Maintenance guide for Nanotalo UHV e-beam evaporator

Mikael Kervinen

October 22, 2020

Preface

The purpose of this guide is to serve as a written document of the regular maintenance process. This guide is based on my personal experience and it should not be taken as the only correct way to do things.

Contents

1 Overview of the system

The Nanotalo ultra-high vacuum (UHV) e-beam evaporator is a custom build system that combines parts from different manufactures. The base pressure of the system can reach below 2×10^{-10} mbar, and the operating pressure is typically 2×10^{-8} mbar depending on the material. The system is equipped with sample oxidation and ion-milling options.

- Main chamber volume: 43 L
- Distance from the crucible to the sample: 40 cm

1.1 Cryopump and He compressor

- Cryopump: Cryoplex CP8
- Compressor: M125 Water-cooled Helium Compressor
- Contact details: Trillium Cryogenics AB
  info@trilliumcryo.se

1.2 E-beam source and power supply

- Unit: Model 568-01 5×4 cc
- Power supply: TT-6
- Company: Telemark

1.3 Ion mill

- Unit: 3cm FC ion source
- Power supply: MPS-3000 FC
- Company: Veeco
- Contact details: Veonis Technologies
  info@veonis.com

Figure 1: Overview of the machine.

2 Evaporator maintenance

The main task of the maintainers is to keep the material crucibles filled up. It is up to the maintainers to keep track of the material consumption. The maintenance frequency will depend on the usage of the materials. Typically it is every 4-6 weeks. The best practice is to schedule the maintenance on Friday. This way the maintenance can last over the weekend which results the least downtime for the users.

There are 5 pockets for the crucibles inside the evaporator. Following arrangement has been established:

1. Changed upon request
2. Ti
3. Al
4. Al
5. Au

Although the pockets have the same volume, the material consumption depends on the material density. Heavier materials last longer while lighter materials evaporate faster. Aluminium is the most used material and therefore there are two crucibles for the material. I have observed that safe limits for aluminium are 650 nm for a new crucible and 1000 nm for an old crucible. The difference is due to the packing density for a new crucible being lower than for an old crucible. In an old crucible, the material pellets have already formed a melt on the bottom of the crucible. Aluminium is always the first material to run out, and the other crucibles can be examined during the maintenance and filled if needed.

Before starting the maintenance make sure that there are new copper gaskets, new crucibles, and enough raw materials. The evaporator should be in a normal state where you would typically find it i.e. main power off, turbo off, and rough pump pumping the load lock.

2.1 Preparing the evaporator for maintenance

1. For safety, turn the R3 fuses to the off position (green color visible).
2. Turn off the high vacuum gauge by first selecting channel 1 and then holding the left arrow button for 3 seconds.
3. Close the valve between the cryopump and the main chamber. This will isolate the cryopump, which is required, since during the maintenance the main chamber will be open to the atmosphere.
4. Open the valve between the load-lock and the main chamber.
5. Close the valve between the load lock and the turbo pump. Make sure the oxidation valve is closed as well. Open nitrogen valves to vent the main chamber.
6. The pressure can be monitored from the Gauge A. Notice that the gauge is at the load lock so there is a delay before the pressure is the same in the main chamber.
7. Once atmospheric pressure (1013 mbar) has been reached, open the load lock door and close the nitrogen valves.

2.2 Filling the crucibles

1. Unscrew the bolts of the 10" flange behind the evaporator.
2. Before the flange is completely unscrewed, attach the flange onto the two rails with the two nuts that are on top of the evaporator.
3. Slide the flange open.
4. At this point, it is a good advice to switch to fresh gloves in order to avoid contaminating the crucibles.
5. Remove the crucibles from the copper pockets using knives to help lift them. Avoid touching the inner parts of the crucibles.

6. Place the crucibles on a table and inspect them. Crucibles where the material has spilled over should be replaced. Don't replace the Au crucible, instead, use a sandpaper or a file to remove the material on the outside of the crucible.

Figure 2: A crucible where the material has spilled over and needs to be replaced.

7. Fill the crucibles and log the amount on the logbook.

8. If you change the crucible 1 to be a new material, you must also program the material parameters to STC-200. Follow the manual of the unit for the procedure.

2.3 Cleaning the pockets

The evaporated material will accumulate around the pockets and it can cause thermal short between the crucible and the pocket, and it can cause the crucible to become stuck into the pocket. Therefore the material has to be cleaned during every maintenance.

1. Use any tools available to clean the evaporated material around the copper pockets.

2. A chisel or a power tool is a very good for removing material.

3. The evaporated material should be cleaned until bare copper is visible. Failure to do so will only make the cleaning more difficult next time.

4. Lots of material will accumulate on the copper covers over the pocket. Make sure to clean those as well. The best practice I have found is to pry the excess material with a sharp chisel.

