GENERAL PROCESS AND OPERATION SPECIFICATION

Lesker PVD 75 E-beam Evaporator

I. SCOPE

a. The purpose of this document is to describe requirements and basic operating instructions for the Lesker PVD 75 Electron-Beam Evaporator. This tool is intended for thin film deposition by e-beam evaporation. Use of this tool is limited to only thin films and substrates that are approved.

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. After power has been disconnected, wait for at least 3 minutes before starting work on the power supply to allow the capacitors to discharge themselves. Use the grounding rod to discharge the capacitors and high voltage feed thru before coming in contact with them. Touch the grounding rod to contacts around the e-gun before performing any work to change crucibles or clean the e-gun surfaces.

e. Use the provided goggles to view inside the chamber during deposition.

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: Table of Recipes, Gun Positions, and Materials

c. Appendix B: Changing the Crystal Monitor

d. Appendix C: How to manage changing the thermal conductivity of the system

e. Appendix D: Changing the Gun Profile

f. Appendix E: Graphite Spacers

g. Appendix F: Shutter Delay Examples

h. Appendix G: Aluminum deposition conditions

i. This tool is intended to be used with a restricted selection of substrate and evaporation materials.

j. Approved evaporation materials. Materials provided by AggieFab as part of normal lab fees are: Al, Cr, Cu, Ti, Ni. For a complete list of other approved materials refer to Appendix A in this document.

k. Approved substrate materials: Glass, Si, PVDF (with no substrate heating).

l. Other substrate and evaporation materials must be pre-approved by the Materials Review Board before running in this tool.

IV. OPERATION

a. Log in by clicking the button on the top right of screen.

b. Click "PC Vent" on the right of screen.

i. Ensure the HV power supply switch is turned off by opening the right door and turning OFF the green switch near the floor.

ii. Click "Yes" when this is done.

iii. Wait for the "Recipe Complete" screen before opening the chamber.

