

Arizona State University NanoFab

LESKER #1 SPUTTERER STANDARD OPERATION PROCEDURE

Rev G

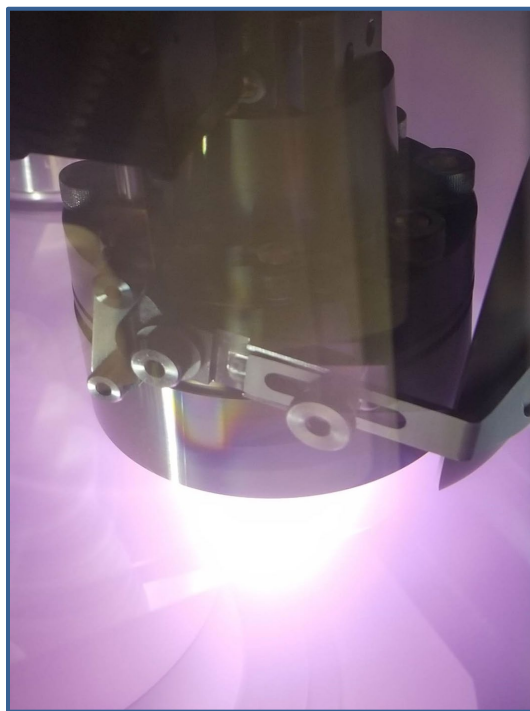


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1.0 Purpose / Scope

- 1.1 This document covers the procedure that should be followed for normal operation of the Lesker PVD75 sputter coater for the purpose of depositing metals & dielectrics on substrate materials that might be used for research purposes. It is suggested that you review this document thoroughly before proceeding with the operation of this tool & always check with staff when using a new target material, to verify if special precautions need to be taken for safety or cross contamination issues.

2.0 Reference Documents

- 2.1 Chemical Safety & Hazardous Waste Management Rules & Procedures Handbook
- 2.2 PVD Series Operation Manual
- 2.3 Kurt J Lesker Circular Sputtering Source Torus 3” HV operations manual

3.0 Equipment / Supplies

- 3.1 Clean room vacuum
- 3.2 Clean wipes
- 3.3 Touch screen stylus
- 3.4 Tweezers/Kapton tape.

4.0 Safety

- 4.1 Follow all safety procedures outlined in the NanoFab Handbook
- 4.2 Follow safety procedures for high voltage when working with DC or RF energy.
- 4.3 Follow safety and handling procedures when working with vacuum systems and target materials.

5.0 Tool Reservation Policies

- 5.1 Only trained users will be allowed to use this equipment.
- 5.2 Recommended to schedule your runs to secure you scheduling and to alert other potential users of the tool.
- 5.3 Our NanoFab 15-Minute rule.
 - 5.3.1 Please start within 15 minutes of your equipment scheduled time or the tool becomes available to anyone. Please place a ‘Tool in Use’ tag when you arrive to indicate use.

- 5.3.2 Please have the equipment available for the next user within 15 minutes after your scheduled time.
- 5.4 Cancellations.
 - 5.4.1 If you cannot meet the equipment schedule, please cancel your iLabs schedule to allow other users to utilize the equipment.
 - 5.4.2 Scheduling on iLabs allows cancellation within 24 hours of your scheduled time. Please email staff if cancellation within 24 hours.
 - 5.4.3 We discourage last second cancellations.
 - 5.4.4 We discourage scheduled equipment no-shows.
- 5.5 Scheduling Overnight runs.
 - 5.5.1 Please complete overnight runs by the following morning at 9am.

6.0 Targets and Magnetron Guns

- 6.1 Please request targets for an upcoming scheduled run at least one hour ahead of time. 'Target requests' would be done using iLabs service requests.
- 6.2 This tool will allow non-Si compatible, 'Dirty' materials such as Au, Ag, Cu and ITO.
- 6.3 This tool has the capability to sputter Al, Cr, Co, Fe, Hf, ITO, Mo, Ni, Si, SiC, SiO₂, Ta, Ti, TiO₂ and W.
- 6.4 Please request by iLabs service request for the loan of our Au target. We will loan the target at the current rate of \$240/gram. Please give us a 24-hour notice. The target will be under your responsibility.
- 6.5 This tool has the capability to run reactive films using O₂ and N₂ gases.
 - 6.5.1 Tool is phasing into the use of TORUS magnetic guns with clamshell shutters.
 - 6.5.2 Magnetron guns.
 - 6.5.2.1 Source 1 (TORUS Targets)-Used for DC or RF Power.
 - 6.5.2.2 Source 2- Used for DC or RF power.
 - 6.5.2.3 Source 3 (TORUS Targets)- Used for DC power or RF power.
 - 6.5.3 Use DC for metallic targets. Use RF power for dielectric or ceramic films.
- 6.6 Please allow gun cooling for films up to 300nm film thickness.
- 6.7 DC films- Maximum of 3Å/sec dep rate allowed.
- 6.8 RF films- Only 0.5Å/sec dep rate allowed due to the fragile bonding of dielectric to backing plate.

