The SPI Sputter Coater Handbook
SPI-Module Sputter Coater with Etch Mode

Coating of Specimens

1. Mount the specimens onto the SEM stub. Keep in mind that many adhesives have high vapor pressure solvents associated with them, and these solvents must be allowed to evaporate fully before sputtering. The stub holder can hold up to 6 specimen mounts.

2. Replace the glass work chamber and place the gold sputter head on top of the chamber.

3. Check to make sure the cords from the top of the sputter head are plugged into the back of the SPI-MODULE Sputter Coater box, and the power cord from the SPI-Module Sputter Coater box is the one plugged into the SPI-MODULE Control.

4. Switch on the SPI-MODULE Quartz Crystal Thickness Monitor power switch.

5. Dial in the density of the evaporant or sputtered material.
   - Gold – 19.4 g/cc
   - Carbon – 2.2 g/cc

6. Use the ‘ZERO’ adjustment to set the display to 0.00.

7. Turn the timer to the required setting. BROKEN

8. Check that the leak valve is fully closed.

9. Switch the SPI-MODULE Control ‘POWER’ switch and the SPI-MODULE Sputter Coater “POWER” switch on. The rotary pump will start immediately and the vacuum will be indicated on the meter.
10. Partially open the gas leak valve to flush the work chamber with argon for about 10-15 seconds. Close the leak valve and allow the work chamber to pump down to approximately $10^1$ millibar.

11. Open the gas leak valve until the pressure just begins to rise and by depressing the test button and adjusting the gas leak valve set the plasma current to 18 mA. A visible discharge should be observed in the chamber.

12. Depress the start button and gold will be sputtered onto the specimen for the time set in step 4. The plasma will automatically extinguish at the end of this period. Turn the mode switch to off to extinguish sputtering.

13. Switch off both “POWER” switches. Admit air into the chamber using the leak valve on the top of the sputter head.

14. Clean both the glass chamber and the quartz crystal thickness monitor with isopropanol.

Use of Etching Mode
The use of etch mode allows the cleaning of contaminants from the specimen prior to sputtering. The etching current is limited by internal resistance. Proceed as for the sputtering, and after the plasma current is set turn the mode switch to etch and initiate the etching by depressing the start button with timer set for an appropriate time. When the etching stops switch to sputter mode and proceed with sputtering.

Replacement of the Gold Target
Disconnect the unit from the power source. Remove the sputter head and disconnect the h.v. leads from the head. Loosen the three allen set screws that retain the outer ring and remove the ring. Unscrew the target retaining ring, this should unscrew easily, however sometimes after extensive use it may be necessary to lubricate the threads with isopropanol. Remove the old target, replace with the new target and reverse the process above to reassemble.
SPI-Module Carbon Coater

Loading Evaporation Sources

CARBON FIBER – The carbon fiber ‘EVAPORATION HEAD’ has two clamps for holding the fiber. A piece of fiber is inserted between these clamps and the two screws locked up. Superfluous fiber can be trimmed off with scissors.

CARBON RODS – The carbon rods (¼” dia.) should be loaded so that the fixed rod terminates in a smooth flat face over the center line of the evaporation head. The fine pointed spring-loaded carbon should be fitted so that it is touching the fixed rod and clamped so that maximum spring tension is applied.

NOTE: Both evaporation heads get extremely hot during evaporation, and subsequent carbon reloading should only be attempted when the heads have cooled down.

Coating of Specimens

1. Mount the specimens onto the SEM stub. Keep in mind that many adhesives have high vapor pressure solvents associated with them, and these solvents must be allowed to evaporate fully before sputtering. The stub holder can hold up to 6 specimen mounts, and is mounted to the stage by a screw.
2. Replace the glass work chamber and place the sputter head on top of the chamber.
3. Check to make sure the cords from the top of the sputter head are plugged into the back of the SPI-MODULE Sputter Coater box, and the power cord from the SPI-Module Sputter Coater box is the one plugged into the SPI-MODULE Control.
4. Check that the leak valve is fully closed.
5. Switch the SPI-MODULE Control ‘POWER’ switch on, and allow the pressure to fall.

6. Ensure that the voltage control is set to zero and the rocker-switch is in the center ‘OFF’ position.
7. Switch the SPI-MODULE Carbon Coater ‘POWER’ switch on.

8. Select position 1 on the back panel-mounted ‘MODE CONTROL’ switch for carbon rod evaporation, or position 2 for carbon fiber evaporation.
9. Select the ‘CONT’ position on the rocker switch and gradually increase the voltage control until the fiber is glowing bright red in color. The vacuum will initially drop, and after a period of time it will regain previous pressure.
10. Select the ‘PULSE’ position on the rocker switch. Turn the voltage control to 7½ or 8 and pulse the pulse button until the carbon evaporation is complete. This is usually indicated by the current ceasing to flow.
11. Immediately after evaporation turn the voltage control back to zero and set the rocker switch to center ‘OFF.’ Admit air into the chamber using the leak valve on top of the sputter head.
12. Clean both the glass chamber and the quartz crystal thickness monitor with isopropanol.
Miscellaneous Information

**Thickness of coating**

Generally a gold film of thickness 100-300 angstroms is used for investigations. An experimentally determined measure of thickness can be obtained from the following equation:

\[ d = KIVt \]

where \( d \) is the thickness of the coating in angstroms. \( K \) is an experimentally determined constant depending on the metal being sputtered and the gas being used based on a distance of approximately 5 cm between the target and the specimen. \( K \) is approximately 0.17 for gold used in conjunction with argon, but falls to 0.07 for gold used in conjunction with air. \( I \) is the plasma current, \( V \) is the voltage applied (1 kV), and \( t \) is the sputtering time in seconds.

Other targets give different sputtering rates (e.g. a platinum target gives approximately half the sputtering rate of gold.)

The sputtering rate will also depend on the cleanliness of the sputtering system. Traces of contamination which can originate from out gassing specimens, adhesives, rubber gaskets, etc. affect the rate of sputtering. Care should be taken to keep the work chamber clean.

**Cleaning the System**

The glass work chamber can be cleaned easily using a dry mildly abrasive material. If a “wet” cleansing material is to be used, it should be isopropanol, which will not affect the vacuum performance of the system.

**SPI-Module Quartz Crystal Thickness Monitor**

The quartz thickness monitor operates by monitoring the frequency of a quartz crystal when loaded with evaporation material deposited onto the crystal. The frequency shift is related to the mass of the material deposited.

When using the equipment it is always assumed that the source to the crystal and the source to the substrate distances are the same. It must be remembered that the thickness of the coatings (from point source) will be inversely proportional to the square of these distances.

The crystal is a 5 MHz “AT” cut plano convex quartz crystal operating in parallel resonance. It will function normally until the total thickness of the deposited material causes the oscillation frequency to be outside the range of the measuring system. At this point, which corresponds to about 2 microns of gold, the digits on the display will no longer alter during deposition. The crystal must then be changed, which is simply done by unscrewing the top plate of the crystal holder.