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Center for Functional Nanomaterials Brookhaven National Laboratory	NUMBER REVISION 2.0
Subject: Kurt J Lesker PVD-75 e-beam evaporator operating procedure	DATE 11/30/2012
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1. Important reminders before you get started:

- This document is not a substitute for system training. No one should use the instrument without proper training and authorization from Aaron Stein, Fernando Camino, Gwen Wright or Ming Lu.
- b. Remember to work safe.
- *c.* Do not attempt to make any changes to any other systems or touch the tool in any way unless authorized to do so by a CFN Staff member.

2. Tool booking rules

- a. We will use the Google Calendar for sign-ups.
- b. At this time, there are no limitations on how often and how long your session can be.
- 3. Basics
 - a. Please keep evaporator workbench and tools clean and in order.
 - b. You can only use materials that you have been authorized to deposit. If you want to try a new material, you need to get permission from a staff member. This may involve doing the deposition together, even after you have been trained.
 - c. Your evaporation run should follow closely a previous run for the same material and similar run parameters. You should know the expected run parameters. It is your responsibility to record and use these parameters. If during a run anything is significantly off these values (e.g, by more than 25%), stop work immediately and find a staff member to assist you. When in doubt, ask!
 - d. This SOP involves MANUAL mode deposition. DO NOT use AUTOMATIC mode deposition.
 - e. Only Users authorized by a staff member can change or refill crucibles. All precious metals (e.g. gold, silver, platinum, palladium) are kept locked up please get a staff member to get them for you to fill crucible if necessary.

- f. It is very important that you can see into the chamber during your process: make sure the mylar window liner is transparent enough. If you have any doubt about it, replace with a new one!
- g. Staff members that will assist you are Ming Lu (x4773), Aaron Stein (x3527), Gwen Wright (x4386), or Fernando Camino (x7606).
- h. Failure to follow these rules and this SOP will result in prohibition from using the evaporator.

4. Loading sample(s)

- a. If you ever need to start the software, the user name and password are both "user".
- b. You should find the machine under vacuum. Hit "Start PC Vent" to vent the machine ("PC" stands for "process chamber"). It will take ~10+ minutes to vent.
- c. To remove the sample platen, first open the sample shutter. You can do this on the "deposition" tab on the software display.
- d. An Allen key is on a hook on the machine which can be used to loosen the screws on the platen.
- Samples can be mounted anywhere on the holder with clips or tape (Kapton tape only).
 Feel free to move the clips as necessary. An Allen key that works with these screws is located on the side of the system. Return the wrench to its box when you are finished.
- f. Be mindful that the shutter does not cover the entire platen keep samples as close to the center as possible.
- g. Once samples are mounted, check to make sure that they are secure and won't fall off when the platen is upside down.
- h. If you are satisfied that the samples are mounted securely, place the platen back in place and screw tight with the Allen wrench. Do not overtighten.
- i. Check the sample shutter for correct operation. Leave it closed.

5. Loading evaporation sources

- a. If necessary and if you are authorized, change or refill the materials you want to evaporate. Otherwise, have a staff member (Aaron, Don, Gwen or Fernando) do it for you.
- b. There is a material list posted on the evaporator, which shows the current materials in each crucible position. Be sure to write the new arrangement down if you make any changes to the materials in the chamber.
- c. Make sure there is enough quantity of material (more than half of the crucible filled). Again, feel free to consult with a staff member.
- d. Crucible position is selected on the "deposition" tab of the software.

6. Chamber inspection and pump down

- a. It is important that you can see through the window during deposition. Make sure the mylar window is transparent (and be aware that more metal will be deposited on the window during your deposition).
- b. If a new mylar window liner is needed, change it by cutting an appropriately sized piece from the roll on the table.

- c. Always assume the old window has some toxic metal (e.g. chromium) deposited on it and treat the mylar as hazardous waste. Place in the satellite accumulation area under the table (white can).
- d. Inspect sealing surface and chamber door O-ring for pieces or lint, thread, etc. Call a staff member is you see any problem. It is best practice to wipe the O-ring with a wipe before pumping down.
- e. In addition, the chamber must be kept clean if there are many flakes of metal in the chamber vacuum them out with the vacuum cleaner.
- f. Inspect thickness monitor for cleanliness. If too many flakes are around crystal, please inform a staff member.
- g. One last thing is to check the life on the crystals in the monitors to make sure that they are sufficient.
 - i. Open the sigma software and check the lifetime on the crystals you will use.
 - ii. If it is above 80%, you are good to go.
 - iii. If any of the crystals you need to use are below 80%, you should probably change it.
 - iv. If you do not know how to change the crystal, or are uncomfortable doing so, ask a staff member to show you/do it for you.
- h. Now you are ready to pump the chamber. Press "Start PC Pump".
- i. The entire pumping process is automated. If after ~15 min the chamber is not in the 10^{-4} Torr range or better, there might be a problem. Please inform a staff member. It will take less than an hour to get into the mid 10^{-6} Torr range and will get into the 10^{-7} Torr range after a couple of hours. Overnight pumpdowns should be well into the low 10^{-7} range.

