M-Probe GLA Procedures

From Google Doc

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Mprobe Restart After Vacuum Loss

- 1. Turn off all electronics, (2505 Memory Interface, Spectrometer Power Supply, Glassman High voltage, Service Physics X-Ray Gun Controller, nti sputter gun controller)
- 2. Turn off alarm on interlock box at bottom of rack and set all three bypass switches on box **up**.
- 3. Push **reset** button on interlock box
- 4. Press **reset** on back of control box over XPS
- 5. If the Neslab circulator is off **Restart** it.
- 6. Check that the transfer arm is full withdrawn.
- 7. If the Cryo's are up to a temperature >80 K or the pressure is higher than 1 x 10⁻⁷Torr.
 - a. Turn off cryo-pump compressor
 - b. If the Turbo pump is on turn it **off** and wait till it stops.
 - c. Turn **on** interlock override on control box.
 - d. Check gate valves to see that 0, 1, 2, 5, and 6 are **open** and 3 and 4 are **closed**
 - e. Turn **on** the turbo pump and wait till it is up to speed.
 - f. Wait till the vacuum is < 1 '10⁻⁶T, this will take 1 tor 2 hours or more.
- 8. If the cryo compressor is off turn it **on**(you may need to turn the cryo switch off and then on).
- 9. Make sure that the cryo pump is working
- 10. After the cryo temperature is down below 25K (about 1 to 3 hours) **close** gate valves 1 and 5 (only 0, 2 and 5 are open).
- 11. On the interlock box set the alarm and three bypass switches to **down**(on)
- 12. Turn **off** interlock override.
- 13. Continue to next section.

Restarting M-Probe X-Rays

- 1 Check vacuum system OK
- 2 Gates 0, 2, and 6 should be **open** with all others closed
- 3 Turn off interlock override on back of control box over XPS if it is on
- 4 Make sure system power switch on the back of the box is **up**
- 5 Turn **on** X-ray gun boxes (bottom two Glassman high voltage and 9603 X-ray spot size controller)
- 6 Check that the Interlock OK LED is on.
- 7 If the vacuum has gotten above 1×10^{-7} Torr or system was vented.
 - a Turn "ramp" knob on spot size controller to **slowest**, fully clock wise (service switch down)

- b Press "start filament" followed by "HV on" and wait for it to read 2kV on the spot size controller box - watch pressure (Filament on LED should turn on, Xfer OK LED on, Panel meter should read I FIL mode and go to ~1.2 A)
 - i Set the "service" switch **UP**, keeping "ramp" knob on **slowest** setting (c.w.) and changing "stand by" to "**operate**" (takes 8 h, watch pressure)
- 8 If the vacuum did not get above 1×10^{-7} Torr:
 - a A Turn "ramp" knob on spot size controller to **fastest**, counter clockwise (service switch down)
 - b Press "**start filament**" followed by "**HV on**" and wait for it to read 2 KV on the spot size controller box watch the pressure
 - c Set the "service" switch **up** and turn "ramp" knob to **fastest**, then press "**operate**" (takes 2 h, watch pressure)
- 9 Once 10kV reached, degas anode by starting X-ray gun on 100 micron spot and slowly increasing the spot size until largest spot does not raise pressure above 2E-8 Torr
- 10 Turn off X-rays
- 11 Turn on spectrometer boxes (top 3, spectrometer power supply, flood gun, memory interface)
- 12 Open software, check "X-ray gun operate" in ESCA control panel
- 13 Switch spot size controller from "manual" to "computer"
- 14 Degas flood gun check "Flood Gun" box in ESCA control panel and increase energy to 5 eV, watch pressure and wait for at least 1 hour
- 15 Turn down flood gun energy, uncheck flood gun box, and close ESCA control panel
- 16 Instrument is ready to use

Venting M-Probe

Check vacuum system OK

Turn on interlock override (switch on back of MProbe on near the gate switch box, make sure X-ray gun is off)

(Are you picking up a dropped stub from the analysis chamber? Make sure you have a 6" copper gasket)

Turn off controller boxes on M-Probe tower, wait 30 min for filaments and anode to cool Close gate 0 and gate 6, Open Gate 1 (gate 2 should already be open)

