

# MBE Procedures

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Note: This manual may contain errors. If something seems wrong, check the official manual, lab notebooks, or contact Omicron.

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# Section 1

## Vacuum Chambers General Operation Procedures

### 1.1 Standby Mode

The MBE is kept in this mode when it is not being used for deposition for several days or more.

1. Ion pump is on. The ion pump is the cleanest pump.
2. Pneumatic gate valve is closed. This prevents risk of venting the system if the turbo pump were to fail.
3. The [FEL, turbo] valve (aka metal valve) is closed.
4. The [FEL, MBE] valve is closed.
5. Turbo pump may remain on or off. It is more convenient to leave it on if planning on doing depositions soon.
6. Water cooling remains running through all four MBE loops.
7. On touch screen, grounding position is selected for heater controller.
8. If need to keep a sample in the MBE, it is kept on the heater stage. If there is a sample (which you care about) in the MBE, then the TSP can't be used (it will contaminate the sample!). If the sample will be taken out anyway, then it's ok to use TSP.

Even without regularly running the TSP, the pressure is expected to stay between 4-6 E-10mB.

## 2 SECTION 1. VACUUM CHAMBERS GENERAL OPERATION PROCEDURES

### 1.2 Loading and transferring samples

#### 1.2.1 Loading in a Sample to FEL

Note: If the cryostat has not yet regenerated, a new sample can't be loaded to the FEL because the turbo needs to continue pumping on the MBE chamber.

1. Keep the ion pump running.
2. Close [FEL,MBE] valve if not already closed.
3. Close pneumatic gate valve if not already closed.
4. Open [FEL, turbo] valve.
5. **Double check that steps 1 and 2 have been done!**
6. Turn off the turbo pump by pressing on "turbo", then "off". Wait for the turbo to spin down and make sure the pressure isn't affected. If the pressure changes significantly, something is very wrong: check the valves/turn the turbo back on if possible.
7. Before opening the viewport, ensure on the touchscreen turbo speed indicator that turbo speed is zero.
8. Open the viewport making sure to pull it outwards straight to not put pressure on the screws. Don't worry about the gasket, it will be left hanging on the screws.
9. Load sample using the special tool. **Ensure that there are no protruding posts on the backside of the sample plate.** Protruding posts are very dangerous because the sample may get stuck in the heater stage and STM. Position sample plate into tool so that the front side of plate (side where want to deposit material) faces the tool handle. Then slide plate into slot in FEL. When insert tool, be careful to not touch the walls of FEL since tool is more dirty than the chamber. Once have inserted into slot in FEL, turn black transfer arm handle 180° ccw which will close the jaw, clamping the sample plate in. Then can **gently** remove the tool (if do it too fast, the plate may fall out or you may hit the wall of chamber with the tool) and sample plate should stay connected in FEL. Rotate black transfer arm handle 90° so sample plate front faces up.
10. Close the viewport. To make sure gasket doesn't fall, start from the bottom. The gaskets should be reused once. If are reusing a gasket, then tighten the nuts as much as possible (with reasonable force). For a new gasket, tighten nuts firmly but not too tight, otherwise will not be able to reuse the gasket.

11. Pump on the FEL by turning on turbo without changing any valve positions. Wait for the turbo to reach full speed. If it is unable to reach full speed, then there is a leak: turn off turbo and further tighten the nuts. For real samples, must pump on the FEL for at least 2 hours before transfer sample onto the heater stage. For blanks, 1 hour is minimum. Even better, is to pump overnight since it will keep the pressure below mid E-9mB range.

### 1.2.2 Transfer between FEL and MBE heater stage

This transfer is the most difficult of the transfers done in the MBE chamber. There is a high risk of the sample dropping due to the heater stage is suspended vertically from the manipulator and when the transfer arm is pulled out, it can vibrate substantially.

1. Since the TSPs can't be used when a sample is in MBE, one TSP should be run before doing the transfer in order to keep the system cleaner. After running the TSP, wait until pressure drops back down before proceeding.
2. Set the manipulator to the correct positions for transfer. As of May 2017, these positions are:  $z = 8.785$ ,  $x = 15.95$ ,  $y = 16.06$ ,  $\phi = 122.3^\circ$ . Set these positions as close as possible since even small changes have a large effect on the smoothness of the transfer.

Note: The positions may change after a bakeout.

- (a) When moving manipulator, adjust  $z$  first ( $z$  is read from the top of the large black moving piece). Rotating in cw direction moves the stage upwards.
- (b) Remember that when turn the brown rotation dial, need to loosen the screw and not let the dial go before tighten screw back! (it has spring in it).
3. **Make sure the FEL has been pumped on for long enough.** Keeping the [FEL, turbo] valve open, slowly open [FEL, MBE] valve. While opening, watch the pressure and turbo pump speed: the pressure is expected to rise up to low E-8mB range but the turbo speed should not decrease. **If turbo speed decreases, immediately stop and close valve.**
4. Now use the transfer arm to transfer the sample to heater stage. The two pins need to fit into the grooves on the top side of the stage.
5. Once have inserted the sample to heater stage, rotate the magnetic coupled handle on the transfer arm by  $180^\circ$  which will release the jaw from the sample.
6. Retract the transfer arm just enough for the jaw to clear the sample tab. **Then close the jaw.** This step is very important and it was specified by Tim at Omicron that the transfer arm alignment with manipulator slightly shifts between open and closed jaw and that jaw should remain closed when inserting and removing the forks except to clear the sample tab. (Following this practice in May 2017 made transfer smoother).

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7. In small steps (with jaw closed) continue pulling back transfer arm and between each step allow the stage to relax to natural position (i.e. not being bent from the vertical). **This is essential to prevent the sample from falling since the removing of the two transfer arm pins from the heater stage is very tight.**
8. Once the jaw has cleared sample tab, close the jaw since this will provide the most smooth retract. When do the final retract, there will still be a small jump, but it will not be as significant since the stage wasn't so far displaced from equilibrium position.
9. After transfer arm has cleared the valve, close [FEL, MBE] valve.

**After transfer to MBE, can NOT open pneumatic gate valve connecting turbo to the MBE for at least 6 hours of continuous operation of the turbo.** The procedure used thus far has been to always wait until the next day to open the pneumatic gate vale.

#### 1.2.3 MBE heater stage to STM transfer

1. Make sure thickness monitor is retracted all the way so it is not hit by the transfer arms.

2. Set manipulator to "heater stage to small wobble stick" position.

**Use the x,y coordinates from FEL to heater stage transfer done on 11/1/16 (see lab notebook).**

3. Pick up sample with wobble stick:

- (a) Remove the wobble stick support and move wobble stick magnetic coupler (never rotate or push on the wobble stick itself, it can only wobble). Use the slot in the side of wobble stick to align it with plane of sample plate (horizontal).

- (b) Then gently push so there will be the plate tab going in to end slot of wobble stick (we don't see this end slot).

- (c) Rotate wobble stick magnetic coupler by 90°(cw or ccw doesn't matter), while watching the slot which should face vertical when have reached 90°rotation.

- (d) Slowly pull out wobble stick, the plate should follow.

4. Use wobble stick to transfer plate to long transfer arm receptacle.

- (a) Slowly move the wobble stick in and make plate go into the top slot (just under the top stainless steel part) of the long transfer arm receptacle. Do not try to put plate into the bottom of receptacle! This will damage it. There should be a gap btw. bottom of receptacle and the plate.

- (b) Once plate is in the slot, rotate wobble stick  $90^\circ$  (cw or ccw) to decouple it from the plate.
  - (c) Slowly pull wobble stick out, it could catch on the tab so watch carefully. If see it start catching, slightly rotate wobble stick back and forth (i.e.  $10^\circ$  cw and ccw) while pulling out to find the best position for decoupling it from the plate tab.
5. Move manipulator z all the way up to its motion limit (at 0 position). This is so that the stage is not in the way of the long transfer arm.
  6. Gradually move in the long transfer arm to the furthest-out (from MBE) sharpie mark (this means that the black magnetic coupler end that is closest to the MBE just reaches the mark. The end of the transfer arm will now be perpendicular to wobble stick).
  7. Rotate wobble stick  $180^\circ$  so sample deposition side is facing up. Then insert sample into long transfer arm receptacle.
  8. To help the transfer in STM, need to rotate the receptacle cw (from the wobble stick's reference frame).
    - (a) Move wobble stick to the right (when viewing from wobble stick's side) side of the receptacle.
    - (b) Very gently push on this side, receptacle will start to rotate. Keep pushing until see the little bars/locking mechanism on the far left side, lock in place at the next bar. Rotate it by 1 of those locking bars.
  9. Rotate long transfer arm coupler by  $180^\circ$  clockwise (direction is important b/c cw ensures that the opening to plate insertion will never be in a position that the plate can fall out) (be careful not to overshoot, so rotate i.e.  $160^\circ$  first, then do the final adjustment).
  10. Make sure long transfer arm receptacle is horizontal and sample plate is on its bottom side.
  11. Slowly move in long transfer arm to the sharpie mark closest to MBE. This marks spot where sample will be just before valve connecting STM with transfer tube.
  12. Check the pressure on both systems, if pressure is ok (low E-9mB or less), slowly open [MBE, STM] valve.
  13. Move long transfer arm all the way in to the metal stopper.
  14. Transfer sample to the STM using STM wobble stick.