5. Use vacuum cleaner to suck all big metal flakes. Clean also inside of the chamber.

6. Wipe the copper pockets with cleanroom paper and isopropanol. Use plenty of isopropanol so that the paper doesn't leave residue behind. Use a new part of the paper for each pocket.

Figure 3: Left: Pocket with material accumulation. Right: Cleaned pocket.

2.4 Changing the quartz crystal

1. Check the crystal lifetime by turning on the STC-200. It shows as XTAL life in percentage. If the lifetime is less than or close to 50 %, it is a good idea to change the crystal. Then turn off the STC-200.
2. Remove the bolts from the 2 $^{3}/_{4}$" flange. The bolt size is 10 mm.
3. The whole crystal assembly will come out by just pulling.
4. Remove the crystal cover by pulling.
5. At this point remove the copper gasket. The gasket wont fit if the cover piece is still in place.
6. Replace the copper gasket.
7. Replace the crystal. Check the corrent orientation from the old crystal. Put the cover back in place.
8. Check the crystal lifetime. It should be over 90 %.
9. Put the flange back in place and then tighten the bolts. Use torque wrench with 20 Nm.

2.5 Closing the flange

1. Switch to a pair of fresh gloves.
2. Place the crucibles back to their respective pockets. The crucibles should be kept in their respective positions to avoid material contamination.

3. Insert a new copper gasket onto the flange. Hold the gasket on the flange side.

4. Place a bolt on the bottom of the flange and slide the flange close so that the bolt will guide it in place. Try to hold the gasket in place as long as possible for example with a blade of a knife.

5. Once the flange is in place start to screwing in the bolts. Start by placing a few bolts through the bottom holes on the flange. Some bolts have to come from the other side since the e-beam source is in the way.

6. At this point, check the deflection coil power supply. The CC (closed circuit) light should be on, otherwise the deflection coil cables inside the main chamber have detached during the maintenance and they should be reattached.

7. Tighten the bolts to 22-25 Nm using a torque wrench. First tighten them evenly in a zigzag pattern, then go through the bolts multiple times.

8. Start pumping the chamber through the rough pump by opening the turbo gate valve. After the pressure is around 5 mbar, I usually flush the chamber once with nitrogen before pumping it down again.

9. Once the pressure is below 1 mbar the turbo pump can be started.

2.6 Heating the vacuum chamber

The purpose of the heating of the vacuum chamber is to detach gas particles (typically water vapor) from the walls of the chamber. Every time the main vacuum chamber is opened, the chamber must be heated again in order to reach the lowest possible base pressure. The cooling water that circulates the chamber must be turned off and drained. The water lines must be drained since closed water lines with water in them would generate steam and increase the pressure inside a closed system.

1. Close the water valves labelled chamber and e-gun. Close both the input and the output side. Don't close the valves labelled compressor and turbo.

2. Open the two nuts of the chamber water lines on the bottom of the evaporator. After you open both of the nuts, water will drain from the chamber cooling lines. Be ready to catch the water in a container.

3. Open the e-gun cooling line from one spot and let the water pour out. Leave some container to catch the water that might drip out.

4. Open the oxidation valves so that the oxidation line gets pumped as well.

5. Start the heating by connecting the white extension cord to the power socket. Now the evaporator can be left overnight.

2.7 Melting the crucibles and finishing the maintenance

The material in the crucibles has to be a solid melt before it can be used for evaporation. Therefore the pellets must be melted with a low e-beam current. If the material was added only a few pellets this doesn't have to be performed, so this part is usually done only for the aluminium cups.

1. If everything is correct, the pressure should be below 5×10^{-6} mbar. If it's not, there could be a leak.
2. Stop the heating by disconnecting the extension cord.
3. Open the cryopump flange.
4. Start the high vacuum gauge by first using CHANNEL button to select channel 1. Then hold the right arrow for 3 seconds.
5. Close the flange between the main chamber and the loadlock. Close oxidation valves. Stop the turbo.
6. Reinstate the water connections and open the cooling water valves. Notice that other than the e-gun line, the valves shouldn't be fully open.

Figure 4: Correct positions for the water valves. Evaporator should be fully open, others only partially.

7. In a few hours the pressure in the main chamber should be around 5×10^{-8} mbar.
8. Flip the R3 fuses back on, and start the main power supply. Let it warm up for at least 10 minutes.

9. Select the crucible that needs to be melted and start the rate controller and deflection coils.

10. Start the e-beam. For some reason, after maintenance it is very likely that the power supply switches off when starting the e-beam. In that case just restart the power supply and try again.

11. Start increasing the current and monitor the high vacuum gauge constantly. Since the material is new, there will be lot of out-gassing. If the pressure keeps rising towards 10^{-6} mbar, ramp down the current and then slowly back up again.