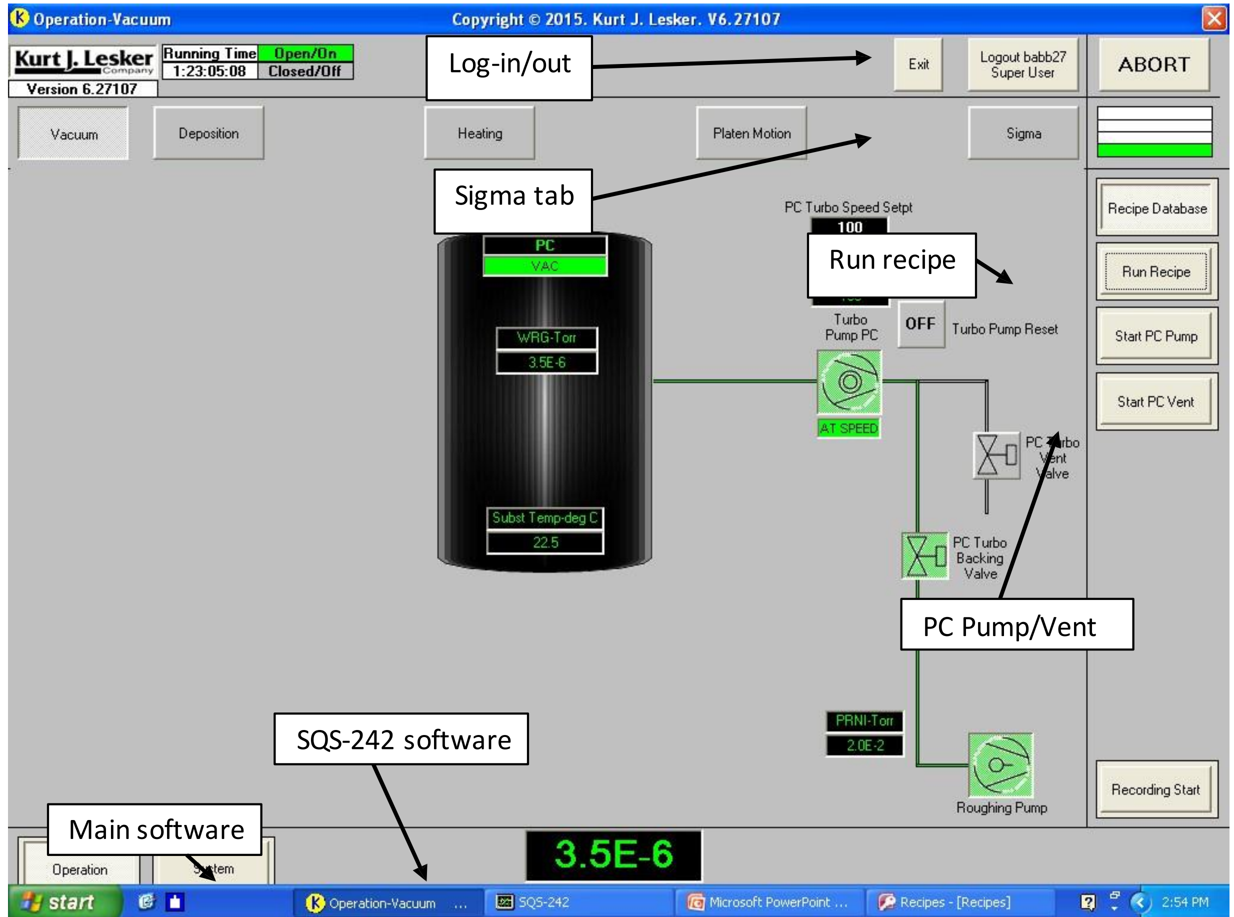


Fig. 1: Main software (CWare) homescreen with major functions labeled

c. Before opening the process chamber (PC) door, hold the grounding wand (left side of tool) in your hand to ensure you remember to use it.

d. Open the door and discharge five places: (See Fig. 2)

i. Two wires underneath the main platform.

ii. Three plates of the main platform.

iii. Other places as wanted (side shields, door shield, etc).

e. If the substrate and target shields do not open, click "Run Recipe" then run the "Load Unload" recipe.

f. Remove the substrate holder and mount your sample: (See Fig. 2)

i. Kapton tape or clips can be used to adhere the sample to the holder.

ii. After adhering the sample, turn the holder upside-down with your hand underneath and shake it gently to ensure the samples will remain there.

g. Place the substrate holder back.

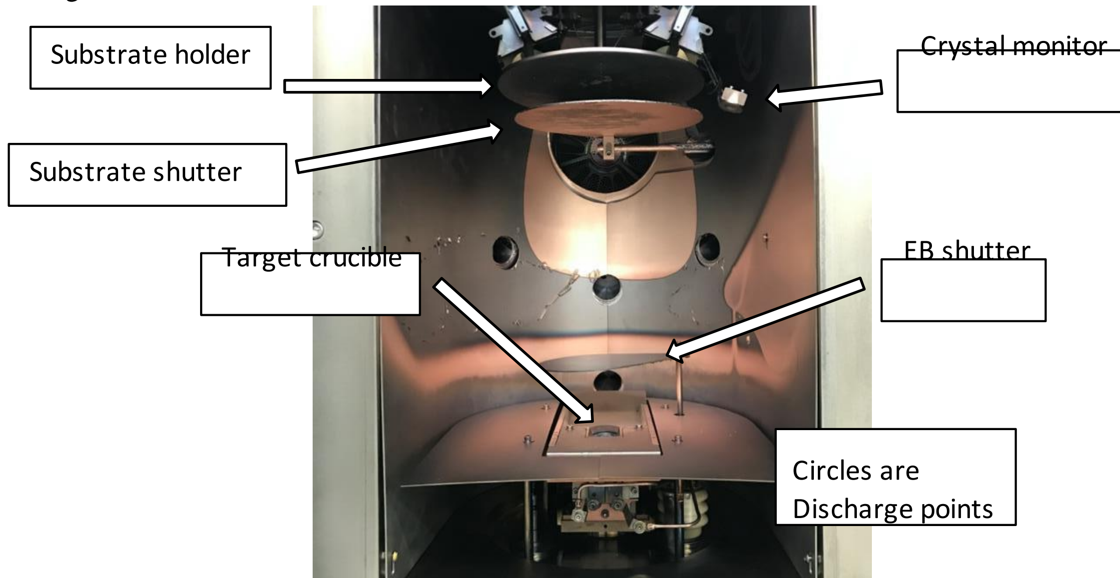


Fig. 2: Process chamber with major functions labeled

h. Prepare target crucible

i. Open the right side door to get to the EB gun controller.

ii. Use the left joystick to go to "Auto/Manual", then use the right joystick to change to "Manual Operation".

iii. Use the left joystick to go to "Set Pocket", then use the right joystick to go to the pocket(s) the materials will be depositing from.

iv. Hold the right joystick to the right while the correct pocket is highlighted. The material name will now say "-Search-" and the crucible indexer will be moving. Hold until "-Search-" is replaced by the new material name.

Fig. 3: EB gun controller screen. Left joystick controls "Set Pocket" and "Auto/Manual" (left side) and right joystick controls the Pocket/Material/Data (right side).

v. Remove the current crucible using tweezers, then wet a cotton swab with IPA and swab the copper part of the pocket. Vacuum the pocket as needed.

vi. Place graphite spacers into the pocket underneath the crucible if needed. Reference appendix E for graphite spacer information and recommendations.

vii. Check crucible for cracks and add target material (if needed). **The maximum crucible fill rate is 75%.**

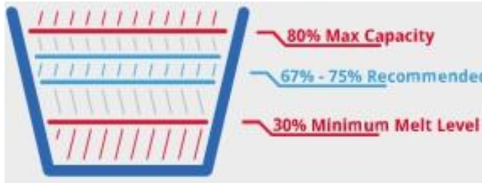


Fig. 4: Fill rate levels

viii. Place the desired crucible back in. This can be a new material or the crucible that was just removed.
ix. Repeat as needed for other pockets.
x. Go back to Pocket 1.
xi. Use the left joystick to go to "Auto/Manual", then use the right joystick to change to "Automatic Operation".