For the reverse transfer procedure, follow the above procedure backwards step-by-step.

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## **Section 2**

# **Deposition Procedures**

Material is sublimated from effusion cells, making molecular beams that are incident (line of sight due to high vacuum) onto heated substrate. Underneath the substrate are effusion cells which have shutters to control when material can reach substrate. The source material is contained in a crucible or as a rod is heated by either resistive heating from a refractory metal (metal that is very resistant to heat and wear) filament wound around a crucible or e-beam heating. In e-beam heating, electrons are accelerated towards source material, inducing heating when they hit. Resistive heating is suitable for lower melting pt. materials while e-beam heating is needed for high melting pt. materials (i.e. tungsten).

### **2.1 E-beam evaporators**

A refractory metal filament is heated by resistive heating and a high voltage is applied between the source material (either source rod or source material pellets in a crucible) and the hot filament. This draws an electron emission current from the hot filament. The emission current hits the source rod (or crucible containing the source), making a hot tip. Once rod/crucible tip gets hot enough, the source material starts to evaporate.

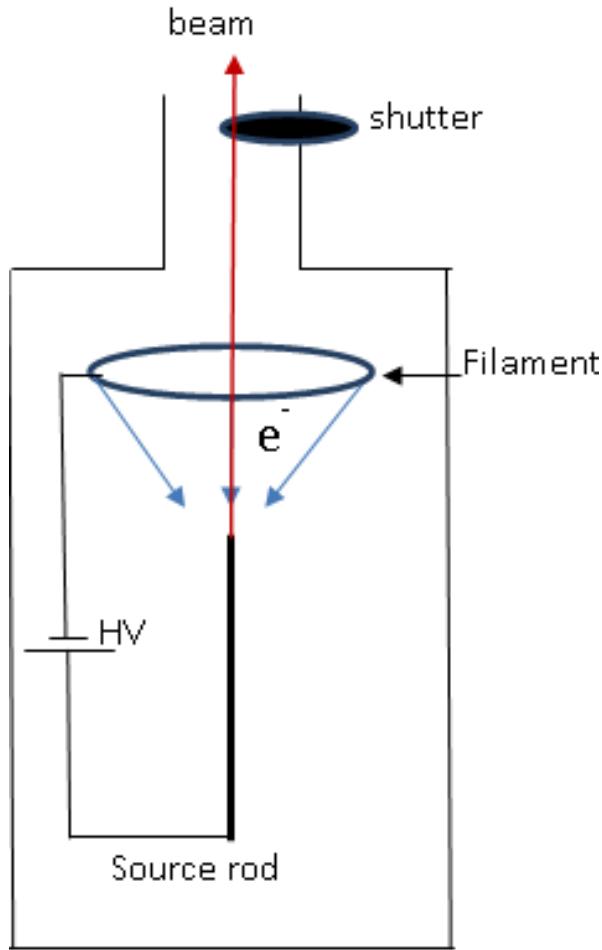


Figure 2.1: Schematic of the e-beam evaporators

### 2.1.1 Currents

It is important to keep track of three currents:

**Filament current ( $I_{fL}$ )**: The current flowing through a circular filament (made of refractory metal, often W) from which the electrons are coming from. We prefer to keep it below 2.2A to lower risk of damaging the filament and extend its lifetime. Between 1.9-2.2A is a good range. Ultimate upper limit is 2.5A, at which the filament lifetime is only 3-9 hours. Our filament is made out of Th<sup>-</sup> doped W.

**Flux current ( $I_{FL}$ )**: Small fraction of evaporated source material will naturally be in ionized state so the current in the beam can be measured. This tells us information about the deposition rate. Current should be  $> +1nA$  when material is being evaporated properly. Different materials have different degree of ionization and thus may have different flux current reading for the same deposition rate.

**Emission current (EMIS =  $I_e$ ):** Current of electron beam going from filament to source. Will be mA order of magnitude. This current depends on FIL to heat up the filament to high enough temperature that electrons start being ejected.

### 2.1.2 Physics of the rod positioning

There are two controlling factors: power density and polarization, with power density being dominant. Also there is the technical limitation of maximum filament current.

**Power density:** If less area of the rod is being bombarded by electrons, the heating is more localized so higher temperature can be reached which results in more flux. If we move the rod too close to filament or even so it is inside the filament loop, then electrons will be bombarding a large area of the rod. This will result in a large EMIS but since heating is delocalized it will not heat up the source material enough for it to evaporate. If we did not have a maximum filament current limitation, then the best position would be as far away as possible from the filament loop so that only the tip of rod gets bombarded with electrons. However, if we position the rod too far away, then not enough of the electrons being emitted from the filament will hit the rod so we will not be able to get a high enough EMIS for flux while keeping FIL under the limit. Therefore the objective is to find the furthest away position where can keep FIL under the upper limit while still resulting in significant FLUX. If we already have  $> 1nA$  FLUX and move the rod further in, while keeping EMIS the same, then FLUX will drop since have decreased power density (increased area of rod that is getting bombarded but not the number of electrons hitting it). If move rod too far in, will not be able to get any FLUX even if increase EMIS since heating is now so delocalized that can't reach the necessary temperature for evaporation.

**Polarization:** Rod is polarized by the electrons hitting it which makes it harder for material to evaporate. Polarization effect is more significant on thin rods. However thin rods result in higher power density. Desired rod thickness is one that has high power density but is thick enough so that polarization is not too sever of an effect. Right now (5/2017) we use a 1.3mm diameter W rods.

### 2.1.3 Operation Procedure

1. Turbo pump should have been running continuously overnight.
2. Make sure water cooling is on, the switches for all 4 MBE lines are open, and water is flowing (check the flow meters).
3. Turn on evaporator controller. (If are doing calibration, also move in the thickness monitor rod to the marking. Do not move it past the marking or the bevel will break). When just turn on evaporator, will see the zero values: FIL=0.12A,

EMIS=0mA, voltage = 4V, Flux = -0.5nA (although the test sheet says +0.24nA, anyway < 1nA is just noise).

4. Open gate valve (will hear loud pop sound) and turn off the ion pump: "Stop HV" on the ion pump controller. The ion pump may also be turned off a bit later, for example right before start ramping the Se evaporator.
5. Need to fill the cryostat with liquid N2 which acts as a pump (make sure that using the in port). During filling make sure dewer hose does not touch any electrical wires. Make sure dewer has enough liquid N2 for the procedure. If run out while doing evaporation, need to turn down evaporator current and voltage and refill dewer.
6. While filling the cryostat can start ramping the evaporators. Once cryostat is full, immediately turn off the dewer valve but leave hose connected since will need to refill it in after about 1hr of operation. Cryostat is almost empty and needs refilling when there is almost no gas coming out the output vent at the right top of MBE chamber.
7. On evaporator controller: first will slowly increase voltage to desired value (i.e. 800V for Sn or 2000V for W). Then start increasing current. At first the current dial controls FIL directly. Can increase FIL current to 1.5A at a moderate pace (i.e. turn slowly but don't really have to wait for system to stabilize since it is well-behaved at this low current). When testing the evaporator for Zr (which is labeled W right now), EMIS went from 0 to 1mA when FIL reached 0.76A so that is the first point where the filament started emitting an electron beam.
8. Once FIL reaches high enough to result in sufficient EMIS (3mA), the controller switches to feedback control and an arrow will now point to EMIS in the display. This means that now when you increase the current dial, you are increasing the set point of EMIS. The FIL current will increase until this EMIS current is reached.
9. Once  $FIL > 1.5A$ , need to slow down in increasing current since there is often a delay between turning the dial and system response. When see FLUX rise  $> 1nA$  which means that the material is being evaporated. Make sure to keep FIL preferably at  $< 2.2A$  in order to extend the lifetime of the filament. For W evaporation, this can be quite difficult and could require moving the rod closer to the filament after each deposition run. Every two small clicks of current dial results in about 0.1mA change in EMIS but there is sometimes up to several minute delay.
10. If are unable to get any significant FLUX, try adjusting the rod position 0.25-1mm forward. This is done with a fine screw manipulator below the evaporator. Decreasing the mm reading means moving the rod closer to the filament. This will first result in a sudden jump in EMIS, since now more electrons hitting the rod

(EMIS is measured as a circuit btw. filament and rod) larger part of rod is in the path of electron beam. Then the FIL will be lowered by the feedback loop in order to bring the EMIS down to the set point. This will allow to safely further increase the EMIS set point to try to achieve a FLUX. Higher EMIS should increase chance that will get a flux (as long as rod is not too close to filament, see below) since there are now more electrons hitting the rod. Example: We have reached FIL = 2.2A but still have no significant FLUX. We adjust rod 1mm forward, which results in FIL to drop to 1.8A. Now we can increase EMIS set point further until FIL reaches 2.2A. If we still have no FLUX try moving rod forward again.

According to the manual, FLUX (aka ion current) is proportional to the evaporation rate and EMIS. At a given voltage, HV and sample position FLUX is directly proportional to the flux of evaporated atoms.

Also from the manual: When initially heat a new rod, almost all rods will melt at the tip and form a ball held by surface tension. Thus, repositioning of evaporate rod may be necessary immediately after initial heating. Can recognize when the rod has significantly shortened in length when “at a given EMIS the high voltage has to be increased in order to obtain the same FLUX”. But in our case for W, we already are using the maximum voltage, so we will probably just see the FLUX drop when use the same EMIS. If this occurs, then need to move the rod further in. Since we do not know how much of rod has melted it is probably best to move it just 0.5mm at a time.