7.0 Operation Procedures

Note: It is recommended to monitor your Sputterer depositions, but you may step away from the tool momentarily. Please do not leave the fab and you will be responsible to complete your own run.

- 7.1 Use touch screen user interface to control settings.
- 7.2 Note: You will be required to reduce the turbo speed to 50% to either perform the Vent or deposition processing.
- 7.3 **Vent chamber.**
 - 7.3.1 On Vacuum screen, view the chamber pressure and the current turbo speed.
 - 7.3.2 Set Turbo speed target to 50% to allow you to Vent.
 - 7.3.2.1 On VAC screen, enter 50% Turbo speed if not at 50%. Insure you do not turn Off the turbo pump.
 - 7.3.2.2 On the GAS screen, depress Gas Injection button to ON (Green).
 - 7.3.2.3 On the GAS screen, enter 4.0 (mTorr) SETP pressure.
 - 7.3.2.4 On the GAS Screen, depress the Ar gas CNTL button to ON (Green).
 - 7.3.3 Monitor Turbo screen for actual Turbo pump speed to 50%.
 - 7.3.4 To turn OFF gas flows.
 - 7.3.4.1 On the GAS screen, Depress Gas Injection button to Off (Green).
 - 7.3.5 On RECIPE screen, Depress Vent button.
 - 7.3.6 Open chamber when Vent Done button is displayed and the chamber is at ATM.
- 7.4 **Chamber preparation.**
 - 7.4.1 Update the materials to be processed on NanoFab run log.
 - 7.4.2 If not done so, replace the mylar viewport protective film.
 - 7.4.3 Inspect chamber for particles or flakes. Vacuum if present.
 - 7.4.4 On the SUBST screen, Open Substrate shutter.
 - 7.4.5 Load substrate(s) on wafer platen. No need to clip or Kapton tape samples down.
 - 7.4.6 On the SUBST screen, Close the Substrate shutter. Note any possible clearance issues.
 - 7.4.7 On SQM Crystal controller, depress XTAL life button to get life remaining on XTAL. If less than 60% XTAL life remaining, notify engineering.
 - 7.4.8 Close chamber door. Please Ensure the substrate shutter is closed.
- 7.5 **Pumpdown of Chamber.**
 - 7.5.1 On the RECIPE Screen, Depress the Pumpdown button.
 - 7.5.1.1 Push and hold the chamber door until pressure reading drops.
 - 7.5.2 Annotate the pumpdown start and end time to 5.0e-5Torr pressure.