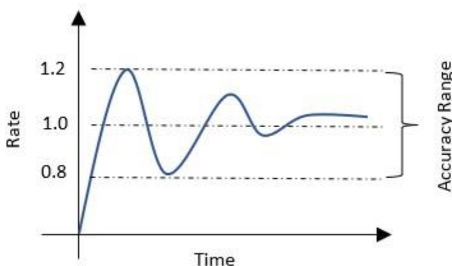
i. Check crystal monitor life:
   i. Select the "Sigma" tab near the top of the screen, then look for "Xtal Qual %". If this is under 70%, change the crystal monitor. Go to Appendix B for instructions on changing the monitor.

j. Check the window film:
   i. Open the window shutter and check if the film is transparent. If it is not, replace with a transparent one.

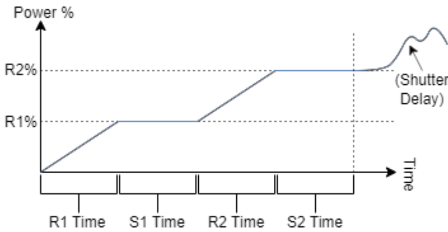
k. Select "Yes" to the "Are you finished loading" recipe step.

l. Click "PC Pump" on the right side of screen.
   i. Push on the PC door to ensure it seals properly.

m. Set up material parameters:
   i. Press ALT+TAB on the keyboard to get to the "SQS-242" application.
   ii. Click "Edit" on top left, then "Process".
   iii. Use the dropdown menu to select which material you will be depositing.
      1. Note: Chromel is Cr-Ni alloy, not Cr. Alumel is Al-Ni alloy, not Al.
   iv. There are a lot of input fields in the edit window, but only a few you need to worry about. Those fields are listed below.
   v. On the "Layer" tab:
      1. "SetPt" is the rate in A/s that the tool will deposit the material (Note: lower thickness films should use a lower rate to improve film quality)
      2. "Final Thick" is the final thickness of the thin film.
   vi. On the "Deposit" tab, the deposition rate stabilization (Shutter Delay) phase can be set up:
      1. "Accuracy" is the range of values the deposition rate can be within to be considered stable (Ex: If the dep. rate setpoint is 1.0 A/s and the shutter accuracy is 20%, the dep. rate can be between 0.8-1.2 A/s to be considered stable. 15-30% is recommended).
      2. "Hold" is the time the dep. rate needs to be within the accuracy range to move on to the actual deposition step. 5-10 sec is recommended.
      3. Note: either decreasing accuracy, increasing the hold time, or both will improve dep. rate stability.



vii. On the “Condition” tab, the Pre-Conditioning phase can be set up:



1. “Ramp1 Pwr” and “Ramp2 Pwr” are the power percentages the EBE will use during the Pre-Conditioning phase. “Ramp2 Pwr” should be set to a percentage that will properly heat up and melt the material and “Ramp1 Pwr” should be less than “Ramp2 Pwr”
2. “Ramp1 Time” is the time the machine takes to get from 0% power to [Ramp1 Pwr]%. “Ramp2 Time” is the time the machine takes to get from [Ramp1 Pwr]% to [Ramp2 Pwr]%.
3. During “Soak1 Time” and “Soak2 Time” the beam stays constant at [Ramp1 Pwr]% and [Ramp2 Pwr]% respectively. The soak times are necessary for the material to properly melt.
4. Recommended values for the Pre-Conditioning parameters are shown in Appendix A.

viii. Do not make any changes to the “RateRamps,” “Source/Sensor,” or “Error” tabs.

ix. After all parameters have been set, click the red X at the top. Select “Yes” if it asks you if you want to set “[MATERIAL] Process” as operating process.

n. Go back to the CWare software and wait for the “PC Pump” recipe to complete, i.e. the chamber pressure reaches 5.00E-6 Torr.

o. When “PC Pump” completes, open the right door and turn on the green HV power supply switch near the floor.

p. Click “Run Recipe” on the right of screen.

q. Choose the recipe you would like to run and click the green area: P[x] [material] [rotation option].

i. P[x] indicates the pocket the material will be deposited from.

ii. [Material] is the material that will be deposited.

iii. If “no rotation” is included in the recipe name, the substrate will not rotate. Else, the substrate will rotate at 20 rpm.

r. Click “Yes” to both questions asking if the HV power supply switch is on and if SQS-242 is launched.

s. Open SQS-242 and watch the following phases. Fill out the logbook while this is occurring.

i. <u>Pre-Conditioning:</u> The material will go through four phases of power ramping as described above to prepare the material for deposition.

