

SputtererAJA

STANDARD OPERATING PROCEDURE

CORAL
Name: *SputtererAJA*

Model
Number: AJA international Orion 5

Location: EML

What it does: Thin film deposition

Introduction: The SputtererAJA is a 4" maximum substrate size, flexible system well designed for low volume R&D use. It has three 300W Guns, two of which are RF for either conductive or dielectric materials and one of which is DC for conductive materials only. Each Gun takes a 2" x 1/4" thick target, and they can be run either sequentially or in parallel. The substrate can be heated to 800C. Sputter gases include Ar for most uses, with N2 and O2 also available for reactive sputtering. There is substrate biasing available for either substrate cleaning before deposition or for film densification during deposition. Film thickness is measured via a quartz crystal monitor (QCM) done on the preliminary 'conditioning' run.

Safety: (A) Emergency Shut-off Procedure

In case of an emergency, such as arcing or overheating of cables, do the following:

Shut off the EMO circuit breakers Mains Power and Vacuum Pumps.

Find MTL Staff.

(B) Warnings about this system

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. This is not an idiot proof, push button tool!

- Never open the chamber when the substrate is over 200C, as the quartz plate and heaters will crack.
- Power up poor thermally conducting targets such as ceramics slowly, and be even more careful on the ramp down, decreasing at 1W/sec, vs 5W/s on conducting targets. Do not use high power on ceramic targets, or they will crack.
- Do not use High Power (>250W) for long periods on any target material; the cables will overheat and melt.
- 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.
- Be absolutely sure your RF plasma has ignited before ramping up power. These guns can survive 60 W without plasma for a short while but at 200W they would be destroyed quickly. Look to be sure, or ask!
- If depositing at >500C, ALWAYS deposit 500A of Ti on the Inconel chuck before the run, or the substrate will alloy with it and explode.
- When pumping down the chamber, push or hold the door shut to ensure there is a good seal.

Procedure: Following are instructions for correct operation of the SputtererAJA.

(1) Preparation

Make machine reservations in CORAL. Allow 1 hour for the initial conditioning run plus time for your run, estimating an approximately 1-2 angstroms/sec deposition rate and 30 min for pumpdown and cooling.

Schedule with Lab Staff to have the correct targets installed before or at the beginning of your run.

Verify with Lab Staff that your substrates are allowed. Low melting temperature / high vapor pressure materials are not allowed with high power!

Preclean your substrate if necessary.

Verify the proper starting conditions, as follows, and when finished, ensure this is the ending state:

Circuit breakers Mains Power (2) and Vacuum Pumps (1) 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 OPEN, not PRESSURE, Mode.

Substrate Rotation is OFF.

MFC gas flow regulators are OFF, with the silver dials pushed in.

Capacitance Manometer (CM) valve should be CLOSED or your pumpdown time will be 30 min longer due to gauge contamination.

Finally, Engage in CORAL

(2) Conditioning & Deposition Rate Determination Run

Pump down the system if necessary:

Turn Vacuum Pumps circuit breaker ON, while holding the chamber door CLOSED. After about 2 min. the interlocks light and the Gun power supplies should turn ON, and the Turbo should almost be up to its operating speed of 1 KHz.

Flowing a purge gas will significantly reduce the water concentration inside the chamber, the major contaminant. At the Pro-Face, press GAS 1, and at the MFC controller, turn to Gas 1, Ar, on the dial and pull the silver knob ON and set it to 12 sccm flow rate. Let the system purge for about 3 minutes, then push the MFC knob OFF and GAS 1 on the Pro-Face.

Open the CM valve, and turn on the ion gauge (IG1).

Within 30 min the required minimum base pressure of below 5×10^{-6} T on the ion gauge should have been met, while the CM and convectron gauges should be at '0'.

Stage height should be lowered to 25, to allow the QCM arm to rotate into the same location and at the same height that the substrate will occupy during the actual run.

Set up the MCM-160, the QCM controller, with the correct parameters for the deposition planned:

Pressing CRYSTAL LIFE should show a reading of greater than 85%, otherwise ask Lab Staff to show you how to change the quartz crystal, then swing the QCM arm into position above the lowered substrate chuck.

You may choose from one of the pre-written material parameter selections, #2-9, as listed

on the panel, by pushing or turning the cylinder knob to display the desired program #, then pushing it again, so the thickness readout screen with μ/s and total A fields are displayed. It is a good idea to check that the parameters are correct against the reference table.