Open gate 5

Turn off IG1 and IG2

Turn off turbo, close gate 2, open gate 3

Allow nitrogen to fill chamber

Replace flange covers Close gate 3 holding door closed, open gate 2, turn on Turbo Allow turbo to pump down transfer and analysis chamber

(Untested Instructions)

Changing M-Probe Anode

- 1. Locate anode replacement kit provided by Service Physics (Includes new anode, replacement screws, new Hawk filters).
- 2. Vent load lock and analysis chamber
- 3. Check to make sure Hawk chiller is off. If not, turn it off now.
- 4. Label and then detach the inflow and outflow lines from the anode at large nuts(1" and 15/16" wrenches required)
- 5. Unscrew anode flange from analysis chamber body. Carefully remove the flange, noting that the anode is attached. You will need to move the braided hose for the sputter gun in order to remove the entire flange part. Discard used Cu gasket.
- 6. Cover anode opening in analysis chamber with Al foil.
- 7. Take anode to a bench.
- 8. Unscrew the golden screws that hold the anode keeper in place with the back of the anode leaning on a clean towel on bench.
- 9. Remove anode keeper and then the anode from housing. This may be difficult. If it does not easily unseat, use a small flathead screwdriver to pry it out or the gasket removal tool.
- 10. Insert new anode puck. Take care to line the indention on the copper side of the anode up with the coolant inlet and outlet. Use new gold screws to connect anode using metal ring (Anode keeper)
- 11. Clear new Cu gasket for analysis chamber with IPA and Install on analysis chamber anode flange.
- 12. Replace anode on analysis chamber. Make sure all threaded studs are screwed in about 4 or 5 turns before putting the nuts on.
- 13. Reestablish vacuum in analysis and load lock chambers. Follow "Restart MProbe" instructions.
- 14. after new anode installation go to Resarting MProbe X-Rays
- 15. After degassing the anode you need to set the spot size.
- 16. adjust spot size- open 9600. push in 800 spot size and turn on box screw on blue potentiometers, also do 500 spot. you will see which one red light will light up after punching spots. CCW ~2 turns.
- 17. press operate with service switch up

(Untested instructions)

Checking for damaged mylar window (obstructs electrons from X-ray gun)

-happens if there's a sudden pressure change in analysis chamber, e.g. if gate is opened while one chamber is at significantly higher pressure than the other

Run an "unscanned" function on gold sample at 0 eV CBE (i.e. static window), Res 4, 1 min Signal-to-background between valence peak and negative binding energies should be > 200:1. If significantly less, the mylar window may be damaged (observed 10:1 signal-to-background on 2018-08-23 when mylar window was completely damaged)

Replace mylar window by venting analysis chamber, removing cap of monochromator, unscrewing bolts, and removing the circular metal holder inside

Detach the two components: outer ring and ring with legs Place ring with legs under new mylar sheet, larger ring on top, and clamp together to sandwich mylar window; remove excess mylar from edges Clean the sides of the chamber where mylar window used to be with tec-wipes and isopropanol, and compressed air

Replace with legs pointing toward analysis chamber

Optional: cleaning the sides with compressed air (be VERY careful as the compressed air can damage the mylar window if aimed too close)

Fixing various M-Probe problems

If Pfeiffer turbo controller says "Error E698," this means there was a loss of communication between the controller and the drive unit. It can be fixed by unplugging the ethernet cable (RS 485) from the back of the turbo controller and plugging it back in. Do it quickly and you won't have to restart the turbo. Just press the right arrow button until the "TMP Power" shows on the readout again.

Sputterer-related

Users should do their best to calibrate the sputter rate for their material, as the sputter rate varies wildly between samples. A rule of thumb that has been thrown around has been 10 nm per minute.

20180614: A combination that was set when turned on: 5 keV, focus 3010, condenser 246 (not present in manual)

M-Probe resolution pass energies

This is measured as the pass energy of the electron that hits the center of the detector in the energy direction during the detector width calibration routine for a particular resolution. You can find these in the software by going to Settings -> Setup Esca and selecting the Configuration tab. The pass energies are PassEvRes1, PassEvRes2, etc.

Resolution	Pass Energy (eV)
1	30.3
2	57.4
3	111.9
4	166.3
5	16.0