

**EML E-Beam Evaporator
AJA ATC
Instructions
Version 1/2019**

E-Beam Evaporator, EML

Standard Operating Procedure

CORAL Name: **EBeam-AJA**
Model: AJA ATC
Location: EML Deposition Room
Purpose: Evaporating Deposition of thin films via electron beam heating
Author: K. Broderick, G. Riggott

Introduction

The AJA ATC is used to deposit thin films, typically up to 1 micron thickness at normal or tilted angles, onto fixed or rotating substrates varying in size from millimeter pieces through 150mm diameter via ebeam evaporation heating. Interchangeable crucibles are available offering a wide variety of deposition materials.

For pieces, multiple depositions can be done of different thickness' or even of different deposition materials, during the same pumpdown.

The substrate can be back sputtered with O₂ or Ar, either before the deposition, to remove organics or thin native oxides, or during the deposition, to change film density or oxygen content.

A Kaufman Ion Mill is also included, and can be used to etch the substrate, at about 30 nm/min, at normal or tilted angles, while either fixed or rotating. This may be used for masked, self aligned etch-depositions or simple masked or bulk etching. Materials such as Al, Al₂O₃, Ag, Au, C, Cr, Co, Cu, Fe, Ge, Mo, Ni, Permalloy, Pd, Pt, Ru, Si, SiO₂, Ta, Ti, TiN, TiO₂, Y and W are available.

Safety

Identify the Emergency Off on the front of the machine.

Ground the crucible area with the Grounding Rod before touching the Pocket area.

Always discuss material or process variations with staff before trying them.

Never put low temperature materials, such as colored lab tape, into vacuum tools which get hot inside.

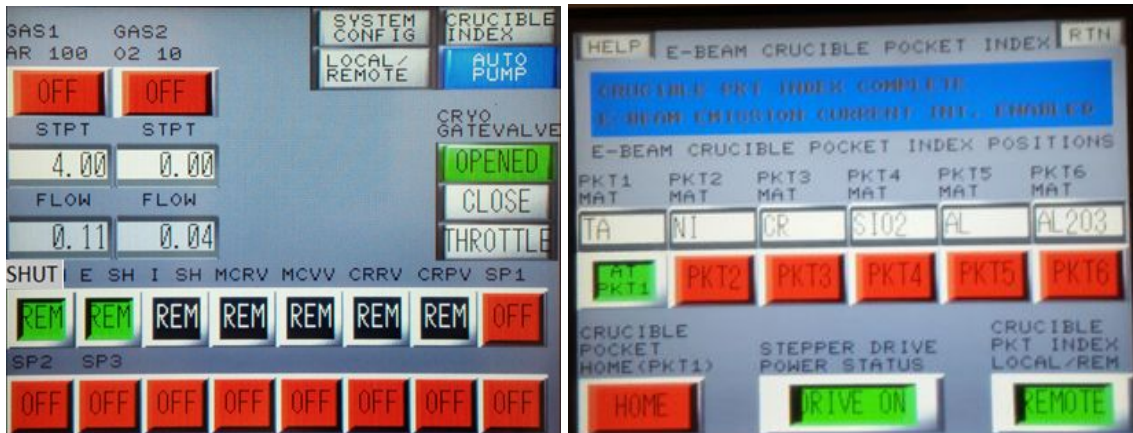
Procedure

Check reservations in CORAL to ensure you have reserved the correct machine at the correct time. Normally, you should allow High Vacuum pump down for at least 1 hour, plus 30 min preparation and unloading, plus deposition time, frequently at a rate of about 1-3 A/s

Engage the tool in CORAL. Note: the tool will not vent the chamber unless engaged. There are three computers: A) ProFace membrane, for pumps, crucibles, and shutters, B) Inficon, for material properties within each crucible, deposition rates, tooling correction factors, and C) Genius handset, to aim electrons into the selected crucible.