1. Ramp 1: Power increases to [Ramp 1 Pwr]% in [Ramp 1 Time] sec.
2. Soak 1: Power remains constant at [Ramp 1 Pwr]% for [Soak 1 Time] sec.
3. Ramp 2: Power increases to [Ramp 2 Pwr]% from [Ramp 1 Pwr]% in [Ramp 2 time] sec.
4. Soak 2: Power remains constant at [Ramp 2 Pwr] for [Soak 2 Time] sec.
5. Check on the material and E-beam throughout the power ramping stage by looking through the window. Ensure the material is heating up and the beam stays lit at each ramp and soak step.
6. Ensure the E-beam is only hitting the material. Refer to Appendix D if it is hitting the crucible or the crucible indexer.

ii. Shutter Delay: This is the deposition rate stabilization phase. The bottom shutter (E-beam shutter) opens to allow the crystal monitor to measure deposition rates. If the deposition rate is too low, the software will automatically increase power to increase deposition rate. Once the deposition rate is considered stable (based on the "Accuracy" and "Hold" parameters in the "Deposit" tab), it will begin deposition onto the substrate.

iii. Deposition: Both shutters (EB and substrate) are now open and there is a clear path between the target material and substrate.

1. In the logbook's column "Power %", note down the power the material is depositing at.

iv. Post-deposition power ramping (turning off): The power will slowly decrease back to 0.0% and hold there to ensure proper cooling.

t. When the "P[x] [material]" recipe is complete, there are two options:

i. To vent the chamber and retrieve your substrate, click "PC Vent" and continue to the next step.

ii. To deposit another material, edit that material's process (as detailed above) and run the respective "P[x] [material]" recipe.

u. Turn off the HV power supply switch and click "Yes" to the question asking if it is turned off.

v. The PC vent process will take about 20 min to complete. Wait for the "Recipe Complete" screen before opening the chamber.

w. Hold the grounding wand in your hand and open the door to discharge the five places as before.

x. Remove substrate holder and sample(s).

y. Remove personal crucible if needed. Go back to Pocket 1 when finished.

z. Place empty substrate holder back.

aa. Click "Yes" to the load/unload question.

bb. Click "Run Recipe", then choose "Post Process Roughing".

cc. Log out of the tool by clicking username in the top right of screen, then "Log out".

VI. SIGNATURES AND REVISION HISTORY

a. Original author of this document: Larry Rehn
b. Original author Title or Role: AggieFab technical manager
c. Date of original: 11 September 2015
d. Revision B notes: Complete rewrite. Changed verbiage to be more succinct. Updated words/steps to reflect GERB protocols. Deleted unnecessary information.
e. Revision C notes: Added Appendix C
f. Revision D notes: Changed recommended gun profile for Ge and Si to "SI10KV" from "META10KV" in efforts to prevent crucible cracking.
g. Revision E notes: Minor changes to verbiage. Minor changes to Appendix A. Created "accuracy" and "hold" graph for visualization purposes.
h. Revision F notes: Added Appendix D, updated/reworded a few procedure steps, updated Appendix A
i. Revision G notes: Added Appendix E and Appendix F, added a procedure step for checking for graphite spacers
j. Revision H notes: Added Appendix G with update Al deposition conditions.

Approvals:

Technical Manager Signature: *Sandra G Malhotra*

Date: *6/7/2024*

Revision History:

Revision	**Author**	**Date**
Original Issue	L. Rehn	
Rev A	L. Rehn	11 September 2015
Rev B	Ethan Morse	20 January 2019
Rev C	Sandra Malhotra	17 March 2021
Rev D	Marcelo Pier	10 May 2021
Rev E	Marcelo Pier	18 May 2021
Rev F	John Slentz	24 October 2023
Rev G	John Slentz	6 February 2024

Appendix A: Recipes, Gun Positions, and Materials

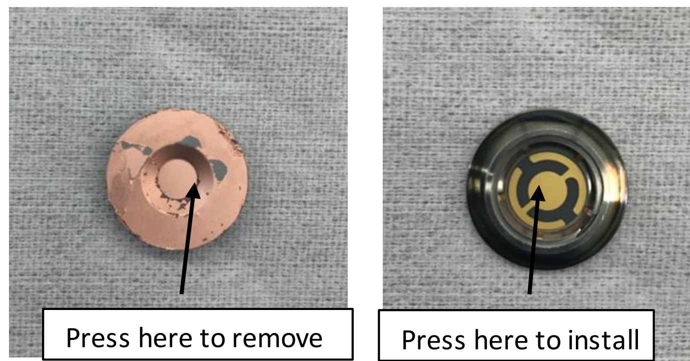
Material	Gun Pocket	Gun Profile	Ramp 1	Ramp 2
			Power/Soak/Ramp	Power/Soak/Ramp
Ag*	3	ALP310KV	6/30/60	12/60/180
Al (existing crucible/melt)	3	ALP310KV	2/60/60	4/60/60
Al (new crucible)	3	ALP310KV	3/60/60	5/60/60
Au	4	P4_M10KV	3/60/90	6/60/90
Cr	1	P1_M10KV	0.7/30/45	1.2/30/45
Cu	2	META10KV	2/90/90	4/90/90
Fe*	4	META10KV	2/30/30	4/60/180
Ge*	3	SI10KV	5/150/150	10/120/120
Mo*	4	META10KV	8/30/30	12/30/30
Ni*	2	META10KV	5/60/60	10/60/60
Pt*	4	META10KV	15/30/90	25/60/90
Si*	4	SI10KV	2/60/30	4/60/30
SiO2*	4	SIO2_6KV	3/30/60	4/30/60
Ta*	4	META10KV	20/60/30	25/60/90
Ti	2	META10KV	5/30/30	8/30/60
TiO2*	4	OXIDE6KV	9/30/60	13/30/60