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- 7.5.3 Monitor pressure to reach at least 5.0e-5Torr pressure for processing. Lower pressures can be used for more quality films.
 - 7.5.4 Update NanoFab run log with material information. (Target position, ID, Target thickness, pump down time, base pressure and enter the current three targets).
 - 7.5.5 Update material program on SQM-160 Crystal controller.
 - 7.5.6 Enter correct material, density, tooling factor and Z ratio parameters.
 - 7.5.6.1 Use Program & Next & rotation knob to update material parameters.
 - 7.5.6.2 Note Tooling factors for guns 1 & 2.
 - 7.5.6.3 Tooling factor for gun 3 is 399%.
 - 7.5.7 Pumpdown recipe completed at 5.0e-5Torr. Depress OK. You may continue to pump the chamber to lower pressures.
 - 7.6 Set the turbopump to 50% for your processing.
 - 7.6.1 Set Turbo speed target to 50% using gas flows
 - 7.6.1.1 On VAC screen, enter 50% Turbo speed if not at 50%. Insure you do not turn Off the turbo pump.
 - 7.6.1.2 On the GAS screen, depress Gas Injection button to ON (Green).
 - 7.6.1.3 On the GAS screen, enter 4.0 (mTorr) SETP pressure.
 - 7.6.1.4 On the GAS Screen, depress the Ar gas CNTL button to ON (Green).
 - 7.6.2 On VAC screen, monitor Turbo speed to get to 50%.
 - 7.6.3 You may use these gas and pressure settings for your processing.
 - 7.7 **RF Back Sputter Process- Optional to pre-clean your substrates.**
 - 7.7.1 Set Turbo at 50%, at selected pressure and that gas flows are ON (See section 7.6).
 - 7.7.2 Ensure the sample rotation is ON.
 - 7.7.3 Turn ON black power button on lower RF power supply
 - 7.7.4 Turn ON RF ON/OFF button. Light should be Blue when OFF. RED when ON.
 - 7.7.5 Open the substrate shutter and turn on the RF. If the plasma does not form near the sample holder, temporarily turn on DC power to a gun to ignite RF plasma.
 - 7.7.6 Slowly increase the RF power to 100W using the up-arrow key. View for cloudy plasma above the substrate to indicate RF started. Watch Timer as Sputter starts.
 - 7.7.7 Adjust the Seren manual matching Tune knob to minimize the reflected power.
 - 7.7.8 The etch rate of thermal SiO₂ is @2.2 nm/min.
 - 7.7.9 When completed, turn down RF power to 0W.
 - 7.7.10 Turn OFF RF ON/Off button and the black power button on RF power supply.
 - 7.7.11 Close substrate shutter.
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7.8 Deposition using DC Magnetron for Metallic materials.

- 7.8.1 Set Turbo at 50%, at selected pressure and that gas flows are ON (See section 7.6).
- 7.8.2 Turn ON Substrate Rotation on the lower panel On/Off switch. Rotation set 31rpm.
- 7.8.3 Determine your intended material, gun and power source for your layer(s).
- 7.8.4 On the DEP screen, Depress Flow SW button to Open on your intended gun shutter.
- 7.8.5 Select the correct DC power supply (DC 1/3 or DC 2) based on the gun you use.
- 7.8.6 Depress Power button to ON, on the DC power supply.
- 7.8.7 Depress DC power supply Output to ON, on the DC power supply.
- 7.8.8 On the Right Display button, select Actual Power and Volt parameters to view.
- 7.8.9 Slowly ramp-up Level knob to desired 1 - 2Å/sec dep rate for Metal materials.
- 7.8.10 IMPORTANT. If gun does not turn ON and has abnormally high or low voltage, please turn OFF the power supply and notify engineering staff if so.**
- 7.8.11 Watch rise in dep rate on the SQM-160 XTAL monitor.
- 7.8.12 Verify gun power operation through the chamber viewport.
- 7.8.13 It is recommended to perform target conditioning for a couple of mins.
- 7.8.14 START of DC Magnetron gun processing.**
 - 7.8.14.1 On SUBST screen, depress SBST shutter to Open shutter (Green).
 - 7.8.14.2 Depress SQM-160 controller Zero button to zero thickness.
- 7.8.15 Monitor processing through to your target thickness on XTAL controller. Watch the viewport, watch for stability of the power and voltage.
- 7.8.16 Record gun process conditions on the NanoFab run log when halfway through process (Dep Rate, Base and Dep pressure, Power, Voltage and Current).
- 7.8.17 COMPLETION of DC Magnetron gun processing.**
 - 7.8.17.1 On SUBST screen, depress SBST shutter to close the substrate shutter.
 - 7.8.17.2 Ramp down the power level on the DC power supply.
 - 7.8.17.3 Turn Off the power supply button.
- 7.8.18 If you are processing multiple layers, proceed back to step 7.6.
- 7.8.19 Turn OFF gas flows.
 - 7.8.19.1 On DEP screen, depress the gun FLOW Switch to close gun shutter.
 - 7.8.19.2 On GAS screen, Depress Gas Injection button to Off (Green).
- 7.8.20 Turn OFF the Substrate Rotation on the lower panel ON/Off switch.
- 7.8.21 Once all the layer(s) are completed, Vent the chamber. Otherwise start next layer.
 - 7.8.21.1 On the Vacuum screen, Depress Vent button on Vacuum Screen.