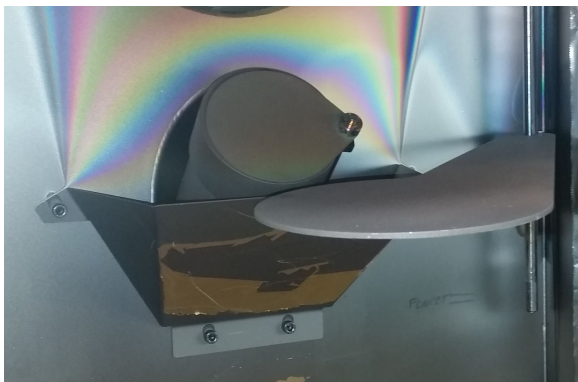
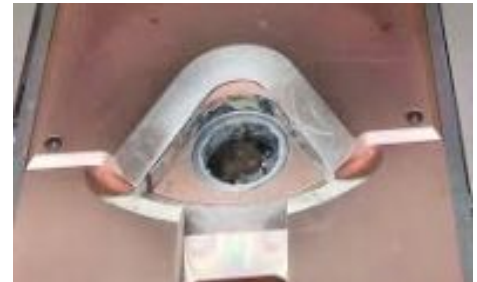
Proface (venting, loading crucibles and substrates, and pumping)

There are three functions from the Proface membrane display, selectable from the Main Menu, in the top right of the display. You can always return to the main menu by touching the small “RTN” button at the top.



ProFace Main Menu, L, and Crucible Index, R. Note Shut and Crucible index are set to Remote, not Local, for deposition.

1. First, you will need to vent the chamber, to load your crucible, and at step 6, you will need to pump down the chamber to vacuum. This is done with the Auto/Pump selection, which needs be pushed twice, and note the second will show a 100 second additional time of venting, past the sensor time.
2. Second, you will load and label crucibles under the “Crucible Index”, where you will find a “Local/Remote” button, which will need be in local mode for loading. When a material is loaded, IMMEDIATELY label the screen! Press Home once every run, then return to “Remote” or the crucible will NOT rotate when needed .
3. Crucibles should be $\frac{1}{2}$ to $\frac{3}{4}$ full, except materials that sublime (Cr, SiO₂) which should be completely full. Fill or remove material from supplies cabinet as needed. Clean debris out of the hearth area with a razor and vacuum EVERY RUN.

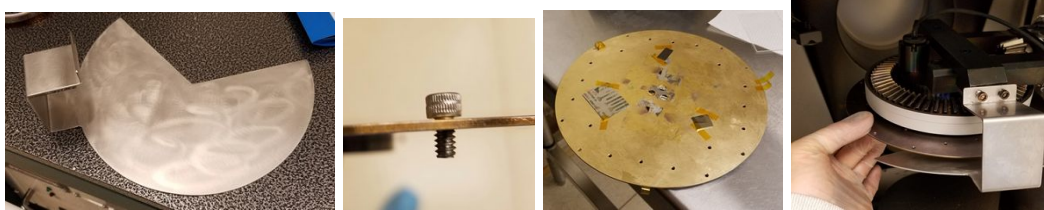


4. a) Verify the shutter for substrate (SHUT) on main menu, by going to Local mode, placing the primary shutter “SHUT” in Local, going back to main menu, and toggling the shutter. It should swing open, exposing the crucible, and closed, going between the crucible and

substrate, at the height of the Kaufman. Now return the SHUT to Remote mode. If using the Ion Mill, I-SH will be tested similarly, and Ion Milling is done over an empty crucible, not a full one, because of particulate ejection..

b) Check shutter Height; normal or low. Set shutter delay to zero if low position, used for 3 wafer planetary or extension.

5. Now load substrates using either clips and screws or high temperature Kapton tape, onto one of these substrate holders:
 - a. the Aluminum 6" holder, magnetically attached, which needs a **Si dummy wafer** installed to prevent its accumulation of excess material. This sample holder may be tilted, and is the only sample holder usable for RF back sputtering.
 - b. One of the two 8" steel perforated disks. These may be considered **the default sample holders**, as they are easily cleanable with a razor blade and vacuum cleaner, and samples may be screwed into place with various clips, or Kapton tape. Also tiltable.
 - c. Multiple deposition capable pieces fixture: Use a steel perforated disk, marked in some fashion, with tape or a screw, and samples placed to match.



Above: shadow fixture, marking screw, 3 different pieces, sample holder placed when fixture is loose, to allow loading room, then tightened last.



If using multiple deposition, the sample holder will be used to rotate, at slow speed (~20%) until the substrate plate indicator for sample #1 is positioned, then it's turned off. The sample is viewed with the light, thru the opened shutter.



