GENERAL PROCESS AND OPERATION SPECIFICATION

Lesker PVD 75 RF Dielectric Sputter

I. SCOPE
   a. The purpose of this document is to describe requirements and basic operating instructions for the Lesker PVD 75, RF Sputter tool. This tool is intended for thin film deposition of dielectric materials by RF Sputtering. Use of this tool is limited to only approved thin films and substrates.

II. SAFETY
   a. Be sure that you are trained and signed off to use this equipment.
   b. Be sure to keep all doors and protective shields in place before operating this equipment.
   c. Use care when operating around high voltage or high current.
   d. Use care when operating in RF mode of operation. Avoid coming near any electrodes or conductors carrying RF energy.
   e. High pressure gas cylinders for this tool are stored in ventilated gas cabinets located in the service corridor. Be sure to ask a staff member for assistance to change out any gases.
   f. If you are unsure about any procedure or indication while operating this equipment be sure to contact a staff member or trainer for assistance.

III. APPLICABLE DOCUMENTS, MATERIALS AND REQUIREMENTS
   a. For more information about the detailed operation of this tool refer to the Lesker factory manual – "PVD 75 Thin Film Deposition System Operation Manual." File name: PVD 75 Manual.pdf.
   b. Appendix A: Editing of Recipes in KJL Software
   c. Appendix B: RF Sputter Example Recipe
   d. Appendix C: Changing Targets
   e. Appendix D: Increasing Deposition Rate
   f. Appendix E: Maximum Power Density of Target Materials
   g. Appendix F: Bonding Targets
   h. This tool is intended to be used with a restricted selection of substrate and target materials.
   i. Approved target materials: Al_2O_3, AlN, Ge, InSb, ITO, $LiTaO_3$, MoO_3, SiO_2, Si, TiN, TaN, ZnO.
   j. Materials provided by AggieFab as part of normal lab fees: Al_2O_3, SiO_2.
   k. Approved substrate materials: Glass, Si, PVDF (with no substrate heating), PET, PS.
   l. Other substrate and target materials must be pre-approved by the Materials Review Board before running in this tool.