## 2.2 Selenium evaporator procedures

The selenium evaporator is made out of two parts: an effusion cell and a cracker zone. The cracker zone is a heated region near the top of the crucible (aka the hot lip). Since Se tends to evaporate in clusters, the cracker zone is used to further crack down the Se clusters which are evaporating. According to literature, temperatures for efficient cracking of Se range from 400-900C. However, due to risk of overheating the Se source (i.e. boiling), for our evaporator another group told us that 400-500C is the limit. The two zones are controlled by two separate controller. We label them EC (effusion cell) and HL (hot lip).

1. Turn off the ion pump. This is essential!
2. Ramp both controllers slowly (15° increments for HL and 10° for EC) to the desired temperature. For all depositions up to May 2017, we have been using 60C for EC and between 135-155C for HL. However, we were probably not using the cracker properly and not actually cracking the Se. Also, it may be ok to use higher temperatures for EC since 100-120C seem commonly used in other groups.
3. When ready for deposition, open the Se shutter (turn 180° from close position).

## 2.3 Co-deposition Overview

For actual growth of films, the following is the general procedure:

1. Assuming the substrate is already ready and on the heater stage, begin heating the sample to desired temperature with resistive heating. (**The displayed heater stage temperature is not the actual temperature of the substrate** therefore must use chart. Omicron has refused to give us a table of values for the heater stage vs. substrate relationship because their measurements are based on an average of measurements taken on many systems. They claim that the error in reading their graph chart is < 5%). This takes some time, so generally allow at least 1 hour to heat the sample before beginning deposition. Also the general rule we've used is to wait for the displayed heater stage temperature to be at the desired value for 30min before beginning deposition to make sure that sample has reached equilibrium with stage.)
2. Begin ramping the e-beam evaporator before Se effusion cell because the e-beam generally needs to be ramped more slowly (especially the Sn one since are using a crucible there).
3. Turn off ion pump and ramp Se evaporator.
4. When everything is at desired conditions, open Se shutter. Wait 5 min. and then open the metal source shutter. (We have also opened both shutters at time and had reasonable results, so unsure that opening Se shutter first is necessary but the idea is to establish a Se pressure in the chamber to prevent pure W or Sn from forming clusters on the sample.)
5. When done with deposition, close both shutters and ramp everything down at reasonable pace (i.e. 10-15° steps with Se) simultaneously. The total ramp down time takes about 10 min.
6. Generally we want to move the sample into STM as soon as possible after deposition to prevent it from getting contaminated by the degassing of the cryostat walls. However, it seems that the cryostat doesn't start regenerating until about 12 hours after deposition, so if necessary can move the sample up to 12 hours later.

## 2.4 QCM Operation

The position of the QCM is adjusted by the bevel. The markings are:

- The closest to MBE sharpie mark is position for when are using the meter. This makes the QCM inside stick out from the metal shield tube so that it can measure a deposition rate and thickness.

- The furthest from MBE sharpie mark is position for when not using the meter. This puts QCM well inside the metal shield tube so it is protected from deposited material.
- The middle sharpie mark is bakeout position.

The reason we don't use the QCM during actual growth is because that would wear it out too fast and it would die. To replace it, need to vent the MBE after which would need to do a bakeout! Therefore we only use QCM to calibrate (choose the proper parameters for deposition) the evaporators individually. This is how the QCM in this MBE design is intended to be used. To use QCM:

1. Move QCM to the closest to MBE sharpie mark. Connect the BNC cable connection that has a small black box along the cable. Can use an adapter to make BNC cable easier to connect. The connection is right in between the in and out water cooling ports of the QCM. Be sure water cooling is running.
2. Turn on the thickness monitor by the switch in the back of electronics stand. Wait a few seconds and it will show some thickness and rate and frequency of QCM.
3. Select the material whose deposition you want to measure. There is a pre-programmed list of many materials with their properties which program needs to calculate thickness and deposition rate from QCM raw data. Also you can make your own favorites list.
4. Press "clear" to make the thickness reading 0.
5. Get the evaporator running at the deposition parameters but with the shutter closed, so no material is being deposited on your substrate yet.
6. Open the shutter on the evaporator and immediately open shutter on QCM which allows it to measure things.
7. For some materials, the deposition slower is faster than the resolution of the rate meter (ie.  $< 0.1\text{A/s}$ ) so the rate meter will always read 0. In this case need to use the thickness meter (which is additive thickness) and divide by the running time to estimate the average deposition rate.

**Currently, as of May 2017, the QCM is unable to detect Sn deposition.** The reason may be due to the geometry of the ports. According to Omicron, the QCM is located 20mm below the point where the beams should meet. For the E-beam evaporators, the beam is quite small, so from some ports it is possible that the beam simply doesn't hit enough of the QCM to result in a reading.

**On June 1, 2017 we switched the positions of the Sn and W evaporator and will retest the QCM.**

### 2.4.1 QCM calibrations

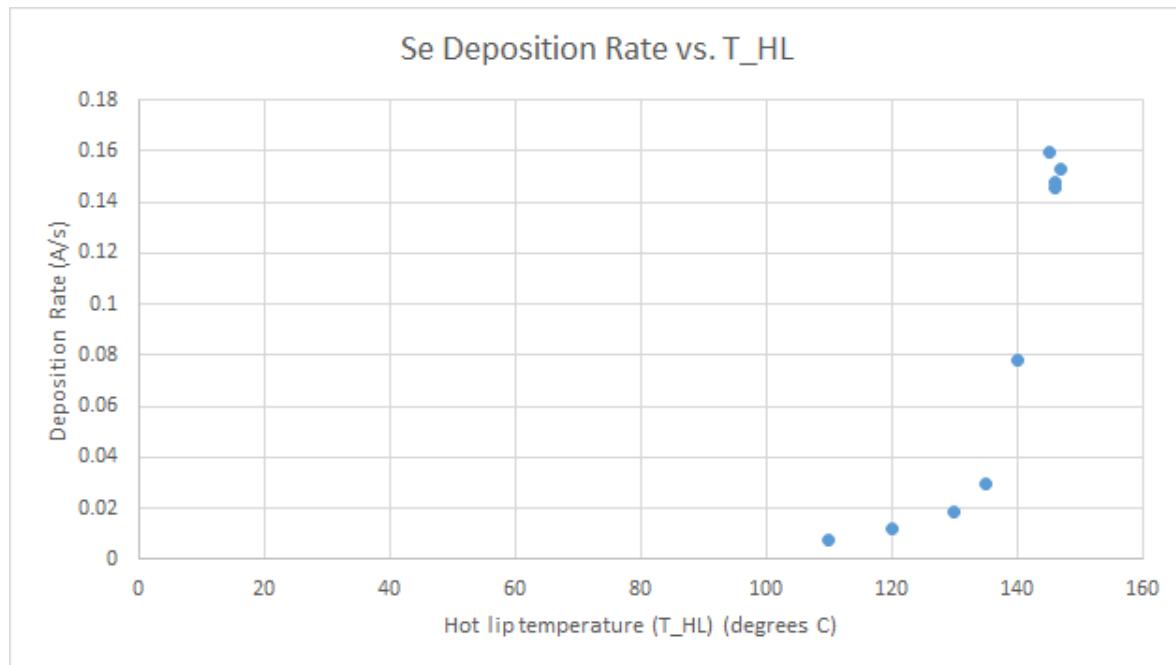


Figure 2.2: Se calibration from 10/14/16. Effusion cell temperature was kept at 60C.