Substrate Rotation. 40 is typical, if used

- Never leave the viewing window shutter open during deposition
- Always test the system with the chamber opened for possible adjustments

- d. Extension, which is attached in place before a 6" holder, with 3 short screws. This attachment has an efficiency of over twice that of the single 6" holder, to save precious metal, but it is too close to the crucible to be used for liftoff. The normal 6" holder is magnetically attached to the extension
- e. the three wafer planetary, which is attached with 3 long screws. This should be set to rotate at a speed of about 40, about 5 - 10 RPM. The planetary has a lower height setting for the substrate shutter, of about 1 cm from the bottom of the shaft, and this position blocks the quartz crystal monitor when closed, so the Shutter Delay (under Deposit Controls) must be set to "0", not the normal "1" minute.

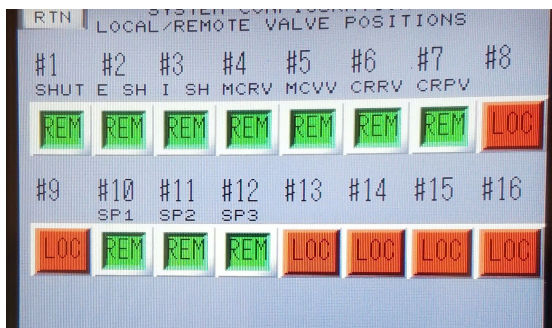
****always return to the normal upper shutter location when finished****

- f. test the substrate shutter by going to Local(vs remote) and tapping SHUT open and closed, return to Remote. Verify crucible index is in Remote.
- g. verify that no gas (Ar or O2) is flowing in the top left of the ProFace main menu
- h. On the Inficon controller, main menu, press "Next Menu" repeatedly until "Sensor Info" appears.
- i. Press "Sensor Info", which displays crystal life. If crystal "Life" reads less than 80%, change it. The crystal closest to you is #2, the one in back is #1.
- j. Update crucible assignments, in Crucible Index.



Cryo Pump Temperature, Normal

- 6. Select Auto/Pump from the main menu, then choose Auto Chamber Pump down, twice. This will pump down the foreline for 1 min, then the chamber for 5 or 10 more min under rough pump, then will switch to high vacuum. You should pumpdown typically for between 30 min to 3 hours. Turn on the high vacuum ion gage, "Sensor 1" on MKS gauge controller , pushing on/off. It will go to wait, then on, if pressure is in the -5 range, or turn off; try again in 2 minutes if needed.



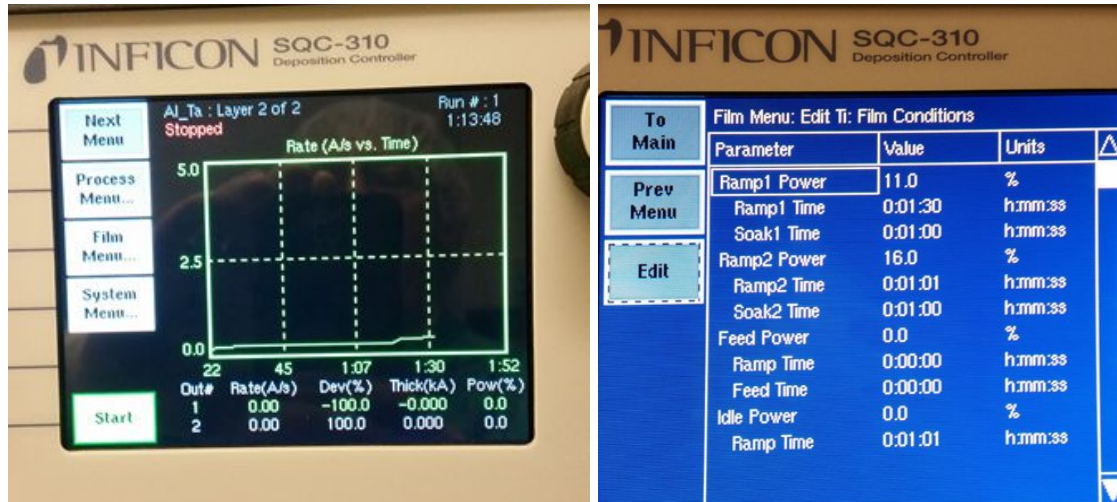
STAFF ONLY: Auto/Pump choices include Vent,Rough and Cool is if temp is less than 30K, needing regen, and Auto Regen is room temp - full-regen. Auto Regen is 30 min purge, then rough cryo to 50mT prior to starting cold head. Before starting Auto Regen, ASSUME valves aren't correctly in Remote mode; see Left. Note 8, Ar and 9, O2, are "local".
MC=main chamber RV=rough valve, VV=vent valve, CR=cryo rough, PV=purge valve. If Genius fails: set data, 2031 admin, HV=8.2KV, set limits top 600, right -450, bot -400, left 500. Verify with new

