Standard operating procedure for the Cooke Deposition Chamber

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Materials not-allowed: In, Pb, Bi, Zn
We can deposit non- metals such as SiO2 via sputtering and e-beam.

E-beam Deposition Standard Operating Procedure

1. Before venting the chamber, have samples and crucibles ready.
2. Make sure that the top/bottom hand screws on the door are released. If you don't it will be difficult to unscrew then ones the chamber is vented and O-ring inside the chamber expands
3. Toggle chamber vacuum control to VENT CHAMBER. The venting process takes up to 10 minutes. When not in use, the chamber should be kept under vacuum.
4. Once chamber pressure has reached atmosphere, open the chamber and toggle the vacuum control to STAND BY. Pull the door handle gently to open the door.
5. Make sure only clean gloves go in the chamber.
   NOTE: Check that the dark shields to the DC and RF sources are in place. If not, metals will deposit on them and cause arcing. If this happens the sources need to be sent out for cleaning and it is expensive!
6. The E-beam hearth is located on the left hand of the side of the chamber. On top of the hearth is the crucible holder. This holds the source crucibles during the deposition
   a. Locate the Sycon SRT-442 crucible indexer on the Cooke Panel on the right hand side. The current crucible exposed in the E-beam hearth corresponds to the number on the display. By rotating the POCKET SELECTION dial to positions 1 through 4, the hearth will expose the corresponding crucible.
   b. TMI provides several source materials. If another material is needed, it must be provided by the user as well as an appropriate crucible. Source material must fit completely in the crucible as material that protrudes from the crucible might catch on the indexer. No not fill crucible more than 2/3.
   c. Note which crucible is in which hearth pocket and what the corresponding number is on the indexer. TMI CURCIBLES CAN BE FOUND IN THE BOX ADJACENT TO THE TOOL.
      NOTE: there is 8,000 V potential difference between the copper feedthrough and the chamber. Make sure that no tools are left in the chamber that touch the two.

7. Fasten samples upside down to the bottom of the carousel located directly above the E-beam hearth using either binder clips of the bolts and tabs located in the yellow plastic bin to the right of the chamber.
8. Program the Inficon SQC-310 quartz microbalance (QCM) for the necessary process. Verify the lifetime of the crystal in use under the SENSOR INFO menu. For more instructions see below.
9. Close chamber door and toggle vacuum control switch to PUMP CHAMBER. Watch the middle pressure gauge and verify that it drops below atmospheric pressure. If it does not, simply grab the chamber door by both ends and squeeze. If this does not cause the pressure to drop, toggle the vacuum switch to STANBY and seek assistance. The chamber will reach 8 x 10-5 Torr in ~ 30 min.
10. Toggle the SOURCE SHUTTER to CLOSED, blocking samples from the source
11. To begin the E-beam deposition
    a. Locate the TT POWER SUPPLY on the lower left hand corner of the Cooke panel. Flip both CONTROL and POWER switches on.
    b. Locate the TT CONTROLLER at the upper left hand corner of the Cooke panel. Toggle the CONTROL POWER on. The AIR/CAB light will brink for two minutes. Wait for this light to stop blinking.
    c. Toggle the DSP SEL switch to FIL (filament) and press the green ON button on the source side (NOT THE HV SIDE). This will heat the filament up and the current through the filament will stabilize to ~20A.
    d. Once the filament current has stabilized, toggle the DSP SEL switch to EMIS (emission) and press the green button on the HV side (it will read 8kV). Rotate the EMISSION dial slowly until a current shows up on the display. Keep increasing until the current is ~ 15 mA.
    e. Look on the chamber, verify that the e-beam is striking the materials in the crucible, not the crucible and not the hearth.
    f. DO NOT TURN ON THE POWER SUPLY CONTROLLER FOUND BELOW THE TT CONTROLLER. ONLY TMI STAFF IS ALLOWED TO OPERATE THIS CONTROLLER.
    g. Toggle the SOURCE SHUTTER up to expose samples to the source.
    h. Use the EMISSION dial to increase current by 5 mA at a time, waiting 15 seconds before increasing again.
    i. Once a deposition rate appears on the QCM, use the rate as a guide for the emission current. SMALL CHANGES IN THE EISSION CURRENT CAN RESULT IN LARGE CHANGES IN THE DEPOSITION RATE!!!
    j. Once the QCM shows the desired thickness, toggle the SOURCE SHUTTER to CLOSED. Slowly decrease the emission current back to zero with the EMISSION dial.
    k. Push the OFF button on the HV side of the TT CONTROLLER, toggle the CONTROL POWER OFF as well.
    l. Flip both CONTROL and POWER switches off on the TT POWER SUPPLY on the lower left hand corner of the Cooke panel.