7. Evaporating

- a. After the system is pumped down sufficiently, you are ready to start the deposition.
- b. Open up the Sigma software and load the settings for the metal you will deposit.
 - i. File>OPEN and find the metal.
 - ii. Then you must hit VIEW>Setup and then OK in the window that pops up.
 - iii. Make sure the numbers have changed.
 - iv. Please note that the tooling factors have been calibrated, but may not be entirely accurate. You may want to calibrate the thickness if you are concerned about deposition accuracy.
- c. On the Lesker software, switch to the "Deposition" screen.
- d. Open the evaporator door (the one on which the screen is placed) to have access to ebeam controls. ONLY TOUCH THE CONTROLS DESCRIBED IN THIS OPERATING PROCEDURE!
- e. Make sure the MANUAL/AUTOMATIC switch in the bottom box (power supply) is in the MANUAL (upward) position. If not, flip this switch upward.
- f. Turn on power supply. First, rotate "e-beam disconnect" circular switch into "ON" position. Then flip upward the main power supply switch. Wait 2-3 minutes for the power supply to warm up.

- g. Verify that the interlock lights (ZERO, WATER, DOORS, VAC, READY) on the upper left side of the "sweep and control" box (second from the top) are all green. If not, please call a staff member.
- h. Check that "emission current" potentiometer is zero (turned all the way counterclockwise). If not, set it to zero.
- i. Turn on high voltage by depressing the controller's ON push-button.
- j. You should read about 7.8 kV and zero current on the voltage/current display. If voltage reading is different by > 10% or if current reading is not zero, turn off high voltage, and call a staff member.
- k. Make sure that the "sweep control" has sweep set to "spiral" (switch located below joystick), that the sweep potentiometers are all in the vicinity of 1.0, and that the center sweep switch is set to "control".
- I. REMEMBER: At any stage of the evaporation, the chamber pressure should not go above mid 10⁻⁵ Torr range.
- m. Slowly increase the emission current to ~5 mA, wait ~1 min while checking chamber pressure. If pressure keeps rising, wait until it starts to drop again. Check deposition rate in thickness monitor. Repeatedly verify that the beam is always inside the crucible, if not, bring down emission current to zero, turn off the high voltage, and call a staff member. <u>CAUTION</u>: WHEN VIEWING A BRIGHT SOURCE, USE TINTED WINDOW FROM WELDING GOGGLES (on workbench) TO AVOID DAMAGING YOUR SIGHT.
- n. Keep increasing emission current in ~ 5 mA increments as described in the previous step. Once you detect a deposition rate (material starts evaporating), pressure will generally increase as the rate increases, unless you deposit a "getter" material (e.g., Ti or Cr), in which case the pressure will actually drop.
- Slowly increase emission current at the rate of ~5 mA/min until you reach the desired deposition rate. Consult the log book for typical currents for a given deposition rate. Typical rates are 1-4A/s.
- p. Once you have reached the desired rate, let it stabilize, and start the actual deposition on your sample. For this purpose, open the sample shutter and "zero" the thickness monitor.
- q. Once the desired film thickness has been deposited, close the source shutter, bring down the emission current slowly, and turn off the high voltage.
- If depositing several layers select another source, and evaporate as explained above.
 Don't forget to load the right material file into "SQM242 Monitor" program and to update it as explained above.

8. Unloading Sample

- a. When you are done with all your films. Check that high voltage is off and that emission current potentiometer is at zero.
- b. Turn off main power (bottom box) and rotate "e-beam disconnect" circular switch into "OFF" position.
- c. To vent the system, simply hit "PC Vent" in the software. This will vent the chamber and make sure the system is in a proper state. It will take ~15 min to vent the chamber.

- d. After venting, remove the platen and your sample. Put the platen back into the chamber when you are done and pump the chamber back down.
- e. Be sure to mark the log book with your name, proposal number, material(s) deposited, chamber pressure, current and deposition rate as well as the total material deposited.
- f. Make sure you are leaving the workbench clean and all tools clean and in order.