*Not tested since 2021

Appendix B: Changing the Crystal Monitor

The EBE uses a 6 MHz crystal monitor (CM) to measure deposition rate and total thickness. As material builds, the monitor's frequency decreases, causing the accuracy to be worse. Monitors with less than 70% life remaining should be replaced with a new monitor.

1. Check the CM life by going to the "Sigma" tab, then looking for "Xtal Qual %". If this is less than 70%, change the CM.
2. Open the chamber door and locate the CM housing near the top of the chamber (right side on EBE1, back side of EBE2).
3. Pull out the circular piece (housing) that holds the CM.
4. Pop the CM out by pressing your thumb on the small circle (the side that was facing down before you pulled it off).
5. Dispose of the old CM in the trash.
6. Place a new CM into the housing and gently seat it using your thumb. The patterned side should be facing up and the all-gold side facing down.
    a. New CM are located in drawer near EBE1. They are in a circular case with the brand name Inficon.
7. Place the housing back into the frame.
8. Check the CM life to verify it is new (>95%).



Appendix C: When changing the thermal conductivity of the system

When you change the thermal conductivity of the system (material, crucible liner, hearth pocket) you must redefine the Ramp2/Soak2 power setpoint. Thermal conductivity of this system determines at what power you get a given rate. When you place a spacer under the crucible, for example, you intentionally limit the thermal conductivity (by adding another thermal interface). As a result, you cannot use the same recipe with the same ramp/soak levels because now it will take less power to make rate. Any time you make changes like adding or removing a spacer you must redefine the ramp/soak power levels.

This is done by running the material up in power until you get the desired rate (1 A/sec) and noting that power. That power then becomes the new Ramp2/Soak2 power level. This way when you are going from the manual power ramp/soaks into the Shutter Delay phase where the PID loop kicks in to control rate using the quartz crystal monitor you will not overshoot or undershoot the rate by too much and you will not waste material.

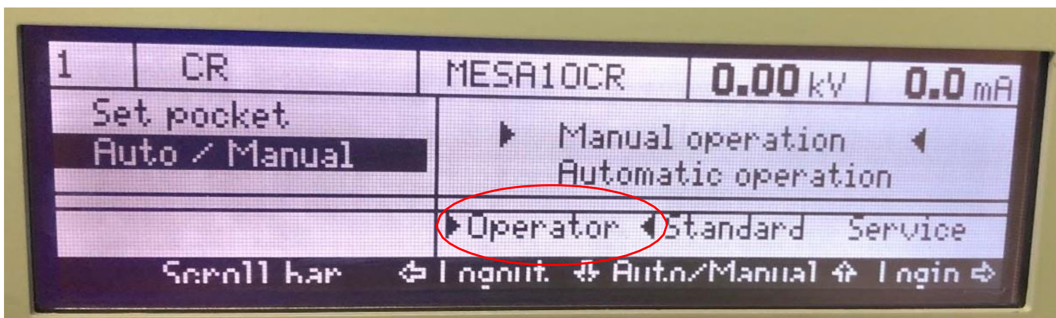
Any time there is a change to the thermal conductivity of the e-gun pocket system (like adding a spacer or removing a spacer or changing the high voltage) the Ramp2/Soak2 power needs to be manually determined and fed back into the recipe.

Appendix D: Changing the Gun Profile

If the E-beam is not centered on the material, it may hit the crucible which will cause cracks to form and decrease the crucible's lifespan. Whenever the E-beam is lit, make sure it is only hitting the material. The gun profile should be changed while the beam is on. It is recommended to change the profile during the power ramping phase since changing the profile during the deposition phase can have various effects on the deposition rate.

