So You're Going to Grow

An Overview of the (old) MBE System and Growth Procedures

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This document is meant as an introduction to the molecular beam epitaxy (MBE) system and the operation thereof in Dr. T. Tiedje's group at UBC (soon to be UVic). Focus will be on the workings of the system and growths of GaAs based III-V alloyed materials. This document is based on a similar document produced in the summer of 2005 by E.C. Young, a grower in the group from 2002-2006. It was largely comprised in the fall of 2005 as I was learning about the system and how to grow dilute nitride GaNAs. It tries to encompass the entirety of the system and its workings from vacuum system to growth procedure; all working parameters of the system are given where known.

Also included are analysis tools, 'Maintain your MBE' and troubleshooting sections. The analysis section describes some of the techniques and equipment we use to characterise the crystals, both in-situ and ex-situ. 'Maintaining your MBE' goes through what needs to be watched for on a daily and monthly basis, as well as a discussion of venting and baking the MBE. The trouble shooting guide is added should problems arise - and they will. To that end, others are encouraged to continue to add to these sections over the years as things change and when new, interesting problems arise - and they will.

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Part I : System Overview

It will be easiest to think of the MBE system as being comprised of several subsystems that can be either internal or external for the purposes of this document. Internal refers to parts inside the vacuum chamber such as the manipulator, trolley line, and the sources; external refers to those parts not in the chamber but associated to it: the vacuum pumps, RHEED, DRS, etc. The vacuum system and sources are complex enough to warrant their own sections.

In what follows, the first four sections will deal with the specific parts of the MBE system and how to use them correctly. The final section, Growth procedure (borrowed largely from E.C. Young), assumes a sufficient knowledge of all of the components covered in the first four sections and their respective functions.

A: Vacuum system

The MBE chamber can be sub-divided into three smaller chambers and the load lock (LL): the prep chamber for pre-growth, the growth chamber (GC) where samples are grown, and the etch chamber for un-growing. The prep chamber contains the load lock where samples are loaded and unloaded, the high temperature heating stage (HTHS) and the trolley line. The growth chamber contains the sources, the cryo-shroud, RHEED gun, flux gauge and the manipulator. The etch chamber simply has a sample holder and input lines for etching chemicals (Cl, Br, etc). Each of these smaller chambers has its own pumping system and each is separated from the others by a gate valve, see Fig.1. These gate valves should only be opened when the pressure on either side is similar and preferably lower than 2×10^{-8}. **The load lock should NEVER be opened at the same time as the valve between the prep chamber and the growth chamber**.

NB: With the more-or-less constant failure of one pump or another - typically turbos - the exact set-up of the vacuum system at any given time may vary from the following description.

i. The prep chamber is pumped ONLY by an ion pump. The gauge controller is located in the first of the three front racks, the pressure should be at most on the order of 10^{-8} under normal operating conditions, typically lower. The load lock has an independent pumping system comprised of a turbo pump backed by a scroll pump. The control for the turbo and the gauge are in the rear stack. The scroll pump is plugged into the green power consol near the

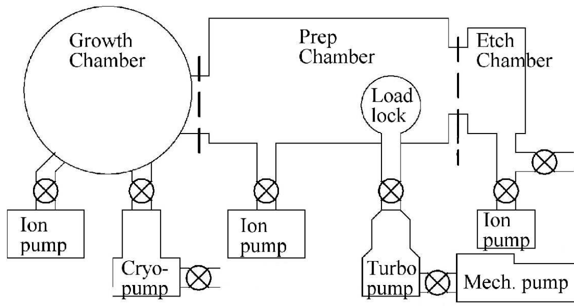


Figure 1: Schematic of the MBE vacuum system

front at the rear of the MBE, the power to the plug is controlled on the front racks, the pirani gauge is also on the front (right side of indicator, maybe now broken Oct 2008). This scroll pump is equipped with an isolation valve, it is recommended that the roughing system always (if possible) has this feature to prevent damage to the turbo/MBE in the event of a power failure.

NB: This mechanical pump up to 2008 was an oil mechanical pump and for large parts of its history was also used to back the turbo pump on the gas lines. This is not recommended as it could lead to contamination of the gas lines. If it necessary to do this again please be careful when venting the load lock, purging it with N, or opening the gas lines that the correct valves are opened/closed and that residual gas has been pumped out of common lines to prevent contamination of the gas lines.