IV.   OPERATION
    a.   Login using username and password.
    b.   Start the "PC Vent".
        i. Ensure the three gas line valves (H_2, O_2, N_2) behind the tool are closed (turned all the way to the right) before venting.
    c.   Once PC is at atmosphere pressure, remove the substrate holder. Be careful not to move the shutters surrounding it.
    d.   Attach your substrate to the holder using Kapton tape or pins.
    e.   Place the substrate holder back into position.
    f.   Uncover and change targets as needed (see Appendix C).
    g.   Close the door and start the "Start PC Pump"
    h.   While the chamber pumps down, find the recipe you need to use.
        i.   If the recipe does not exist, copy a similar recipe and edit the copy (See Appendix A and B).
        ii.   Rename the copy to identify it as your own and press "Update VB" to save it.
    j.   Once "PC Pump" is complete, the process gas valve(s) can be opened if needed. Go behind the tool and open the appropriate valve(s) (turned all the way to the left).
    k.   Go to the "Gas" tab, then under MFC2, click on the "Corr. Factor" box and enter the appropriate value for the process gas you are using:
        i. $H_2 = 1.01$
        ii. $O_2 = 0.993$
        iii. $N_2 = 1.00$
    l.   Click "Run Recipe" (right side), choose your desired recipe, then click the green area.
        i   Running the recipe could cause the software to crash. If this happens, close the software using Task Manager then restart it.
    m.   Allow the recipe to run. Confirm that the plasma lights when the recipe reaches the ignition step.
        i   If the plasma does not light, try restarting the recipe.
        ii   If the issue persists, notify a staff member.
    n.   When the process has completed, close the process gas lines and vent the chamber. Remove your samples (Be careful! The substrate holder may be hot), cover the targets back up with aluminum foil, and pump down the chamber again. Once the chamber is pumping, you can log out.
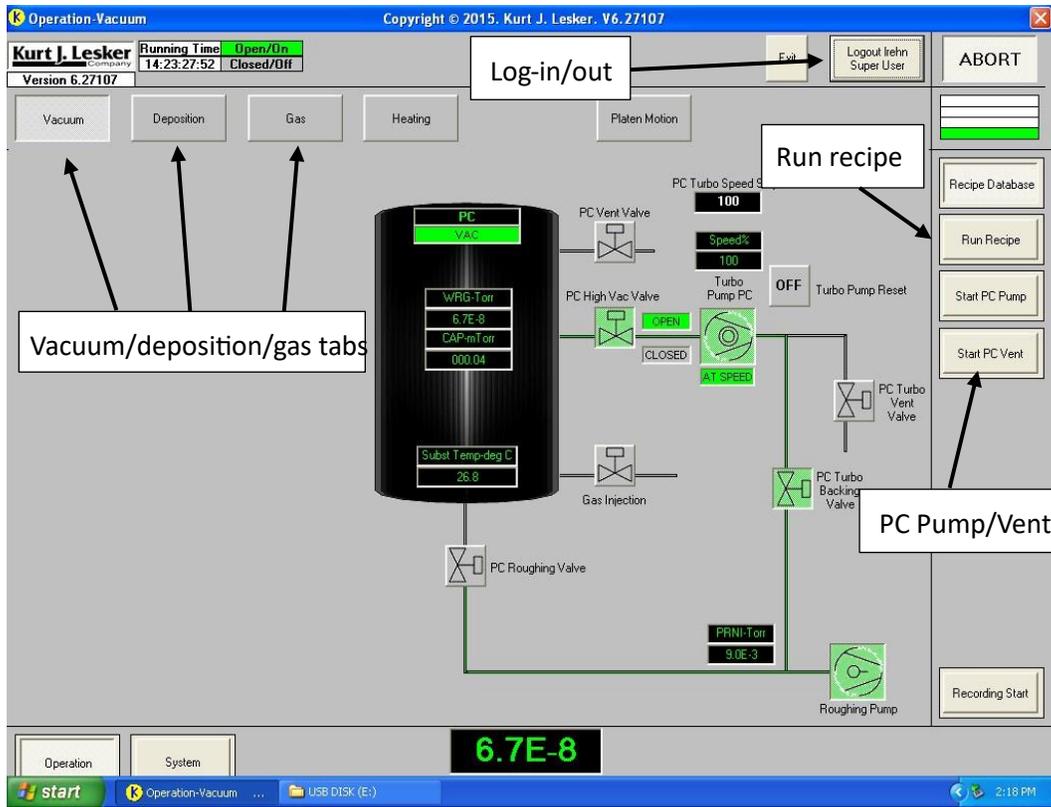
Fig. 1: Main vacuum screen showing pumps and valve positions during normal operation under vacuum.
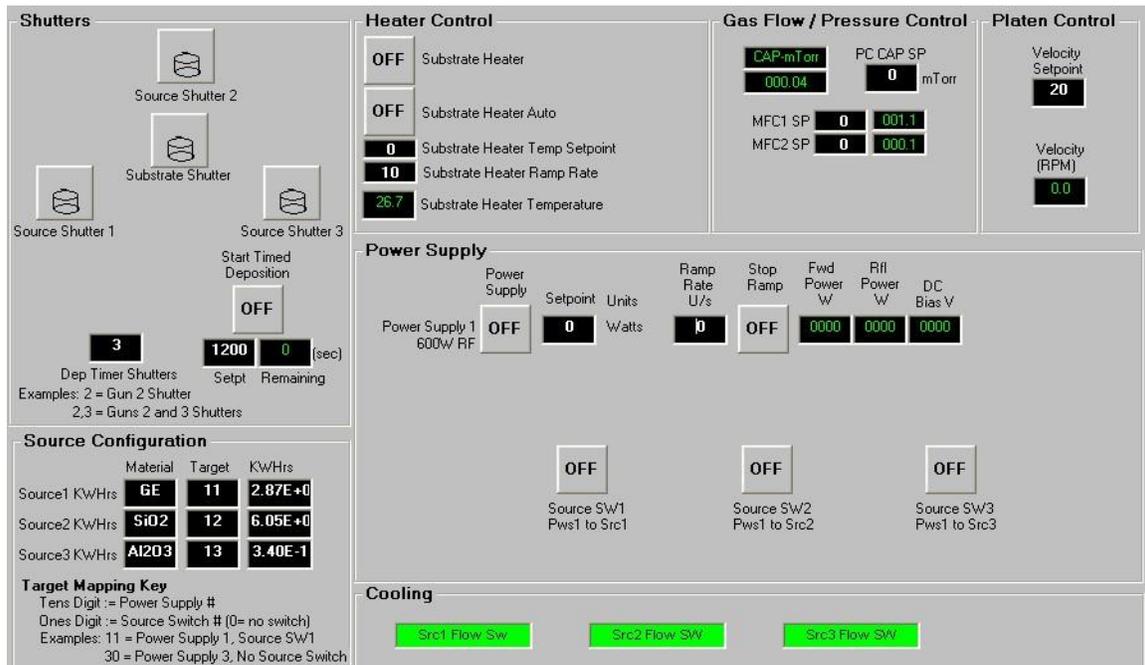
Fig. 2: The deposition screen showing shutters, source targets and configuration, heating, power supply status, gas flow, and platen (substrate holder) motion.