12. Toggle chamber vacuum control to VENT CHAMBER. Once vented, toggle vacuum control to STANDBY. Do not touch anything in the chamber with dirty gloves.
13. Exercise caution when removing samples and crucibles from the chamber! There could be residual heat from the deposition process.
14. Once samples and crucibles have been removed, close the chamber door and toggle the vacuum control to PUMP CHAMBER, again verifying that the pressure drops below the atmosphere before leaving the instrument. IF THE VACUUM SEAL DOES NOT FORM AND THE PUMP IS LEFT RUNNING, IT WILL GET DISTROYED.

<u>Note:</u> At the top of the chamber there are some heaters that can be used to clean-up the chamber. In order to clean the chamber set the Temperature Control to OUTPUT ENABLE. It is set to 120°C. When 120°C is reached the light goes off. As it heats up one can see how the pressure rises. This operation should be done under supervision from TMI staff. The heater cannot be operated at the same time with the DC and RF- it will blow a fuse!

RF Sputtering Standard Operating Procedure

1. Before venting the chamber, have samples and crucibles ready.
2. Toggle chamber vacuum control to VENT CHAMBER. The venting process takes up to 10 minutes. When not in use, the chamber should be kept under vacuum.
3. Once chamber pressure has reached atmosphere, open the chamber and toggle the vacuum control to STANDBY.
4. The RF sputter head is located in the rear of the chamber on the right behind the DC sputter head. First change out the sputter target:
    a. Make sure only clean gloves are used inside the chamber.
    b. For easier access, the DC sputter head may be rotated down for this procedure
    c. Tilt the RF sputter head toward the chamber door and remove the dark space shield on the sputter head by loosening the three hex bolts on the shield. There should be an appropriate hex key to the right of the chamber.
    d. Remove the retaining ring holding the target down with a flat head screwdriver also located to the right of the chamber.
    e. Replace target and refasten the retaining ring. Tighten the screws till just hand tight. DO NOT OVERTIGHTEN!
    f. Replace the dark space shield, choosing the appropriate notch on the dark space shield to produce a 1-2 mm gap between the inner tip of the shields and the top of the target. Tighten the screws to secure the dark space shield and set the sputter head vertically.
    g. The sputtering targets can be purchased from Kurt J Lesker; diameter: 3" and they can be up to 1/4 inch thick.

NOTE: The shields should always be in place (even if the DC or RF sputtering are not used). If not, metals will deposit on them and cause arcing. If this happens the sources need to be sent out for cleaning and it is expensive.

5. Fasten samples upside down to the bottom of the carousel located directly above the sputter head using binder clips or Kapton tape. A test microscope slide can be put under the sputter head first to verify material deposition and then the samples can be rotated above the sputter head using the sample rotation controls. Writing on the glass slide with a marker provides an easy visual indication of deposition.
6. The sample to sputtering head distance is a crucial parameter for uniform deposition and varies from material to material. It should be set to at least 1 inch
7. Close chamber door and toggle vacuum control switch to PUMP CHAMBER. Watch the middle pressure gauge and verify that it starts dropping. If it does not, simply grab the chamber door by both ends and squeeze. If this does not cause the pressure to drop, toggle the vacuum switch to STANDBY and seek assistance. The chamber will reach 5x10-5 Torr in ~ 30 min.
8. To begin RF sputtering
    a. Verify the chamber pressure with the high vacuum gauge (top pressure display) and the settings of the Ar mass flow controller. The flow is set to 50 sccm, but can be adjusted to other values, if needed.
    b. Toggle the GAS FLOW and the CHAMBER THROTTLE switches up. Wait for the pressure to stabilize.

