CORAL/NEMO Name: Sputter-AJA-ChamberLoad

Location: Bay 8U
Purpose: Deposition
Contact: Gary Riggott, Kurt Broderick

Contents

Introduction 1
Safety 2
User Qualifications and Responsibilities 2
Operating Procedures 3
Loading Deposition Material 3
Conditioning Run 4
Deposition 7
Shutdown 8
Appendix 9
Advanced Process Capabilities 9
Tips and Tricks 9

Introduction

The Sputterer-AJA-ChamberLoad is a sputter deposition system capable of processing samples from small pieces up to 4" diameter wafers. This system has the broadest range of available targets for sputter deposition at MIT.nano and allows for deposition on a wide variety of substrates. The system has a sample stage that can rotate for uniformity and can be heated by quartz lamps up to 500C. The substrate can also be biased for substrate cleaning before deposition, or for film densification during deposition.

The Sutter-AJA-ChamberLoad has 3 deposition guns which accept 2" diameter targets up to ¼" thick. All three guns are connected to 300W generators, there are two RF generators for either conductive or dielectric materials and one DC generator for conductive deposition materials. The guns can be run independently or simultaneously for co-sputtering.

Deposition rates can be measured as part of a preliminary conditioning run using a built-in quartz crystal monitor (QCM). Pure material sputtering is done with Argon (Ar) gas, but the system can also do reactive sputtering by adding a low percentage of Nitrogen Nitrogen (N_2) or Oxygen (O_2).



Figure 1: AJA-Chamberload in Bay 8U

Safety

Hazards present on system are high voltage, RF radiation, and pressurized gases. In case of a machine emergency, press the Emergency Stop button to power down the tool, and notify a member of the staff. The power supplies on the tool are capable of supplying 300W of power to the deposition sources. This system is designed with a full array of electrical interlocks that should at no time be defeated or overridden. Do not attempt to open or remove any panels as you risk exposing yourself and others to these hazards.

- Mechanical pinch/crush points exist with the loadlock lid and reaction chamber lid. Exercise care when loading/unloading samples and carriers, and when changing targets in the chamber.
    - Ensuring your feet are planted firmly under your shoulders while lifting can make lifting the lid easier and help to prevent strains or other injuries.
- Do not remove any panels or attempt any repairs on the system.
- If the tool is behaving dangerously or making loud noises, press the red EMO button, alert the staff, and shutdown the system on CORAL/NEMO. DO NOT attempt to re-energize the tool!
- Never open the chamber when the temperature is over 250C: the quartz plate and heater block will crack.
- Power up ceramic or insulating targets slowly, even more slowly on the ramp down, decreasing at 1W/sec.
- Do not use High Power (>250W) for longer than 30 minutes on any target material; the cables will overheat and melt.

User Qualifications and Responsibilities

This is a manually controlled machine, so you must be careful not to choose a destructive sequence and be aware of the machine status at each step.

- All processes must have PTC approval, and the specific details of the deposition conditions should be verified with staff prior to running a deposition.
- Users must ensure the carrier is in the chamber when running any deposition, including deposition test rates. Without the carrier in place, material would be deposited onto the quartz heater window which will reduce heater efficiency and shorten its lifetime.
- Do not use high power (>250W) on ceramic targets, or they will crack.
- Verify RF plasma has ignited before ramping up power. High reflected power will result in damage to the guns. Plasma can be verified by viewing the load and tune capacitor positions, about 30-70% of full scale, and/or visually through the viewport.
- For high temperature depositions (>400C) use the dedicated Inconel chuck that has been pre-coated with 500Å of Ti as a diffusion barrier. Otherwise, the chuck will alloy with the film/substrate and break.
- At the end of the process, users are expected to leave the system pumped down before disengaging from CORAL.
- If there are problems while operating the machine, report them immediately in CORAL so staff can address them in a timely fashion.

Operating Procedures

Make tool reservation in CORAL/NEMO. Allow 1 hour for a conditioning run, a minimum of 1.5 hours for pump down (unless depositing fairly non-reactive metals and/or if you don't require a good vacuum), plus your run time. Estimate 0.2-0.5 Å/sec for ceramics or reactive sputtering and up to a maximum of 1.5 Å/sec for metallic targets, beyond which target micro melting can cause blotchy depositions.
Verify the proper starting conditions exist before starting:

- Circuit breakers 'MAINS POWER' and 'VACUUM PUMPS' are **ON**.
- TEMPERATURE CONTROL and HEAT dials are **OFF**.
- Pro-Face target shutter & MFC shutoff is in **MANUAL VALVE** mode, submenu LOCAL. On this screen, no MFC's or shutters are selected.
- VAT pressure controlling gate valve is **OPEN**, not PRESSURE mode.
- MFC gas flow regulators are **OFF**, with the silver dials pushed in.
- Capacitance Manometer (CM) valve should be **CLOSED**. This is a 2" bellows valve on the left side of the chamber, protecting the pressure gage from contamination.
- Magnetic target materials (Fe, Ni, Co, etc.) must have the iron plug/blank installed behind them. The standard magnets *MUST* be reinstalled after deposition. Magnetic material targets ***CAN NOT*** be left in the tool after you have finished your run.
- Ceramic targets *MUST* be connected to RF power (*not* DC). RF power cables are light brown and the DC cables are black.

Loading Deposition Material

1. Engage in CORAL/NEMO.
2. Turn the VACUUM PUMPS circuit breaker **OFF** to vent the system.
3. After verifying that the chamber is no longer under vacuum (atmospheric at approximately 7.6e+2 Torr), carefully open the chamber.
4. Put the protective dish in place over the chamber to prevent items being lost inside the chamber.
    - The dish should have 2 Allen wrenches, a torx driver, and a razor blade to hold the copper mesh in place.
5. Open the shutter on the gun that you wish to use.
6. Unscrew the collar and carefully separate to remove the sputter target.
    - Be mindful of the copper mesh behind the target when removing.
7. Replace with a new sputter target, placing the copper mesh behind. Tighten the collar screws snugly, but do not overtighten, and close the shutter. Repeat for any additional target changes.
    - Use the magnets provided on the side of the tool to identify which sputter targets are currently installed on each gun.

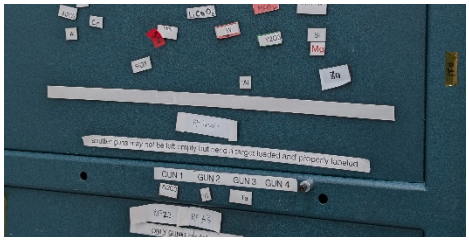


Figure 2: Material magnet board on the AJA-Chamberload

8. Open and close the shutter 1-2 times to verify functionality and that the shutter fully closes before continuing.
9. While the chamber is still open, verify the sample holder chuck is in place and covering the quartz heater window.
10. Check QCM crystal life on the FTM-2400.
    - Press **XTAL LIFE** and the readout will tell the current life. QCM life should be 85% or higher.

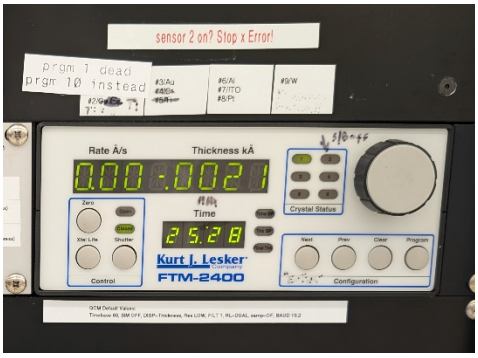


Figure 3: FTM-2400 module to check QCM life.

11. After all required targets have been installed. Return unused targets back to the storage cabinet and remove the protective dish from the chamber. You are ready for a conditioning run.

Conditioning Run

1. Open up the bottom front panel and lower the stage to 25. This allows the QCM arm to rotate into the same location and at the same height that the substrate will occupy during the actual run.

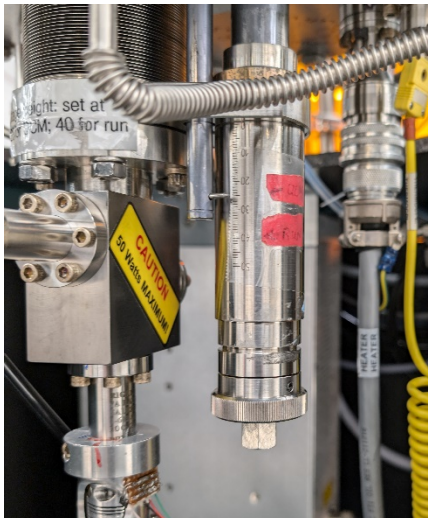


Figure 4: Stage adjustment located under the tool chamber.

2. Load your sample into the chamber on the stage.
    a. If you need to add foil to the QCM arm in order to cast a larger shadow (so that your samples do not receive deposition during the rate determination step), then do so now.
3. Swing the QCM arm into position above the lowered substrate chuck.

a. The lever to move the arm into place is on the left side of the tool by the CM valve.

