

## I. Preparations

1. Turn on the WATER COOLING for both the turbo pumps and the sputtering guns, by turning the large blue and white valves in the front of the machine.
2. Check to make sure all 4 of the smaller blue and white valves for the sputtering guns are open.
3. Check that the Ar (and He if necessary) valve is open on the gas cylinder beside the orange equipment rack.
4. Check that the butterfly valve on the condensation chamber is open.
5. Open *Q-pod* program (Desktop) and load the configuration for your sputtering target.
6. Turn on roughing pumps for Deposition chamber (blue) and Condensation chamber (red).
7. Once the pressure reaches 5 mbar (Dep.) and  $10^{-1}$  mbar (Con.) you can switch on the turbo pumps using their respective switches on the equipment rack (E-rack). Since this pumping procedure can take a while you may want to pump overnight, and you may want to bake-out the chamber to reach UHV.

## II. Power Supply and Mass Flow Controller General Instructions

1. Set DC power:
  - a. Select Gun 1 by scrolling with C knob to Gun 1, then depress C knob.
  - b. Set desired power by scrolling to power set point, depress C knob, adjust with C knob, and then depress C again to select the power.
  - c. Scroll back to Gun 1, select it. The number 1 and TG should flash. DC gun is ready.
2. Set RF power:
  - d. Should be auto-set at 30W, want 50W.
  - e. Use up arrow to increase to 50. RF gun is ready.
3. Turn on *MKS 247 4 channel readout*.

- f. Pull out toggle switch then flip up to on position.
4. Set desired gas flows.
  - g. Select the channel you want to set with the "display channel" knob.
    - i. Channel 1: 2" Ar gas (don't change)
    - ii. Channel 2: 3" Ar gas
    - iii. Channel 3: 3" He gas (don't change = 50 – 100)
  - h. For each channel there are two toggle switches, don't touch the right one.
  - i. Flip and hold the left one in the up position and turn the screw immediately to the right with small screwdriver to change flow.
  - j. The set-point flow should be displayed in the readout if the display channel is set to the same one you're changing.

## II. Loading the sample

1. Wear gloves! Never let your skin touch the inside of the chamber.
2. TEM grids or Si wafer should be mounted on the Al plate with SEM tape. Fasten with screw.
3. Close door tight. Make sure vent on load lock turbo is closed.
4. Turn on roughing pump by turning on the power strip (fan and turbo controller will also go on). Let pump to  $10^{-1}$  mbar range then flip the Load Lock Turbo switch on E-rack.
5. Let load lock reach  $10^{-6}$  mbar before opening gate valve.

*Note 1: Never tighten vent screw with anything other than fingers!*

## III. Background Pressures

1. Load lock needs to be at or below  $10^{-6}$  mbar to open gate valve.
2. Inner Cond. Chamber should be in the  $10^{-6}$  mbar range.
3. Outer Cond. Chamber and Dep. Chamber should be in  $10^{-7}$  mbar range.

## IV. Operation

1. Open all gate valves.
2. Insert thickness monitor and start Q-pod software. Make sure it is facing towards the cluster source or RF source – whichever rate you want to measure.
3. To start the cluster source, set DC power 100 W to start.
4. Open 3" Ar and 3" He (top two) valves on E-rack, wait for the chamber pressures to equilibrate.
5. Turn on (A button) DC power supply. Know it's on by actual values displayed in second column under "ACT". When off these read zero.
6. Can change both Ar gas flow and power to fine tune the deposition rate measure by the thickness monitor. For best results try 0.1 – 0.25 A/s. Use the rate to estimate the thickness of your film – for best results use 2-4 A per layer.
7. Deposition time = Thickness  $\div$  Deposition Rate

8. After you know the deposition conditions remove the thickness monitor from the cluster beam and insert sample holder.
9. Insert sample holder in vertical position to deposit clusters, align the green line with the word "side" next to it with the gap in the stopper.
10. When done, turn off DC power supply using B button.
11. Close He and Ar valves on front panel (top two).
12. Open 2" Ar (bottom) valve on front panel – don't have to wait for pressure to equilibrate.
13. Turn on RF power supply. BLUE light means supply is OFF, RED light is ON. Make sure reflected power is <5W displayed as "REF".

