

MN Nano Center -- University of Minnesota

Standard Operating Procedure

Equipment Name: Temescal E-Beam Evaporator

Badger Name: K3 Evaporator Temescal **Revision Number:** 9
Model: Temescal **Revisionist:** L. von Dissen
Location: Bay 3 **Date:** 6/16/2021

1 Description

The electron beam evaporator is used to deposit thin films onto substrates. Evaporation is done under a high vacuum in a bell jar chamber. Evaporation is achieved by heating a source with an electron beam. As the source material evaporates, it forms a thin film on the samples.

2 Safety

- a. If the building alarm sounds anytime during a run, TURN OFF THE POWER SUPPLY CIRCUIT BREAKER and leave immediately.
- b. When viewing the e-beam inside the chamber, wear UV glasses or use the chamber window Polarizer to minimize the light, to prevent eye damage.
- c. If there is water leaking out of the machine, or any immediate danger, press any of the red EMO buttons (one located on the bottom of the equipment control board stack, and one built in on the front-left of the temescal)

3 Restrictions/Requirements

- a. Must be a qualified user on K3 Evaporator Temescal.
- b. Materials are restricted to those found on the material parameter sheet. Any other materials besides those listed must be approved by NFC staff.
- c. Fill out the Temescal Google Log Sheet before/after your run.

4 Required Facilities

- a. Process chilled water

5 Definitions

- a. Ion gauge -- measures the pressure of the chamber while under high vacuum (i.e. while using the cryo pump)
- b. Hearth -- located inside the chamber consisting of 4 pockets which holds the materials to be evaporated.
- c. Lift-off Fixture -- the fixture that holds your substrates inside the chamber (rotation optional).
- d. Scissor Shutter -- Two half-moon shaped metal blades that are used to prevent metal deposition during the pre-deposition step and also after the metal deposition has been completed.
- e. View Shutter -- A viewport shutter located in the front of the chamber. When this is opened, it allows the user to see the e-beam source/pocket throughout the run.

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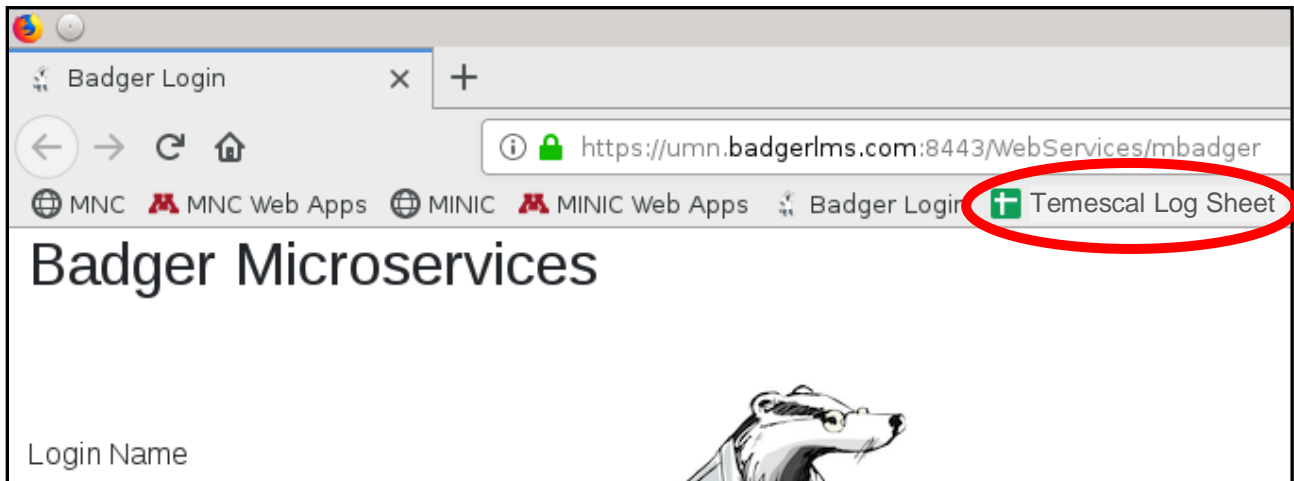
6 Setup

