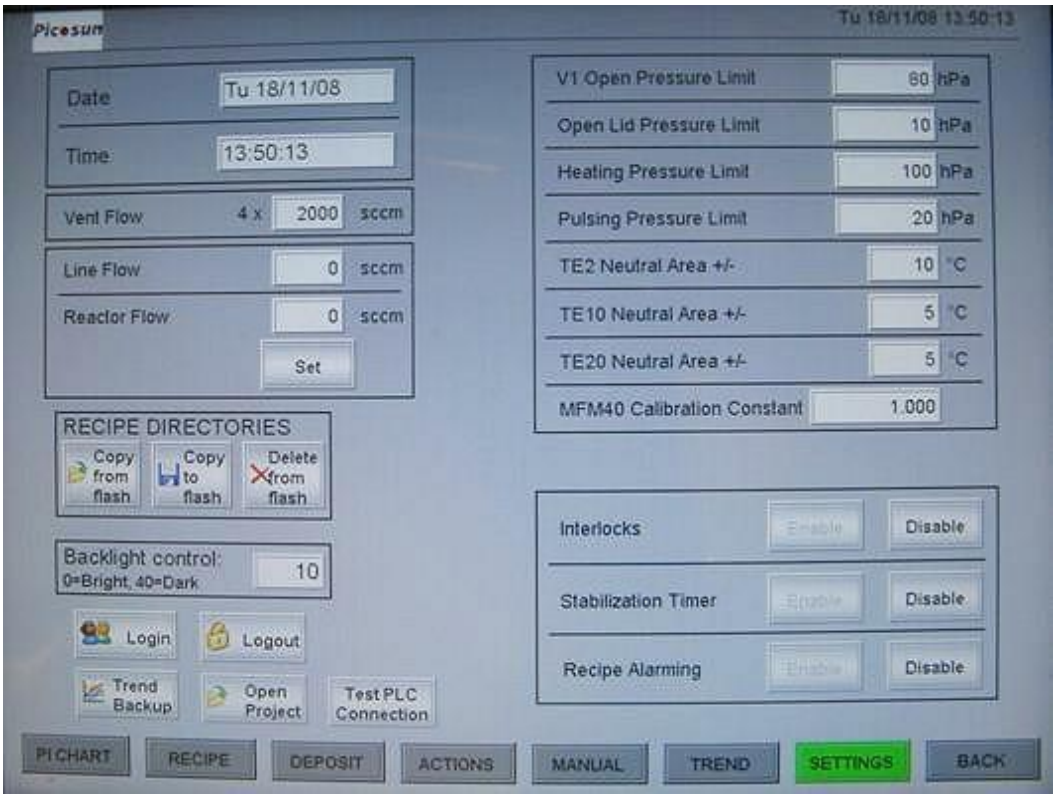


Figure 4 - Settings Screen

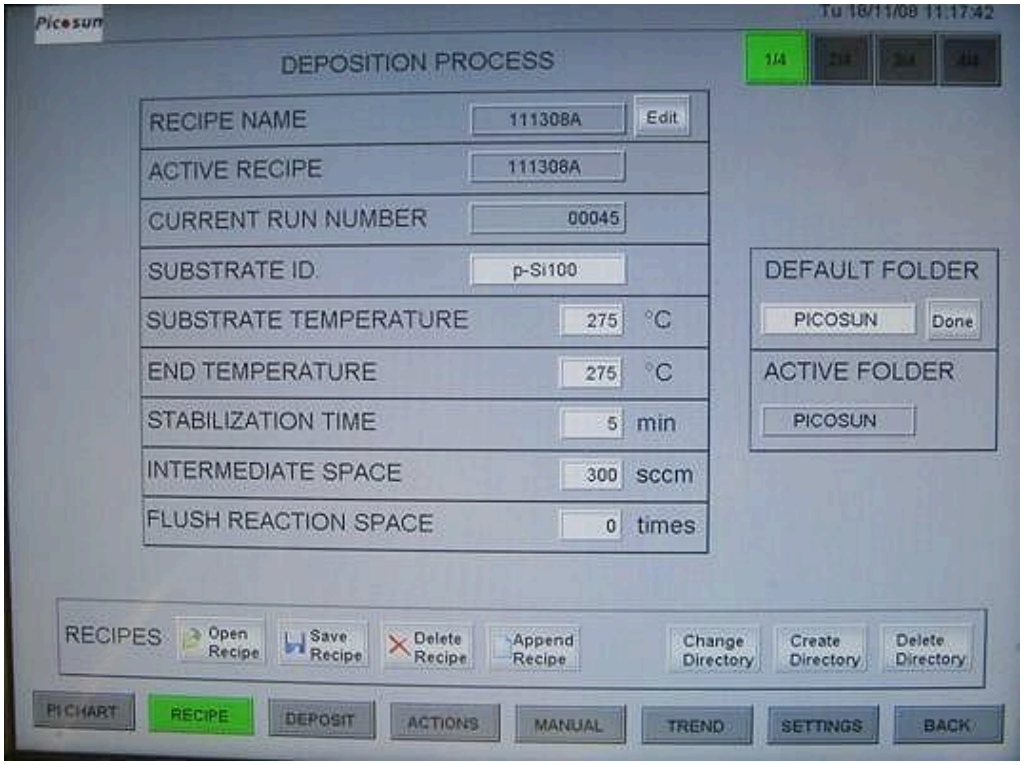