mirror, Ti, manual, sweep to 0, run 10mA (cuts off with menu choice-restart as needed) return to lissajous sweep, auto mode, test again.

Cryo Regen (Auto problematic): local CRRV CRPV x 10min, then CRRV only 20 min, then both off, (s/b <50mT) both to remote. Pump Rough & Cool Coldhead ON

STAFF USE ONLY

Inficon (Identifying materials' properties, deposition ramps and rates, pocket location, and feedback loops)



Inficon Main Menu, Left, and Film Conditions, under Film Menu, Right

The Inficon controller is used as a material database interfacing with your specific requirements, with menu choices following.

- **Film Menu:** density and other standard values are stored, to which you assign a **crucible pocket location**:
 - Select material from list, press Edit
 - Turn wheel down to select **Pocket #**; update correctly.
- Film Conditions: Material specific ramp up and ramp down heating profiles. Verify these are correct, compared to the *Film Condition Values* on last two pages of this SOP
-
- **Deposit Controls:** (under film menu) After ramp up 1 and 2 are done, either opening shutter immediately, if using 3 wafer planetary, or typically after 2 min under deposition rate targeting, the normal situation.
 - **Shutter Delay** means “target the desired deposition rate”, for the time listed, (typically 1 min)
 - **Capture** means “allowable error % to end shutter delay time”, (typically 10%) from targeted value. In not met, system shuts off.
 - **Control Delay** means delay after deposition rate is reached, to improve quality of critical first layer interface. Use 2 min for first level

adhesion layer, to allow gettering and moisture desorption; 1 min subsequent layers, minimum, more if high surface area crucible fill.

- **Process Menu:** Start Mode Automatic
Initial Rate = Deposition Rate (typ 1-3A/s)
(faster rate is rougher, but slower rate has more time for contamination)
Either Sensor 1 or Sensor 2 Must be ON
Final Thickness = your required thickness
Max Power from the table at end of this document
(all unused fields are at value 0)
- **System Menu:**
- **System Tooling=** ratio of material received at substrate to QCM sensor, depends on fixtures.
 - Standard 6" holder system tooling is **1.58**
 - Extension is **3.75**
 - 3 Wafer Planetary is **2.1**
 - Caution!! system tooling can be adjusted at the film level as well. Leave that at 100% (unless otherwise specified) or the effects will multiply!!
- **Rate Filter Alpha** is similar to phase lead, or integration from PID, and can generally be set to **.25**, although for materials which sublime, like Cr and SiO₂, a smaller value like .1 is better



Verify the sensor 1 and 2 are operational, via the Inficon, From Main menu, press Next Menu, until you find the "Sensors Info". These Quartz Crystals are 6 mHz, declining frequency, and are replaceable by pulling down on the sensor face, and they should be replaced when less than 80% life is displayed, or if they are reporting failure.

Sensor 1 (back) and 2 (front) aiming down towards crucibles. Rings pull down for crystal replacement.

Genius Handset (swept area before run)

The remote handset is used only for controlling the swept area and x, y beam positioning, with other functions such as power controlled by the Inficon or ProFace.