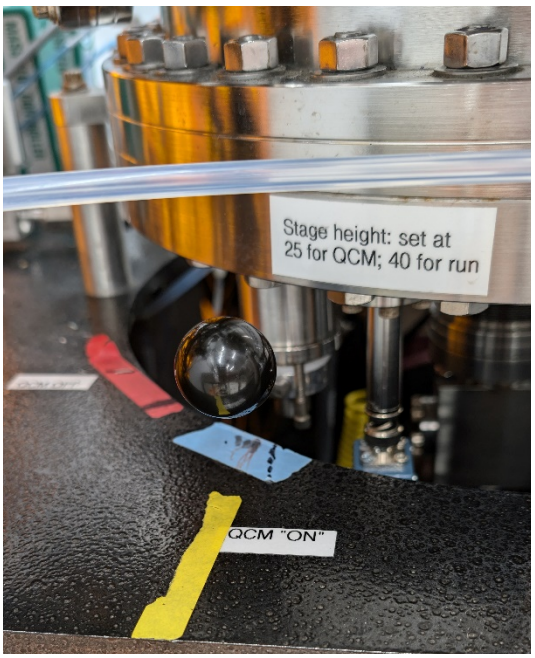


Figure 5: QCM position lever located on left of chamber.

4. Carefully close the chamber lid and turn the VACUUM PUMPS circuit breaker **ON**.
5. Press **Gas 1** on the Pro-Face panel, and pull out the MFC switch to turn on the argon flow. Set it to 5 ±1sccm while the chamber is pumping down, this speeds up pump-down time. Let the argon flow for about 5 min.



Figure 6: The AJA MFCX to control flow of gasses into the chamber.

6. Once the chamber pressure gets below 1e-2 Torr, open the CM valve located near the QCM lever.
    a. At this point, the argon flow should be off.
7. After opening the CM valve, reset the timer on the FTM-2400.
8. Wait for the pressure to get below 2e-3 Torr, then turn on the ion gauge with the switch labelled **IG1**.
    a. Turning on the ion gauge at pressures above this limit may damage the gauge.
9. Base pressure of ~1e-5 Torr should be reached in about 90 minutes.

10. Once base pressure is achieved, the green “Interlocks” LED by the “vacuum pumps” breaker should turn on. This turns on the power supplies to the guns.
    a. If the light does not come on, try disengaging and re-engaging in CORAL/NEMO.
11. Select one of the material parameter selections #2-9 by pushing its respective button or by turning the cylinder knob to display the desired program number. Verify the parameters are correct against the reference table. Push the knob again so the thickness readout screen with rate (Å/s) and total thickness fields are displayed.
    a. If depositing a non-standard material not listed as #2-9, use **Program #10** to enter parameters. Select **Program #10** and press the dial so the word "**Density**" appears.
    b. Turn the knob to the correct density from the table and push **NEXT** so "**Tooling Factor**" appears.
    c. Tooling Factor should be set at 100% when the substrate is at the standard stage height of 40. Push **NEXT** so “**Z-Factor**” appears.
    d. Turn the knob to enter the correct Z-Factor, and push **NEXT** to save it. If unknown, use 1.0.
    e. Other variables are not used in manual deposition mode. Press **Program** to return to the thickness capture.
    f. When selecting a program, if both “Crystal Status” buttons are on then the selected channel is dead and cannot be used.
12. Verify the Argon MFC (**MFC #1**) valve is enabled at the Pro-Face touch screen, and is selected with the gas selector knob (see Figure 6). For reactive sputtering, open other MFC valves as needed.
13. Set MFC flows so the combined flow of all gases is 12 sccm.
14. Set the VAT controller from OPEN to PRESSURE Mode, and select the proper setpoint for plasma strike.
    a. Standard: use **Setpoint #4** (30 mT) for DC or RF strike.
    b. Setpoint #2 (10 mT) or Setpoint #3 (20 mT) may also be used for DC strike.
15. All guns are preset to start at 60W, and if another value is displayed initially, please notify staff.
16. Gun 1 (DC) can send output to DC cable #1 (Default) or #4. Cable #1 runs through the Sparc-le unit, which back-pulses to remove native oxides from the sputter target which can lower the deposition rate.
    a. To switch to output 4, push the 1” cylindrical dial in once to adjust power, and twice to switch output cable.
    b. Verify the DC power inverter is set at ACTIVE or ON if doing reactive sputtering.
    c. To strike plasma, push the “**ON**” button. After a short delay, the ACT output value equals the set value. PLASMA will be lit and ARC will not be lit.
    d. Close the view port shutter after plasma has been established if it is open.
    e. On the VAT controller, change to deposition pressure to 3mT (**VAT Setpoint #1**).
    f. Adjust power by twisting the rotary dial to achieve desired deposition rate (between 1-2 Å/s).
17. Guns 2 and 3 (RF) have dedicated outputs per unit.
    a. To strike plasma, press **RF ON/OFF**, the button light will switch from blue to red.
        i. The probability of striking plasma is much greater when the target shutter is open.
    b. Look to confirm if plasma is lit by inspecting the reflected power (REF). It should be “0”. Verify the Automatic Matching Controller positions are in the 30-70% range.
    c. On the VAT controller, change to deposition pressure and verify plasma is still lit.
    d. Close the view port shutter after plasma has been established.
    e. Adjust power by twisting the rotary dial to achieve the desired deposition rate.