SIGNATURES AND REVISION HISTORY

      a.   Author of this document: Ethan Morse
      b.   Author Title or Role: Student Technician
      c.   Date: 17 March 2020
      d.   Rev. 1: Added operation steps, appendices B-F.
      e.   Rev. 2: Updated instructions to reflect current procedures

Approvals:

    Technical Manager Signature:_____

    Date: _____

Revision History:

Revision	Author	Date
Original Issue	L. Rehn	
Rev 1	Ethan Morse	17 March 2020
Rev 2	John Slentz	5 June 2023
Rev 3		

Appendix A – Editing of Recipes in KLJ Software:

Editing Recipes
Note: You can only edit recipes if you are the owner or if the owner has opened access to you for editing. However, the recipe can be copied and then edited. The owner of a file cannot be changed and only the owner or a system administrator can delete the file.
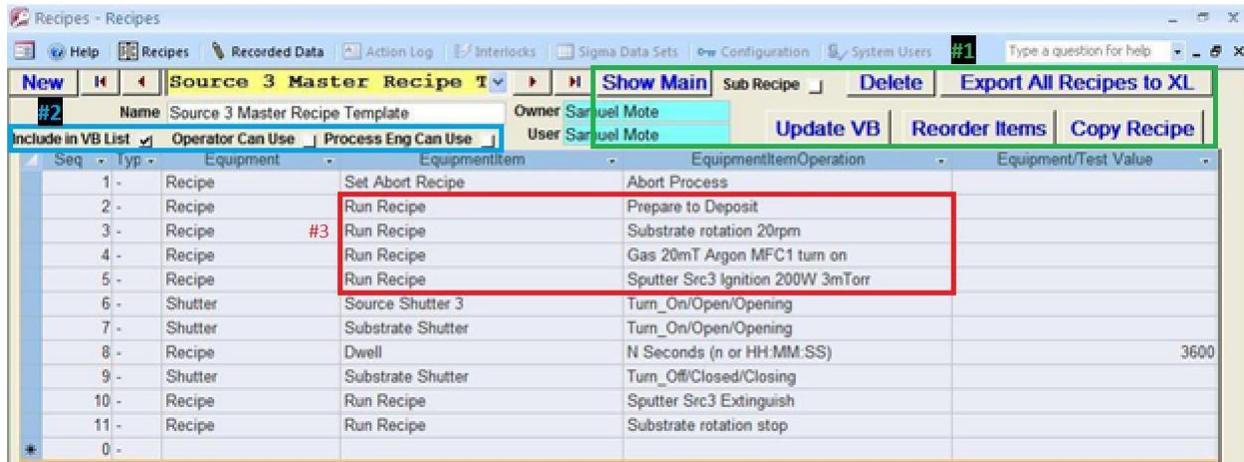
Fig. 3: Recipe editing using KJL's VB software