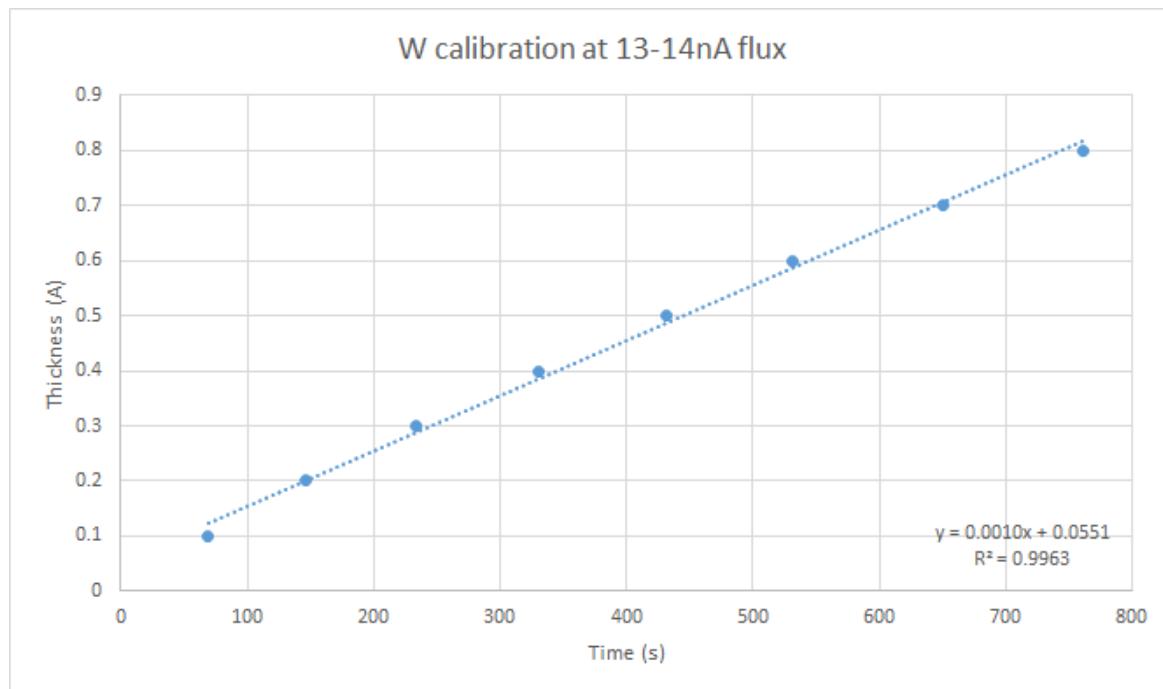


Figure 2.3: W calibration done on 10/20/16

## Section 3

# HOPG Substrate Preparation

### 3.1 HOPG Substrate and Sample Plate Compatibility

The holder we use with the threaded posts and screws in standard configuration (sample plate, then post and nut to hold post, then holding leaves and another nut), can only accommodate HOPG substrate that is > 1mm thick. This is because the nuts that go under the screws are 1mm thick so this is the minimum substrate thickness to be able to hold substrate in place. If we use anything thinner, will either need to remove the nuts that are between sample plate and leaves or add a spacer (i.e. another thin piece of HOPG). It seems risky to remove the nuts since they provide additional support for the posts.

Also the  $10 \times 10$  SPI substrate needs to be cut in half by the machine shop. The HOPG substrates could have a preferred cleaving orientation, so it might be wise to check the cleaving first and specify to machine shop in which direction to cut. We cleave the sample by pulling off tape lengthwise. **Make sure to let machine shop now that this is a very brittle sample. They have a different method of cutting it.** If they don't know what we gave them, the cut quality may not be so good (personal experience).

### 3.2 Cleaving

As of May 2017, the following is working procedure for cleaving HOPG: Use regular Scotch magic tape. I found that the adhesion of the Kapton vacuum tape is poor resulting in an incomplete layer being removed by the tape.

1. Put HOPG on a kimwipe and stick a piece of tape to it.
2. With back of tweezers (tape tweezers to finger), very gently and applying no downward force rub the tape in different directions for 2-3 minutes. The purpose is too remove bubbles and improve adhesion between tape and HOPG.

3. Peel the tape very slowly (over course of 30-60sec).
4. After cleaving, do not touch the HOPG cleaved surface with anything. To remove any possible tape residues, sonicate in acetone and IPA for 5 min each.
5. Let the HOPG dry, then carefully install the substrate in the substrate holder.

Note: The cleaving will never be perfect. Will always have non-smooth areas, terraces. However should see sample is relatively flat, no large chunks sticking out, and have some flat terraces. Usually 1 careful cleaving is enough to achieve this.

### 3.3 Assembling sample plate

This is difficult to describe in words. The best way to do it right is to pay attention to how you disassemble the existing plate. Briefly, insert each post and screw it in a little, but make sure it doesn't protrude from the other side. Then add a nut to the post and use the special molybdenum tool to tighten it and the post. Then add the leaves, and the nuts on top to hold the leaves down. **Do not touch the parts or sample with stainless steel tweezers or other tools since we are concerned about Ni contamination.** If can't find a suitable tool, can wrap the stainless tool in foil before using it, but it's not too convenient.

**Never sonicate the assembled parts together. It damages the threads.** If parts need cleaning, need to sonicate the individual parts.

### 3.4 Annealing

Once we load the newly cleaved HOPG to the heater stage we prepare/further clean substrate before deposition by annealing it using resistive heating. If planning to do deposition on the same day, make sure to move manipulator to deposition position before start annealing. If not doing deposition the same day, manipulator position is not important as long as it not closer to the evaporators than the deposition position ( $z=95\text{mm}$ ).

1. Select *resistive heating* in the bias controller touchscreen menu. Turn on resistive heater. Press *prev* and make sure that current is 0.
2. Gradually increase current until the base plate temperature (displayed temperature) is 350C. (This corresponds to the substrate temperature of about 450C.) When increase current, need to wait a bit to give the temperature time to warm up.
3. Anneal for 30-60min at 350C.

4. Increase current further so temperature goes to 375C. Anneal for 30min.
5. If planning on doing deposition the same day, gradually tune current down to the value necessary for deposition. It is safe to let sample be heated for some time (up to hours) at deposition temperature before deposition if you need time to prepare things. Otherwise gradually tune current to zero. **Do not move the manipulator or attempt to take a sample out until the heater stage displayed temperature falls to  $\leq 100^{\circ}$ .**





## Section 4

# Maintenance

### 4.1 E-beam evaporator maintenance

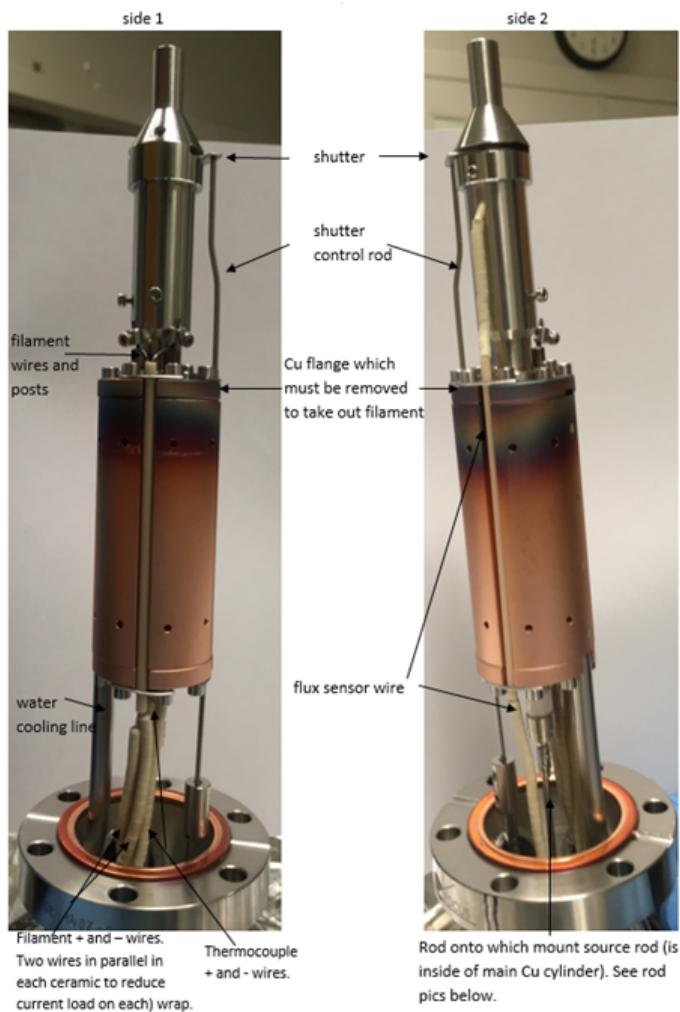


Figure 4.1: Tungsten evaporator components

### 4.1.1 Filament Replacement

This is described in detail in the e-beam manual. Follow everything except we have changed the design of how the filament loops around the posts to make the spot-welding job easier. We add two tiny loops around each post which help hold the filament in place while spot welding. It is still quite difficult to attach; in the past we always asked Reza for help. **The working spot weld settings are 3.6V, 14ms using spot welder to Ghosh lab.**



Figure 4.2: Left: partially disassembled e-beam evaporator. Middle: Original attached e-beam filament. Right: Re-designed filament attachment.

### 4.1.2 Replenishing the source material

#### Tungsten rod

The source rod (our W rod was 40mm in length and 1.3mm diameter) is mounted at the end of the metal rod which gets inserted into center of the evaporator. Source rod is inserted 5mm into hole and held in place with 2 screws. When inserting and removing this rod setup must be careful to not hit the walls of evaporator so nothing gets bent. After using W rod for several months, we can clearly see the result. At the tip is small ball of W formed (1mm diameter, this is in contrast with the 2-3mm diameter ball that the manufacturer says will form). Also a little lower can see significantly thinner diameter of rod. This happened when rod was inserted too close to the filament and electrons were bombarding the side of the rod instead of tip. To move source rod closer/further from filament the screw gauge outside of evaporator is moved which moves a bevel on which end this whole rod setup below is attached. The whole setup seen below moves as one piece.

