

## Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

### Thermionics Thermal Evaporator

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*These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.*

*Before using the MCC, please review the MCC User Handbook available through our website.*

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#### **THERMIONICS THERMAL EVAPORATOR INSTRUCTIONS Cr/Au, etc.**

Revised – 2/5/2019 by E.J.Basgall, Ph.D.

Users are required to provide their own W boats and precious metals (Au, Pt, etc. 99.99% minimum purity). The CRF has a limited supply of inexpensive metals (Cr, Al, etc.). Please check with Dr. Basgall, before your session, that the metal you need is available. ([ebasgall@coe.drexel.edu](mailto:ebasgall@coe.drexel.edu)). A list of suppliers is at the end of this document.

**System Start/Finish state: LOGON to iLab at nearby PC.**

**Check logbook for any previous user problems...Login your starting time, metals and target thicknesses. Use a separate line for each metal.**

Main power switch (lower center panel) – **ON**

Front left upper panel - Power switch – **ON** (Yellow light)

Vacuum gauge reading **750 torr**

Hoist indicator green light – **ON**

All Accessories – **OFF**

Sample plate removed and on the table.

**Water flow to QCM – ON/OFF - chiller/recirculator.**

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Check water level in tank, top off with dist H<sub>2</sub>O, if needed. Alarm will beep if level is too low.

**DO NOT adjust the water flow regulator.** It is set at ~0.25 GPM. (~1.0 L/min)

**CHECK Vacuum pump oil level** (remove front panel to access the Rotary Pump sight glass).

Top off with #19 Pump Oil, if needed.

Pump oils are located in room 106G in lower cabinet to the right of the sink.

**Do not forget to power OFF the chiller when finished.**

**Brief Description:** The Thermionics VE-Series of thermal evaporators has two thermal stations, a water-cooled Inficon Quartz Crystal Thickness Monitor and digital thermometer. The vacuum system is fully automatic with fast turbomolecular pumping.

It does not have a shutter or automatic deposition termination. Deposition is halted by turning off the High Voltage at the remote controller.

It is generally used for low melting point metals (Cr, Au, Al...) with W boats as the source. Each boat should be able to undergo 5-6 evaporations before breaking. W boats become brittle with use. Handle used boats as little as possible to extend their useful lifetime. Return any unused CRF metals and broken boats with residual metals to Dr. Basgall.

Do not mix metals in boats. A boat used for Au evaporation should only be refilled with Au...

Gloves must be worn anytime you are handling anything that will be in the chamber.

Powering OFF/ON main power (lower panel switch) will start a timer for the hoist.

It will not activate until the preset time has passed.

#### **Operation:**

**1. Raise the chamber to access the thermal boats and to load your sample.**

**Vacuum clean the inside of the chamber before and after each use.**

**Use the cleanroom vacuum to clean up the inside of the chamber. Do this BEFORE loading either samples or metals. Be sure that the sealing 'O' ring on the bottom of the chamber is clean and seated in the groove.**

**Vacuum clean the base and sidewalls of the chamber both before and after using. The metal end of the vacuum hose may be used as a scraper.**

**2. If the sample holder plate is not out, raise the chamber and remove it from the mounting post at the top, inside the chamber. Twist the plate CW(clockwise) and pull down to remove it from the bayonet mount.** Attach your samples to be coated to the flat side of the circular sample holder plate. Use the screws and clips provided to secure your samples. The samples will face down when mounted inside the chamber.

Kaptan tape can also be used. Your samples should not be able to "fall off" the holder. Remove any left over Kaptan tape from the plate when you are finished.

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**3. Re-attach the sample holder with your samples to the post inside the top of the chamber. Push up and rotate the plate CCW(counter-clockwise) until it clicks into place and stops.**

**4. Load the thermal boats with the appropriate metals.**

The front post set is source A – Use this set for Au. The rear post set is source B – Use this set for Cr and all other metals.

Use a new boat if you are using a different metal. To change W boats: Bolt head size is 9/16".

Loosen both bolts, remove the old boat, replace it with a new boat. Align boats centered between the Cu washer and the Cu post. Tighten bolts firmly but not too tight. Finger tight is good enough. Do not put strain on boats or they will break when heated.

**5. Set-up the Inficon QCM (Quartz Crystal Monitor).**

Turn on power. After the self check is finished, press the PROG button to go into “program” mode. Use the UP and DOWN arrow keys to navigate through the menu options. There is an indicating cursor that moves with the UP and DOWN arrow keys.