#1:
- Show Main/(Show All): Toggles the yellow recipe list between showing only Main Recipes or both Main and Sub Recipes. (The button displays the opposite of what is being used. For example, when the button reads "Show Main", the recipe list will be showing all.)
- Sub Recipe Check Box: Defines Recipe as a Sub Recipe; Default as Main Recipe
- Delete: Remove Recipe file
- Export All Recipes to XL: Used only by system administrators
- Update VB: Save all recipes that have been edited
- Reorder Items: Move lines of the recipe
- Copy Recipe: Create new recipe file with all of the same specifications currently selected. You will become the "Owner" of the new file.

#2:
- Include in VB List Check Box: (Default checked.) If checked, and if the recipe is a main recipe, it will be made visible in the yellow recipe list when Show Main is selected. If not checked, it will only show when Show All is selected.
- Operator /Process Eng Can Use Check Boxes: (Default unchecked.) These boxes specify what groups are given access to editing the recipe file. If unchecked, only the owner and system administrators can edit it.

#3:
If the "Equipment/Item" column reads "Run Recipe", then the next column will specify what Sub Recipe is being called. Sub Recipes contain most of the editable parameters of a process. By clicking on the Sub Recipe name, a drop-down box will appear that will allow you to select which specific Sub Recipe to run. Other "Equipment/Item" commands specify an action to be taken within the Main Recipe.

Editing Sub Recipes
Sub Recipes are where most parameters are defined.

Fig. 4: Recipe editing with the equipment/test value parameter highlighted

Equipment/Test Value: This is where the parameters of a Sub Recipe within a process are defined and edited. The same owner permissions are required as in a Main Recipe for editing.

If there are a set value and a check value for the same parameter, make sure to change both values together to keep the logic true. For example, if the power is set to 200 W and the check is set to greater than 190 W originally, and you want to drop to only 100 W set point, make sure that the check point is reduced similarly to greater than 95 or something close.

Appendix B: RF Sputter Example Recipe

Refer to the **PVD75 Operation Manual** (page 57-58) for an example and explanation of steps required for a typical DC Sputter recipe.

In general, an RF Sputter recipe will look like the following:
1. Set the abort recipe to ensure a safe abort process.
2. Rotate the substrate (optional)
3. Obtain desired sputter pressure and gas mixture, e.g. 20 mTorr with 95% Ar and 5% O2
4. Light the plasma and ramp to peak power
5. Decrease pressure
6. Clean the target surface (source shutter open, substrate shutter closed)
7. Deposit (source shutter open, substrate shutter open)
8. Turn everything off: source power, gas flows, and substrate rotation.

There are other optional steps you can add within the recipe to change the film, such as:
- Substrate heating
- Different deposition rates
- Different pressures
- Different gas environments

See the next page for detailed and explained steps of a common main recipe and its sub-recipes.

Main Recipe: Source 1 SiO₂ 5% O2 10mT

Table 1: Example of a main recipe using multiple sub-recipes

Seq	Type	Equipment	EquipmentItem	EquipmentItem Operation	Equipment/ TestValue	GRST
1	-	Recipe	Set Abort Recipe	Abort Process		
2	-	Recipe	Run Recipe	Prepare to Deposit		
3	-	Recipe	Run Recipe	Substrate rotation 20rpm		
4	-	Recipe	Run Recipe	Gas 20mT Ar /O2 MFC1/2 turn on 5% O2		
5	-	Recipe	Run Recipe	Sputter Src1 Ignition 250W SUB 10 mT		
6	-	Shutter	Source Shutter 1	Turn_On/Open/Opening		
7	-	Recipe	Dwell	N Seconds (n or HH:MM:SS)	00:05:00	
8	-	Shutter	Substrate Shutter	Turn_On/Open/Opening		
9	-	Recipe	Dwell	N Seconds (n or HH:MM:SS)	00:30:00	
10	-	Shutter	Substrate Shutter	Turn_Off/Closed/Closing		
11	-	Recipe	Run Recipe	Sputter Src1 Extinguish SUB		
12	-	Recipe	Run Recipe	Gas Supply turn off		
13	-	Recipe	Run Recipe	Substrate rotation stop		