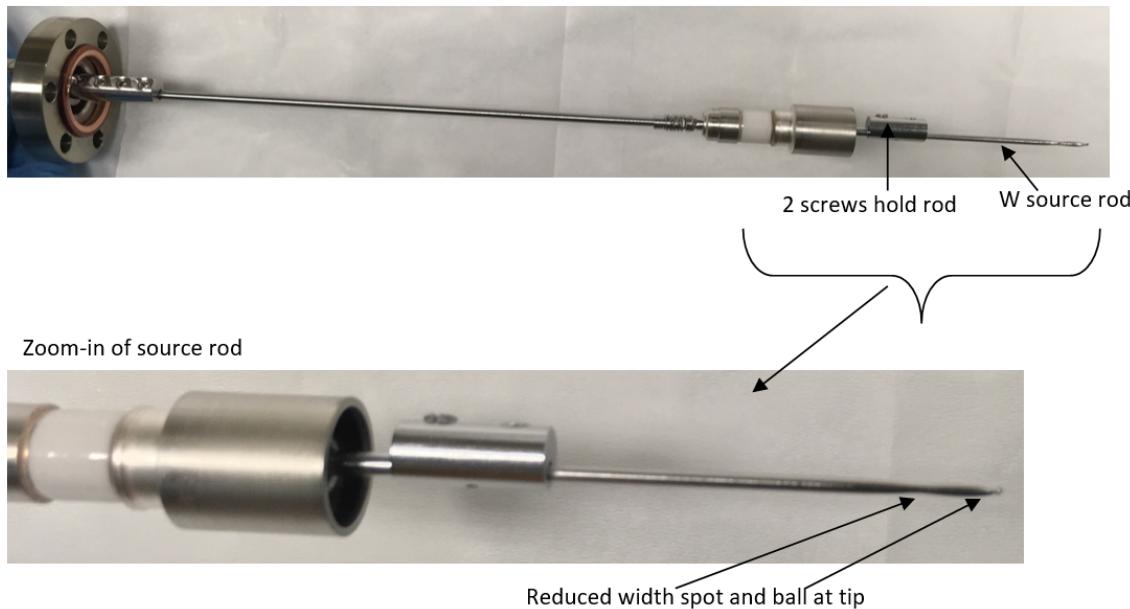


Figure 4.3: Tungsten rod setup

### Tin pellets

Fill the crucible about 1/3 full with pellets. This is to ensure that pellets don't fall out when slightly tilt evaporator as needed to insert it back into MBE port. Also ensures that no liquid Sn flows out of crucible. **Do not significantly tilt the evaporator or lay it down if it has source pellets in it!**

## 4.2 Removing a dropped sample

Taking out a dropped sample (as done on 10/27/16)

1. Get the mirror kit from the machine shop. Use the straight, extendable rod and the larger square mirror. The connections of mirror to rod and spring loaded (push back at rod end, insert mirror connection and release). Wipe rod and mirror with acetone and IPA, then wrap rod and back side of mirror with foil.
2. Open the large port on the side of the MBE (this port's face is vertical). Put the old gasket back and hold it in place by wrapping some foil around parts of it and the outer parts of port flange.
3. Open the medium sized port on the bottom (closest to water cooling manifold wall) of MBE.
4. Make sure QCM is all the way retracted and z manipulator all the way up. Turn on the 2 LED lights at the viewports.

5. Now have 1 person carefully move the mirror into the large side port. Other person lays on the floor and looks up through the bottom port at the mirror. Person with mirror moves mirror around so other person can see the bottom of the chamber and try to find the sample.
6. Once see the sample, procedure to take it out depends on where it is:
  - (a) If sample is in one of the bottom ports, then it is easiest. Simply open that port and sample will fall out.
  - (b) If sample is on the chamber floor, and there is a large enough port nearby (i.e. the one you are looking through at the bottom), then can take out the sample by hand. Must wear clean gloves and either wrap arm with clean foil or wear special shield (Andrew has), like cleanroom shield up to the elbow. It is easier to take out sample by hand than with some tool, since hand can feel the sample and grab it.
  - (c) If sample is near a port that is too small to fit hand into or not near a port/evaporator, then need to use a tool to move sample to a port and drop it into there. The other rod from the mirror kit (the flexible rod) can serve as the tool. Wipe this with acetone and IPA and wrap with foil. Then bend in the necessary shape and try to move the sample to a port by inserting the tool through one of the other opened port. It is useful to also make the mirror stay in place (easiest way is to simply put bunch of foil at exit point of the side port where mirror is, so mirror is resting on the foil) and use it to see what you are doing. Once have pushed the sample into a port, just open that port to take it out. Remember to change gaskets on all ports you have opened before re-tighten them for the final time before bakeout. These ports are to the main MBE system, so we do not want to re-use the gaskets since a leak is risky.

## 4.3 Degassing a new Ta crucible

Degassing New Ta crucible: A new crucible must be degassed before using. Crucible is installed without any source material into evaporator. Then evaporator is run which heats up the crucible and cleans it from dirt/impurities. The parameters values refer to those observed when we degassed a crucible for first time in Sn evaporator on 9/23/16.

1. Turn on water cooling (can do this day before to be able to check for leaks and save time).
2. Connect liquid N<sub>2</sub> hose. Make sure that using a full dewer (it is very inconvenient if L N<sub>2</sub> runs out mid-process). Start filling cryostat. It will need to be periodically refilled during the process.

3. Turn on evaporator controller. If it doesn't turn on, check the MBE interlock screen on touch screen to make sure that "activate devices" is selected.
4. Shutter can be kept either open or closed. We usually keep it closed b/c if keep shutter open the liquid N2 is used up much more since more heat gets transferred to the MBE chamber.
5. Since evaporator filament was exposed to air when filled the crucible, first need to degas the filament: Keeping HV (high voltage) off (therefore there will be no e-beam hitting the crucible), gradually ramp up FIL (filament current) to 2.0A in increments of 0.1-0.2A. Will see jumps in FLUX (flux current) as filament degasses. Wait to make sure FLUX is decreasing before further increasing FIL.
6. Now can degas the crucible. Tune FIL down to 0. Gradually ramp up HV to 2000V. Then gradually increase FIL until either there is a rise in MBE chamber pressure or EMIS reaches 5mA. When we did it, a jump in pressure occurred when FIL reached 1.0A at which EMIS was still 0mA. Wait for pressure to stabilize before continuing to increase FIL. Pressure might not go back down to initial levels, but as long as it stays constant for some time, can keep going. Keep pressure under 1e-7 during entire degassing procedure.
7. Keep increasing FIL using small (i.e. 0.05) steps if have pressure instability. When FIL reaches 1.5A, EMIS increases from 0 to 0.1mA. Flux rose to > 1nA when EMIS reached 0.2mA and FIL=1.55A. Flux will rise to higher values when increase FIL more, but generally should then decrease since once dirt has been removed from surface, there should not be more dirt coming off. Flux was 2.75nA when EMIS reached 1mA. Note that there will likely be a quite significant delay (up to several minutes) between turning the current dial and EMIS increasing especially during initial stages of degassing. We think this is due to that there is an initial degassing of the filament itself, after which it is more efficiently emitting electrons which results in the EMIS rise. Also we notice that after a while the FIL decreases a little, again indicating that it is being cleaned and improving as we proceed.
8. Controller switches to EMIS control (using a setpoint and feedback) when EMIS reaches 1.5mA. Reaching to 5mA will take some time since there is a lot of initial degassing occurring. At EMIS = 2.2mA, MBE pressure rose to mid  $10^{-8}$  from starting pressure of low  $10^{-9}$ s. After reach 10mA EMIS, can increase EMIS much faster since no longer have such large pressure jumps. Continue increasing EMIS in 5mA increments (5, 10, 15mA) and record all observed parameters right when reach that specific EMIS current and later after the parameters have stabilized. Since the flux generally continues to slowly decrease over a long time, we defined "stabilized" as 3 minutes after initially increasing to certain EMIS value and all other parameters are stable.

9. The goal was to increase EMIS up to the maximum possible 150mA which with the 2000V HV would provide 300W of power to the crucible. This was the recommendation that the manufacturer gave us for degassing the crucible. However, after reaching 115mA, we observed very high flux current, reaching  $\mu A$  level and not decreasing. We were concerned about evaporating the crucible itself (fortunately when we took out evaporator, we confirmed that no damage was done to the crucible), since contaminants should result in a decreasing flux after the initial increase. Even when decreasing EMIS to 100mA, we still observed a gradually increasing FLUX of  $\mu A$ . Therefore we stopped degassing at this point.
10. Gradually tune current down to zero, then ramp voltage down. Turn off evaporator. Need to wait for the crucible to cool before vent the system and take out evaporator to fill it with source material. Keep the water cooling running while the evaporator cools. Best to keep it running overnight.



# **Section 5**

## **Venting and Bakeout**

### **5.1 Flushing Water Cooling Lines**

This must be done before bakeout to prevent from water boiling in the pipes (and if need to turn off the chiller while cryostat is still cold to prevent from water from freezing in the pipes).

1. Open air valve behind AFM computer. Feel flushing tube end to make sure air is flowing.
2. Open water supply and return valves for the closed loop. This is used to relieve water pressure when all other loops will be closed.
3. For each MBE cooling loop:
  - (a) Connect air tube (white) to water input line (green label) and the clear tube to water out line (red label). Close corresponding supply and return valve. Set up a container to collect the water.
  - (b) Turn grey switches on water input and output lines 180° to flush position: plastic piece should point towards flushing tube connection ports. Water will flow for a few minutes. For regular flushing, wait 5-10min. For flushing before baking need to flush much longer (greater than 30min each line) to get all water out. Once done, disconnect the flushing tubes and leave the grey switches in the flush position.
4. Once all MBE cooling loops are flushed, can turn off chiller if needed. For bakeout, it's best to keep water running through the closed loop to reduce risk of corrosion.