**Select FILM.** Enter “1” to choose film #1 (Cr) parameters, enter “2” to chose film #2 (Au). Move to the density window display with the DOWN arrow. Press PROG to exit or the UP and DOWN arrows to check, add or change additional film and density info. Refer to the chart below. Z Ratio = 1.0, Tooling Factor = 100.0. If the FILM number is blinking it has not yet been saved. Press E to enter and save.

Pressing ZERO will reset the thickness and timer readings. The timer is displayed at the lower right of the Inficom.unit screen. This is a convenient timer for stepwise heating cycles.

#### **Check the crystal lifetime**

XTAL will display the % of crystal life used. The crystal sensor should be replaced when it reaches 20-25%. Contact Ed Basgall for instructions on how to replace the crystal sensor.

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**TABLE 1. Inficon settings, MPs and recommended current values for thermal evaporation.** (Contact Dr. Ed Basgall for additional metals data). Refer to the wall chart containing physical properties of many metals.

Film #	Metal	Density (g/cc)	MP (C)	Amps
1	Cr	7.19	1857	160-170
2	Au	19.32	1064	100-110 1gm=250nm
3	Al	2.70	660	100
4	Ti	4.5	1660	150-160
5	Cu	8.92	1083	100-120
6	SnO2	6.95	1630	150-160?
7	Ag	10.49	962	80-90
8	Pb	21.45	1768	@70=3A/sec?
9	Ni	8.91	1453	100-?

6. Lower the chamber (guide it down with your hand to be sure it is centered) and turn on the MECHANICAL PUMP SWITCH..

The chamber should pump down to about  $7 \times 10^{-5}$  torr within 5 minutes.

7. Once the chamber is pumped down into the  $10^{-5}$  range, turn on the HV power to the electrodes. The yellow "Power" light and green "Ready" light should come on, both on the front main panel and remote.

8. Select source B for Cr or source A for Au. Use the remote unit "EMISSION" knob to ramp up the current. Be sure the knob is turned all the way CCW, then energize the remote by pressing the ON button. The red "ON" lights should illuminate on both the front main panel and remote.

Bring the current to 40A for 30 secs, then 60A, for 30 secs., then 80A for 30 secs...continue ramping up the current 20A/30secs until the Inficon rate monitor reads 1 - 3 angstroms/sec.

Refer to the above table for recommended current values for different metals. Monitor and record the chamber pressure during deposition for future reference.

9. Once the target thickness is reached, turn off the current by pressing the OFF button on the remote. Turn the current CCW all the way back down to "0". The green "ON" and yellow "START" lights will illuminate. Record the final thickness in the logbook and note the XTAL reading.

10. Repeat steps 7, 8 & 9 for the second metal. (Eg: Au.).

11. Once finished with all depositions, turn OFF the mechanical pump. The system will cool down and autovent to 750 torr within 10 minutes. Hoist light should activate within 15 minutes.

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**While waiting for the chamber to vent** - Turn off all accessories...Inficom QCM, thermometer, HV supply. Clean up any mess you made. **Fill in the paper Log** and record your ending time and final XTAL reading. The jar can be raised only when the green indicator light is on above bell jar switch. It is controlled by a timer. **(If the main power is cycled OFF/ON the timer will restart.** Plan on a 15 min wait.)

Please make a note in the log if you experienced any problems.

CAUTION! The sample holder plate will be HOT for about 10 more minutes after venting.

Remove your samples and leave the holder plate out on the console next to the chamber.

**12.** Lower the chamber but do not pump it down.

**13. LOG OFF the Thermal Evap in FOM, LOG OUT of FOM.**

### Ordering information for precious metals and W boats

W boats - thermal evaporation

Electron Microscopy Sciences - #73816-B6 5/pk @ \$34.00

<http://www.emsdiasum.com/microscopy/products/vacuum/pumps.aspx#73816>

Precious Metals -

Kurt Lesker - Au pellets, 99.99% or 99.999% pure call for pricing.

[http://www.lesker.com/newweb/deposition\\_materials/depositionmaterials\\_evaporationmaterials\\_1.cfm?pgid=au1](http://www.lesker.com/newweb/deposition_materials/depositionmaterials_evaporationmaterials_1.cfm?pgid=au1)

<http://www.lesker.com> Phone (800) 245-1656

International Advanced Materials - Au pellets 99.999% pure, call for pricing.

<http://www.iamaterials.com/materials/gold.htm>

(845) 352-5800 or [info@iamaterials.com](mailto:info@iamaterials.com)

Refining Systems - Precious metal foils and powders, call for pricing

<http://www.refiningsystems.com/>

Phone (702) 368-0579 Fax (702) 368-0933 or [adayani@earthlink.net](mailto:adayani@earthlink.net)

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