

INSTRUCTIONS FOR EMS 550X SPUTTER COATING DEVICE

I. SPECIMEN PREPARATION

A. Dry specimen. This can be done by either air drying, with HMDS, or by critical point drying.

B. Mount sample onto a specimen support stub:

1. The most common type of stub is composed of aluminum. If you intend to do elemental x-ray micro-analysis (or EDS) on your specimen you might consider using a carbon/graphite stub instead.
2. In most cases, the use of double-stick carbon tape is sufficient. If another solvent-based adhesive is used (e.g. nail polish, silver colloidal paint, etc), allow it to dry out thoroughly before sputter coating.
3. Pre-pump any large porous or gaseous samples in another vacuum chamber ahead of time to reduce the length of pumping time required on these types of specimens.

II. SPUTTER COATER OPERATION

A. Place specimen/support stub into the chamber:

1. Gently raise the sputtering head and carefully remove the glass chamber.
2. Place your specimen/support stub(s) into one of the holes on the holder.
3. Carefully replace the glass chamber making sure it is seated properly on the lower side of the chamber.
4. With two hands, carefully close the top sputtering head. Again, make sure the sputtering head is seated properly onto the glass chamber.

B. Switch power **ON** with switch located on right rear of instrument. The **STOP** button and the L.E.D display window should be illuminated.

Appearance of display window at this step:

Press ENTER to change parameters Press START to run when ready
Settings: 25mA, 00:02:00 H:M:S

C. **OPEN** the argon tank using the main tank valve only. **DO NOT** adjust the regulator setting.

D. Press the **STOP** button. This will purge the chamber with argon gas for 30 seconds as indicated on the display window shown below:

Bleeding Gas into Chamber
Time Remaining 00:00:30 H:M:S
Vacuum : 2x10 ⁻¹ mbar

E. When the display has returned to the initial layout (see display in step II B), press the **START** button. Watch the vacuum reading. Vacuum values should decrease. If they do not, check the glass chamber seal for leaks or simply apply gentle pressure to the sputtering head to help seat it properly on to the glass chamber.

F. With the **Pump Hold** feature enabled, the instrument will pump to **1 x 10⁻¹ mbar** (bleed point) as normal, then the **Pump Hold** feature will activate. **Pump Hold** time is set for 10 minutes. The display should look like this:

Pump Holding, Press a key to continue		
Time Remaining	00:10:00	H:M:S
Vacuum : 1x10 ⁻¹ mbar		

G. Allow the vacuum reading to drop to **7x10⁻² mbar** and then push the **Enter** button to override any remaining pump hold time. Should the enter button not be pushed, the sputter coating cycle will begin after the pump hold time has expired.

H. After pushing the enter button or after the pump hold time has expired, argon gas will “bleed” in to the chamber causing a slight increase in the vacuum reading. After approx. 15 seconds, the sputter coating process will begin and a violet plasma should be visible inside the chamber. The vacuum reading should show **1 or 2 x 10⁻¹ mbar**. If necessary, you may need to adjust the needle valve at the left rear of the instrument to achieve a stable vacuum of **1 or 2 x 10⁻¹ mbar**.

Appearance of display window at this step:

Coating Sample at 25 milliamps		
Vacuum	Current	Time Remaining
1x10 ⁻¹ mbar	25 mA	00:01:38 H:M:S

I. Allow the sputter coating process to finish. After sputter coating, the chamber will vent automatically to atmosphere with argon. Venting will take 30 seconds to complete.

J. Remove specimen support stubs:

1. After chamber reaches atmosphere, gently lift sputtering head off of glass chamber and remove the glass chamber.
2. Remove your stub(s) with the SEM forceps. **DO NOT** use your fingers.
3. Carefully replace the glass chamber making sure it is seated properly on the lower side of the chamber.
4. With two hands, carefully close the top sputtering head. Again, make sure the sputtering head is seated properly onto the glass chamber.

K. **CLOSE** argon main tank valve.

L. Turn sputter coater **OFF**.

M. **Be Sure to Record Your Sputter Coating Time in the Log Book.**

III. ADDITIONAL INFORMATION

A. To Check or Modify Parameters

1. From the main display menu (see display in step II B) press **ENTER** to select the change parameters menu. The screen below should appear in the window:

UP and DOWN key to ALTER ENTER to accept, STOP to return <i>Variable Name</i> <i>Current Value</i>
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2. For most parameters, the value is shown along with the variable name. Use the **UP** and **DOWN** arrow keys to change the values as required. When the desired value is shown in the window, press the **ENTER** key to accept that selection. To exit from the parameter editing menu, press the **STOP** key.

