VCR Ion Beam Sputter Coater

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Sputtering Process and Rates

The VCR Ion Beam Sputter Coater produces ultra-fine grain coatings suitable for high resolution SEM and TEM. Platinum, for example, has a grain size of 1-3 nm and coating granularity only becomes apparent at 100,000 – 120,000 magnification.

<table>
<thead>
<tr>
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<th>Approximate Sputter Rates</th>
<th>Typical Sputter Durations</th>
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<tbody>
<tr>
<td>Platinum</td>
<td>2 Angstroms/minute</td>
<td>6 minutes</td>
</tr>
<tr>
<td>Tungsten</td>
<td>1 Angstroms/minute</td>
<td>15 minutes</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.5 Angstroms/minute</td>
<td>15-20 minutes</td>
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The Ion Source emits a fine beam of ions and energetic neutrals onto a Target of deposition material. The material is then sputtered off the target surface onto the specimen without thermal or photon effects. The sputtering process is conducted under high vacuum.

The target holder can accept up to six individual target materials. Target selection is accomplished with a dial on the outside of the chamber.

During coating, the specimens undergo a complex rotating planetary motion – thus allowing irregular surfaces to be uniformly coated.
Vacuum System

As mentioned above, the sputtering process is conducted under high vacuum. The diagram below shows the main components associated with the vacuum system. There are several RULES you should follow when operating this system:

1. **NEVER** turn on the Diffusion Pump unless its Cooling Water Supply Valve is open.
2. **NEVER** operate the ION Source unless its Cooling Water Supply Valve is open.
3. **NEVER** allow Backing and Roughing Valves to be open simultaneously.
4. **NEVER** allow Roughing and Main Valves to be open simultaneously.
5. **NEVER** vent the Chamber when the Main Valve is open.
6. **NEVER** open the Roughing Valve if the Chamber is at high vacuum, even if the Main Valve is closed. You should always close the Main Valve and at least partially vent the chamber before opening the Roughing Valve; otherwise, oil can be pulled into the Chamber from the Roughing Line.
Loading the Sputter Chamber

1. Before starting, check the following: Diffusion Pump (ON); Main Valve (CLOSED); Roughing Valve (CLOSED); Backing Valve (OPEN); Range Select switch (OFF); Power switch (OFF) to power supply up on the shelf. If they are found in other positions, contact one of the staff.

2. Samples to be coated must be attached to either of the two types of specimen holders. Three standard pin-type SEM mounts can be held and secured with set screws in specimen holder type A. Flat chips or other unmounted samples can be secured with double-stick tape to specimen holder type B. It is a good idea to secure a small rectangular piece of white “POST-IT” to one of the holders to serve as a record of the coating.

3. OPEN the Chamber Vent. The chamber is vented when the TC2 Thermocouple Vacuum Gauge reads at atmospheric pressure. Check this by testing the lid to determine if it will lift off. At this step we are venting the chamber by opening the line to the N2 tank.

4. Remove the lid from the unit. Stop the flow of N2 going into the chamber by CLOSING the Chamber Vent. Note: During the humid, summer months you may leave the chamber vent open, allowing N2 to flow through, preventing excessive water moisture from entering the chamber.

5. Turn on the HV1010 VCR Power Supply (sits on the shelf) to rotate the sample head to allow you to load samples. You may insert a piece of white indicator paper between the sample and the sample stub as an aid in determining the amount of coating that will be applied during your run.

6. Put the samples into the machine making certain they are secure and will not fall into the machine. Wrap a single layer of tape around the specimen stub to build up the diameter of the stub, making it more secure in the sample head.

7. Put the cover back on and center the top unit inside the red circle. The viewing window should line up with the path of the argon beam.

8. CLOSE the Chamber Vent.
Sputter Coating

1. CLOSE the Backing Valve, and then OPEN the Roughing Valve to “rough down” the chamber to <50 milliTorr. Monitor this with the TC2 thermocouple vacuum gauge. **Rule: The backing and roughing valves should never be BOTH open.**

2. Close the Roughing Valve, and then open the Backing Valve.

3. OPEN the Main Valve slowly and wait for the TC2 Thermocouple Vacuum Gauge to reach ~20 milliTorr.

4. Set the High Vacuum Gauge range select to ZERO. The needle will drop below and then come back to the zero position.

5. Turn ON the High Vacuum Gauge (push the “READY / METER READ” button). Make certain that the “READY / METER READ” light is on. If the right light stays on, the gauge is read to use. If the right light is not on, high vacuum has not been obtained...consult with the staff.

6. Wait until the High Vacuum Gauge goes down to $5 \times 10^{-5}$. The High Vacuum Gauge might be erratic at first depending on the sample. If it continues to behave erratically or fails to give a reading at all – consult Appendix A. If the gauge seems to be working properly but you are not able to obtain a vacuum level of $5 \times 10^{-5}$ after 10-15 minutes, someone may have altered the flow rate of the Argon --- consult with one of the staff.

7. The Power Supply sits on the shelf above the coater. Turn the Power ON. Turn the High Voltage ON by depressing the red button. The voltage reading should be ~ 1.5 kV.

8. Turn the Current Voltage Control knob until the gauge needle reacts; then set the knob at the point marked by the red dot. You should now be able to see the argon beam within the chamber.

9. Set the timer (sits on the shelf) for the desired amount of coating. **Note:** This VCR coater does not require cooling water through the unit, but if your run is extensive, for example, >20 minutes, then you should contact staff to inquire about opening the cool water line.