To change the gun profile, follow these steps:

1. Open the right-hand door and grab the gun controller.
2. Use the left joystick to navigate to the "Auto/Manual" menu.



3. If "Operator" is selected (as shown in the picture above) you will need to login. If "Service" is selected, skip to step 5.
4. Push the right joystick to the right to login. Use the password 2031 to enter service mode. DO NOT USE THE PASSWORD 3210! This could reset the gun controller and delete a lot of data.
5. Leave the controller in automatic operation.
   a. If it is switched to manual, the beam will turn off.
6. Once you are logged in, use the left joystick to navigate to the "Set Data" menu. Once it is highlighted, push the left joystick to the right to enter the "Set Data" menu.
7. When you are in the "Set Data" menu, use the left joystick to navigate between the settings and the right joystick to adjust each setting.

   a. "Position" is used to adjust the location the beam hits.
   b. "Amplitude" is used to adjust the width and height of the beam.

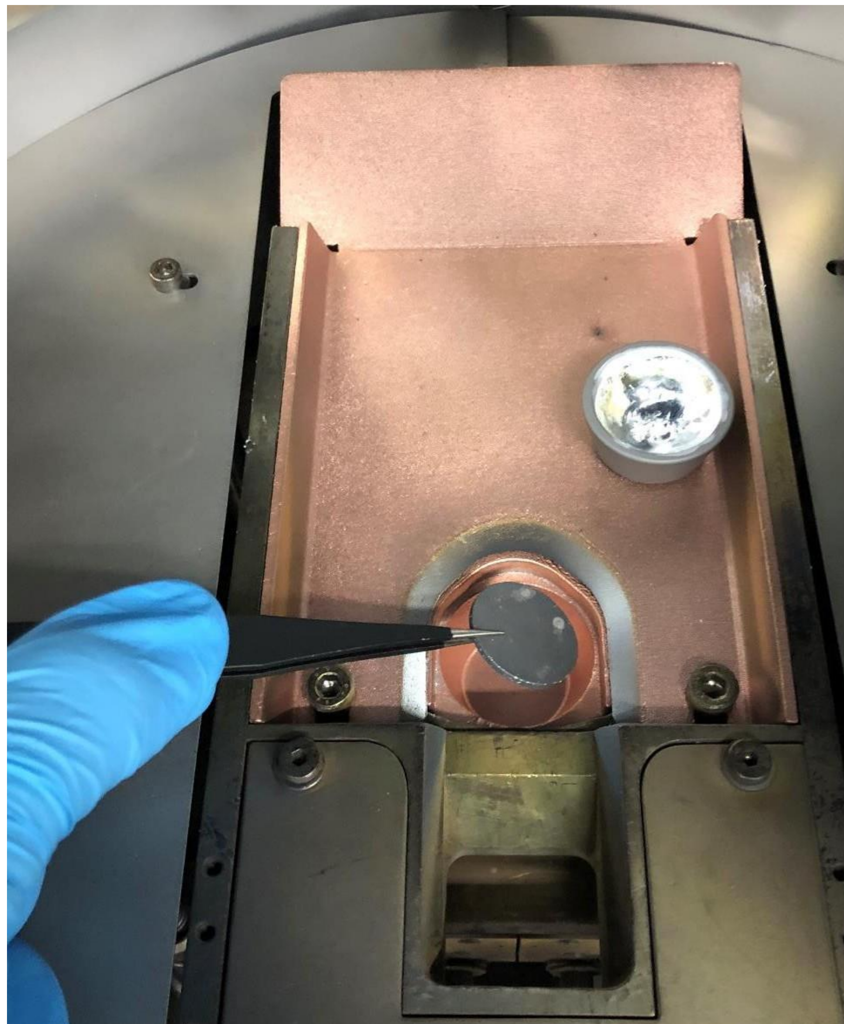
   c. Please only change these two settings. Do not adjust the other settings.
8. While you change the position and amplitude of the beam, be sure to watch the beam through the window and visually confirm the beam is not hitting the crucible or crucible indexer.
9. Once the beam has been adjusted so that it is only hitting the material, use the left joystick to navigate to "Save / Load" and then "Save Data"
10. Push the right joystick to the right to save the profile.

Appendix E: Graphite Spacers

Graphite spacers (also called shims) are vital to ensure your material will heat up enough to be evaporated and deposited onto your sample. They sit between the crucible and crucible indexer. The crucible indexer is water cooled, so if a material that needs a spacer does not have one during a process, the material might not heat up enough to evaporate. To the right are some spacer recommendations. If the material being used is not present, use 1 spacer. Spacers are located in the small bin labeled "SPACER" on the shelf next to the Lesker tools.

