E-Beam evaporation facility

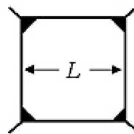
General overview of E-beam facility

E-Beam evaporation is a physical vapor deposition (PVD) technique whereby an intense, electron beam is generated from a filament and steered via electric and magnetic fields to strike source material (e.g. pellets of Au) and vaporize it within a vacuum environment. At this point they will traverse the vacuum chamber, at thermal energy (less than 1 eV), and are used to coat a substrate positioned above the evaporating material. Average working distances are 300 mm to 1 meter.

Since thermal energy is so low, the pressure in the chamber must be below the point where the mean free path is longer than the distance between the electron beam source and the substrate. The pressure is typically around 3.0×10^{-4} Torr or lower.

One of the E-beam applications is to deposit tiny Ohmic contacts on corners of grown sample which is required for Hall and Vander Paw measurements. There is a specific sample holder for these applications which provide tiny holes on corners while cover the rest of surface. We need to cut the samples with the **exact size** of the sample holder and then screw the lid to be mounted in chamber.

Sample with corner contacts



This device includes a chamber which is vaccumed by a cryo pump followed by a rough pump. It has an ion gauge to read the pressure inside the chmber.

There are two crucible to lead source material (one of them is out of work) and a shutter to cover the samle holder during adjustment. With the source material placed in the crucible a filament below the crucible is heated. By applying a large voltage, electrons are drawn from the filament and focused as a beam on the source material by several bending magnets. The beam is swept across the surface of the source material to heat all of the material. Chamber is also cooled down by water during evaporation precess.

A crystal can measure the thickness of the deposited thin film in-situ. When the stable evaporation rate is detected by crystal, the shutter can be opened to coat the substrate with desired thickness.

How to operate E-beam evaporation

To load sample holder, we need to vent the chamber first using N gas. Light up vent valve key on remote control. Open the N line with 20 psi pressure. After venting chamber, light down the vent valve key again.

1- Light up the hoist key on remote board followed by turning on the hoist key on panel.
2- Mount in the sample holder and and desired target materials regarding your purpose (you may use the below table). E-beam has 4 cricibles to provide maximum number of 4 target material; however, at the moment, rotator from place D to A is not working properly. Therefor, we use A, B and C or B, C and D in order for max of 3 targets and while going from D to A, we help the rotation by hand.

Materials for low-doped (<10^{18}) samples contact evaproation

P-GaAs Ohmic	Ti (0.8 kA^0) + Pt (1 kA^0) + Au (1.8 kA^0)	30s aneal at $450^{0\,C}$
N-GaAs Ohmic	AuGe (1 kA^0) + Ni (0.28 kA^0) + Au (1.5 kA^0)	120s aneal at $400^{0\,C}$
N or P GaAs shotky	Cr (0.25 kA^0) + Au (1 kA^0)	

kkkkkkkkk

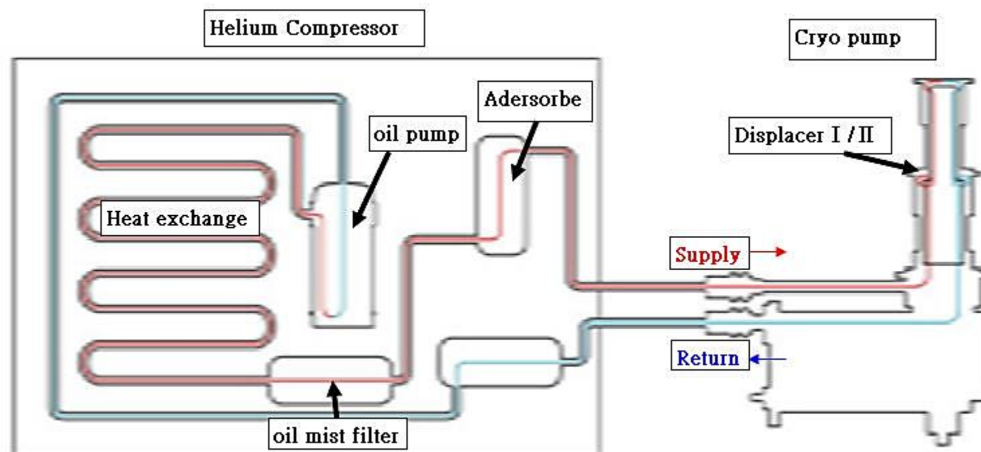
3- Close the chamber by turning the hoist key on control panel toward off position then light down the hoist key on remote control.

- before closing chamber, check that the mirror which is used to see the target surfaces is in good position regarding the chamber window to be seen from outside while depositing process.
- Clean the contact surface of chamber and buttom plate if there is any dust.

