Cleaning the Aixtron BM

The Aixtron BM should be cleaned on an annual or biannual basis depending on tool usage. Ideally, there should be separate sets of quartzware for all substrates to prevent contamination and decrease downtime, but this clean may be conducted between substrate types as well. Pages 214-223 and 254-265 in the Aixtron BM manual should be consulted during disassembly and reassembly for valuable pictures and diagram.

1. With the chillers and gases off, vent and open the chamber.
   (a) Open the pecvd software and login. The password for the GUEST account is "graphene," case-sensitive. The keys are sticky, so watch to make sure all the letters are entered.
   (b) Click on the close pump valve button.
   (c) Click on the vent valve button, and wait for the green LED near the enclosure to turn on after a minute.
   (d) Carefully open the enclosure. Unscrew the clamp and lift the chamber door fully open. Push back the door to ensure that it is fully open before proceeding.
2. Put on a second pair of gloves. Disassemble the bottom heater.
   (a) Using just your hands, unscrew the four ceramic bolts on the top of the bottom heater. They should be fairly loose. As you remove the pieces, you should wrap each set (or large piece) in its own cleanroom wipe and separate into plastic bags.
   (b) Lift the quartz heater top ring off.
   (c) *Carefully* lift the graphite cathode up. Wrap this piece in a cleanroom wipe and place separate from all the other pieces due to its fragility.
   (d) Slowly lift the quartz heater dome. The thermocouple on the north end of the dome may be sticking to the tube, so watch it as you lift.
   (e) Remove the loose ceramic washers (8).
   (f) Use the provided torque screw (0.4 N*m) with the 2.5 mm head to remove the bottom heater. Wrap this piece in a cleanroom wipe and place separate from all other pieces due to its fragility. Keep the metal screws (4) separate.
      i. Put your fingers on the heater near the screws while you remove them to minimize forces on the heater.
      ii. Remove the rectangular heat spreader as well and place with the fragile graphite pieces.
   (g) Lift off the steel heater reflector shield. This will not be cleaned in aqua regia or SC2, so it may be good to put it in a separate location.
   (h) Lift off the quartz reflector cover.

(i) Carefully lift off the three quartz tubes (one on the thermocouple, two on the Mo rods).

3. Change your gloves. Disassemble the top heater.
   (a) Holding your hand under each part, unscrew the twisted quartz legs (4). Keep the screws and washers separate.
   (b) Remove the thermocouple by pulling it out from the base.
   (c) Use the provided torque screw (0.4 N*m) with the 2.5 mm to remove the top heater. Wrap this piece in a cleanroom wipe and place separate from all other pieces due to its fragility. Keep the metal screws (4) separate.
      i. Put your fingers on the heater near the screws while you remove them to minimize forces on the heater.
      ii. As this graphite heater is vertical rather than horizontal, it may be useful to have a second person to hold the heater as you unscrew it.
      iii. *Carefully* lift the heater out.
   (d) Remove the quartz tubes from the four anode rods and the two Mo rods.
   (e) While holding the quartz showerhead fixture, use your hands to remove the ceramic screws (4) holding it vertically. It may be useful to have a second person to hold the fixture while the first removes the screws. Lift the showerhead fixture away.

4. Wipe all metal, quartz, and ceramic parts down with an IPA-soaked cleanroom wipe. This will remove some of the carbon contamination, but evaporated metal may remain.

5. Clean the quartz and ceramic components with dilute aqua regia in an exhausted wetbench to remove metal deposits. Stainless steel tweezers or teflon tweezers can be used with aqua regia.
   (a) Put on normal corrosive PPE (blue chemical apron, inner latex gloves, chemical-resistant gloves, a second pair of vinyl gloves, safety glasses, face shield).
   (b) Turn on the DI water faucet and leave running while working with aqua regia.
   (c) Fill a large pyrex beaker with DI water and set next to your work station. This water will be used to quench a reaction if it starts going wrong.
   (d) Pour 2 parts DI water : 3 parts 37% HCl : 1 part 70% HNO_3 by volume into a large pyrex beaker in that order. Slowly add the acids to the water as mixing aqua regia is an exothermic reaction.

(e) Slowly submerge the parts into the beaker. This will need to be done in several batches. For many of the small parts, it may be necessary to use a teflon piece holder to dip them in and out. The quartz showerhead may also serve as a colander of sorts for smaller pieces.

(f) After 10 minutes, all of the copper or nickel should be removed. Very carefully transfer the parts to the water bath using tweezers, piece holders, or gloved hands (in the case of the quartz showerhead, which does not fit entirely into a large pyrex beaker and hangs over the edge). Soak the pieces (and tweezers or piece holders) in DI water for at least 5 minutes.

(g) Carefully pour aqua regia mixture into a separate hazardous waste container as it contains dissolved metals. Also pour the first rinse bath from the cleaned pieces into the same waste container.

(h) Rinse the pieces with the DI water gun.

6. Clean the quartz and ceramic components in SC2 in an exhausted wet-bench to clean any particles left behind from the aqua regia mixture. Teflon or delrin tweezers can be used with SC2.