B. Setting Operating Parameters

1. There are a number of user programmable options that can be altered by the front panel keys. When these parameters are modified, they will be saved and in force the next time that the instrument is turned on.

Parameter	Allowable Value	Description of Parameter
Coating Current	0-50 mA in 5 mA steps	Plasma current, default value is 25 mA
Coating Time	0-4 min in 1 second steps	Time of coating, default value is 2 min.
FTM Enabled	yes or no	Film Thickness Monitor, default is NO
FTM Type	external or internal	default is External
FTM Mode	automatic or manual	default is Automatic
Material	gold or other	default is Gold, density = 19.32g/cm³
FTM Terminate		default is 005.nm
FTM Tool Factor		default is 1.0
Pump Hold Enabled	Yes / No	Extra pumping time is enabled or disabled. Default setting is YES
Pump Hold Time	0-8 hr in 5 sec steps 00:00:00 H:M:S	Set amount of extra pumping time, default time is 00:10:00 (10 minutes)
Stage Rotate Enabled	Yes / No	Rotate stage during coating, default setting is YES

C. Pump Hold Feature

1. The **Pump Hold** feature is used for one of two reasons:
 - a. If it is required to outgas a specimen more than would be possible during the normal automatic cycle, then select **Pump Hold Enabled** in the parameter menu. This will prevent the cycle from continuing until either a key is pressed or the stored **Pump Hold Time** elapses, whereby the cycle will continue in the normal

manner. The vacuum should eventually achieve better than 7×10^{-2} mbar.

b. If it is required to use the EMS 250 Carbon Coating Attachment (see Instructions for EMS 250 Carbon Coating Attachment). If this is the case, then select **Pump Hold Enabled** and select a fairly long **Pump Hold Time**, perhaps longer than 30 minutes. The EMS 550X is now used as a vacuum chamber for the carbon evaporation head.

2. When **Pump Hold** is enabled, the instrument will pump to 1×10^{-1} mbar (bleed trip point) as normal, then the **Pump Hold** feature becomes active. The display should look like this:

Pump Holding, Press a key to continue
Time Remaining 00:04:36 H:M:S
Vacuum : 8×10^{-2} mbar

3. The Pump Hold time has a default value of 10 minutes but it can be programmed for up to 8 hours. As stated above, the instrument will continue pumping until either the time elapses or a key is pressed. The cycle will continue as normal.

INSTRUCTIONS FOR USING THE EMS 250 CARBON COATING ATTACHMENT

I. SPECIMEN PREPARATION

- A. Dry specimen. This can be done by either air drying, with HMDS, or by critical point drying
- B. Mount sample onto a specimen support stub:
1. The most common type of stub is composed of aluminum. If you intend to do elemental x-ray micro-analysis (or EDS) on your specimen you might consider using a carbon/graphite stub instead.
 2. In most cases, the use of double-stick carbon tape is sufficient. If another solvent-based adhesive is used (e.g. carbon colloidal paint), allow it to dry out thoroughly before coating.
 3. Pre-pump any large porous or gaseous samples in another vacuum chamber ahead of time to reduce the length of pumping time required on these types of specimens.

II. COATER OPERATION

- A. Place specimen/support stub into the chamber:
1. Gently raise the sputtering head and carefully remove the glass chamber.
 2. Place your specimen/support stub(s) into one of the holes on the holder.
 3. Carefully replace the glass chamber making sure it is seated properly on the lower side of the chamber.
 4. **DO NOT** close the top sputtering head. For carbon coating, leave sputtering head open or in the upright position.
- B. Load carbon in to carbon coating head:
1. Unit is stored behind sputter coater under a large plastic beaker.
 2. Stand carbon head on sputter coater while loading. Rubber feet should be down right now.
 3. Carefully load a small section of braided carbon rope in to electrodes and trim off excess with scissors.
 4. Carefully invert carbon coater and place on top of glass vacuum chamber. Rubber feet should now be pointing upward.
- C. Switch power **ON** with rocker switches located on right rear of **BOTH** units. The **STOP** button and the L.E.D display window should be illuminated on the EMS 550X unit (left-hand unit) and the 12V ac light and the 12V dc light should be lit on the EMS 250 unit (right-hand unit).