### **5.2 Venting**

Ion pump and ion pressure gauge should be off well in advance (day before for ion pump, 1 hour for ion gauge) of venting because when they are on, they contain hot

filaments which could oxidize when exposed to air. **Also should pump the FEL with turbo pump so that will be able to open [MBE, FEL] valve and vent them together.** Wenzhi told me to run turbo pump overnight before venting? Why do you need to pump system down when goal is to bring to atmosphere?

1. If not already done so, flush the water cooling lines (see flushing procedure) turn off chiller and remove the cooling lines.
2. Retract the crystal thickness monitor rod to the bakeout marked position.
3. Check all valve positions:
  - (a) Pneumatic gate valve open
  - (b) [MBE, FEL] open
  - (c) [FEL, turbo] closed

Note: This is different from the Omicron MBE manual which has mistake.

4. Vent the system (click on turbo pump icon on touchscreen, then vent)
5. Make sure ion gauge is off: when it's off, the left display (MBE system pressure) will not display a reading. If it is not off, need to turn it off and wait 1 hour for filament to cool down before venting.
6. Venting is done with in-house N2 since we do not want fill the MBE with air. Open the N2 valve behind the AFM desk and to the left of the compressed air valve. Make sure to use N2 and not compressed air! Open the valve at end of N2 pipe (white) and adjust the regulator until feel proper N2 pressure: want a gentle N2 flow since venting slowly is safer. Insert the N2 pipe into black pipe which comes out of turbo pump.
7. On touch screen press: *turbo pump → vent → yes*. This turns off both roughing and turbo pump. Turbo takes some time to slow down. The system will gradually fill with N2 and can hear the soft air flow sound going in through the turbo. **Need to wait until the soft noise is almost gone which means the system is vented.** The small pressure reading in right display reads it near the turbo pump, so it doesn't indicate pressure inside MBE. It can be used for rough estimate of if pressure is rising or falling. When the air flow sound disappeared, pressure showed 2.8e0mB. The flexibility of the bevel on the manipulator can also help determine if system has been vented enough.

### 5.3 Removing evaporators

1. Need 2 people for this. Wearing clean gloves, use 2 wrenches (size10) to unscrew the nuts that hold the evaporator in place. When removing last nuts, one person

needs to hold the evaporator. Very carefully pull the evaporator straight out of the system (it is quite long and need to not hit any part of it against system). Make sure to not hit the wobble stick when standing up!

For the W evaporator, its source is a rod so can carefully put the metal protective tube/shield onto evaporator (1 person should hold evaporator steady), making sure to not hit components against the shield, and use the nuts to hold it in place. Wrap around near evaporator-shield connection point with large Kimwipes and then foil. Then can lay the W evaporator down, supporting it on the shutter and electrical connection "legs". **If the Sn or Se evaporator has source in it, can never lay it down!**. Once person must always hold it upright until the source crucible is removed.

2. After have placed shielded evaporator in safe and stable location, need to cover the evaporator opening in MBE system using a flange. First wipe it with IPA and then put a new Cu gasket into the slot.
3. Attach the cover with the same nuts used for evaporator to the opening. For tightening components on vacuum systems need to ensure that do it evenly. Here dealing with small components so it is not so strict. Must make sure that you are always turning in the correct direction (tightening and not loosening) because if loosen after tightening and then tighten again, this could result in leakage. Correct direction for tightening is clockwise when looking at the nut or bolt from the side that's facing out. First hand-tighten all the nuts. Then go in a crisscross pattern and tighten about 1/4 turn. Then go around in circles, gradually tightening each nut about the same amount. Do not apply too much force when tightening. Due to space limitations, it seems easier to hold the bolt in place while tightening the nuts from the bottom. Make sure to not hit the wobble stick which will be right above your head!
4. If planning on baking the system soon, before pump down, temporarily close [FEL, MBE] valve and install a blank (need for post-bakeout degassing) to the FEL. **Then before even tightening the viewport, remove the magnetic coupler from the FEL transfer arm while system is still vented.** This way if the blank falls (which has good chance of happening) while trying to remove coupler, can just open view port, put it back in the jaw, and try again. If where to fall when already pumped down, would need to vent FEL, then pump it down again after put sample back.
5. After the cover(s) are on, need to pump down the system. It is not good to leave the system at atmosphere pressure even though is filled with N<sub>2</sub>. Should pump down using the same valve positions described in step 3. To pump down:
  - (a) Turn off N<sub>2</sub> flow but don't disconnect the tube from turbo pump, otherwise it lets air into pump which could make it harder to pump.

- (b) Turn on turbo pump. Will at first hear a loud noise of the roughing pump motor. This noise seems louder than when turn on turbo pump on a system already at vacuum but is probably because of the higher pressure difference/- more work the pump needs to do when pumping from atmosphere. The turbo will turn on and speed up automatically until it reaches max speed. This should take less than 15 minutes. At max speed the turbo current should be about 1.65A.
- (c) After 30min pumping, can turn on ion gauge: At top control buttons press the book button, then display will say “emission auto?” then press checkmark button. Foreline pressure (right display) reading was 5.8e-1 at this time. MBE pressure (left display) was 1.2e-6 after 1 hour pumping. MBE pressure should drop to below 8E-7mB 2hrs after start pumping.

## 5.4 Bakeout

### 5.4.1 Procedure

Must follow all steps carefully!

1. Make sure maintenance work on evaporators is completed and source materials installed. Make sure a clean blank Mo plate is in FEL and no sample plate in MBE heater stage. Since there is high risk of the FEL transfer arm to jump when remove coupler and therefore for the blank (recall that we install a blank onto the transfer arm in FEL before bakeout, so that after bakeout can easily transfer it into MBE for degassing. Recall that for degassing things need a blank on the heater stage to protect it.), it is not good idea to first pump down FEL, and then try to remove coupler (we did it this way before). If blank falls, then need to vent FEL, reinstall blank again and then it might fall again.

Way to do it:

- (a) Vent FEL together with the MBE by having [MBE,FEL] valve open, but [FEL, turbo] metal valve closed (since want there to be only 1 pathway for venting (through the gate valve (touchscreen controlled)).
- (b) Open FEL viewport and install blank to transfer arm (make sure that have already taken out any samples/sample plates from the heater stage!).
- (c) Gently close viewport using same gasket and just few nuts (this is so that if blank falls while taking off the coupler, it does not fall out of viewport).
- (d) Now slowly remove the coupler. If blank falls, it's not a big issue, just open viewport again and reinstall blank and try again.
- (e) Once ready to pump down MBE, pump it down along with FEL, by keeping the valves in the configuration described above.

2. Start pumping down MBE+FEL by turning on turbo pump. Keep ion pump off. Make sure pneumatic gate valve is open, [MBE chamber, FEL] is open, metal valve [Turbo molecular pump, FEL] is closed. System needs to reach good vacuum (mid e-8mB or better) before start baking.
3. Thoroughly flush water cooling lines with air. Must flush significantly longer ( $> 30\text{min}$  each line) than for regular shut-down of water chiller since water vapor in lines during bakeout can cause damage(see details in Section 5.1).
4. Disconnect water cooling lines including the thicker lines above the T-shaped connectors with valves (these lines need to be slit with knife to be able to remove them).
5. Make sure all evaporator electronic connections are disconnected. (For safety, unplug corresponding power supplies before removing Se evaporator cable connections).
6. Use IPA to clean the chamber surfaces and viewports.
7. Move thickness monitor in to avoid it being hit by bakeout panel. For bakeout need to move in the protrusion so the bakeout wall doesn't hit it. The middle sharpie mark is bakeout. We don't move it all the way in because that put unnecessary (in the sense that the moving in part of the way already ensures that it doesn't hit the bakeout wall) stress on the bevel (bevel gets compressed a lot).
8. Cover viewports with 3 layers of foil to protect during bakeout.
9. Remove the plastic handle for metal valve [Turbo, FEL].
10. Remove the two black magnetic couplers from transfer arms.
11. Make final checks before install bakeout panels:
  - (a) **Are you sure the water lines are flushed?**
  - (b) Make sure no stray objects are lying on the system and all viewports are covered with foil.
  - (c) Valves are in correct position.
  - (d) All electrical wires that need to be removed are removed (LEDs, evaporator connections, the wires that connect to the manipulator, etc)
  - (e) Evaporator shutters are closed.
12. Install baking panels (see installation order below) and 2 heating blankets (for transfer arm and linear transfer line to STM). Connect power for the heating blankets.

### Panels setup



Figure 5.1: Order of panel assembly (read left to right).

13. Once system reaches good pressure (i.e. mid e-8mB), can start bakeout but first double-check the following things: a. Turbo pump is operating at full speed b. Ion pump and TSPs are off. c. Emission current on the ion gauge is set to automatic mode (see degassing ion gauge for details about using the controller).
14. Start bakeout with set temperature of 105C if have selenium source in the chamber. If don't have selenium source, can use a higher temperature (i.e. we used 120C on 6/2/2017).
15. Pressure in MBE chamber will start rising. Foreline pressure (the one in small display window) will nearly stay constant (measured near the pump) at 5.5 to

5.9e-1mB. It is expected to take about 90min for system to reach the 105C bakeout temperature. Should monitor pressure closely during this first period. Pressure will most-likely reach between 2 to 5e-6mB.