1. Set the procedure the tool should follow if it needs to abort. This always needs to be the first item in a recipe.
2. Check chamber pressure to ensure it is low enough to begin deposition.
3. Rotate the substrate at 20 rpm. This improves film uniformity across the sample.
4. Increase chamber pressure to 20 mTorr with 95% Ar and 5% O2. [See sub-recipe explanation below.]
5. Ignite the Source 1 plasma, increase power to 250 W, then decrease pressure to 10 mTorr. [See subrecipe explanation below.]
6. Open the Source 1 shutter to begin target cleaning.
7. Clean the target for 00:05:00 (5:00 min). This sputters off any contaminants on the target surface.
8. Open Substrate Shutter to begin deposition.
9. Deposit for 00:30:00 (30:00 min).
10. Close Substrate Shutter to prevent further deposition on substrate.
11. Slowly decrease Source 1 power until it turns off.
12. Turn off the gas flow and seal the chamber.
13. Stop rotating the substrate.

Sub Recipe: Gas 20mT Ar/N2 MFC1/2 turn on 5% O2
Table 2: Example of gas flow sub-recipe using 95% Ar and 5% O2 at 20 mTorr

Seq	Type	Equipment	EquipmentItem	EquipmentItem Operation	Equipment/ TestValue	GRST
1	-	Recipe	Set Abort Recipe	Abort Process		
2	-	Valve	PC High Vac Valve Opened	Check_On/Open/Opening		AT[1]
3	-	Pump	PC Turbo Speed Setpt	Set Value = n.nn	35	
4	-	MFC	MFC1 Ratio	Set Value = n.nn	95	
5	-	MFC	MFC2 Ratio	Set Value = n.nn	5	
6	-	Valve	Gas Injection	Turn_On/Open/Opening		
7	-	MFC	MFC1 SP	Set Value = n.nn	0	
8	-	MFC	MFC2 SP	Set Value = n.nn	0	
9	-	Recipe	Dwell	2 Seconds		
10	-	MFC	MFC1 Mode	Set Value = n.nn	4	
11	-	MFC	MFC2 Mode	Set Value = n.nn	11	
12	-	Gauge	Capman Pressure SP	Set Value = n.nn	20	
13	-	Gauge	Capman Pressure	Check Pressure > n.nn	19.5	AT[2]
14	-	Pump	PC Turbo Speed	Check Value <= n.nn	37	T[3]

[1] TimeOut for Wait = 1. TimeOut Message = Hi Vac Valve Not Open. Goto Sequence No if Timeout = 999.

[2] TimeOut for Wait = 300. TimeOut Message = Failed to Reach SP in Set Time. Goto Sequence No if Timeout = 999.

[3] TimeOut for Wait = 500.

1. Set the procedure the tool should follow if it needs to abort. This always needs to be the first item in a recipe.
2. Check if the High Vacuum Valve is opened.
3. Set the turbo pump speed to 35%.
4. Set MFC1 (Ar) ratio to 95%.
5. Set MFC2 (O2) ratio to 5% (of the 95% flow rate during flowing).
6. Open the gas injection valve to allow gas to flow into the chamber.
7. Reset MFC1 flow value to 0 standard cubic centimers per minute (SCCM).
8. Reset MFC2 flow value to 0 SCCM.
9. Hold (dwell) for 2 sec.
10. Set MFC1 to Mode 4. Mode 4 will flow the required amount of gas to match the capman pressure setpoint. Since the capman setpoint is 0, the flow is 0.
11. Set MFC2 to Mode 11 (one-one). Mode 11 on MFC2 will slave MFC2 to MFC1, so MFC2's flow will be 5% of MFC1's.
12. Set the capman pressure to 20 mTorr. MFC1 will begin flowing Ar and MFC2 will begin flowing O2.
13. Check if the capman pressure is >19.5 mTorr. If yes, it is essentially 20 mTorr and will continue.
14. Check if turbo pump speed is low enough to begin plasma ignition.