*Note 3: Sometimes the RF plasma doesn't strike, you can tell by looking for the purple light under the load lock or seeing a high reflected power ~20-30W. If this happens just turn off the RF power and turn it back on again. Always check to make sure the plasma has struck before inserting the sample. The easiest way is to make sure the REF power is 0 when ON.*

14. Insert the sample holder face up. Align the green line with the word "up" next to it with the gap in the stopper.
15. Since the thickness monitor is less sensitive to C atoms, use the following guidelines for depositing the C films. Success has been had depositing at 50 W for 1 min for intermediate composite layers, and 2.5 min for a Carbon cap layer.
16. Remove sample holder, turn off RF power supply, and close the Ar valve.
17. When you're done with the sample, retract the holder back into the load lock and close the gate valve between the load lock and the deposition chamber.
18. Switch off the load lock turbo pump.
19. Wait for the rotation speed to drop to zero AND for the pressure inside the load lock to return to the  $10^{-2}$  mbar range.
20. Open the vent screw to bring load lock to atmosphere. Don't unscrew it completely! Just enough to hear air flowing through.
21. Turn off the roughing pump.
22. At atmospheric pressure you can then open the door, and remove the screw fastening the holder to the rod (make sure you are wearing gloves).
23. Take out the holder with your sample on it.

## **V. Managing the sputtering rate**

Some of the trends we have seen so far include:

1. Gases: Set the He between 50 and 100 at the start of your deposition and DON'T change it afterward! Every time the He is changed during the deposition process the rate is lost and it is hard to recover. The Ar flow rate directly controls the sputtering rate, begin with a low Ar flow rate depending on the aperture size (see chart above) and increase until the desired sputtering rate is found. Ideally, 0.1 – 0.3 A/s works best.
2. Power: In general, to achieve the same sputtering rate at high and low power, use a lower Ar flow rate at high powers. This is more noticeable when larger apertures are

used. If the Ar flow rate is held constant while the power is increased, the sputtering rate increases.

3. Aperture: The pressure difference necessary to produce a cluster beam that reaches the substrate is the goal, and it can be achieved with much less gas in the case of the smaller apertures. Larger apertures requiring larger operating pressures will stress the turbo pumps significantly, and should be used with caution and continuously monitored as they can destabilize more frequently during operation.
4. Target: The DC gun has a magnetic materials array which means it is optimized for 1/8" Fe and 1/4" Co targets 3" in diameter. These do not have the same racetrack, so be mindful of which you are using when you make a composite target.

Aperture	Target	Power	Ar flow	He flow	Stable Rate (A/s)	Stable delay (s)	Turbo
4 mm	1/8" Fe	Low (50)	150	50	0.1	10	very stable
			200	50	0.2	10	very stable
		Hi (175)	134	50	0.1	10	very stable
			150	50	0.2	10	very stable
Fe80W20	Low (25)	457	50	0.11	10	very stable	
		Med (100)	225	50	0.11	12	very stable
		Hi (200)	180-200	50	0.11	10	very stable
5.5 mm	1/8" Fe	Low (50)	687	50	0.1	15	stable
7 mm	1/4" Co	Med (100)	550	100	0.5	15	unstable
7 mm	FeMn	Med (100)	1000	100	0.4	10	unstable
		Hi (150)	650-1000	100	0.12	10	unstable
		Max (200)	370	100	0.7	10	unstable