16. Use a webcam to monitor pressure during rest of bakeout. The first about 9 hours are most dangerous and require more frequent checking. **Note that the pressure may start decreasing a bit (water molecules have been removed) and then increase again (probably selenium is being evaporated from chamber walls).** See 6/2/17 bakeout log for example of this.
17. As bakeout continues, dirt/impurities are being removed from the system and pressure will gradually drop. Monitor pressure periodically. Once pressure drops to 8e-8 can probably stop bakeout. Most likely this will take 60-72 hours of baking.

#### 5.4.2 Bakeout Logs

ADD EXCEL GRAPHS HERE

## 5.5 Immediate Post-Bakeout Procedures

Right after bakeout is stopped:

1. Degas ion pump: a. Turn on ion pump controller (at very bottom of stand) and press *start HV* on controller touch screen. It will automatically select an HV at 7kV. Will see pressure spike up to e-6 or e-5 range at which point immediately press *stop HV*. Then repeat turning on and off but each time should be able to leave ion pump on for longer intervals (pressure will be more stable). Eventually should be able to leave it on for 5-10min with stable pressure.
2. Degas TSPs: Overide interlock (since degassing causes pressure rise and system will not allow to operate things otherwise). Then press “activate devices”. Once temperature cools to < 100C, start degassing TSP filaments (3 of them).
  - (a) TSPs are controlled by same touch screen controller as ion pump at bottom of stand. Select 1 of the TSP filaments (1,2, or 3) and hold “start TSP” until the display reads “firing”. It will automatically ramp up the current and then ramp it back down. Pressure will go up, might reach 1e-6mB during the ramp.
  - (b) Degas filaments 1 at a time at least 3 times. Use cycles of 1 ramp for each of 3 TSPs before doing a second ramp, instead of doing repeated ramps for only 1 individual TSP. 1 ramp cycle takes about 1min.
3. Degas ion gauge:

- (a) Keep override interlock enabled and devices activated. Keep TSPs and ion pump off. This puts a higher current through the 2 filaments of ion gauge to help clean it. On ion gauge controller at top of stand, press book icon until reach *ion gauge menu* → press checkmark to select this. Press book icon until reach *filament* and see which filament is selected → press *x* to go back to menu. Press book icon until reach *emission* → use arrows to find *medium degas* → press checkmark to select. Repeat for the other filament (there are 2 filaments since 1 is a backup, only 1 filament is used to read the pressure at a time).
  - (b) To repeat for other filament, need to turn switch pressure reading to that filament: Press checkmark and *x* at same time to turn ion gauge off. Then press book icon → *ion gauge menu* → checkmark to select. Book icon → *filament* → arrow to chose other filament → checkmark to select → *x* to go back to main menu. Book icon → *emission* (this allows to choose emission current used → checkmark to select *auto*). Wait until see a pressure reading show up again, now the other filament is on. Now proceed as before to verify that desired filament is on and degass it. One filament degassing takes 10min.
  - (c) Change the settings to use the filament that used before did degassing: book icon → *filament* → arrow to chose filament → checkmark → *x* to go back to main menu.
4. Now since have degassed the major things, can turn on ion pump. If turn on a bit later is also ok.
  5. Once system temperature reaches 80C, remove the small round covers to speed cooling.
  6. While ion pump is still off, run TSP filaments one by one for 1 ramp every 30min. 1 ramp takes just 1 min and will auto shut off. Will still have some degassing occur and pressure could rise to low e-7 range. Running TSPs will help improve the vacuum. After finish running TSPs, remember is disable the interlock override so that the safety feature is enabled.
  7. Once temp. reaches 70C, open flaps on flexible bakeout panels and heated tubes but do not remove them.
  8. At 60C, remove all flexible bakeout panels and tubes.
  9. At 50C, remove all metal bakeout panels. Remember to finish running all 3 TSPs for 1 ramp cycle. By this time, pressure should have reached low e-9 range.
  10. Wait until most system components are only slightly warm to the touch before doing any other work on the system. While waiting, can connect the electronic cables for the evaporators and the LEDs for the viewports.

11. Once pressure reaches low e-9mB range, will need to perform more degassing. All of these are not possible to do the same day as finishing bakeout. Generally allow about a week to finish all the degassing. **Before any degassing, make sure that there is a blank on the heater stage!**. To transfer a blank to stage, need to first put back the black magnetic coupler onto the transfer arm as follows:
  - (a) Close [FEL,MBE] valve (so when reinstall coupler don't accidentally push transfer arm into MBE).
  - (b) Open [FEL,turbo] metal valve to let turbo pump on FEL.
  - (c) Slowly put the black coupler onto short transfer arm. After on at some distance, arm will begin to move. Move in slowly until feel the resistance of forks touching the valve. Gently push on coupler to try to install coupler a little bit further in. Sometimes coupler will couple before it is far enough on the transfer arm. In this case, do not apply too much force, pushing arm against valve. To fix this, when move the blank into MBE heater stage, can apply some force when arm is locked to heater stage to push coupler in further. There is less damage risk, pushing against heater stage (since it's made for that) than pushing against valve.
  - (d) Close [FEL,turbo] metal valve and close [FEL,MBE] valve.
  - (e) While waiting for system to cool, reconnect the evaporator electronic connections.
  - (f) Once ion pump is relatively cool, close main gate valve (touchscreen) so system is running only on ion pump. Leave turbo running since will need it for degassing.
  - (g) Need to wait for system to cool (can judge by heater stage temperature, readout on touchscreen or touching various system components) before transfer blank to heater stage.
12. Degas evaporators (see procedure).
13. Degas heater stage (see procedure). **Make sure the evaporators have been degassed first, otherwise they will contaminate the heater stage!**
14. Run TSPs, 1 filament at a time, to help reduce pressure which will have increased due to degassing. To run: override the interlock and press *activate devices* on the interlock screen on touch screen. Then on very bottom touch screen, select which TSP filament want to run, hold "start" until displays "firing". TSP will automatically run and shut off. When run TSPs, the pressure will increase fast and then decrease. This pressure is due to the titanium being sublimated which then absorbs other particles and sticks to the chamber walls.
15. When pressure reaches 1-2e-9mB, turn on ion pump (press *start HV*). If pressure spikes up too high, immediately turn off ion pump.

16. If were able to turn on ion pump successfully, close gate valve on touchscreen (this is valve btw. MBE chamber and turbo pump). Now the system will be pumped only by ion pump. With the MBE on ion pump only, should be able to reach e-10mB pressures (without L N2 filling).

## 5.6 Degassing Se Evaporator

First, the power supplies need to be reconnected:

1. Need to have 3 boxes: the large grey Eurotherm box and 2 small boxes (which work together as 1 power supply + temperature controller is a separate box). Connect the EC (small box) connections to the Se evaporator protrusion that is labeled EC. Make sure the 2 terminals (metal loops) do not touch when are fixed in place! Need to tighten gently but at the same time make sure that the terminals do not move.
2. Connect the connections from Eurotherm box to the other protrusion on evaporator. When connecting the terminals polarity doesn't matter. Be very gentle when tightening the screws. The connections are very delicate and bend very easily! Connecting the power supplies is inconvenient due to the small space available near the Se evaporator.
3. For power cords: connect the 2 small boxes (power supply and temperature controller) to the surge protector and connect surge protector to a socket on the short wall of the room. Connect the large Eurotherm power supply using the orange extension cord directly to a socket on the long wall of the room (under the large Cutler-Hammer power breaker). This is so that are using different circuits and don't overload anything.

Now can begin the degassing:

1. On EC controller (small boxes) on the Solo temperature controller, at first set the voltage set dial to 1 (read in the small window of dial, the main knob should line up at 0). Setting to 1, makes the maximum voltage = 3V (or 10% of 30V). Then when reach higher temperatures, if see that actual temperature doesn't follow fast enough, increase the voltage set dial gradually. Once reach close to 60C, have the voltage set at 2. If initially set the max voltage too high, then temperature might overshoot in an uncontrolled way.

For bottom large (Eurotherm) controller, hear the “heating power output limitation” is equivalent to the voltage set on small controller. At first, set this to align with the sharpie mark. Later will need to increase it gradually to be able to heat to higher temperatures.

2. Gradually increase temperature on both controllers in 5-10C increments. If there is significant degassing at lower temperatures, then wait a while before further increasing temperature. It is better to degas as much as possible at lower temperatures at which the Se will not be evaporating than try to do all degassing at high temperatures where are also consuming the Se. On the Eurotherm controller, will need to adjust the heating power knob (increase it past the sharpie mark) for there to be enough power to maintain the desired temperature. Otherwise the temperature actually begins to decrease! But do not put too much heating power because that will result in temperature to rise too fast and overshoot.
3. In 2016 bakeouts, we degassed in the range 50-60C for top controller and 110-145C for bottom controller. Increased top by i.e. 2C increments and bottom by 5C increments. Want stay in this temperature range for less than 1 hour, otherwise will be consuming too much Se source. Therefore, do not wait too long between incremental increases. Stay at the max setting: 60C and 145C for about 10min, then gradually tune it down. **Note: Degassing procedure probably needs to change if plan on using much higher cracker temperatures.**

During degassing pressure may rise significantly since Se is easy to evaporate. Pressure may rise to e-8mB range. If pressure rises to high e-8mB, need to be very careful and perhaps tune down the temperature. (When degassed on 10/13/16, pressure stayed in the e-9mB range even at the max temperature settings of 60C and 145C).