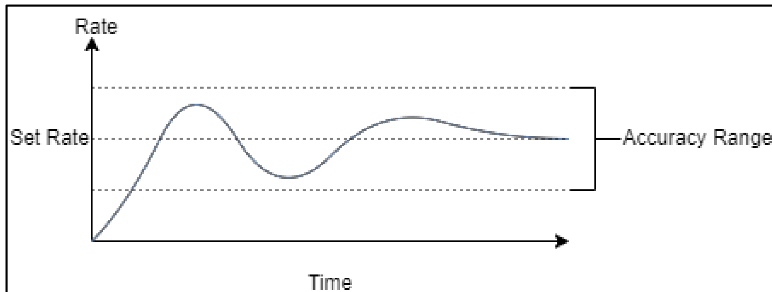
Material	Number of Spacers
Chromium	0
Aluminum	1
Titanium	1
Gold	2
Any other material	1

Below is a picture of a staff member placing a graphite spacer into the crucible indexer:



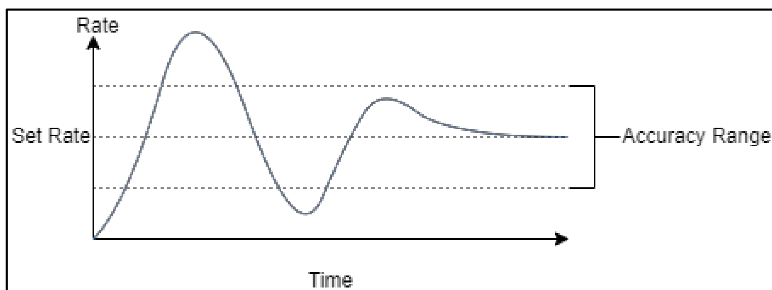
Appendix F: Shutter Delay Examples

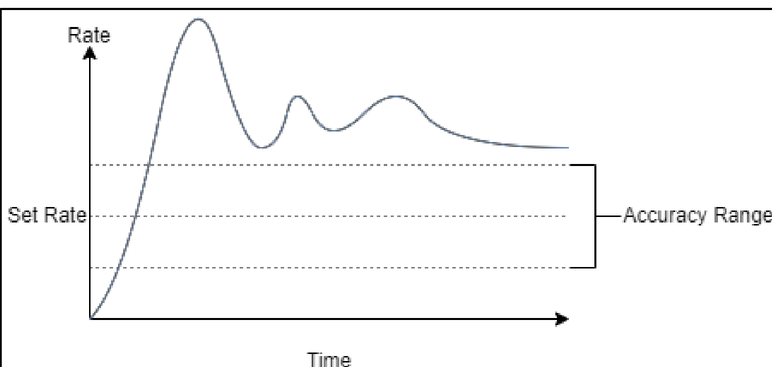
The Shutter Delay step ensures the EBE Lesker can stabilize the rate of deposition. If the EBE cannot stabilize the rate, the recipe will abort as the tool is unable to reach the correct rate. There are several reasons the Shutter Delay step may fail. Below are some examples and explanations of different Shutter Delay scenarios.



Ideal Shutter Delay:

In this example, the deposition rate quickly rises into the accuracy range and stays within the range. This allows the deposition to start quickly and wastes minimal material.

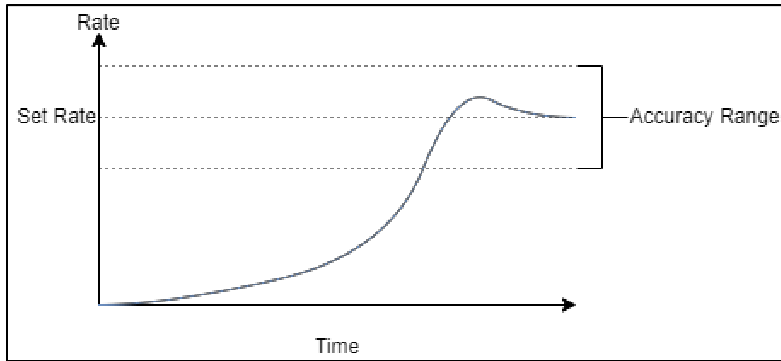


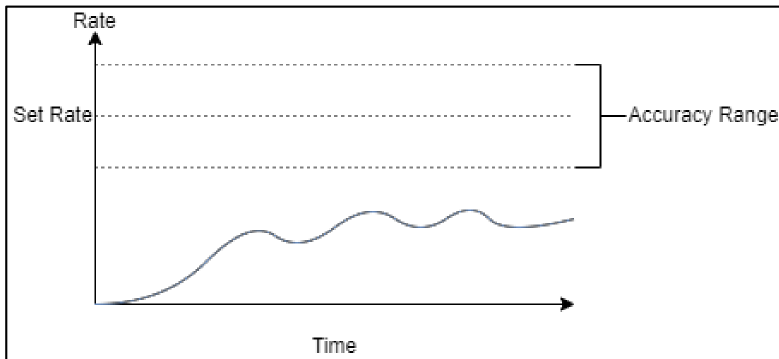


Overshoot:

In these examples, the deposition rate overshoots the accuracy range. The power must decrease so that the rate can fall. In the first example, the rate eventually stabilizes. In the second, the rate does not stabilize, and the Shutter Delay fails. Generally, overshot indicates that the material is too hot and evaporating too much. Solutions include:

- Ensuring proper graphite spacer usage (reference Appendix E)
- Decreasing Pre-conditioning ramp powers and/or times
- Adjusting the amount of material in the crucible without going over 75% full