- a. Enable the “K3 Evaporator Temescal” in Badger.
- b. Turn the **ION GAUGE FILAMENT** off by ‘bumping’ the switch up (assure it is off by checking that the LED located by the switch is not lit).
- c. Press the **VENT MODE** button on the pump controller to vent the chamber.
- d. The chamber will reach atmospheric pressure and be ready to open in approximately 5 minutes. Monitor the pressure on the vacuum gauge located to the left rear of the chamber. When the vacuum gauge has reached zero, wait a minute or two, then lift the chamber cover fully open (Note: it can be very heavy to lift, so if necessary, ask assistance from another user or staff member).
- e. While the chamber is venting, you can begin to fill out the “**Temescal Log Sheet**” (see next steps below).
- f. Users are no longer entering process / run data into a physical log sheet. Instead, users are now required to enter this information electronically within a Google Sheet (online spreadsheet). There is a link to this spreadsheet (titled: “**Temescal Log Sheet**”) on every Badger station, which is located as a shortcut / link on the Mozilla Firefox Bookmarks toolbar (see steps below).

If you are unable to access the Google Sheet for some reason (e.g. the link doesn't work, there is something wrong with the Google Sheet, the Google Sheet is full, etc.), then you should enter your process / run data in the Temescal's physical log book (located behind the pocket/hearth indexer). If any of these problems do occur, please report a problem in Badger so we can address the issue, as we want to minimize any entries made in the physical log book.

- g. Whenever you are ready to start entering log sheet information, go to a Badger Station and **open** the Mozilla Firefox browser. There should be a shortcut for this browser on the desktop.

Once Mozilla Firefox opens up, click on the “**Temescal Log Sheet**” bookmark link to open up the Google Sheet (see image below).



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h. The “Temescal Log Sheet” should look like the following (see image below):

	A	B	C	D	E	F	G	H	I	J
1										
2		Temescal Log Google Sheet								
3										
4		Name:	Lage von Dissen		<u>Layer 1</u>	<u>Layer 2</u>	<u>Layer 3</u>	<u>Layer 4</u>	<u>Layer 5</u>	<u>Layer 6</u>
5		Date:	5/18/2020	Pocket #:	4	3				
6		Time In:	14:15	Material:	Ti	Au				
7		Time Out:	16:35	Rate (Å/sec):	2	4				
8		Pump Time:	45 min	Thickness (Å):	200	2000				
9		Tooling Factor (%):	26.1							
10										
11										
12					*** Precious Metal Weights *** (Remember to do a "Supply Checkout" in Badger)					
13					Starting Au Weight (grams)	Ending Au Weight (grams)				
14		Crystal % Life (Before run)	11		162.5	161.2				
15		Crystal % Life (After run)	14							
16					Total Au used (milligrams):	1300				
17										
18					Comments: This is an example log sheet entry for demonstrational purposes only. Gold was not actually used in this run. If users want to enter/record additional information about their run, they can do so within the "comments" box in their own log sheet entry below.					
19										
20										

- i.** Scroll down or use the “**Page Down**” key on the keyboard to find the first empty log entry, and then you can start entering information for your current run. Enter your information in the appropriate white-colored boxes in the Google Sheet. **The entries are saved automatically.**
- j.** If you are using the gold source, lift the cover off of the scale that is located to the left of the bell jar. Turn on the scale and then immediately place the cover back on the scale, to ensure it is zeroed properly. **If you are not using gold**, then you can skip ahead to step ‘L’.
- k.** Remove the gold source (and the two buttons that are underneath the gold source) from the plastic jar and place them on the scale. Record the combined weight of the gold source and the two buttons in the “Temescal Log Sheet” (“Starting Au Weight”).