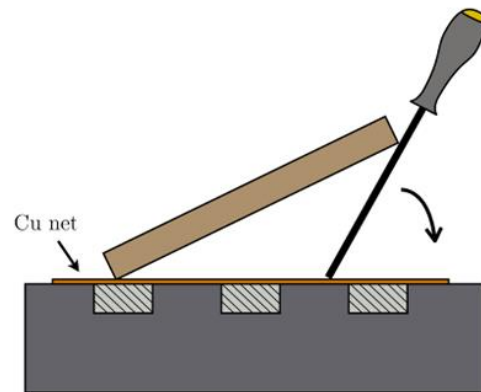
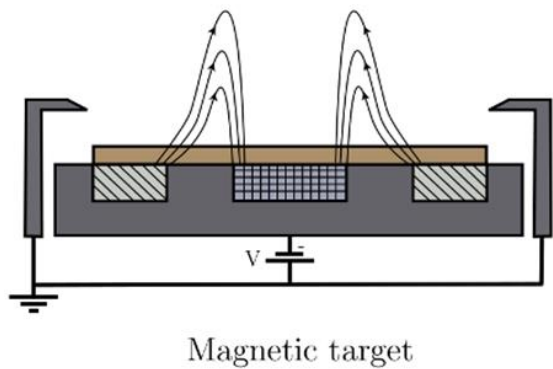
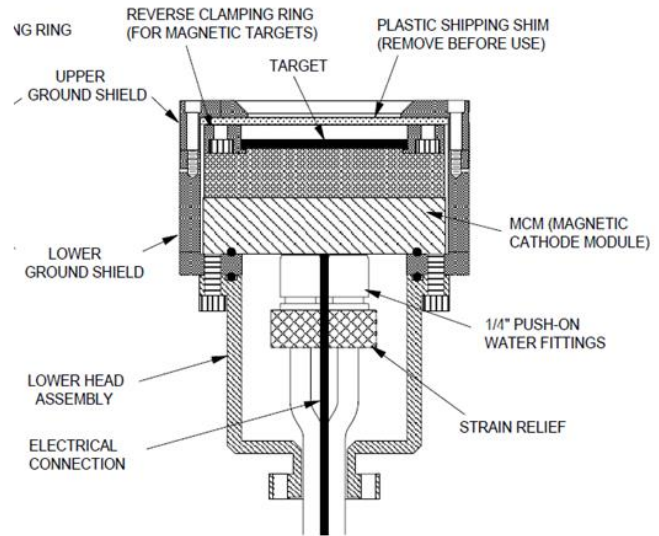
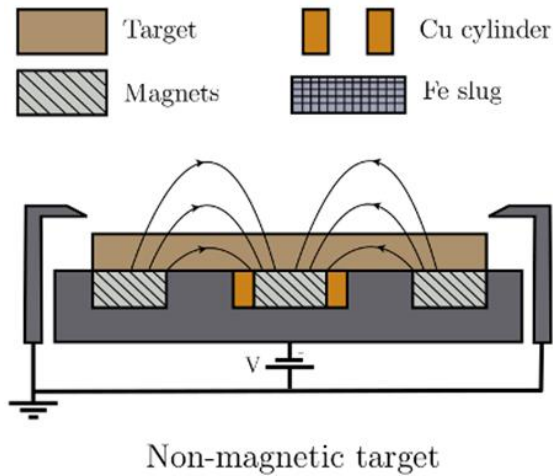
Racetrack Dimensions		
	1/4" Co	1/8" Fe
inner D (mm)	35.00	21.90
outer D (mm)	46.75	30.26
width (mm)	5.88	4.180

## VI. When done for the day

1. Close ALL gate valves.
2. Turn off power supplies.
3. Turn off all turbo pumps on E-rack – unless you wish to pump them overnight.
4. Wait for pressure in load lock to return to 10<sup>-2</sup> mbar range to vent and remove sample (see section IV 20-24).
5. When Con. And Dep. Turbo pumps fall back to 40% of their normal speed (66% = 550 Hz), you can backfill the chambers with Ar gas through the MFC.
6. Backfill to about 10 mbar in each chamber. Leave load lock under vacuum.
7. Close the water cooling valves on the front of the system.

## VII. Changing target

1. The gate valves should all be closed, and the chamber containing the Gun you must remove should be vented.
2. Make sure DC power supply is off and disconnect the BNC gun cable along with the water lines and Swagelok gas connection.
3. Remove left and right clamps on 8" KF flange and insert hand clamp near top of the flange. Remove the remaining top and bottom clamps and hold the opposite side of hand clamp with free hand. Remove the gun from the chamber taking care not to bang it on the inside of the chamber. And set it on the Styrofoam platform on the nearby table.
4. Remove the chimney and ground shield by using the appropriate hex key to remove the two opposing screws on the front face, while leaving the third screw in place (its purpose is to prevent the gas from escaping from the shutter hole when a shutter is not in use). Set them aside for later cleaning.
5. Remove the centering ring by unscrewing the six hex screws.
6. It can be difficult to remove the target if it is a magnetic material in a magnetic gun. Often, the only way to do this is to slide the target sideways using a flathead screwdriver as a lever until enough of the target overhangs for the screwdriver to be safely wedged between it and the gun assembly. Then pried up from the target on one side bringing it to a nearly perpendicular position where it can be grasped and pulled away from the gun.
7. The 3" gun will usually be in the "magnetic" array configuration, while the 2" gun should be in the "non-magnetic" array. Both guns are magnetron sputtering and thus use magnets but these terms refer to the target material being either magnetic or non-magnetic. The difference is that in the magnetic array the center magnet is an Fe slug with high magnetization that allows more field lines to escape the target. Non-magnetic materials can be sputtered in magnetic guns; however, this is not recommended since the rate will be higher and the target will not last as long.
8. Magnetic targets should be 3 inches in diameter and either 1/8" (Fe) or 1/4" (Co) thick, while non-magnetic targets should be 1/4" thick and either 2" (RF gun) or 3" (DC gun) in diameter depending on the gun used.
9. Targets consisting of an insulating material or a semiconductor should have a copper backing plate to improve the heat conduction during deposition. The total thickness, target material plus backing plate, should be 1/4".
10. A copper net should be placed underneath the target on the 2" gun, but is not necessary on the 3" gun as long as the material is a good conductor.
11. If necessary, change the magnetic configuration.
12. This is necessary if a non-magnetic target needs to be used in a gun configured for magnetic materials or vice versa. The 3" gun will almost always be configured for magnetic materials.
13. In a gun configured for magnetic materials, the center magnet has been replaced by an iron slug. The difference between a normal gun and one configured for magnetic materials is shown the figure above.