Default is 8.2KV, with both left and right side in Automatic mode, and Pocket 1 material SiO₂ displayed, with Data:



Oxide 6KT. Default settings, not user adjustable. Normally beam swept area should be full sized, touching circle at the corners in display, but for refractory or high temperature materials, such as W, or Ta, the beam should be reduced to <25% of the area. *Return to full sized after doing reduced area run, please!*

To adjust swept area or beam positioning:

- a. Press Menu, making display offer Manual choice on left.
- b. Toggle down on left, causing Manual choice on right
- c. Toggle down on right
- d. Accept Manual mode by pressing Menu
- e. Adjust area with left toggle switch
- f. Adjust impact center with right toggle switch
- g. Press Menu, return to Automatic left and right, accept with Menu, when done

Operations:

After system loaded, pumped down to desired base pressure, verify:

Film Menu pockets on ProFace and Film Menu Match, and ProFace is in Remote for Shutters and Crucibles, ie green color buttons.

Recipes for Films match the Film Condition Values table.

Tooling Factor is correct for substrate holder type. If using Planetary, shutter has been lowered and shutter delay is "0", otherwise delay is up to about 2 min and error 10- 20%, and control delay is about 1 min. See materials table at end for specific values. Start is set to Automatic, not Manual

Rate is up to 3A/s, lower for thin films and much lower for small ebeam write lithography liftoff. Final Thickness is in KA: for ex 100 nm is 1000 A.

Press the Green HV safety on the Remote handset.

Press start on the Inficon.

The first thing that should happen is the Proface should rotate to deposition material 1.

Always watch to make sure the deposition starts, and the shutter opens, because if there's an error, like crystal failure or not reaching targeted deposition rate, the machine will shut off with only a transient error display

Verify the current is going up on both the remote (shows mA, 500max) and Inficon (shows % of max) proportionally.

Open the viewport 30 degrees and view the location where electrons are hitting the material inside the crucible, causing it to heat and emit visible light. Please advise staff if it is aimed poorly or appears incorrect. The EBeam can melt itself easily, so pay attention, please! Depositions can be easily aborted with the Red Button on the Handset, or the Stop Button on the Inficon.

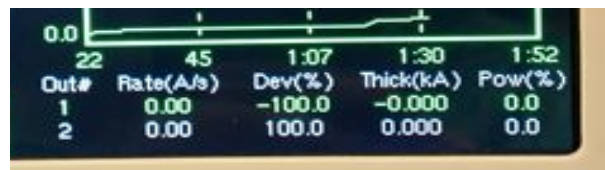
Close the viewport unless you are looking through it. These cost \$1000 to replace if coated with metal!

Watch deposition rate during Ramp Ups and Soaks, and when switching to Deposition Rate Targeting, look for the Deposition Rate displayed on the bottom of the Inficon, and beware of errors such as Maximum Power Reached.

***During ramp and soak 1 and 2, VIEW Deposition Rate!!!**

***Make Sure Deposition Starts; if it aborts, it will only show the wrong Thickness, no other error!**

***If Deposition Rate ever goes over 3A/sec, abort the run, and lower the soak power so Pre-Deposition Rate STAYS less than 3A/S, then PEN CHANGE the SOP!**



In such a case, look through the viewport, and if all looks well, and it is just a little off target rate, press the Inficon Next button to make the “Quick Edit” menu appear. In this menu, you may change deposition rates and maximum power settings before the machine shuts off.

Verify Material 1 Deposition Thickness displayed on the bottom of the inficon is the value you intend (or slightly higher, as the deposition sensor will capture material during ramp down phase, when the substrate is blocked with the Substrate Shutter.

- **Caution: If Max Power exceeded error appears, STOP and THINK! Either you are on the wrong crucible, or your shutter is blocking the Sensor, or possibly a normal situation like heat loss out of the crucible from splashed conduction paths is occurring. Is Crucible index on REMOTE?**

The ProFace will automatically rotate to each subsequent material, and the Inficon will drive current similarly.

Ten minutes after the end of the last material layer, when crucibles aren't too hot, vent the system under ProFace Auto Pump, press Auto Chamber Vent twice. Ground the hearth with the grounding rod before unloading crucibles. Return to default sample holder 6" stage.



Unload your samples, vacuum any debris left in the chamber, and Pump Down the chamber, and disengage.

Kaufman Ion Milling:

To etch into your substrate, at the rate of perhaps 4-20 nm/min, use the 6" sample holder tilted towards the Kaufman Ion milling source, at 55 degrees. This will accelerate Ar into the sample, causing etching of the substrate. To protect crucibles from Ion Milled debris, leave an empty hearth pocket, without crucible, exposed during Ion Milling!