f. If plasma does not strike in step 17b, *do not proceed*. Never increase power unless plasma is present otherwise the gun will be damaged. See the appendix for additional measures to get the plasma to strike.

18. Open the gun shutter and monitor the deposition rate as power is slowly increased. Limit maximum power to 250W.
19. Perform the conditioning run for 2-4 minute to allow the QCM to reach thermal equilibrium and native oxides to be removed from the targets. Note the stabilized deposition rate.
20. After the deposition rate appears to stabilize, clear the timer on the FTM-2400 and record the amount deposited in **100 seconds**. This will give you the dep rate in **Å/min** to 2 significant figures and will allow for further target degassing.
    a. Use this dep rate to determine how long your run needs to be for your desired film thickness.
    b. $Run\ Time\ (sec) = \frac{Desired\ Film\ Thickness\ (nm)}{Calculated\ Deposition\ Rate\ \left(\frac{\text{Å}}{\text{sec}}\right)} * 10\ \left(\frac{\text{Å}}{nm}\right)$
21. After you have the dep rate, close the shutter then move the QCM to the "**OFF**" position
22. If your process requires you to heat your sample, you may turn the heater on now.
    a. The heater should be ramped at no more than 100°C per 5 minutes (20°C/min).
23. Unless depositing non-reactive metals, such as gold, always spend at least 5 minutes pre-conditioning metal or ceramic targets to remove oxides and moisture before starting a deposition.
    a. Always condition sensitive targets such as ITO for at least 10 minutes before starting a deposition.
    b. Au, Pd, and Pt only need about a minute of pre-conditioning.

Deposition

1. Ensure the QCM is in the OFF position and raise the chuck height to 40.
2. Start stage rotation using the power supply under the chamber.
    a. Simply flip the switch to **ON** and the stage will start rotating.
    b. Rotating your substrate produces a more uniform film.
3. Reset the FTM-2400 clock to zero to time your deposition, or use a timer of your choice.
    a. Ignore any data shown in the Rate or Thickness field on the FTM-2400. With the QCM arm "Off", the data is meaningless.
4. Open the shutter and simultaneously start the timer.
    a. Deposition on your sample has started. Let the process continue for the time required to achieve your film thickness.
    b. At this point it is a good idea to record important parameters (RF/DC, pressure, target material, etc.) so that you may refer to them if/when you use the tool again for a similar process.
5. Close the shutter at the end of deposition when your timer has lapsed.
6. Ramp down power by **1W/sec** for ceramics and **5W/sec** for metals down to **60W**.
7. Turn off the Gun Power when down to 60W. Wait **5 minutes** for the target to cool.
8. Push **OFF** on the MFC controller(s) to close the gas valves.
9. Switch the VAT from PRESSURE to **OPEN** Mode.
10. Lower the stage height to **25**.
11. If this is your only/last layer, then continue to remove your sample. Otherwise, repeat the process for each material you will be depositing.
12. **CLOSE** the CM valve.
13. Shut off the Ion Gauge to prevent damage.

14. Turn the VACUUM PUMP circuit breaker **OFF** to start venting. After 3 minutes the chamber door can be opened.
15. With clean gloves, remove the chuck with your samples. Unload the samples from the chuck and replace the chuck into the system, or return the chuck to the storage cabinet if it was swapped out.
16. Close the lid and turn the VACUUM PUMP circuit breaker **ON** to pump down the chamber.

Shutdown

Verify the proper standby conditions before disengaging from CORAL/NEMO:

- Circuit breakers MAINS POWER and VACUUM PUMPS are **ON**.
- Temperature Control and Heat dial are **OFF**.
- Pro-Face target shutter & MFC shutoff is in **MANUAL VALVE** mode, submenu LOCAL. On this screen, NO MFC's or shutters are selected.
- VAT pressure controlling gate valve is set to **OPEN**, not PRESSURE mode.
- Substrate rotation is **OFF**.
- All MFCs are **OFF**.
- CM valve is **CLOSED**.