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- l.** Open the **SHUTTER** (by flipping up the shutter switch on the control rack).
- m.** Load the metal sources which are to be evaporated into the desired **HEARTH** pockets. There are 6 pockets available for use (Note: you can choose/indicate the pocket number by using the hearth rotation controller located to left of the chamber). To make things easier to remember, load the sources in the order they will be deposited (i.e. Layer 1 in Pocket 1, Layer 2 in Pocket 2, etc.).
- n.** Close the **SHUTTER** (by flipping down the shutter switch on the control rack).
- o.** Load the substrates onto the **LIFT-OFF FIXTURE** with the samples facing down. There are various holders/clips available to accommodate different sized substrates, located in the black plastic boxes on top of the control rack.
- p.** Turn on the **DEPOSITION CONTROLLER**. The **T/X** light should blink 2 or 3 times, and then stop blinking. If the light continues blinking, the thickness monitoring crystal needs to be replaced. To confirm this, press and hold down the **T/X** button, and check the second group of numbers displayed in the center (if >15 , replace the QCM crystal).
- q.** Open the viewport shutter (small window located in front of the vacuum chamber) and adjust the polarizer if needed to verify that you can see the source/pocket. You want to ensure that you can see the source well before closing the chamber.
- r.** Close the chamber cover slowly and carefully.
- s.** Press the **AUTOMATIC MODE** button on the pump controller to start the pump-down sequence.
- t.** After about 5 minutes, the pump controller display will read a "TC1" value of 1mT. Once this value is reached, turn the **ION GAUGE FILAMENT** back on by bumping the switch up (the LED located by the switch should light up). If the LED doesn't light up, you may have to bump the switch up again.
- u.** Wait until the pressure drops to at least 4.0×10^{-6} Torr prior to deposition (while you are waiting for the desired pressure, you can program your layer parameters).
- v.** The word **PROGRAM** should be lit up near the right of the deposition controller.
- w.** The **CONTROL POWER** rocker switch should be set to **AUTO**.
- x.** You **MUST** press the red **ENTER** button after each parameter value is entered.
- y.** **RISE TIME**: is the time spent to ramp from zero power to **SOAK 1 POWER**.
- z.** **SOAK TIME 1**: is the desired hold time after reaching the **SOAK 1 POWER** setpoint.
- aa.** **SOAK TIME 2**: half of this time is spent ramping up the beam from the **SOAK 1 POWER** setpoint to the **SOAK 2 POWER** setpoint, and the remaining half of this time is the desired hold time after reaching the **SOAK 2 POWER** setpoint.
- bb.** **RATE**: this is the evaporation rate in $\text{\AA}/\text{sec}$. Do not go above 5, unless evaporating Aluminum (then you can go as high as $10 \text{\AA}/\text{sec}$).

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- cc.** TOOLING: A calibration ratio for proper thickness measurement by the QCM crystal. This number is posted on the front of the chamber; Tooling = 45.0%
- dd.** SOAK 1 POWER: first power setpoint used to pre-heat and de-gas the metal within the crucible
- ee.** SOAK 2 POWER: second power setpoint used to pre-heat and/or melt the metal within the crucible prior to deposition.
- ff.** MAX POWER: this setting prevents the beam from exceeding this power set point (even if the desired deposition rate is NOT achieved), and this helps to prevent damage to the equipment, or to the e-beam evaporation source.

To reduce the risk of damaging the metal source and the scissor shutter, you may choose to temporarily set the **MAX POWER** to the same value you used for your **SOAK 2 POWER**. Then, if you forget to open the shutter immediately after the Deposition Controller gets to the **DEPOSIT** step, the beam power will not increase beyond the **SOAK 2 POWER**.

If you choose to follow this advice, then once you get to the **DEPOSIT** step and open the shutter (which occurs later in this SOP), you can toggle the **KEYBOARD / LOCK** switch to re-enter the **PROGRAM** mode, and then change the **MAX POWER** to the value listed on the Material Parameters Sheet. Then you can toggle the **KEYBOARD / LOCK** switch one more time to disable the **PROGRAM** mode.

- gg.** IDLE POWER: always set at zero (0).
- hh.** GAIN: this controls how fast the beam power adjusts over time in order to reach the desired deposition rate. The lower the gain value is, the longer it will take for the beam to ramp up/down as needed. Typically, users program a gain value that is between 1 and 10.
- aa.** THICKNESS 1: This is the desired thickness that you want for your evaporated film, and is measured in kilo-angstroms (kÅ). Example: “5.000” = 5000Å = 500nm = 0.5 micron. The THICKNESS 2 parameter is not used, and so it doesn’t need to be programmed.
- bb.** DENSITY and Z-RATIO are characteristics of the evaporated material. These numbers are posted on the equipment control rack and on the Material Parameters Sheet.
- cc.** SOURCE/ SENSOR: always set at 1/1.
- dd.** If you have more than one layer to program, you can program another layer by pressing the white round button corresponding to the correct layer number (located next to the **START**, **ZERO**, and **STOP** buttons). Then you can program the parameters for that layer just as you did with the first layer, etc.
- ee.** Toggle the **KEYBOARD / LOCK** switch. This should cause the **PROGRAM** indicator light to turn off on the deposition controller.
- ff.** Wait for the chamber to pump down to the desired vacuum by observing the Ion Gauge. Maximum allowable pressure for deposition is 4.0×10^{-6} Torr, and we recommend going to a lower pressure if time permits ($\leq 2.0 \times 10^{-6}$ is ideal).