If depositing a material not listed as #2-9, write in new material parameters ONLY on Program #1. Select Program #1 and push the dial in causing the word "Density" to appear, then spin the knob to the correct density from the table and push NEXT so "Tooling Factor" appears. This is the ratio between deposition rate at the sample and substrate, which is 100%, which is correct when the substrate is at the stage height of 40. Push NEXT enter the correct Z-Factor and push NEXT to save it. Other variables are not used in manual deposition mode so return to the thickness capture screen by pressing Program.

Dummy Run:

Open the MFC shut off valve(s) at the Pro-Face touch screen by touching MFC #1 (Ar) and possibly #2 (N₂) or #3 (O₂).

Set the MFC flow rates by turning the black knob to the desired gas, pulling out on the selected gas dial, and turning to the target flow. The combined flow of all gasses should be 12 sccm, ie 12 sccm of Ar, or 10 of Ar and 2 of N₂ / O₂, to properly load a turbo pump of this size.

Set the VAT to PRESSURE Mode, and select setpoint #2, 3, or 4 for DC ignition, and #4 for RF ignition. These are 10, 20, and 30mT settings, respectively.

Set the relevant Gun power supply to 60W. For Gun 1 (DC), push the 1" cylindrical dial in and turning until the wattage is selected, pressing in, and turning to 60. Press it again, and turn until Gun 1 is selected, and press to make '1' flash. Make sure the DC power inverter is set at ACTIVE or ON if doing reactive sputtering. For RF Guns 2 or 3, just arrow up or down on the power supply panel to set 60W.

Ignite plasma by pressing the "ON" button for the relevant gun. Verify there is plasma by looking through the viewing port. There is a short delay with Gun 1, and it is somewhat difficult to see because of the angle, but if you look closely you can usually see plasma in a 2mm hole on each gun's shutter. If plasma doesn't light within 30sec, turn gun power off, and either increase the pressure, briefly open the shutter on the Pro-Face, or increase power and try again. Never allow the power to remain on without plasma, especially at higher power settings. Reduce pressure to setpoint #1, 3mT, after ignition. Practice now, not with your real substrate in place! 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. With the shutter open, monitor the deposition rate as you increase power.

Deposition rates with the DC gun are higher than the RF guns for a given metal and power, and RF-oxide deposition rates are the lowest, less than 1 μ/s , but limit maximum power to 250W maximum anyway. 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 1 – 2 mT, which reduces speed dampening gas collisions, allowing faster deposition, at the cost of uniformity. If necessary, deposition of the same material from two guns simultaneously can effectively double the rate. Calculate how long your actual deposition will take now and write down the power & pressure settings in the log book.

Close the view port shutter after ignition has been established so it stays transparent. This shutter is only magnetically attached, so do not twist aggressively or it will detach and fall onto your sample stage.

After determining your deposition rate at a certain power/pressure setting, and having run

it for a minimum of 3 minutes if it is a readily oxidizing metal, 1 minute otherwise, start the power ramp down sequence by lowering power by 1W/sec for ceramics and 5W/sec for metals, and close the shutter. Turn off the Gun Power when reaching 60W, then wait 5 min, to allow cooling before venting.

After cooling, push the MFC controller(s) OFF, and shut the MFC valve(s) on the Pro-Face.

Switch the VAT from pressure to OPEN Mode.

Spin CLOSE the CM valve.

Pull the Vacuum Pump Circuit Breaker OFF, to start the venting. After 1 minute the vacuum interlock light and the 4 power supplies should turn off, and after 3 min the chamber door should be able to be opened.

(3) Deposition

Swing the QCM to the back, in the OFF position.

With clean gloves, remove the Inconel chuck, place your substrates on it, and promptly reload it into the chamber, making sure the chuck is well seated by spinning it slightly. Try to limit the chamber exposure to humid room air.

Immediately start the Pumpdown sequence above. (Flip Vacuum Pump circuit breakers ON, then after interlock pressure is met, open the CM valve, Ar purge 3 min, and turn on the Ion Gauge)

Set the stage to the deposition height of 40, to match the QCM deposition rate.

When 5×10^{-6} T base pressure is met, turn on the Stage Rotation to full speed.