Appendix

Advanced Process Capabilities

- **Substrate Bias** can be used to pre-clean or etch-back the substrate at low power (10-45 W). Oxygen (O2) can be used to remove organic residues, or Argon (Ar) for etch-back of native oxides. After the system has reached base pressure, set the desired power on the Substrate Bias Power Supply (SBPS) using the up/down arrow, then press **RF ON**. The SBPS is manually tuned: make slight adjustments to the large LOAD and TUNE dials on the left unit to minimize the reflected power.
    - Substrate bias can also be used to densify a deposited film by leaving the SBPS on during the normal deposition process. However, the normal deposition rate will be reduced due to the non-zero etch-back rate.
- **Substrate Heating** can prevent condensation and incorporation of contaminants and residual gases in the deposited film. Heating can also affect thermal stress on films that may subsequently be exposed to high temperature environments. Temperatures up to 800°C are available and heating above 400°C requires a 500Å Ti deposition on the Inconel chuck to prevent alloying damage. Additional reservation times will be required to allow the substrate to cool before venting. This is required to prevent oxidation of substrates and targets and to prevent damage to the system.
    - Note the temperature correction chart posted by the heater.
    - Never heat substrates that decompose at higher temperatures (such as organic materials).
    - Minimum stable temperature of the heater is 200°C.
    - The temperature control unit is circuit breaker enabled with a separate ON/OFF switch. The temperature is selected by the thermocouple readout up/down buttons.
- **Reactive Sputtering**. RF sputtering of oxides and nitrides can be very slow (less than 1 Å/sec). Argon with low percentage Oxygen or Nitrogen can assist with sputtering of these materials. Faster deposition rates can be achieved by reactively sputtering a pure source metal (using **DC Gun #1 only**) with Argon and a higher percentage (20%+) of reactive gas (O_2 or N_2). For example, depositing TiO_2 from a Titanium target using Argon and Oxygen; the Ti should react with O_2 in the plasma and at the substrate, if Substrate Bias is being used. Gun #1/Output #1 is required due to the close proximity to the Argon inlet (reducing the probability of contamination of the target by the reactive gas) and to utilize the Sparc-le unit (DC polarity pulse inverter) which is in line with that gun. The Sparc-le unit must always be active when reactively sputtering.
    - Note: Because Gun 1 is located near Argon inlet, it will have a lower partial pressure of N_2 or O_2 during Reactive Sputtering.
- **Co-Deposition** is the deposition of multiple materials from different guns at the same time. For this process all guns strike plasma at the same time (at 30mT), then pressure is reduced to 3mT for power to be ramped up before the simultaneous 3 min pre-deposition on closed shutters before the actual deposition begins. There may be some interference between guns, causing visibly non-uniform plasma flows. This will result in a uniform deposition because of the generally neutral, non-charged nature of the deposition material.

Tips and Tricks

- Lower the stage height to 25 before moving the QCM arm into position, or you will hit the chuck. Likewise, move the QCM out of the way before raising the stage to the deposition height of 40.
- Before closing the chamber, use a fab wipe to clean the O-ring with isopropanol to ensure there is a clean seal.

- During pump-down, flowing a purge gas can promote the desorption of moisture from surfaces inside the chamber more quickly than just pumping a static chamber. Using the Pro-Face controller, press GAS 1 (Argon), and at the MFC controller pull the rotary dial out and adjust to 12 sccm flow rate. Let the system purge for about half of your total pump down time, then push the MFC dial in to shut off GAS 1. Leave the Pro-Face GAS 1 enabled.
- If you encounter trouble striking plasma:
    - Try opening the shutter for the gun for 5 seconds. This allows a longer Ar+ ion path length, increasing the energy and ionization probability.
    - Try turning off the power, manually moving the capacitors to approximately 50% on the scale by pressing the min/max buttons, then turn power back on and try again.
    - Never increase power above 60W unless plasma is confirmed.
- With the plasma lit, ramp up the power slowly if the target is a poor thermal conductor or ceramic. The power can be ramped up more quickly if depositing a metal.
- If your deposition rate is too slow at maximum power, you can lower the run pressure with the arrows next to the Pressure Mode SETPOINT buttons, down to 2 mT, which reduces speed-dampening gas collisions, allowing for faster velocity Ar atoms. If necessary, deposition of the same material from two guns simultaneously can effectively double the rate.
- If depositing **Multiple Layers**, guns not in use may be left ON with the shutters closed to allow fast processing. Lower the idle gun power(s) if layers take more than 5 minutes each.