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7 Operating Instructions

- a. Make sure the Hearth is set to the correct pocket. Select the correct pocket by sliding the thumbwheel switch up or down as needed on the **HEARTH CONTROLLER** (which is also called an “Indexer”).
- b. Flip up the circuit breaker on the **H.V. POWER SUPPLY**.
- c. Turn the **SOURCE CONTROLLER** on by flipping up the rocker switch.
- d. All four interlocks should have their corresponding green LEDs lit. If not, the beam cannot be turned on. The four green LEDs that should be lit are:
 - Water
 - Transformer
 - Vacuum
 - Doors (closed)
- e. Turn the **SWEEP CONTROLLER** on. If you are facing the front of this controller, you will find the rocker switch located on the upper left side of the back of the unit.
- f. Turn the **HIGH VOLTAGE** on by pressing the **H.V. ON** button.
- g. Turn the **FILAMENT CURRENT** on by pressing the **FIL-ON** button.
- h. Turn on the sweep controller current by pressing the **ON** button located on the right side of the touchscreen display.
- i. To change any of the parameters, press the **PRM** button located on the right side of the touchscreen display. Press the corresponding parameter you wish to change (either the X or Y offset value, or the Sweep amplitude), and then press the up or down arrow buttons on the remote controller until the desired value is selected. Zero out the X and Y offsets for a safe, initial position for the beam.
- j. The sweep pattern selected should be set to option 1 (so “Circle” is displayed).
- k. Change the sweep amplitude to the value listed on the material parameter sheet.
- l. Turn the **ROTATION** on by toggling the switch up on the control rack.
- m. Start the process recipe by pressing **START** on the deposition controller.
- n. Open the View Shutter to see the source/pocket inside the system. Once the beam is visible (usually by SOAK 1, otherwise for sure by SOAK 2), center the beam on the SOURCE by adjusting the X and Y offset values on the **SWEEP CONTROLLER**. The beam should not touch the pocket or crucible liners, but rather it should only be on the source metal you are evaporating. Check the source periodically, to ensure that the beam remains in a good position and to ensure that the source looks stable. If it doesn't look stable, hit the **STOP** button on the deposition controller, and get an NFC staff member for assistance).

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- o.** When the cycle reaches the **DEPOSIT** step, OPEN the **SCISSOR SHUTTER** to begin depositing metal on your samples. If the shutter is not opened immediately after reaching the DEPOSIT step, the metal source and shutter may be permanently damaged.

If you chose to temporarily set your MAX POWER to the same value as your SOAK 2 POWER, then once you get to the DEPOSIT step and open the shutter, you can toggle the KEYBOARD / LOCK switch to re-enter the PROGRAM mode, and then change the MAX POWER to the value listed on the Material Parameters Sheet. Then you can toggle the KEYBOARD / LOCK switch one more time to disable the PROGRAM mode. Now the beam power should begin to increase slowly and the deposition should proceed as usual.

- p.** After the desired thickness is reached, the beam will automatically shut off. Close the **SCISSOR SHUTTER** and then press **STOP** on the **DEPOSITION CONTROLLER**.
- q.** If you programmed more than one layer in your process recipe, wait 5 minutes to cool the metal source before rotating the hearth to another pocket. After rotating to the next desired pocket, press **START** on the deposition controller, then select the appropriate layer number, and then press the **START** button again. Be sure to re-adjust the SWEEP CONTROLLER parameters as needed (X/Y offsets, sweep amplitude, etc.).
- r.** Turn off the DEPOSITION CONTROLLER when you are finished depositing all layers.

8 Shutdown

- a.** Press the **FIL-OFF** button on the **SOURCE CONTROLLER**.
- b.** Press the **H.V. RESET** button on the **H.V. POWER SUPPLY**
- c.** Turn off the circuit breaker for the **H.V. POWER SUPPLY**.
- d.** Turn off the **SOURCE CONTROLLER**.
- e.** Turn off the **SWEEP CONTROLLER**.
- f.** Turn off the **ROTATION**.
- g. IMPORTANT:** WAIT AT LEAST 10 MINUTES BEFORE YOU VENT so that the metal sources can cool adequately.
- h.** Turn off the **ION GAUGE FILAMENT** by bumping the switch up. This should cause the red LED (located next to this switch) to turn off.
- i.** Press the **VENT MODE** button on the pump controller.
- j.** The chamber will reach atmospheric pressure in approximately 5 minutes. You may monitor the pressure on the vacuum gauge located on the left-rear side of the chamber. When the vacuum gauge has reached zero, wait another minute to ensure that the system is vented, and then open up the chamber lid carefully.