Ion Mill gun, with I-SH closed. Note Substrate Shutter is UNDER Ion Mill Gun.

Operation

To use this, after desired high vacuum base pressure is achieved:

First preset the voltage and current for approximately (record your values) -set values on display by selecting the box with the button, ie "cathode" then spinning the "Setpoint Adjust" dial above the cathode display

*Accel display will flash when above 2mA(set) but will go to ~10mA-record this value!

** 30sec etch time, 30sec off for substrate cooling wait, repeat

Driving voltages/currents, and results. Please update on paper or email Kurt...

Cathode V/A	Discharge V/A	Beam V/mA	Accel V/mA(set)/mA (actual)	Neutralizer V/A	Emission mA	Etch Rate film/mask nm/min
9/ 7	40/ 0.65	600/45	120/1.6/9	9/ 11.5	40-70	SiO2 10-25
8.5/1.16	40/0.59	600/46	120/2/8.0	10.7/12.1	46.2	Au/Ag 60 Resist 33

Operation:

1. Turn OFF the Ion Gauge and Turn ON the Ar at about 3.5 sccm.
2. CLOSE the Cryo Gate Valve for about 30 seconds, to allow pressure to increase to ~2mT, then go to THROTTLE to allow pressure to stabilize.

3. Turn ON Kaufman Controller, see below, and check values. Adjust as required.
4. OPEN the I-SH, ion mill shutter then push BEAM ENABLE. If it doesn't ignite, briefly (1-2 sec) close I-SH to allow the Ar flowing through it to build local pressure, then open I-SH and proceed. Open the side viewing port if you wish to see the ion mill plasma.
5. Forgetting to open the shutter will cause an error and destroy the shutter!



Some notes on Ion Milling:

Accel mAmp has two displays, the Limit, bottom left, and the Set Value, bottom 2nd from right. The set value should be set to 4-15% of beam current, so about 2mA. Help 26 fault, pressure too low. Is gas flowing and throttle valve active?

To Proceed with EBeam evaporation at this point:

- Close I-SH
- Return to normal substrate holder angle
- Turn off Ar
- Open Cryo
- Turn on Ion Gauge

Back Sputter RF

Substrate sputtering can be done with either O₂ or Ar, to remove organics or etch slightly, respectively. This is ONLY available with the standard Aluminum 6" holder!

Operation

After achieving desired base pressure, flow desired gas, from ProFace main menu, Top Left. On same menu, Top Right, is option to close the pump or close partially (Throttle). Ideally, the Back Sputtering is done at ~3-5mT, so if throttle is selected with gas flowing, the chamber pressure should be about in this range. Gas flow may be adjusted if needed.



Main Menu, ProFace, L, showing gas, and RF Generator, R, for Back sputtering

To light plasma:

1. flow gas as desired, sufficient for 3-6 mT on Throttle mode
2. close cryo gate valve, causing pressure to rise.
3. when about 20 mT chamber pressure, rotate dial to FP, press to change, and set FP RF Power value to about 25W, and press to accept setpoint.
4. Light Plasma with RF Output button. Look in side window to verify plasma.
5. Switch immediately to Throttle valve on the ProFace.
6. Organic strip is often 1 minute, although significant process variations are used.

Turn off Gas, RF Power, and Open cryo gate valve when finished, and proceed to normal evaporation

When processing is complete and the system is ready for the next user, you may disengage from the tool in CORAL. Please be sure to enter accurate processing information into the CORAL dialogue as this is used for billing and tool maintenance.

Film Menu/Material choice/Film Condition “Values” (pg 1 of 3)