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- 7.8.22 Pumpdown the chamber after processing. Depress the Pumpdown button on the RECIPE screen. Push and hold the chamber door until pressure reading drops.
- 7.9 Deposition using RF Magnetron for Dielectric or ceramic materials.**
- 7.9.1 Set Turbo at 50%, at selected pressure and that gas flows are ON (See section 7.6).
- 7.9.2 Turn ON Substrate Rotation on the lower panel On/Off switch. Rotation set 31rpm.
- 7.9.3 Determine your intended material, gun and power source for you layer(s).
- 7.9.4 On the DEP screen, Depress Flow SW button to Open on your intended gun shutter.
- 7.9.5 Select Lesker RF power supply and auto match network.
- 7.9.6 Depress black Power button to ON, on both units.
- 7.9.7 Depress RF power button to turn ON. Indicator light should be Red.
- 7.9.8 Slowly ramp Up RF power using Up arrow to ensure plasma is on. Dial RF UP to the lower 0.3 to 0.5 Å/sec dep rates for dielectric or ceramic targets.
- 7.9.9 IMPORTANT. If gun does not turn ON and has abnormally high or low voltage, please turn OFF the power supply and notify engineering staff if so.**
- 7.9.10 Monitor gun output through the chamber viewport.
- 7.9.11 Watch dep rate on the SQM -160 XTAL monitor.
- 7.9.12 When dep rate and RF is stable.
- 7.9.13 It is recommended to perform target conditioning/cleaning for a couple minutes.
- 7.9.14 START of RF Magnetron gun processing.**
- 7.9.14.1 On SUBST screen, depress SBST shutter to Open shutter (Green).
- 7.9.14.2 Depress SQM-160 Zero button to zero thickness.
- 7.9.15 Record gun process conditions on the NanoFab run log when halfway through process (Dep Rate, Base and Dep pressure, Power, Voltage and Current).
- 7.9.16 COMPLETION of RF Magnetron gun processing.**
- 7.9.16.1 On SUBST screen depress SBST shutter to close the substrate shutter.
- 7.9.16.2 Depress RF power button on power supply to turn Off.
- 7.9.16.3 Depress black Power button on both units to turn Off.
- 7.9.16.4 On DEP screen, depress the gun FLOW Switch to close gun shutter.
- 7.9.16.5 On GAS screen, depress Gas Injection button to Off (Green).
- 7.9.17 Turn OFF Substrate Rotation on the lower panel On/Off switch.
- 7.9.18 Once all the layer(s) are completed, Vent the chamber. Otherwise start next layer.
- 7.9.18.1 On the Recipe screen, Depress the Vent button.
- 7.9.19 Pumpdown the chamber after processing.
- 7.9.20 On the RECIPE screen, Depress the Pumpdown button. Push and hold the chamber door until pressure reading drops.
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7.9.21 End your process on the NanoFab log system.

8. Dielectric Film Processing

8.1 Sputtered Indium Tin Oxide (ITO) using Lesker In_2O_3/SnO_2 , 90/10 wt% 3" target.

8.1.1 Pump the chamber to at least 3.0×10^{-7} Torr base pressure. Lesker1 equivalent to an overnight pumpdown.

8.1.2 Use RF sputter process with a dep rate of $0.5 \text{ \AA}/\text{min}$. Please Ensure the target is properly conditioned for a 100 \AA thickness with the substrate shutter still closed.

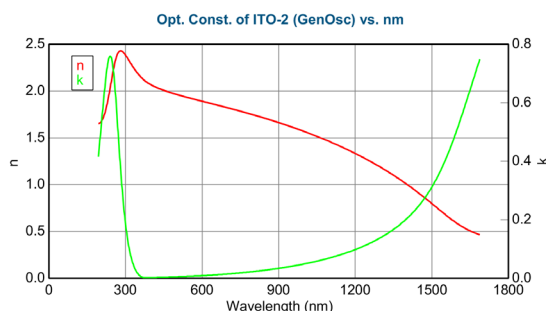
8.1.3 4mTorr process pressure.

8.1.4 No additional O_2 will be required for this process.

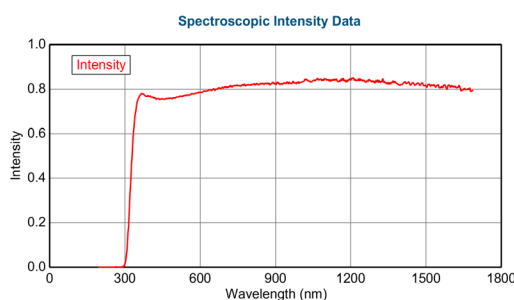
8.1.5 Expect a darkened film on glass post deposition.