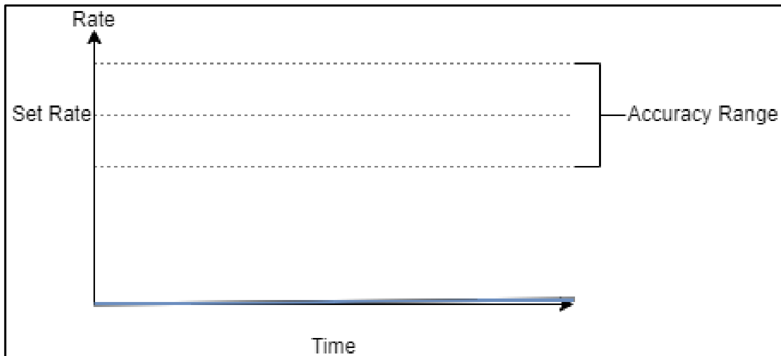




Undershoot:

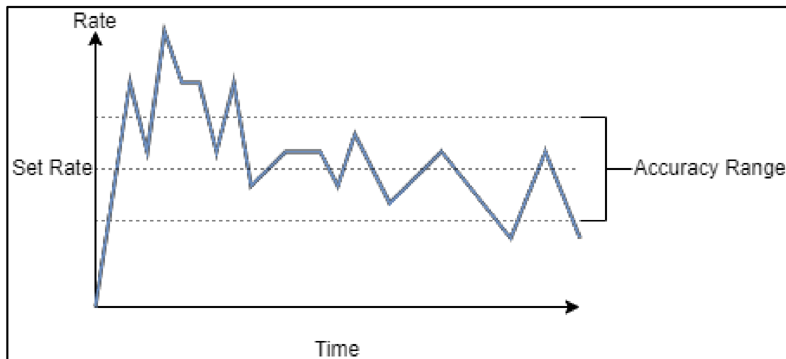
In these examples, the deposition rate undershoots the accuracy range. The power must then increase so that the rate can rise. In the first example, the rate eventually increases into the accuracy range and stabilizes at the setpoint. In the second example, the rate never reaches the accuracy range, and the Shutter Delay fails. Undershot usually indicates the material did not heat up enough to reach the desired rate. Solutions include:

- Ensuring proper graphite spacer usage (reference Appendix E)
- Increasing Pre-conditioning ramp powers and/or times
- Adjusting the amount of material in the crucible without going over 75% full
- Adjusting the gun profile (reference Appendix D)
- If you are using Chromium, try replacing the old Chromium with new pellets.



Beam Turned Off or Material Not Melted:

If the Shutter Delay step looks something like this example, the e-beam might not be lit. This will happen if the gun controller has an error. Look inside the chamber to check if the beam is lit. If not, check the gun controller for errors and ensure it is set to automatic operation. If the beam is lit, then this is a case of undershoot and the solutions listed above may help.



Bad Crystal:

If the rate during the Shutter Delay step is spiking up and down rapidly, the crystal monitor is faulty. This can occur even if the crystal life percentage is above 75%. To fix this issue, replace the crystal monitor.

Appendix G: Aluminum deposition conditions:

It is not advisable to set the dep rate more than 2A/s because that dep rate will require greater power consumption, which could also lead to greater power oscillations and damage to crucibles from thermal shock. Users should add two or three new 1/8" diameter and 1/4" long Al pellets in the crucibles before their run if the melt level in the center is below 50% (due to core drilling in the center). AggieFab also provides 1/4" diameter and 1/4" long Al pellets, but those are bigger, and if the users are not careful and add too many of those, the crucible may overflow with the Al melt. It is, therefore, better to use the smaller 1/8" diameter and 1/4" long pellets. The X-Y beam position settings can be subjected to minor changes. Both the amplitude and frequency can remain as it is.

For E beam 1:

Dep rate 1A/s	Dep rate 2A/s		X	Y
Ramp 1 power = 3%	Ramp 1 power = 3%	Position	-10%	5%
Ramp 1 time = 45sec	Ramp 1 time = 45sec	Amplitude	45%	60%
Soak 1 time = 45sec	Soak 1 time = 45sec	Frequency	20Hz	16Hz
Ramp 2 time = 30sec	Ramp 2 time = 30sec			
Soak 2 time = 30sec	Soak 2 time = 90sec			

For E beam 2:

Dep rate 1A/s	Dep rate 2A/s		X	Y
Ramp 1 power = 3%	Ramp 1 power = 3%	Position	-10%	-5%
Ramp 1 time = 45sec	Ramp 1 time = 45sec	Amplitude	55%	75%
Soak 1 time = 45sec	Soak 1 time = 45sec	Frequency	20Hz	16Hz
Ramp 2 time = 30sec	Ramp 2 time = 30sec			
Soak 2 time = 30sec	Soak 2 time = 60sec			