Appearance of 550X display window at this step:

Press ENTER to change parameters Press START to run when ready
Settings: 25mA, 00:02:00 H:M:S

D. **OPEN** the argon tank using the main tank valve only. **DO NOT** adjust the regulator setting.

E. If you want the stage to rotate during carbon coating, make sure the coating time on the 550X display window reads 6 minutes or more. If you don't want the stage to rotate, leave the coating time at 2 minutes and then make sure the **Pump Hold** feature is enabled and is set for at least **15 minutes**. See the **Section III: Additional Information** if you need help locating or changing these settings.

F. Press the **START** button. Watch the vacuum reading. Vacuum values should decrease. If they do not, check the glass chamber seal for leaks or simply apply **gentle** pressure to the sputtering head to help seat it properly on to the glass chamber.

G. At a vacuum reading of **1 x 10⁻¹ mbar**, outgas the carbon rope by doing the following:

1. Rotate heat shield over your sample (if desired)
2. Push the **Outgas button**
3. Slowly turn the **Outgas Current Knob** clockwise until the rope begins to glow a good **orange color** and hold for approximately **60 seconds**. Keep watching the rope while you are doing this. You will need to make constant adjustments to the current knob during this 60 seconds in order to maintain this **orange glow**. Otherwise, it tends to get brighter over time. Reduce the current knob if necessary. While you are doing this, the vacuum reading will go up (e.g. maybe **3 or 4 x 10⁻¹ mbar**).
4. After ~60 seconds, turn **Outgas Current Knob** down and push the **Outgas Button** again to disengage.
5. Remove the heat shield from your sample while the system continues to pump down.

H. At **7 x 10⁻² mbar** evaporate the carbon rope. For quick evaporation, do step 1. For slow and controlled evaporation, complete steps 2 through 6.

1. For quick evaporation, push and hold the **Evaporate Button** until bright glow stops.
2. For slow evaporation, first push the **Outgas Button**
3. Slowly increase current with the **Outgas Current Knob** until the rope is bright white (~35-40 Amps). The goal here is to keep it as bright as possible with minimal amount of sparking. If the carbon rope begins to spark, reduce current slightly but try to maintain a bright carbon source at all times.
4. Hold at this level for as long as possible (~15 seconds to 1 min). After this, increase the current to the maximum to finish off the rope.
5. Once the rope is finished, reduce the current to zero with the **Outgas Current Knob**.
6. Push the **Outgas Button** again to disengage.

I. Push the **STOP button** twice and allow the chamber to vent. Venting will take 30 seconds to complete.

J. Remove specimen support stubs:

1. After chamber reaches atmosphere, gently lift carbon coating head off of glass chamber and stand coating head rubber feet down on to sputter coater unit.
2. Carefully remove glass chamber.
3. Remove your stub(s) with the SEM forceps. **DO NOT** use your fingers.
4. Carefully replace the glass chamber making sure it is seated properly on the lower

side of the chamber.

5. With two hands, carefully close the top sputtering head. Again, make sure the sputtering head is seated properly onto the glass chamber.
6. Clean up coating head by removing any unused carbon rope from electrodes and brushing off any carbon debris. **CAUTION: ELECTRODES MAY BE HOT!!** 
7. Place carbon coating head back behind sputter coater and cover unit with large beaker

K. If you made changes to the sputter coating time in the beginning, please reset this to **2 min.**

L. **CLOSE** argon main tank valve.

M. Turn **OFF** both sputter coating units.

N. **Be Sure to Record Your Sputter Coating Time in the Log Book.**

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FTM Type	external or internal	default is External
FTM Mode	automatic or manual	default is Automatic

Material	gold or other	default is Gold, density = 19.32g/cm³
FTM Terminate		default is 005.nm
FTM Tool Factor		default is 1.0
Pump Hold Enabled	Yes / No	Extra pumping time is enabled or disabled. Default setting is YES
Pump Hold Time	0-8 hr in 5 sec steps 00:00:00 H:M:S	Set amount of extra pumping time, default time is 00:10:00 (10 minutes)
Stage Rotate Enabled	Yes / No	Rotate stage during coating, default setting is YES

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Pump Holding, Press a key to continue Time Remaining 00:04:36 H:M:S Vacuum : 8×10^{-2} mbar
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