8.1.6 Post ITO bake- Place your already coated glass substrate on an already heated hot plate at 300°C for 30 minutes. ITO Film will lighted up.

8.1.7 Expected 4-point probe resistivity of 65nm film is $\sim 20 \Omega \text{cm}^2$.

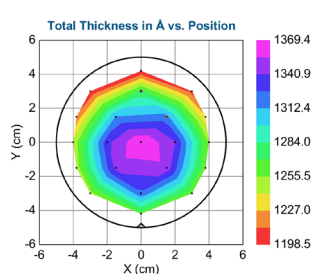


65nm film ITO Index and Absorption

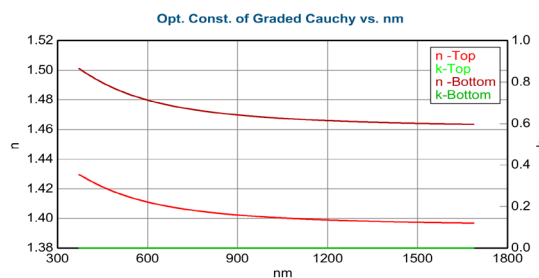


65nm film on glass ITO transmission%.

- 8.2 Sputtered Silicon Dioxide (SiO₂) using Lesker SiO₂ 3in target.
- 8.2.1 No additional O₂ gas is require during processing.
- 8.2.2 Use RF sputter process with a dep rate of 0.5Å/min. Please insure the target is properly conditioned for a 100Å thickness with the substrate shutter still closed.
- 8.2.3 4mTorr process pressure.
- 8.2.4 SiO₂ film 128nm (09/06/18) on silicon data.
- 8.2.4.1 Woollam Ellipsometer film avg Index (*n*) is 1.4458 (@632.8nm.
- 8.2.4.2 Woollam ellipsometer film avg Thickness is 1276Å.
- 8.2.4.3 Woollam Ellipsometer 21pt. non-uniformity is 13.4%.

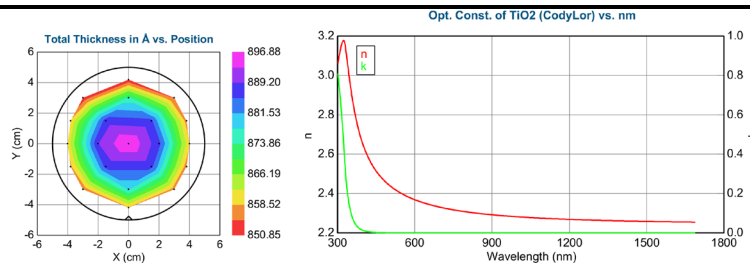


SiO₂ film thick map



SiO₂ film Graded Index(Cauchy)

- 8.3 Sputtered Titanium Dioxide (TiO₂) using Lesker TiO₂ 3in target.
- 8.3.1 No additional O₂ gas is require during processing.
- 8.3.2 Use RF sputter process with a dep rate of 0.5Å/min. Please ensure the target is properly conditioned for a 100Å thickness with the substrate shutter still closed.
- 8.3.3 4mTorr process pressure.
- 8.3.4 TiO₂ film 87nm (02/20/19) on silicon data.
- 8.3.4.1 Woollam Ellipsometer film avg Index (*n*) is 2.358 (@632.8nm.
- 8.3.4.2 Woollam ellipsometer film avg Thickness is 869.04Å.
- 8.3.4.3 Woollam Ellipsometer 21pt. non-uniformity is 4.90%.



87nm TiO₂ film thick map

87nm TiO₂ film Index and Absorption

9.0 Revision History

Effective Date	Originator	DESCRIPTION OF REVISION	Issue
04/11/06	Paul Boland	Initial Release	A
04/11/07	Jon Martin	Updates & improvements due to new materials	B
03/27/15	Clarence Tracy	Updates and change in contamination protocol	C
10/23/15	Clarence Tracy	New touch screen control system	D
11/06/18	Jaime Quintero	Checklist version of operating procedures.	E
05/21/19	Jaime Quintero	Updates, Process and Film updates	F
08/23/19	Jaime Quintero	Reservations and Cancellation policies	G