Open the MFC(s) at the Pro-Face, and set the MFC(s) at the controller dial, to a combined 12sccm flow.

Proceed to ignite Gun plasma, at Pressure Mode 2, 3, or 4, and 60W, then reduce to Pressure Mode setting #1. Keep the shutter closed until after the power has been ramped up to the targeted power, then wait an additional 3 minutes if it is a readily oxidizing metal, 1 minute otherwise, for target cleaning time, then open the shutter for the calculated time of deposition. Pressing Zero on the QCM will give you a count up clock.

Shutdown is the same as with the Conditioning & Characterization run, above:

Ramp down power by 1W/sec for ceramics and 5W/sec for metals

Turning off the Gun Power when down to 60W, then wait 5 min to allow cooling

Turning OFF the MFC valves at the Pro-Face and at the MFC controller

Set the VAT to OPEN Mode, CLOSE the CM valve

Shut the Vacuum Pump Circuit Breaker OFF

Unload your samples, and immediately either reload further samples or if this is your last run, pump down the chamber and return the stage height to 25.

(4) Shutdown

Verify the proper ending or standby conditions:

Circuit breakers Mains Power (2) and Vacuum Pumps (1) 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 OPEN, not Pressure Mode.

Substrate Rotation is OFF.

MFC gas flow regulators are OFF.

CM valve is CLOSED.

Disengage in CORAL.

(5) Further Capabilities and Complex Depositions

Substrate Bias is used to Pre-Clean or Etch-Back the substrate, at between 10 and 45 W, with O₂ for organic descum or Ar for etchback, after base pressure is met, set the power with the substrate bias power supply up/down arrow, and press RF ON. This unit is manually tuned, with the goal of minimizing reflected power by very slight adjustments to the large Load and Tune dials on the left unit. Etch Back is supported by leaving power supply is turned on during the normal deposition to density the deposited film, as well, but it will reduce the resulting deposition rate due to the non-zero etch back rate.

If depositing **Multiple Layers**, guns not in use may be left ON with the shutters closed to allow fast processing. Lower idle gun power if layers take more than 5 minutes each.

For **Co-Deposition**, all guns are struck 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 not result in a non-uniform deposition because of the generally neutral, non-charged nature of the deposition material.

Substrate Heating can prevent condensation of contaminants and reduce the incorporation of gas in the film, and can reduce thermal stress on films subsequently exposed to high temperature environments. Temperatures up to 800C are available, but all applications above 500C require a 500A Ti deposition on the Inconel chuck to prevent alloying damage. Always let the substrate temperature cool before venting, to prevent oxidation of substrates and targets and to prevent damage to the quartz heating apparatus. Note the temperature correction chart posted by the heater, and never heat low temperature substrates. Minimum stable temperatures are above 200C. The temperature control unit is circuit breaker enabled with a separate ON/OFF switch, and the temperature is selected by the thermocouple readout up/down buttons.

Reactive Sputtering. RF sputtering of oxides and nitrides can be very slow, at less than 1 Å/sec, in predominantly Ar gas, perhaps with 10% O₂ or N₂ to make up for the lower sticking probability of these species. To enable faster deposition rates, we can reactively sputter a pure source metal in **DC Gun # 1 only**, in an ambient of Ar and a significant (20%+) concentration of reactive gas, depositing TiO₂ from a Ti target, for instance, when sputtered in Ar and O₂. The Ti should react with O₂ in the plasma and at the substrate, if Substrate Bias is being used. Two enabling technologies are first, the Ar is specifically sourced at Gun #1, to reduce the probability of contamination of the target by the reactive gas. This would otherwise render it both non-conducting, causing a plasma failure, and contaminated, and second, is a DC polarity pulse inverter, the Sparc-le unit, which is in line with the DC power supply. This unit positively charges the target at 20kHz. This selectively removes non-conducting (oxidized) regions from the target. Always run this actively if reactively sputtering. It also removes native oxidation from targets which have been out of use. While RF guns could be used to deposit conducting metals into a reactive

ambient gas mixture, they don't have a direct Ar feed to prevent target metal contamination. The reactive gas injectors aim right above the substrate, away from the targets.

[A table with a history of run parameters](#) is viewable. You may also [add your run results to the table](#). Send Kurt an e-mail if you want to modify/update/edit any previously recorded results.
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