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- k. Open the **SHUTTER** (by flipping up the shutter switch on the control rack).
- l. Remove the substrates and the sources (BE CAREFUL, as the metal sources may still be warm to the touch, and if so, wait a few minutes to let them cool further).
If you used the gold source, please re-weigh the source and the two buttons, and record this weight in the “Temescal Log Sheet” (“Ending Au Weight”). Also, remember to perform a “Supply Checkout” in Badger (instructions for this are found at the bottom of this page).
- m. Close the **SHUTTER** (by flipping down the shutter switch on the control rack).
- n. Close the chamber lid slowly and carefully.
- o. Press the **AUTOMATIC MODE** button on the pump controller to start the pump-down sequence.
- p. Finish filling out the “Temescal Log Sheet”.
- q. After about 5 minutes, the pump controller display will read a “TC1” value of 1mT. Once this value is reached, turn the **ION GAUGE FILAMENT** back on by ‘bumping’ the switch up (the LED by the switch will now be lit).
- r. Disable the “K3 Evaporator Temescal” in Badger.

9 Badger Supply Checkout (for gold users)

*** Precious Metal Weights *** (Remember to do a "Supply Checkout" in Badger)	
Starting Au Weight (grams)	Ending Au Weight (grams)
162.5	161.2
Total Au used (milligrams):	1300

In Badger, click on the “**Supplies**” menu, and select “**Check Out**”. You will then notice a supply list that appears on the left side within the Badger window. Scroll down the list and click on “evap gold”. Then you will see that supply name appear in the “**Supply**” field box. In the “**Quantity**” field box, enter the number of milligrams of gold that was used (which was 1300 mg in the example above, as indicated in the “Temescal Log Sheet”). Click on “**Save & Close**”.

Close the “Temescal Log Sheet” tab/window on the Badger Station when you are finished entering your run data.

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10 Problems/Troubleshooting

- a. The system is not pumping down- If the samples have moisture on them, i.e. water or photoresist, the chamber will take longer to reach the desired pressure. If the samples are dry, there may be particles on the O-ring preventing a good seal. Vent the system and wipe off the O-ring with a wipe soaked with either methanol or IPA (**DO NOT USE - ACETONE**) and try to pump down the chamber again.
- b. The T/X light keeps on flashing when the Program Board is turned on- This indicates that the crystal that monitors the thickness needs to be changed by a staff member or the replaced crystal has been installed improperly.
- c. The metal will not evaporate- This may happen for a number of reasons:
 1. Check to see if the hearth has been rotated to the correct pocket/material.
 2. Re-check the parameters entered on the program board. Flip the rocker switch back to Program and press the corresponding white buttons and check the original entries. If an entry is not correct and needs to be changed, press the CLEAR button. Enter the correct parameter and press the red **ENTER** button. (Toggle the keyboard switch to save these new parameters).
 3. If the cooling water has been left on for an extended period of time without evaporating any metal, the source metal may be too cold. Turn off the cooling water for at least 30 minutes to allow the metal to warm up and retry the evaporation process again.
- d. There is no beam visible when program board has reached SOAK POWER2- This may happen for a number of reasons:
 1. The **SWEEP CONTROLLER** has most likely been set too far in either the X or Y position. On the **SWEEP CONTROLLER**, press **PRM**. Press the X offset and return it to 0.0 by moving the arrow in the up or down direction. Return the Y offset to 0.0. At this point, the beam should be in view and any further X/Y offset adjustments should be minimal (you should NEVER have to exceed ± 2.0 on the X/Y offsets to center the beam). If problems persist, press the **STOP** button on the **DEPOSITION CONTROLLER** and notify the process staff for assistance.
 2. With the **View Shutter** open, adjust the sacrificial viewport film by rotating the "Film" dial **CLOCKWISE** (you should NEVER have to turn the dial more than a 180° turn; if you do, you are wasting film). If this sacrificial viewport film becomes heavily coated with metal, it can make it difficult to see the metal source.
 3. Make sure that the polarizer on the chamber window is not rotated to minimize the light coming through; try rotating it 45°.