12. Monitor the crucible from the mirror. Before the material is melted, it's hard to see the e-beam spot. Trust the previous settings that the spot is close to the center. Once the material melts, a dark spot appears in the center of the crucible (or at the e-beam spot). Let the material melt and increase the current slowly until the crucible has even dark color. Afterwards, ramp down the current and shut down the e-beam source.

13. Repeat the process for all of the filled crucibles. Typically this means only the aluminium crucibles. If the crucible was filled with only a few pellets they will melt quickly when the first user uses that material.

14. As a final step, evaporate titanium on the chamber walls. Titanium is very reactive element, therefore it can trap gas particles from the chamber and lower the chamber pressure. I usually evaporate around 5 nm of titanium.

3 Miscellaneous maintenance

3.1 Regenerating the cryopump

The cryopump works by trapping gases by freezing them on the cryopump surface. Therefore, the cryopump looses it effectiveness the more material is accumulated on the pump. To fix this, the cryopump needs to be regenerated by warming it up to the room temperature and pumping the gasses out. This does not have to be done often, maybe once a year. One indicator that the cryopump needs regeneration is that the cryopump temperature has risen. Typically it is 11.0-11.1 K, but if it's clearly higher than that, I would consider regenerating the cryopump.

1. Turn off the cryopump from the on/off switch at the back of the compressor. Press the button firmly to the off-position.

2. Open the cryopump flange. The main chamber pressure has to be low enough ($< 1 \times 10^{-4}$ mbar) and the route to the turbo pump has to be open.

3. Keep pumping the main chamber and the cryopump with the turbo pump.
4. Once the temperature of the cryosurface is close to the room temperature, isolate the cryopump and turn the compressor back on. It will take about 1.5-2.0 h to reach the coldest temperature again.
5. Once it's cold and the main chamber pressure is low enough, open the cryopump flange.

3.2 Dropped sample holder

When the sample holder is transferred to the main chamber, it is possible to mishandle the transfer such that the holder drops into the main chamber. This can be easily avoided with careful handling, which means that dropping the sample holder is always due to user's carelessness. Such behaviour leads to downtime for all of the users and it is not acceptable. Always talk with the user to find out what went wrong. First instance can be looked through the fingers but if it happens a second time, remove the booking rights from the user.

1. Isolate the cryopump by closing the cryopump flange.
2. Close the turbo gate valve, open UHV valve, and vent the chamber with nitrogen.
3. Open the 6" top flange that houses the sample stage. Tilt the sample stage to high angle to help the sample stage fit through the opening.
4. Make a mark on the flange to be sure that it is connected back in the same orientation.
5. Lift the flange. It may require some pulling and nudging to get it out.
6. Check where the sample holder is, then insert your arm through the opening and grab it.

3.3 Sample exchange rod

The screw at the end of the exchange rod is a part that can wear out. Therefore it is made replaceable. To avoid getting the sample holder and the rod stuck together it is very important to specify the users not to tighten the parts too tight but leave a small gap in between them. If the screw needs replacement the workshop can make a new piece fairly quickly. Tell workshop that the prints for the part can be found at 'NEMS/evaporator rod screw'. The screw should be made from stainless steel so that it is the same material as the holder and they don't wear each other out. To change the screw just unscrew the bolt on the side and slide it out from the end.

3.4 Polishing the mirror

With time, the mirror looking down at the crucibles becomes more and more foggy. Therefore, once in a while it should be cleaned. The mirror can be removed by opening the 6" top flange. Then it is simple lifted from its place. The mirror can be polished with sandpaper and metal polishing paste. When placing it back use a laser light or a flashlight to align the reflection at the cups.

3.5 Water cooling system

As long as there is no leak, the water cooling system does not require regular maintenance. However, once in a while, the filter on the wall at the input line can be cleaned. Best time to clean it is during cryopump regeneration. Stop the water pump, and close the input and output water valves at the wall. Use the tool to open the water filter. It is possible to clean the filter by just keeping it under a running water.

3.6 Opening the big top flange

The big 12" top flange may need to be opened sometimes. To open it, first open the 6" top flange and remove the mirror. Then open the flange for the quartz crystal and remove the crystal assembly. After that, undo the screws holding the top flange in place, and the flange can be lifted.