10. Note the Current and Voltage and write it the log book.

11. If the equipment detects instability, the circuit breaker goes off with a “pop”, and the beam goes off. Should this occur, simply turn DOWN the Current/Voltage Control knob, turn OFF the High Voltage, and turn OFF the Power. Then turn them back on. Frequent recurrences of this problem means the ion beam gun needs an overhaul. NOTE IN LOG BOOK AND NOTIFY STAFF.

12. After your coating time is done, turn DOWN the Current/Voltage Control knob, turn OFF the High Voltage, and turn OFF the Power.
Removing Samples from Chamber

1. Turn OFF the High Vacuum Gauge to protect it.

2. CLOSE the Main Valve. Check that the Backing Valve is OPEN and the Roughing Valve CLOSED. Then OPEN the Chamber Vent.

3. Lift the lid and take the samples out.

4. CLOSE the Chamber Vent. CLOSE the Backing Valve. OPEN the Roughing Valve and wait for the TC2 thermocouple gauge to reach 50 milliTorr.

5. CLOSE the Roughing Valve and OPEN the Backing Valve.

6. Check that the Main Valve is CLOSED with the Diffusion Pump ON.
Appendix A

VCR High Vacuum Gauge Conditioning Procedure

Occasionally the high vacuum gauge will behave erratically or fail to give a reading at all even though you have followed all procedures correctly and are obviously at high vacuum. To whip it back into shape…

1. Turn High Vacuum Gauge OFF.
2. CLOSE Main Valve.
3. OPEN Chamber Vent and admit just enough N₂ to raise chamber pressure to between 200 and 500 milliTorr.
4. CLOSE Chamber Vent.
5. CLOSE Backing Valve.
6. Open Roughing Valve.
7. Turn ON High Vacuum Gauge.
8. When chamber pressure reaches 100 milliTorr, depress the Ready/Meter Read button and hold it down. You will hear a humming/buzzing sound as ionization occurs inside the vacuum gauge tube. Keep pressing the button until the chamber pressure drops below 50 milliTorr.
9. Then, CLOSE the Roughing Valve.
10. OPEN the Backing Valve.
11. OPEN the Main Valve.
12. Check for high vacuum as usual. On rare occasions it will still be erratic and you may have to repeat this procedure, but usually the gauge will now be stabilized.
Appendix B

Diffusion Pump Overheat Alarm

The Diffusion Pump needed to achieve high vacuum in this instrument is cooled by the Nils Hasselmo Hall central chiller loop. If this loop loses pressure, even briefly, or the coolant temperature rises too high, the Diffusion Pump will begin to overheat. The thermal circuit breaker will then open to turn off power to the pump heater coil, and an alarm will sound.

If this happens, simply close the main valve if it’s open, and turn off the Diffusion Pump switch on the front panel, because it will still be illuminated as if the pump is still being heated. The alarm will turn off.

Contact a staff person if possible so that they can check with the building engineers or managers to find out what the problem is. You can call 6-0911 (Nils Hasselmo Hall Reception Desk) to try to get help. If no one is available, simply wait 15-20 minutes for the pump to cool, and then try the following steps to restore normal operation:

1. Look behind and underneath the machine and locate the base of the Diffusion Pump cylinder, with the cooling coil spiraling around it, on the left side of the machine (if you were viewing the machine from the front)…note that it has a small, rectangular gray box with a button in the middle of it, facing toward you. The button is the reset switch of the thermal circuit breaker. It can only be reset if the pump has cooled sufficiently.

2. Push on the button with the wooden dowel rod. If you hear a click, the circuit breaker has been reset and you can turn the diffusion pump back on. You will need to allow another 15-20 minutes for it to reach operating temperature.

3. If it doesn’t click, wait even longer and try again in a few minutes to reset it. Or, if the alarm sounds again as the pump is heating up, the problem remains with the cooling loop and will have to be solved before the machine will operate.
Start-Up of Cold System

(Note: The system is normally left running in “STANDBY” mode (see Step 6, below) with the Main Valve closed, the Backing Valve open, and the Thermocouple Vacuum Gauge switch at TC1 or TC2. The mechanical pump will then be “backing” the diffusion pump through the open backing valve. So, Steps 1-6 in “Start-UP” apply ONLY if system has been shut down.

1. All Vacuum System Valves should be closed and all switches off. All Cooling Water Valves to the diffusion pump (valves A, B, C and D) should be open. These valves have yellow handles which are parallel to the direction of the piping when open. In addition, there are two Cooling Water Valves to the ion gun (valves E and F). These two valves are normally closed. They can be opened when sputtering runs are over 20 minutes in duration. Staff should be consulted in this instance.

2. Turn Main Power switch ON

3. Turn Mechanical Pump switch ON

4. Turn the Thermocouple Gauge switch ON; switch the Thermocouple Gauge Selector to FORELINE position

5. When Thermocouple Gauge reads below 50 milliTorr, OPEN the Backing Valve.

6. When Foreline pressure reads below 50 milliTorr, turn Diffusion Pump ON. Wait 15 minutes for Diffusion Pump to heat. Foreline pressure should drop below 20 milliTorr. Leave in this position until you’re ready to sputter coat.

System Shut Down

1. CLOSE the Main Valve

2. Turn Diffusion Pump OFF. Allow pump to cool for at least 15 minutes.

3. CLOSE Backing Valve

4. Turn Mechanical Pump OFF

5. Turn Main Power OFF

6. Vent the Forepump by OPENING the M.P Vent Valve on front of the evaporator until hissing stops

7. CLOSE the M.P Vent Valve