c. Toggle the power on the RF tuner, PI-II-CE. Verify that both switches are set to AUTO and that the needle is set in the middle of the range. If not set to manual and adjust. At the end set again to AUTO. Do this on both indicators.
d. On the RF VII module, turn on the power to the unit with the lower right hand switch.
e. Make sure that the power knob is set to zero, then push the ENABLE button.
f. Slowly turn the power knob clockwise. Eventually the incident power will begin to increase. Bring incident power up to 40-60W and wait for the RF tuner to minimize the reflected power.
g. Visually confirm that the Ar plasma has been struck and then toggle the CHAMBER THROTTLE switch down.
h. Increase the incident power up to the desired wattage, while making sure the reflected power is never more than 10 % (ideally it will be 1-2W the entire time). If the reflected power exceeds 10% immediately lower the incident power to 0W. Re-engage the THROTTLE switch up and try increasing the power again. If similar issues are experienced forming a stable plasma, turn off the RF VII and seek assistance.
i. Once deposition has been achieved, while looking in the chamber, rotate the chamber carousel such that the microscope slide is transitioned out of the plasma and sample have been rotated under the sputter head. Rotation controls are located on the lower left corner of the Cooke control panel.
j. Measure the deposition rate: Time your deposition for the given conditions and use the optical profilometer to measure the film thickness.
k. Once finished, rotate the incident power knob counterclockwise back to zero. Push the ENABLE button and turn off the power to the RF VII.
l. Turn off the tuner and toggle the gas flow switch off.
Note: The THROTTLE valve is located into the vacuum line that goes from the turbo to the deposition chamber. If this is closed in the THROTTLE position the pumping is reduced.
Note for aluminum deposition: Since Aluminum oxidizes the power should be increased initially to 300-400W until we see the deposition start. Then go down to 320W and use rotation to bring the desired substrate above the RF sputtering head.
Note: if the reflected power cannot be controlled below 10%, vent and check
    - make sure the target does not touch the shield
    - after removing the dark shield check to see that there are no deposits at the base
    - remove target and check it the back of the target is clear
    - lastly increase the target to sample distance

9. Toggle chamber vacuum control to VENT CHAMBER. Once vented, toggle vacuum control to STANDBY. Verify that only clean gloves are going into the chamber.
10. Exercise caution when removing samples and targets from the chamber! There could be residual heat from the sputtering process. Never attempt to remove anything if heat can be felt
11. Once samples and targets have been removed, replace the retaining ring, and dark space shield over the sputter head. Close the chamber door to toggle the vacuum control to PUMP CHAMBER, again verifying that the pressure drops below atmosphere before leaving the instrument. IF THE VACUUM SEAL DOES NOT FORM AND THE PUMP IS LEFT RUNNING IT WILL GET DISTROYED.

DC Sputtering Standard Operating Procedure

1. Before venting the chamber, have samples and crucibles ready
2. Make sure that the top/bottom hand screws on the door are released. If you don't it will be difficult to unscrew then ones the chamber is vented and O-ring inside the chamber expands
3. Toggle chamber vacuum control to VENT CHAMBER. The venting process takes up to 10 minutes. When not in use, the chamber should be kept under vacuum.
4. Once chamber pressure has reached atmosphere, open the chamber and toggle the vacuum control to STANDBY.
5. The DC sputter head is located in the front of the chamber on the right in front the RF sputter head. First change out the sputter target:
    a. Make sure only clean gloves are used inside the chamber
    b. Tilt the DC sputter head toward the chamber door and remove the dark space shield on the sputter head by loosening the three hex bolts on the shield (do not pool them all the way out). There should be an appropriate hex key to the right of the chamber
    c. Loosen the top two screws of the retaining ring holding the target down with a flat head screwdriver also located to the right of the chamber.
    d. Insert the target and refasten the retaining ring. Tighten the screws till just hand tight. DO NOT OVERTIGHTEN!
    e. Replace the dark space shield, choosing the appropriate notch on the dark space shield to produce a 1-2 mm gap between the inner tip of the shields and the tiop of the target. Tighten the hex bolts to secure the dark space shield and rotate the sputter head so that it is vertical

NOTE: The shields should always be in place (even if the DC or RF sputtering are not used). If not, metals will deposit on them and cause arcing. If this happens the sources need to be sent out for cleaning and it is expensive!