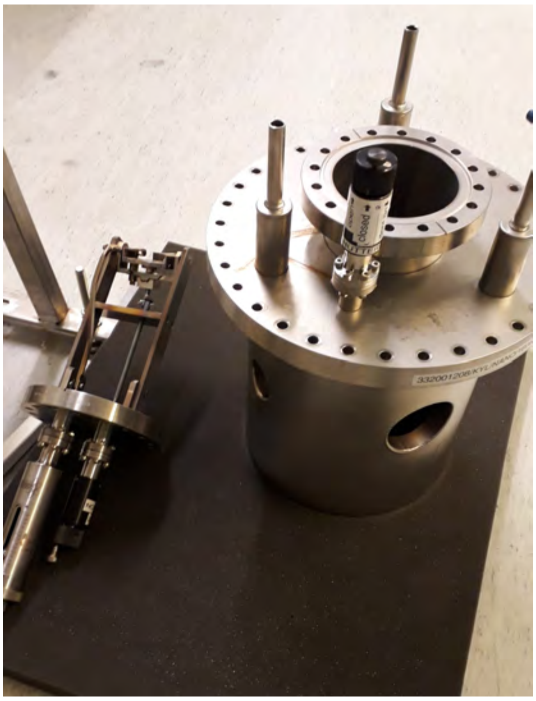

Figure 5: Top flange removed. Inside there is a hollow wall that can be cooled with liquid nitrogen.

4 Ion mill maintenance

Ideally, the ion mill does not need any regular maintenance. However, there will be situations where it is required to open the ion mill unit to fix a problem. Most commonly there is a short between the acceleration grids that needs to be removed, or one of the filaments needs to be replaced. The ion mill maintenance can be performed separately from the evaporator maintenance. Opening the ion mill unit is fairly simple since the unit is only connected to the load-lock.

4.1 Opening the ion mill

1. Vent the load lock like you would do in a sample exchange.
2. Detach the power cable by rotating it open.
3. Open the bolts of the 6" flange that houses the ion mill unit.
4. Before the bolts are completely off, detach the Ar gas line below the green valve.
5. Unscrew the last bolt and carefully lower the ion mill unit.

4.2 Disassembling the unit

The unit is best handled sideways on a table. Put all the parts on a cleanroom paper in the order that you removed them so that you can reassemble the unit as well. Refer to the manual for help when reassembling or disassembling the unit. Disassembling the unit is pretty self explanatory but I will detail the general steps. Check the manual for the item names and drawings.

1. Remove two thumb nuts that hold the neutralizer filament in place. Then remove the filament.
2. Remove the rest of the parts all the way to the two insulators.
3. Remove the two thumb nuts that are holding the cover shroud.
4. Lift the cover shroud. You may need to nudge it little bit and hold down on the body of the unit.
5. Before the grid assembly can be lifted, the grid mount ring needs to be detached from the body.
6. Loosen the screw that connects the grid mount ring, then slip free the mount ring strap.
7. Now the accelerator grid assembly can be freely lifted.

8. Unscrew the three screws that hold the grid stack together. Notice that in the grid mount ring, one of the wings is broken.

4.3 Cleaning and reassembling the unit

1. Clean the accelerator grids with sandpaper and isopropanol on a cleanroom paper.
2. Do the same for the grid spacer and the insulators. The short can be caused by a conductive coating on the edges of the insulators, so use sandpaper to get rid of the coating.
3. Clean everything possible in the unit by wiping it with IPA.
4. Reassemble the grids with the help of the manual.
5. Reassemble the whole unit.
6. Use a multimeter to check the connections at the power socket. All of the different pins should be open circuits between each other and to the ground. If they are not, something is touching a wrong place in the assembly. However, there are two pins for the cathode, which are of course short circuited.

4.4 Closing the ion mill

1. Replace the copper gasket.
2. Place the ion mill on the flange and make sure that the orientation is the same as before.
3. First tighten some bolts to hold it in place and then tighten the rest of the bots. Finally use the torque wrench at 22-25 Nm.
4. Put some vacuum grease on the Ar gas tube and then tighten the fitting back into its place. It may require some force to keep it in place until the threads start to bite.
5. Attach the power cable.
6. Pump the load lock with the turbo pump. The pressure should reach 5e-5 when the Ar valve is open and 5e-6 when the valve is closed.

5 Shopping list

Here is a list of things that are commonly purchased for the evaporator. All items are available at

- Contact details: Kurt J. Lesker
  www.lesker.com

5.1 Materials

Part No	Description
EVMAL50EXED	ALUMINUM PELLETS, Al, 99.999% PURE, 1/8" DIAMETER X 1/8" LONG, 100 GRAMS

5.2 Other consumables

Part No	Description
GA-1000	GASKET, COPPER, 10" FLANGE, 8.743"OD,8.007"ID 10/PKG
GA-600	GASKET, COPPER, 6" FLANGE, 4.743"OD,4.006"ID 10/PKG
GA-275	COPPER, 2 3/4" FLANGE, 4.743"OD,4.006"ID 10/PKG
EVCEB-22INT	INTERMETALLIC CRUCIBLE LINER, 0.885" TOP OD X 0.595" HIGH X 0.093" WALL, 15 DEGREE WALL ANGLE, 4CC POCKET VOL, 2.1CC LINER VOL