Material	Al fresh	Al (old)	Al2O3	Ag	Au	C	Cr	Co	
Ramp 1 Pwr	10	15	10	6	15	15	5	10	
Ramp 1 Time	3	4	2	2	3	2	2	5	
Soak 1 Time	1	1	1	1	1	1	1	2	
Ramp 2 Pwr	12	22	14	9	20	25	7	15	
Ramp 2 Time	2	2	1	1	1	1	1	2	
Soak 2 Time	1	1	1	2	1	1	1	2	
UNUSED ROWS	X		X	X	X	x	X	X	
Idle Power	0		0	0	0	0	0	0	
Ramp Time	2	3	0	1	1	0	0	2	
Notes: tooling factor, swept area								1hr min pumpdown 0.5A/s max	
density	2.7	2.7	3.97	10.5	19.3	2	7.2	8.9	
z-factor	1.08	1.08	.336	.529	.38	3.3	.305	.33	
Process Menu/ Max Power	40	65	25	40	65	45	14	35	
Shutter delay (max allowed) **	2	2	1	2	2	1	1	2	
Capture (error allowed)	10	10	10	10	10	15	10	10	
Control delay (gettering/ stabilizing time)	2	2 min	2 min	1 min	45s	1	1 min	1min	

Film Conditions

Deposit Controls

** For extender or planetary sample loading, always use 0 shutter delay!

PID values P=50, I=0.7, D=0.0, and Film Tooling=100%

Film Conditions = Preheating, Deposit Controls = Sensor feedback

Film Menu/Material choice/Film Condition “Values” (pg 2 of 3)

Material	Cu	Ge	Fe	Mo	Ni		Pd	permalloy	Pt	Ru
Ramp 1 Pwr	10	8	7	30	18		9	12	25	20
Ramp 1 Time	2	1	2	2	2		2	2	3	3
Soak 1 Time	1	1	1	1	1		1	1	3	2
Ramp 2 Pwr	15	13	10	45	25		15	14	30	29
Ramp 2 Time	2	1	1	2	1		1	1	2	2
Soak 2 Time	1	1	1	1	2		1	1	2	1
UNUSED	X	x	X	X	X		X		X	x
Idle Power	0	0	0	0	0		0		0	0
Ramp Down	1 min	1m	1m	1m	2 min		2 m	3 min	2 m	2 m
Misc					2A/sec max				0.7A/s max	0.5A/s max
density	8.9	5.35	7.9	10.2	8.9		12	8.7	21.4	12.3
z-factor	.44	.52	.35	.257	.33		.36	1	.245	.18
<i>Process Menu</i> Max Power	60	40	65	70	70		35	35	65	50
Shutter delay max allowed	2	2	2	2	2		2 m	2 min	2 min	2m
Capture %error allowed	10	10	10	10	10		10	10	10	10
Ctl delay -gettering	30 s	30 sec	1 m	1 min	1 min		30 s	30 s	30 sec	30 s

Film Conditions

Deposit Controls

**** For extender or planetary sample loading, always use 0 shutter delay!**

If max power is exceeded; likely either shutter position closed is blocking sensor or wrong crucible is indexed. Watch during ramp 2 and soak 2!

If Deposition rate is **over 4A/sec, abort the run**, make *soak 2* power lower, and start again. Pen change this sheet, too, or send a note.

Film Conditions = Preheating, Deposit Controls = Sensor feedback

Material	Si	SiO2	Ta	Ti	TiN	TiO2	W	WO3	Y
Ramp 1 Pwr	15	2	50	11	6	15	50	3	2
Ramp 1 Time	3	1	4	2	2	2	4	2	2
Soak 1 Time	1	1	1	1	1	1	2	1	1
Ramp 2 Pwr	20	3	30	16	8	20	60	4	4
Ramp 2 Time	1	1	1	1	1	1	1	1	1
Soak 2 Time	2	1	1	1	1	1	1	1	1
UNUSED	X	X	x	X	X	X	X	x	x
Idle Power	0	0	0	0	0	0	0	0	0
Ramp Down	1m	0	2min	1min	0	0	2 min	0	0
Misc nf, swept area			½ area sweep		Store in N2!	1.39 tool factor	Smallest swept beam area		Store N2 box
density	2.32	2.65	16.6	4.5	5.43	4.26	19.35	7.16	
z-factor	.71	1	.26	.63	1	.4	.163	1	
<i>Process Menu/ Max Power</i>	45	9	80	40	15	40	95	10	8

Shutter delay(max allowed)	90s	1min	90s	2m	2 min	1min	3min	30s	1min
Capture (error allowed, %)	10	10	10	10	10	10	20	10	10
Ctl delay (gettering stabilizing)	1	30s	30s	2	30s	30s	1min	30s	1min

Film Conditions

Deposit Controls

**** For extender or planetary sample loading, always use 0 shutter delay!**