6. Fasten samples upside down to the bottom of the carousel located directly above the sputter head using either binder clips or the bolts and tabs located in the yellow plastic bin to the right of the sputter chamber. A test microscope slide can be put under the sputter head first to verify material deposition and then the samples can be rotated above the sputter head using the sample rotation controls. Writing on the glass slide with a marker provides an easy visual indication of deposition.
7. The sample to sputtering head distance is a crucial parameter for uniform deposition and varies from material to material. It should be set to at least 1 inch.
8. Close chamber door and toggle vacuum control switch to PUMP CHAMBER. Watch the middle pressure gauge and verify that it drops below atmospheric pressure. If it does not, simply grab the chamber door by both ends and squeeze. If this does not cause the pressure to drop, toggle the vacuum switch to STANDBY and seek assistance. The chamber will reach 8 x 10-5 Torr in ~ 30 min.
9. To begin DC sputtering
    b. Verify the chamber pressure with the high vacuum gauge (top pressure display) and the settings of the Ar mass flow controller; The flow is set to 50 sccm, but can be adjusted to other values, if needed.
    c. Toggle the GAS FLOW and the CHAMBER THROTTLE switches up. Wait for the pressure to stabilize.

d. On the DC HIGH VOLTAGE CONCEPTS LLC module, turn on the power to the unit with the lower left hand switch. Before doing this make sure that both the power and voltage knobs are turned counter-clowise all the way.
e. Push the HV ON button.
f. There are two displays and two LEDs. One for the voltage and one for the Power. Slowly turn the Voltage knob clockwise to increase the potential bias as indicated by the voltage display. At a high enough potential, the plasma will spark and the Power LED will light up. Once it does, slowly increase the Power knob to increase the wattage of the plasma. This will control the deposition rate. Good starting: 290V and 70W.
g. Visually confirm that the Ar plasma has been struck and then toggle the CHAMBER THROTTLE switch down.
h. Increase the incident power up to the desired voltage. Be aware that at higher powers the plasma will become unstable. If this occurs, lower the power back to the stable point. If the power is insufficient for sputtering, reengage the THROTTLE switch up and try increasing the power again, this time with more Ar gas in the chamber
i. Once the deposition has been achieved, while looking in the chamber, rotate the chamber carousel such that the microscope slide is transitioned out of the plasma and samples have been rotated under the Cooke control panel.
j. Measure the deposition rate: Time your deposition for the given conditions and use the optical profilometer to measure the film thickness.
k. Once finished, rotate the power knob clockwise back to the incident power of zero. Rotate the Voltage knob back to zero and push the OFF/RSER button and turn off the power to the module.
l. Turn off the tuner and toggle the GAS FLOW switch off.
Note: The THROTTLE valve is located into the vacuum lines that goes from the turbo to the deposition chamber. If this is closed in the THROTTLE position the pumping is reduced.

9. Toggle chamber vacuum control to VENT CHAMBER. Once vented, toggle vacuum control to STANDBY. Verify that only clean gloves are going into the chamber
10. Exercise caution when removing samples and targets from the chamber! There could be residual heat from the sputtering process. Never attempt to remove anything if heat can be felt
11. Once samples and targets have been removed, replace the retaining ring, and dark space shield over the sputter head. Close the chamber door to toggle the vacuum control to PUMP CHAMBER, again verifying that the pressure drops below atmosphere before leaving the instrument. IF THE VACUUM SEAL DOES NOT FORM AND THE PUMP IS LEFT RUNNING IT WILL GET DISTRYED.

Instructions for using the Inficon SQC-310 Deposition Controller

Setting up a new process

First, create a new film

1. If necessary, press the "next menu" button until you see 'film menu' in the list. Press the "film menu" button
2. If you do not see the material you are interested in depositing in the list, scroll to an empty slot and press 'create'. If the material is already in the list, skipt to step 5.
3. Press "edit name" and change the name to the materials of interest
4. Press 'edit' and scroll down to material using the scroll wheel. Press scroll sheel to select the material and scroll through the list to find your material
5. Exit to the main menu

Next, create a new process

1. If necessary, press the "next menu" button until you see 'process menu' in the list. Press the 'process menu' button
2. Scroll to an empty slot and press "create"
3. Press "edit name" and change the name to indicate the deposition method and the material of interest
4. Press "select" and then "edit" and then "insert new". Choose your material from the list and press "insert normal"
5. Press "edit" and scroll down to the sensors. Turn on the corresponding sensor
    - SENSOR 1 for Ebeam evaporation
    - SENSOR 2 for RF Sputtering
    - SENSOR 3 for DC Sputtering
6. Exit the main menu

Selecting a previous made process

1. If necessary, press the "next menu" button until you see 'process menu' in the list and press this button. Use the scroll wheel to highlight your process and "select"
2. Exit the main menu
3. If you do not see the indications on the screen for deposition rate and thickness, press "next menu" until you see "next graph" in the list
4. Press "next graph" until the deposition rate and thickness is displayed
5. Press "next graph" until zero is observed in the list
6. The deposition monitor is now ready