14. Use the magnetic tool (black elongated magnet) to remove the middle magnet and the copper cylinder surrounding it.
15. The iron slug has a long screw sticking up in its middle to make handling easier.
16. Place the iron slug in the middle hole and remove the screw.
17. To remove an iron slug from the center of a gun, fasten the screw in the center and pull it straight out; it might be necessary to use a tool to get a better grip.
18. When placing a magnet in the center, make sure its polarity opposes that of the surrounding magnets. That is, if all the surrounding magnets has a north pole pointing upwards, the middle magnet should have its south pole pointing upwards, and vice versa.
19. Placing a magnetic target should be done carefully; the magnetic force can be strong and it is easy for a piece of the glove to get stuck underneath the target. To prevent this, one can make use of a screwdriver; first place the target on its edge, then use the screwdriver to slowly lower the target down onto the copper net, without using any fingers. See figure below.
20. Since magnetic materials are thinner, an adapter ring should be placed between the target and clamping ring.
21. When replacing the clamping ring, make sure that the screws are tightened evenly; the plasma is very sensitive to the distance between the clamping ring and ground shield.

22. Use a magnet, placed inside a glove, to remove any flakes on the outside of the gun before replacing the ground shield. By placing the magnet inside a glove, any flakes can be removed by simply removing the glove.
23. Replace the ground shield after having checked its interior for flakes or loose material. Also look at the inside edge of the opening (the part closest to the target during deposition, see figure above) so that there isn't a too thick layer of sputtered material; if the ground shield has been used for a long time without cleaning, a thick layer of material forms on the inside edge, decreasing the distance between target and ground shield. This will eventually affecting the ability to strike a plasma.
24. Replace chimney.
25. Before evacuating the chamber, measure the resistance between the target and ground shield using a multimeter; it should be in the range of a few megaohms. If it is only a few ohms, there is a flake somewhere on the inside of the ground shield. If the resistance between the target and the bottom ground is still ohmic, even with the ground shield removed, there could be a flake in the small gap where the ceramic insulators are located; see the cross section sketch.
26. For insulating materials, the resistance can be measured from underneath the chamber, between the center pin and outer part of the coax-cable connected to the gun.
27. Re grease the O-ring and thoroughly clean the gun before reinstalling it on the sputtering chamber. Reversing steps 1-6.
28. Insulating materials must be sputtered using an RF power supply.
29. Metals can be sputtered using an RF power supply, but the rate is going to be much lower; better to use DC power if possible.
30. Common reasons for why it will not work and the solution:
  - a. Flakes shorting the gun. Chamber needs to be opened and flakes removed.
  - b. The matching box used for RF-sputtering is not set properly for the given process. Set both dials to the up position, turn on the RF power, turn one dial at a time and reduce the reflected power as much as possible each time you turn a dial until the reflected power is zero. Never turn the source dial while the power is on! Don't keep power on for longer than 1 min if reflected power is  $> 5$  W.
  - c. The DC power supply is stuck. Turn the power supply off, wait a moment, then turn it back on.
  - d. The clamping ring has been tightened unevenly. Chamber needs to be opened and the clamping ring tightened evenly.
  - e. There might be impurities or an oxide layer on top of the target preventing a DC plasma. To prevent this, change cables and sputter for a while using an RF plasma, then change back.
  - f. Cable is not fastened correctly. Check the cable.
  - g. Gate valve is closed. Open the gate valve.