4- We need to evacuate the chamber up to 10^{-2} torr by mechanical pump (rough pump) followed by cryo pump up to 10^{-6} torr through following instruction:

- Light up the mechanical pump key on remote control and **after few minutes** open rough and fore valves by lighting up keys on remote control. (**Note:** mechanical pump is full of oil so we need to turn it on for a while (few minutes) before opening either fore valve or rough valve. Otherwise, if the chember pressure is lower than the connecting lines, the mechanical pump oil will be sucked into the chamber)
- When TC1 gauge reaches 100 mbar, close the rough and fore valves by lighting down the keys on remote control.
- to operate cryo-pump, first open the water drain line followed by water supply line which is used to cool down compressor. Then, turn on the compressor power botton.

- Note: compressore is designed to operate between certain range of He pressure under operation or rest status. the pressure is checked using the gauge attached behind compressor. If either of these limits is exceeded, some protecting internal circuits will cutt of the power and it means that we need to purge compressor.
  Compressor He pressure limits: 110 psi for operating status / 230 psi for rest status.
  Compressor has 2 slow-burn fuses behind the device to protect internal circuits. In the case of high current, the fuse blows up and the device is cut off. (generally we have two different kinds of fuse: fast-burn and slow-burn. Fast-burn fuse is a tiny piece of wire which can be seen by eye when it is burnt. Slow-burn fuses are like spiring and are used in those devices which start to operate by high current but the current falls to smaller value after a while for steady operation. To see whether the fuse is burnt or not we need to apply ohm-meter on end contacts. If ohm-meter shows a huge value (around mega ohm) it means that the fuse is burnt (open circuit).



5- Wait for cryo-pump to evacuate at least for …… min then turn on the ion gauge moniture to track the pressure. We need to reach the pressure of 2.5×10^{-6} torr before evaporation step.
   (**Note:** ion gauge bulb is directly connected to the main chamber. Any pressure above 10^{-6} can explode the ion gauge bulb. So keep eye on pressure to not exceed max pressure of 9×10^{-6} torr).
6- Turn on the power supply of evaporator by depressing the power botton on high voltage box. High voltage of about 6kV is applied across the tungestan filament to produce electron beam while crusible, chamber and all other components inside the chamber are grounded. (**Note**: avoid being in vicinity of high voltage supply while operation)
7- Open the chamber cooling water line to cool down the chamber. The water drain line is always open.

(the chamber is cooled down for two reasons: 1st to help the vaccume to be remained on desired level while evaporation since cool chamber make free particles more susceptible to stick to the hamber wall. 2nd to avoid overheating while filament is getting hotter and hotter)

8- Light up "e-beam evap" switch on remote control.
9- Switch the key to "on" status on. This key apply the HV on filament.
10- Turn the indicator to select to desired target source (b, c or d) and make sure that the target is aligned. (You may need to adjust it by hand through underneath handle gently)
11- Push the high voltage botton (on) and set the number on 6 kV.
    (try to go up to 6 kV almost quickly since there are some problems in HV system which may cut off HV automatically if this is done slowly)
12- Turn on crystal controller monitor and adjust density and z-ratio of the target material which is evaporated according to the provided table using following instruction:
    Press PG (program).
    Set the cursor on density and then enter number.
    Set the cursor on z-ratio and then enter the number.
    Press PG again to go back to monitoring page.

- When a voltage is applied to a quartz plate, the piezoelectric effect causes the plate to bend. If the voltage is removed, the quartz's elasticity makes it mechanically vibrate at its natural frequency and, while flexing, give out microvolt piezoelectric signals. As material condenses on the monitor, the crystal's mass changes, whereby changing its natural frequency. If a thin quartz crystal is combined in a special "free running" oscillator circuit, the RF voltage output from the crystal can be amplified and used to determine its frequency. Comparing the crystal's frequency an instant ago and its current frequency, and using an appropriate algorithm, gives a measure of the mass deposited and, from the time interval, a measure of deposition rate and thickness.
- A material's acoustic impedance, often quoted as 'Z', is density x longitudinal velocity of sound. When the Z value is compared to that of quartz it is quoted as 'Z-ratio'.
  To learn more about crystal controller:
  http://www.lesker.com/newweb/process_instruments/processequipment_technotes.cfm

Often used target materials in E-beam					
Material	**Density (gr/cm^3)**	**Z-ratio**	**Material**	**Density (gr/cm3)**	**Z-ratio**
Au	19.3	0.381	Ni	8.91	0.331
Cr	7.2	0.305	Al	2.7	1.08
AuGe	14.68	0.381	Ti	4.6	0.628
Mo	10.2	0.257	Pt	21.4	0.245

13- Push gun 1 filament on (in gun control 1 section)

14- Press “start” on crystal controller monitor to read the realtime deposited thickness.
To set the tickness to zero, press: stop, zero, start in order.
***Note:** through all folowing steps, track the ion gauge pressure not to exceed 8-9×10^{-6}. High pressure can explode ion gauge bulb, oxidize tungestan element with particles in chamber and help the HV to arc.