Figure 5 - Recipe Screen No.1

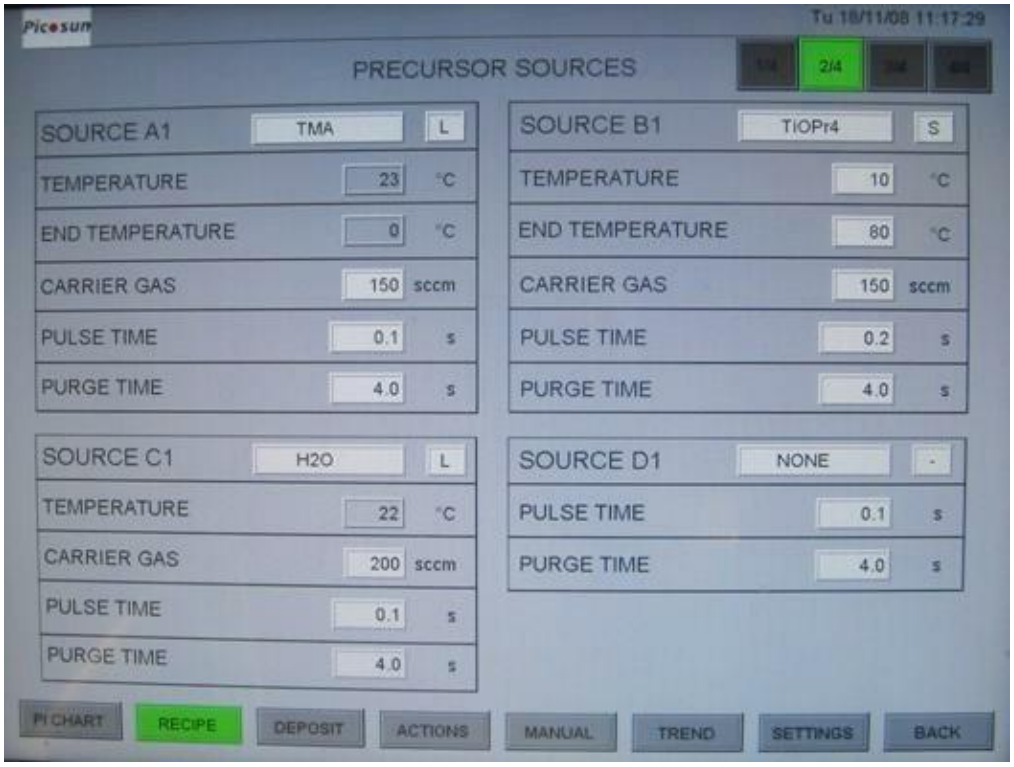


Figure 6 - Recipe Screen No.2