## 5.7 Degassing E-beam Evaporators

The tungsten E-beam evaporator will be degassed twice. The first time, no water cooling and no HV is used. This is to degas the filaments. During the second degas, we use both the water cooling and HV and degas the evaporation source. The Sn evaporator will be degassed only with water cooling because Sn has high vapor pressure and low melting point.

### 5.7.1 Degassing Tungsten evaporator without water cooling and HV

**This does not apply to Sn evaporator when have Sn in the crucible! If you do this, you will melt or evaporate all the Sn!**

This is also the procedure for any other e-beam evaporator where the source is a rod or for degassing an evaporator where have an empty crucible (i.e. just installed a new crucible). **During this we keep HV off!** This is extremely important, since we have the water cooling off. Otherwise we will overheat the evaporators.

1. Turn off ion pump to avoid potential damage due to high pressures from degassing.

2. Make sure evaporator shutters are closed, otherwise too much heat is introduced into the MBE chamber.
3. Make sure that have properly connected the electronic cables to the evaporators: red goes to HV (the rod), black to flux monitor terminal, 4-pin connector to 4-pin port, grounding cable should be put under the nut on the flux monitor port protrusion and nut gently tightened with wrench.
4. Will degas without water cooling so evaporator can reach a high temperature (i.e. 250C) enabling to remove contaminants. This means must make sure that HV is kept off! Otherwise an electron beam will heat crucible/rod and thus evaporator to unsafe temperatures (the temperature reading on evaporator controller does not accurately portray temperature near crucible or rod when that is heated by an electron beam).
5. Gradually increase FIL current to 1.6A. (If need to degas two evaporators without water cooling (this will be rare), it is most time efficient to degas both evaporators at the same time.)
6. Wait for evaporator temperatures to rise which will cause pressure to rise. When did degassing on 10/5/16 on W evaporator, at first there was significant flux which later went to <0nA. This was probably the filament degassing, since we did not degas filament on this evaporator after changing it. The reason that we see no flux when degas the entire evaporator is that the filament locally reaches much higher temperatures than the evaporator body. These high temperatures of filament are enough to cause the dirt on filament (if haven't degassed it previously) to ionize and register a reading on the flux monitor. The evaporator body reaches just 200-300C which is not enough temperature to ionize the dirt coming from it.
7. During degassing, keep pressure below 9e-7mB. If pressure rises above this, decrease FIL to 1.5A or 1.4A. If temperature is rising too slow, can increase FIL in 0.1A increments. Note that there is a delay between increasing FIL and temperature increase. Once temperature reaches 220-240C and pressure stabilizes and starts decreasing, degassing is complete. (Manufacturer suggests to tune FIL to 2A and just wait 1-2hrs for temperature to rise to 200-300C, but we find this causes too fast temperature rise which causes too high of pressure so we use lower FIL currents). Unlike the STM thermal evaporators, here only 1 ramp up cycle is necessary to degas.
8. Gradually tune down FIL to zero. Evaporators will take a while (several hours to cool).

### 5.7.2 Reconnecting Water Cooling

The stainless steel pipe going down from evaporator is where hook the supply (green) lines. The pipe going out horizontally is where hook the return (red) lines.

Note: We have 2 types of plastic pipes: metric standard (about 6mm outside diameter) and US standard (1/4" or about 6.25mm diameter). The metric pipes fit the grey connectors on the tubes. The US pipes fit the orange connectors. Don't mix them up or will have leak! If try to insert US pipe into metric connector will feel a lot of resistance. When inserting into the proper connector, should just be able to easily insert then push and feel a click as it locks in place. The manifold uses all US pipes. The Se evaporator uses metric tubes and tube end has adapter with thread. These screw onto evaporator water cooling ports which are labeled as in and out.

### 5.7.3 Degassing E-beam Evaporators with water cooling, L N2 cooling, and HV on

**This is done after bakeout and degassing tungsten w/o water cooling (and w/o HV).** Both e-beam evaporators can be degassed at the same time.

1. Turn on turbo pump the day before plan to do this degassing. Keep ion pump on for now.
2. Make sure water cooling is running properly.
3. Make sure turbo pump has been running. Open gate valve: this will result in a pressure spike (i.e. into low e-9mB range) which is normal. The pressure should fall back down to e-10mB range after fill the cryostat.
4. Turn off ion pump to protect it from high pressures/contaminants due to degassing.
5. Fill L N2 cryostat which acts as a pump to help maintain low pressures during degassing. It also keeps the chamber walls cool. This is important since there is concern that using evaporators w/HV on radiates a lot of heat which would heat up the chamber walls and could damage the coating on the walls. The company suggests to always use L N2 when using evaporators with HV on.
6. Gradually tune HV to 2000V for W and 800V for Sn. Slowly increase FIL until get a stable FLUX of 10-20nA for W and about 100nA for Sn. This will occur after the dirt from the rod has been degassed and is slight evaporation of rod occurring. On 10/12/16, W reached 10nA FLUX at FIL = 1.88A, EMIS=26.6mA. Reached 17nA FLUX at FIL=1.90A, EMIS = 28.0mA and held at this setting for 15min. Pressure stayed in high e-10mB or 1e-9mB range throughout the degassing.
7. Run at this setting with stable flux for 15min. Do not run longer because are evaporating and consuming the source material here. While running, refill cryostat if necessary (if no vapor coming out of outlet, then need to fill).
8. Tune down FIL and HV.

## 5.8 Degassing heater stage

1. If evaporators have not yet been degassed, do that first. We degas evaporators before heater stage since there is no cover that can protect stage from dirt so it gets contaminants on it while degassing the evaporators.
2. Make sure there is a blank installed on MBE heater stage.
3. Make sure MBE pressure is in low e-9mB range before attempt to degas heater stage. If pressure is in mid e-9mB range, could still try degassing but pressure will most-likely rise into unsafe levels ( $> 5e-7mB$ ) before are able to reach 915C. It might make the second degas attempt easier though. Do NOT fill cryostat, it must remain at room temperature. Pressure will likely not be low enough if the evaporators have just been degassed. Need to wait until evaporators cool before try degassing heater stage.
4. Make sure evaporator shutters are closed, to protect evaporators from dirt coming off the heater stage which is above them.
5. Make sure ion pump is off to protect it from any high pressures and dirt coming from the degassing. To turn off press *stop HV* on ion pump controller.
6. Make sure viewport metal shutter is closed to protect viewport from the high temperatures.
7. On touchscreen go to “bias selector” screen and choose “resistive heating”. Then turn on heater control box, press preview, make sure see 28.7 voltage value (+/- 0.2) and 0.02(or close) current value. If values are different, then do not proceed further. Press *out* on heater controller: this turns on the output to the heater stage. Push *fine* to be able to have better control when turn the dials.
8. Tune the current up in 0.5A steps (or lower increments if pressure increases too much). Voltage will increase automatically. Heater stage temperature is displayed on the touch screen. Temperature will increase as increase current. Keep pressure below 5e-7mB, but preferably in mid-e-8mB while current is below 8A.
9. At first as increase current in 0.5A steps, should observe increases in pressure, followed by decreasing back down to the value before did the increase. Once heat sample stage to about 600C, pressure will most likely gradually creep up without decreasing. When see pressure gradually creep up, just keep increasing the current after waiting for a few minutes. The continuous pressure increase is because higher temperatures, stage is also significantly heating the MBE chamber walls and they will start degassing also. Since there is a large amount of walls, pumps will not be able to overcome this degassing. This is why it is important to start the heater stage degassing at a low base pressure.

10. Continue tuning up current (while keeping pressure below 5e-7mB), until heater stage reaches 915C. The current needed will most-likely be 10.66A. Do not allow heater stage to heat above 917C (1190K) since the absolute upper limit that the heater stage can withstand.
11. Degas at 915-917C for 3-5 minutes. Note that the temperature tends to slowly increase even after have already set the current to 10.66A so monitor the temperature closely. If at any point it becomes > 917C, decrease the current immediately. Pressure is unlikely to decrease and may even creep up. This however does not mean that degassing is ineffective. Pressure is high because the heating of heater stage to such high temperature heats the MBE chamber walls which start degassing a lot also.
12. Gradually tune down current to 0. Can tune down faster than tune up, but still do it at a controlled speed so pressure and temperature doesn't fall too fast. Can turn off "fine" to tune down, but in that case, turn knob very slowly.
13. To shut-off: on heater controller, deactivate *out* button, deselect *fine*, turn off controller box. On touch screen *bias selector* screen, select *grounding*.

Some other notes:

At 200C, sample plate will already glow an orange color. When heater base reaches > 400C, plate will glow bright orange color. At 680C, plate becomes even more bright, orange-yellow color. At 765C, plate glows almost white at the center, but still bit of orange. At 870C, sample plate glows very bright white/pink color.

Note: The temperature displayed on touchscreen is at the heater baseplate. Temperature of sample is significantly higher and maxes out at 1670K when baseplate reaches the 1190K limit. There is a reference chart in the manual.

Maximum allowed current for the heater is 12A. Do not exceed this or will damage the filament. Manual states that usually 11A is sufficient to reach the maximum heater baseplate temperature. We have found repeatedly that current of 10.66-10.67A is sufficient to reach 915-917C.

If degas for a second time (i.e. in the case that the first attempt reached too high pressures before could reach 915C), then pressure should behave much better this time and therefore can increase the current a little faster and in 1.0A increments up to the level that reached upon first degas attempt.