15- Increase the current slowly while checking the target material image in mirror untill you can see the glowing spot.

16- By adjusting the beam position, try to center the the spot in the crucible. Then adjust the sweep size to fill the circle. Frequency range depends on the the material source. For example, it seems that higher frequency is better for Pt.
(Note: To align the beam on target material properly, three adjustable items of beam position, sweep size (amplitude) and frequency has been designed. These factors change the current in electrical magnets in order to adjust the produced magnetic field magnitude and configuration to properly converge electron beam on target material)

17- After beam adjustment done, increase current to get **0.2, 0.3 A/S** deposition rate.

18- After beam adjustment and getting stable deposition rate, set the crystal thickness to zero and open the shutter. Fill the below table every few minutes for records.

	Time (min)	material	Deposition rate (A^0/s)	Thickness (kA)	Emission curent mA (Ec - mA)	Filament current (Fc - mA)	HV (kV)	Pressure (torr)
Example →	1	Ti	1.6	0.099	125	650	6.1	3.5e-7

19- After getting desired thickness, close the shutter and stop the crystal thickness measurement.

20- To change the source element do the following step:
Decrease the current to zero.
Gun 1 off
Close the sweep
Set frequency to zero
Set high voltage to minimum turnable possible degree (less than zero)
Turn the HV off
Wait for crucible to be cooled down and solidified (at least 10 min)
Close the water-in line
Turn the crusible to select next material source
Open the water-in line again

Start from step 13

21- After final evaporation, repeat step 22 folowed by turning the switch off on control panel and light down the E-beam switch on remote control.
22- Wait 20 min for high voltage to be cooled down and then turn of the power key.
23- Close the water-in line which cool down the chamber.
24- Close the gate valve.

To take out sample holder:

25- Make sure that the gate valve is closed then turn off cryo pump compressor. Close the cooling water-in line first followed by water-out line. (to avoid water accumulation)
26- To vent chamber, light up vent valve switch and then open the N gas line (pressure) into the chamber. After feeling gas to be flewed out of lower ring, light down the vent valve on remote control.
27- Light up hoise key on remote control, depress hoise key on control panel and then turn the hoise switch to lift up the chamber. (do the last part step by step to avoid chamber to bump into internal components). Take out sample holder and source materials while wearing gloves.
28- Turn the hoise switch again to lower the chamber slowly folowed by turning of hoise keys on controla panel and remote control.

To shut down the device:

29- Turn on the mechanical followed by opening rough valve to vaccume the chamber upto 10^{-2} torr. Then close the rough valve.
(Generaly we need to keep chamber under vaccume when we don't use it. At room temperature, the water molecules are very sticky and stick on the inner side of the chamber wall in several layers. Water is evacuated too slowly and the next evacuation will take too much time. Therefore, we keep the chamber under vaccume)

30- We need to vaccume cryo-can using mechanical pump. After turning of the cryo pump, we need at least one hour for condensated particles on carbon plated to evaporate and pressure to be increased slowly. After an hour, turn on the mechanical pump and then open the fore valve to enable the mechanical pump to vaccume cryo-can for ???
31- We need to dry water lines of compressor to avoid rust. To do so, open the water hoises from wall and pour out water into a buckle. Then connect one of these hoses to N cylinder and let the N flow in while another one is in the buckle near the ground. Increase the N pressure gently until making sure that all hoses are dry. disconnect the hoise from cylinder and leave them in a safe place on the ground.
32- Reconect the original line to N cylinder and make sure that the cylinder is closed befor leaving lab.

Fuse in the control panel / fuse of hoising chamber

To know more about appropriate materials to provide ohmic contact you these links provide useful information:

- H P Meijs, Doctoral Thesis: Ohmic Contacts on p-GaAs and p-In0.53Ga0.47As, Solid State Electronics Department, Faculty of Electrical Engineering, Technical University of Eindhoven, The Netherlands, 1992.
- For GaAs contacts: http://materion.com/~/media/Files/PDFs/Advanced%20Materials%20Group/ME/Challenge%20of%20Applying%20Ohmic%20Contacts.pdf
- A survey of ohmic contacts to III-V compound semiconductors: (n-p of GaAs, InP, GaN) https://crn2.3it.usherbrooke.ca/guide_sb/procedes/contacts/ohmic-III-V_Baca.pdf

Sputtering and evaporation sources for are available from Materion Technical Materials: http://materion.com/Businesses/TechnicalMaterials.aspx