

EML Sputter Deposition
AJA Orion 5
Version 4/2016

CORAL Name: *SputtererAJA*

Type: 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 sputter depositions. It has three 300W Guns, two of which are RF for either conductive or dielectric materials and one of which is DC for conductive deposition materials. Each Gun takes a 2" x 1/4" thick target, or 1/8" if of brittle material, and they can be run either sequentially or in parallel. The substrate can be heated to 500C. Sputter gases include the typical argon, as well as N₂ and O₂ for reactive sputtering. There is substrate biasing available for either substrate cleaning before deposition, either with Ar for etchback or O₂ for organic strip, or for film densification during deposition. Film thickness is measured via a quartz crystal monitor (QCM) done on the preliminary 'conditioning' run, with time solved based on a constant rate assumption.

Safety:

(A) Emergency Shut-off Procedure

In case of an emergency, such as a water leak on top of the tool, do the following:

Shut of 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 a push button tool!

- Never open the chamber when the substrate is over 250C, as the quartz plate and heaters will crack.
- Power up ceramic or insulating targets slowly, even more slowly on the ramp down, decreasing at 1W/sec, vs 5Å/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. Plasma can be verified by viewing the load and tune capacitor positions, with good tuning within 20% of halfway, or 30- 70%

- If depositing at $>400\text{C}$, ALWAYS use a dedicated Inconel chuck, which has been pre deposited with 500A of Ti as a diffusion barrier, or the chuck will alloy with the film, or the substrate, and break.
- When pumping down the chamber, first isopropyl fab-wipe the O-ring on the side door briefly to ensure there is a good seal, as there is a particulate trap in the bottom of all side doors.

(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 Å/sec deposition rate and 30 min for pumpdown and cooling.

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. Discuss with lab staff prior to training.

Verify the proper starting conditions exist before starting, 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.

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

Capacitance Manometer (CM) valve should be spun closed. This is a 2" bellows valve on the left side of the chamber, protecting the pressure gage from air (water) contamination.

(2) Conditioning & Deposition Rate Determination Run

Engage in CORAL

Vent 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 reduce the water concentration, the dominant contaminant, inside the chamber more quickly than just pumping a static chamber. At the Pro-Face, press GAS 1, and at the MFC controller, pull the rotary dial Gas 1, Ar, 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 membrane GAS 1 enabled.

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

In about an hour, the required minimum base pressure of the 10^{-6} T on the ion gauge should have been met, while the CM and convectron gauges should be at approximately '0'. If they show a significant variation, notify staff of calibration requirement.

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 $\text{Å}/\text{s}$ 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:

Start by making sure the QCM is centered over the chuck, which needs be at the lower height of 40 mm (It was built inverted, so 25 is the upper or deposition height)

Open any needed other MFC shut off valve(s) at the Pro-Face touch screen by pressing #2 (N2) or #3 (O2). Ar should still be enabled.

Set the MFC flow so the combined flow of all gasses is 12 sccm, ie 12 sccm or 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.

ALWAYS IGNITE at 30mT, and typically deposit at 3mT.

All guns are preset to start at the correct value of 60W, and if another value is displayed initially, please notify staff.

Note: Gun 1 is the location of the Ar input, so will tend to have a lower partial pressure of N2 or O2 if Reactive Sputtering.

Gun 1, DC, can send output to DC cable#1 or #4. OUTPUT 1 is Default, designated by flashing #1 and it always goes thru the Sparc-le unit, which changes the deposition rate, depending on its setting of ON/INTERMITTENT (default) or /OFF. This unit back pulses to remove contamination from the sputter target, typically native oxides from metal targets, which lowers the deposition rate. To switch to OUTPUT 4, push the 1" cylindrical dial in, once to adjust power, and twice to switch output cable, making output 1 or 4 flash, as desired. Make sure the DC power inverter is set at ACTIVE or ON if doing reactive sputtering. To light plasma, push the "ON" button, and after a short delay, you will see the ACT output value equals the set value, PLASMA will be lit and ARC will not be lit. You may now go to deposition pressure, typically setpoint 4, 3mT, and then adjust power by twisting the rotary dial. DC targets deposit faster than RF on the same power level.

Guns 2 and 3 are RF with one output per unit. To light plasma, press the RF ON/OFF, and it will switch from a blue to red light on that button. Immediately look to see if plasma is indeed lit by inspecting the REF (reflected, or out of tune) power. It should be "0". Additionally, inspect the Automatic Matching Controller, which shows the position of two capacitors, on a 0 to 10 scale. They should both be in the 30-70 range. If plasma is not lit, according to these indicators, do not proceed, but keep trying to light plasma, by these adjustments. First, simply open the shutter for the gun the power supply is attached to, for 5 seconds. This allows a longer Ar⁺ ion path length, increasing the energy and ionization probability. If this doesn't cause the Load/Tune to respond correctly, turn off power, and manually move the capacitors to approximately 50% on the scale by pressing the min/max buttons, then try again. Never increase power unless plasma is confirmed. If plasma is lit, proceed to lower pressure, confirming plasma is still lit, then ramping up power at the appropriate rate.

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. RF-oxide deposition rates are the lowest, less than 1 Å/sec, but try to limit maximum power to 250W maximum anyway or the target may break. 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 faster velocity Ar atoms. 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.

After determining your deposition rate at a certain power/pressure setting, after waiting 5 minutes for the sensor to reach temperature equilibrium and native

oxides to be removed from the targets, 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.

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. Raise the chuck height to 40mm.

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 10⁻⁶ T base pressure is met, open the MFC(s) at the Pro-Face, and set the MFC(s) at the controller dial, to a combined 12sccm flow. Follow the same conditions as your dummy calibration run for the same rates!

Shutdown is the same as well.

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 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.

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