(a) Put on normal corrosive PPE (blue chemical apron, inner latex gloves, chemical-resistant gloves, a second pair of vinyl gloves, safety glasses, face shield).

(b) Fill a large pyrex beaker with DI water and set next to your work station.

(c) Pour 5 parts DI water : 1 part H_2O_2 : 1 part 37% HCl by volume into a large pyrex beaker in that order. Add the acid carefully as this is an exothermic mixture.

(d) Slowly submerge the parts into the beaker. This will need to be done in several batches. For many of the small parts, it may be necessary to use a teflon piece holder to dip them in and out. The quartz showerhead may also serve as a colander of sorts for smaller pieces.

(e) Carefully transfer the parts to the water bath using tweeers, piece holders, or gloved hands (in the case of the quartz showerhead, which does not entirely fit into a large pyrex beaker and instead hangs slightly over the edge). Soak the pieces (and tweezers or piece holders) in DI water for at least 5 minutes.

(f) The SC2 mixture and any rinse baths can be poured into the AWN drain.

(g) Rinse the pieces with the DI water gun.

7. Dry the items at 80C in an oven for an hour to remove excess water. You will want to put down fresh aluminum foil to protect the quartzware from the bottom of the oven.

8. Wipe everything down with an IPA-soaked cleanroom wipe. Take care to wear fresh gloves to avoid getting fingerprints on the quartzware.

9. Bake the quartzware and ceramic pieces in the Vulcan 3-1750 furnace located next to the Aixtron BM to remove carbon deposits. There is enough quartzware that it will take at least 3 days to finish baking everything.

   (a) Open the exhaust flue to 0.06 inches of water.

   (b) Flip the green switch on.

   (c) Open the door to the oven, which should automatically stay open. It may not stay open, in which case you should work with a second person to keep it open.

   (d) Load the quartzware into the oven and close the door. As a precautionary measure, do not let the quartzware touch other pieces or the sides of the oven when loaded into the oven.

   (e) Using the numberpad, press 2 and then press enter. Press the green start button. This will start program 2, which ramps up to 950C at a rate of 4C/min and holds for one hour.

   (f) The program will begin ramping down but will not ramp fully down. After several hours, the furnace will be stalled around 850C. Press the red stop button to escape program 2. Quickly press 3 using the numberpad, then hit enter. Press the green start button. This will start program 3, which will carefully bring the quartzware and ceramic pieces down to 50C at 4C/min.The program does not need to run for the full six hours as three of the hours are holding steps at 50C; it can be stopped early at that point using the red stop button.

   (g) Once the furnace is at 50C, press the stop button the end the program. Unload the oven. It may be necessary to wipe some pieces with an IPA-soaked wipe if there is visible ceramic dust on them.

   (h) Repeat two or three times to accomodate baking all the quartzware.

   (i) Switch off the power switch and close the exhaust flue.

10. Reassemble the top heater.

    (a) Having a second person hold the quartz showerhead fixture to the instrument, gently screw and hand tighten the ceramic bolts to fix it in place. Loosen the ceramic bolts by one turn. This will prevent them from breaking as the neighboring materials expand during heating.

    (b) Slip the quartz tubes over the four anode rods.

    (c) Using graphite washers and the small metal screws, put the four twisted quartz legs back onto the anode rods of the top heater. The legs should be loose enough to allow you to move them around with unscrewing further but tight enough to stay in place once moved. Face them away from the heater.

(d) Slip the two quartz tubes over the Mo rods.

(e) Have a second person *carefully* hold the graphite top heater in place. Note that the legs should be clockwise instead of counterclockwise. Using the provided torque screw (0.4 N*m), screw the heater in with the graphite washer and screw. Hold the edge of the heater to minimize the forces put onto it.

(f) Attach the thermocouple. Bend the thermocouple so it touches the edge of the graphite top heater.

11. Reassemble the bottom heater.

(a) Put two quartz tubes onto the Mo rods and one quartz tube onto the top thermocouple.

(b) Carefully set the quartz reflector cover onto the bottom heater, lining the edges up with the rods.

(c) Screw the four metal studs into the four corners to help with alignment.

(d) Set the stainless steel heat reflector down, aligning its four holes over the edges. Make sure that the bottom and top thermocouples are able to poke through the holes.

(e) Fit the quartz heater stage over the heat reflector such that the rectangular divot is facing up.

(f) Place the rectangular heat spreader into the divot.

(g) *Carefully* screw the graphite heater into the Mo rods using the provided torque screw (0.4 N*m). Hold the edge of the heater to minimize the forces put onto it.

(h) Place two ceramic washers over each metal stud (8 washers total). This will prevent the quartz heater dome from placing pressure on the graphite heater.

(i) Carefully lower the quartz heater dome over the assembly, watching the position of the top thermocouple.

(j) Place the graphite cathode onto the quarter heater dome with its tabs rotated 45from the metal studs.

(k) Place the quartz heater top ring over the cathode such that the cathode's tabs sit in the slotted grooves.

(l) Remove the metal studs from the setup and replace with ceramic screws. Hand tighten and then loosen by one turn. This will prevent the screws from breaking when the materials expand.

12. Run a growth that has been known to work. Confirm the presence and quality of graphene using the Horiba Labram.