Sub Recipe: Sputter Src1 Ignition 250W SUB 10 mT

Table 3: Example of plasma ignition sub-recipe with 250 W peak power and 10 mTorr pressure

Seq	Type	Equipment	EquipmentItem	EquipmentItem Operation	Equipment/ TestValue	GRST
1	-	Recipe	Set Abort Recipe	Abort Process		
2	-	Power Supply	Power Supply1 Output Setpoint	Set Value = n.nn	0	
3	-	Source	SW1	Turn_On/Open/Opening		
4	-	Power Supply	Power Supply 1	Turn_On/Open/Opening		
5	-	Power Supply	Power Supply1 Output Setpoint	Set Value = n.nn	50	
6	-	Power Supply	Power Supply1 Output Voltage	Check Value > n.nn	20	AT[1]
7	-	Power Supply	Power Supply1 Ramp Rate	Set Value = n.nn	2	
8	-	Power Supply	Power Supply1 Output Setpoint	Set Value = n.nn	250	
9	-	Power Supply	Power Supply1 Output Power	Check Value > n.nn	230	AT[2]
10	-	Gauge	Capman Pressure SP	Set Value = n.nn	10	
11	-	Gauge	Capman Pressure	Check Pressure < n.nn	10.5	

[1]   TimeOut for Wait = 5. TimeOutMessage = PWS1 Plasma Not On. Possible short circuit, pressure too low, etc. Goto Sequence No if Timeout = 999.

[2]   TimeOut for Wait = 200. TimeOutMessage = PWS1 Power < 250 W. Goto Sequence No if Timeout = 999.

1. Set the procedure the tool should follow if it needs to abort. This always needs to be the first item in a recipe.
2. Set power supply power to 0 W, in case it was non-zero before.
3. Turn on Switch 1 (SW1). Because all three sources (S1, S2, S3) are connected to PWS1, a switch needs to be on to designate which source to power. SW1 will allow Source 1 to be turned on.
4. Turn on Power Supply 1 (PWS1). No power is being applied yet.
5. Set PWS2 output to 50 W. This will ignite the plasma.
6. Check output voltage. If less than 20 V, there is probably no plasma. Lack of plasma can be due to a short circuit or the pressure being too low. Sometimes there is a plasma even with 0 V.
7. Set the PWS1 ramp rate to 2 W/s. See suggested ramp rates in Appendix E.
8. Set final PWS1 power to 250 W. It should take $(P_{final} - P_{initial})/(\text{ramp rate}) = (250-50)/2 = 100$ sec to reach the peak value.
9. Check if PWS1 power is greater than 230 W. If yes, it is close enough to 250 W that the pressure can be lowered.
10. Set capman pressure to 10 mTorr. The pressure will immediately begin to decrease.
11. Check if capman pressure is less than 10.5 mTorr. If yes, continue to next step (this will go back to the main recipe).

Appendix C: Changing Targets (pp 63-64 from the PVD75 Operation Manual):

TARGET CHANGING

Installing/changing targets:

1. Loosen the 3 screws using a hex wrench supporting the dark space shield (DSS) and remove the shield (see Figure 7).
2. Remove the 4 screws using a flathead screwdriver and remove the rings. Be careful not to drop the screws or rings.
3. Remove the target and place it back in its box.
   a. When removing a target of magnetic material, carefully slide the target to one side and pick it up (do not attempt to pry magnetic targets from the cooling well. This may result in permanent damage to the cooling well).
4. Place a new target on to the source, ensuring all parts are perfectly clean.
   a. If installing a magnetic target, take extra care that fingers or parts of a glove do not get pinched between target and source as the magnets are very powerful.
5. Tighten the 4 hold-down ring screws evenly. Do NOT overtighten – this can break the target.
6. Place the DSS back on. The DSS has three machined slots, which ensure .080" dark space on top of the 1/8", 3/16", and 1/4" targets. Position the DSS on the smallest slot for thick targets and one of the larger slots for thin targets.
7. Tighten the screws evenly and enough to prevent the DSS from moving.
8. After installing a target, you can check for a short by running the recipe "Source [number] Short Test". Take a multimeter and place one lead on the target and the other on the DSS. This should be an open circuit.
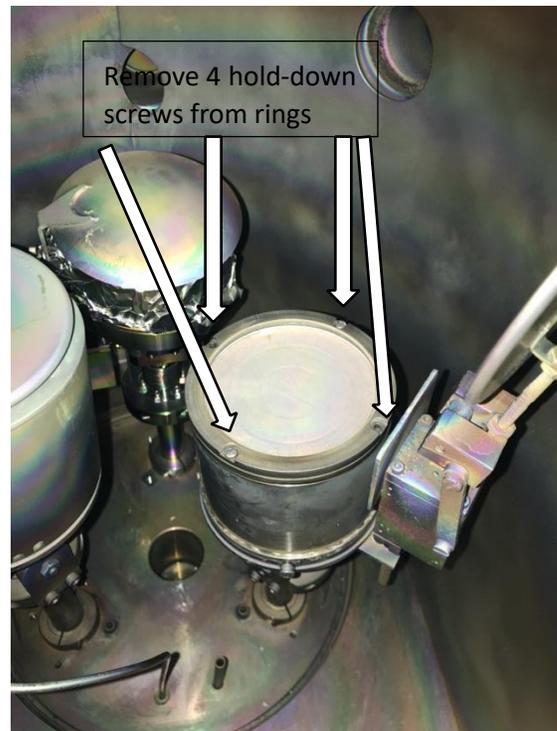
Fig. 5: Sputter gun with DSS on (left) and DSS off (right).

Appendix D: Increasing Deposition Rate

There are three ways to increase the sputter deposition rate: decreasing throw distance (distance from target to substrate), increasing power, and decreasing chamber pressure. Decreasing Throw Distance

The deposition rate and throw distance are related by the inverse square law: Rate = 1/(distance^2). While the substrate position cannot be changed, the target position can be moved up. Ask a staff member to help you with this. There are a few advantages and disadvantages of shorter throw distance.

Advantages:

- Reduction in tensile stress of growing film
- Improved film adhesion
- Higher density films Disadvantages:

- Worse film thickness uniformity
- Substrate melting due to higher heat (caused by target proximity)
- Increase in compressive stress of growing film
- Substrate outgassing
- Underlying film damage due to electron bombardment
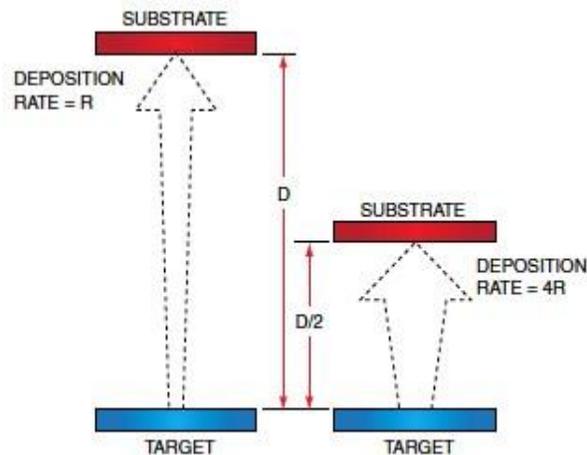
Increasing Power

Increasing power increases the accelerating voltage of ions, which increases the number of atoms "knocked off" of the target for each incoming ion (called sputter yield).

Maximum power density must be considered, or else damage to the target, sputter gun, and possibly substrate may occur. You can find maximum power density of DC-sputtered materials in Appendix E. It is important to not go near the maximum power, but rather 10-20% below it to prevent possible damage.

Decreasing Chamber Pressure

Decreasing chamber pressure will cause an increase in deposition rate for two reason:

1. The mean free path within the chamber is less, so more sputtered atoms will reach the substrate surface.
2. Ion accelerating voltage will increase as pressure decreases, causing a larger sputter yield.

Appendix E: Maximum Power Density of Target Materials

This is a guideline only These are initial maximum power levels and power densities. This is not to replace common sense. If you see a target overheating or the results of a target overheating do NOT increase the power further. Also, if the target survives a max power limit the max power can be increased over these values. This chart Serves ONLY to get you started. It is always a BAD idea to work at the maximum rated power.

Maximum power densities will be determined by the target material and how it is made into the target shape, the melting point for that material, the thermal conductivity of that material, the efficiency of the cooling circuit in the sputtering gun, how the target is fixed into the sputtering gun, and structural strength of the target.

Target Material	Max Power on Torus-3	Suggested Ramp Rate
Indium Tin Oxide	140 Watts	0.3 W/s
Titanium Dioxide	140 Watts	0.3 W/s
Zinc Oxide	140 Watts	0.3 W/s
Zinc Alumina Oxide	140 Watts	0.3 W/s
Silicon Dioxide *	400 Watts	0.3 W/s
Aluminum Nitride	140 Watts	0.3 W/s
Aluminum Oxide	140 Watts	0.3 W/s
Lithium Niobate	140 Watts	0.3 W/s
Silicon Nitride	140 Watts	0.3 W/s

* Do not bond SiO2. It is best to run unbonded to achieve higher power levels.

Appendix F: Bonding Targets

Some insulator targets are bonded to a copper (Cu) backing plate to allow them to be used even after they crack due to high power, since unbonded targets cannot be run after cracking.

Two types of bonding materials are used: indium (In) and elastomer. In has a relatively low melting point of 157 °C, which is easily reached when power is applied to the target. In is used because most materials are relatively unreactive at 157 °C and there will be no diffusion of In into a target. In's ductility also allows for thermal expansion of the target and copper backing plate, preventing target cracking. Elastomer can also be used, but poses possible contamination issues when heated over 200 °C.

Despite careful ramping procedures and no chamber heating, In has been found to melt and flow out the sides, causing a premature short circuit. If this occurs, use a sharp razor to carefully trim the excess In off and try again. If the Cu backing plate and target become separated, you cannot use the target anymore.

Targets generally crack due to poor thermal conduction, large temperature gradients from the front side to the back side (front side is significantly warmer than the back side), and high clamping force of the rings (see Appendix C's Fig. 5b). The only way to improve thermal conduction is to ensure the Cu plate sits as flat as possible on the Cu cooling well (the top of the sputter gun).
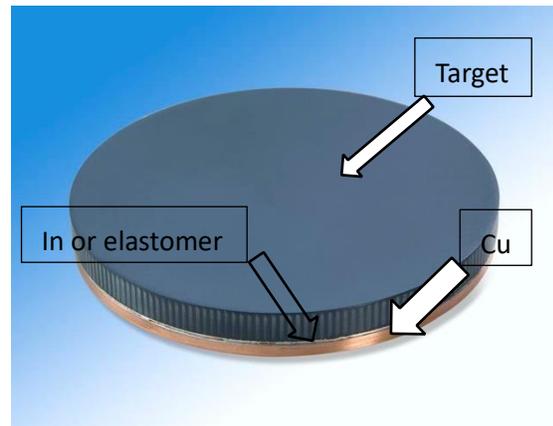
Lesker's chief process engineer recommends to run targets unbonded unless they absolutely have to be bonded. The ways to minimize target cracking are:

1. Low ring clamping force. Turn these barely to tight such that the target doesn't move.

2. Use the recommended ramp rate or lower. Thermal shock can occur, cracking the target.

3. Use a power well below the peak power of 140 W.

Contact an AggieFab staff member if you have any questions about target bonding.