NB: As of 2008 the MBE is now fully oil pump free, let's try to keep it this way! This means pumps, bellows, valves, etc, etc.

ii. The growth chamber is pumped by an ion pump and by a cryo-pump. The growth chamber gauge is in the first stack on the MiG. The pressure should be on the order of 10^{-8} when significant time has passed since the previous growth. When the shroud is cooled to LN2 temperatures the pressure should drop well into the 10^{-9} range. During growth the pressure will rise into the 10^{-7} range.

(a) The growth chamber cryo-pump can be regenerated through lines attached to the gaslines turbo pump. Again, be careful when venting the gaslines to other systems to avoid contamination - it is recommended

that the valve to the gaslines be mostly closed when the cryo-pump is regenerated.

(b) There is also a titanium sublimation pump in the growth chamber - it is not used very frequently and typically only when there are leak problems. Its controller is in the first stack.

NB: This pump was removed in 2007 because there was a leak in the ceramic feed through.

iii. The etch chamber is pumped by a small ion pump as well as a turbo (missing). As of the fall of 2005 the etch chamber is de-funct/off-limits due to pump problems (i.e missing turbo).

NB: As of the spring of 2007 the etch chamber was removed along with the gate valve to it - all was replaced by a simple 8“ blank flange.

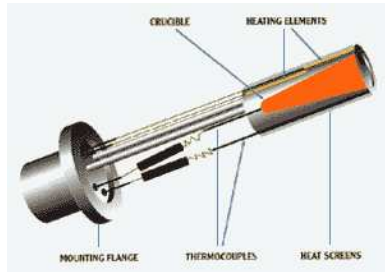


Figure 2: Image of a standard Knudsen cell consisting of 4.5" flange, cooling lines, heating filaments, thermocouples, heat shielding and crucible. The cells we use are more or less the same as shown here.

B: Sources

The VG-80 we use has 8 cell ports on the growth flange, they are: (clockwise from top) DRS in, In/Si, N plasma, Ga, Al, Bi, LS in, As. Gallium, Al and Bi are all Knudsen cells and operate more or less the same (see 2); the silicon/indium cell is a dual Knudsen cell from e-science. Most use PBN crucibles with the exception being Ga which now uses a carbon sumo crucible. The As source is a 2-zone cracker, basically a bucket of hot (400°C) arsenic and then a very hot (1000°C) cracker to produce As_2. Each cell is described in more detail bellow.

NB: After each bake the cells should each be outgassed (typical grow temps +50C for 30min+) and the fluxes of each of the sources has to be measured as a function of cell temperature, this data is recorded in the MBE log book. These fluxes are used to calculate growth rates and times for subsequent growths. After significant time (2 months) the source fluxes should be re-checked, there has been a recent trend to try to check fluxes more often, i.e. after most growths.

NB: The following temperatures quoted for growth conditions refer to standard GaAs growth conditions for approx $1\mu m$/hr. Different materials may require different conditions, most notably GaAsBi and GaNAs.

i. **As and As cracker:** This is a solid source, As sublimates as As_4 and is then thermally cracked to produce active As_2. It is held at 200°Cand ramped to 400°Cfor growth. The As cracker has to be maintained at a higher temperature than the As source. The controlling variac is maintained at 20 % (450°C) and ramped to 75 % (1000°C) for growth - if you monitor the output voltage then maintain at approx 11V and run at 48V. When you start ramping up

the As set the cracker to 50% and when it reaches 300°C set the cracker to 75%

NB: The As cracker thermocouple has been undergoing a slow failure over the past few years and the TC value is not to be trusted - for each vent/bake-out the value tends to decrease, this is not a very big problem but maybe it should be fixed someday.

NB: the actuator on the As valve is connected to the needle at the end of the As escape tube via a single loose set screw (lose so it does not rub against the inside of the tubulation), we recently had to fix the setscrew as it came lose - this is noticeable as you can no longer open/close As and the rod on the rear bellows of the set-up is very mobile.

ii. **Ga:** Gallium's melting temperature is 30°C, it is maintained at 400°Cbetween growths. It is ramped to around 950°Cduring growth. It is the rate limiting element in the growth, its temperature is varied to raise or lower the growth rate. ** Growth rate calibration info here **

iii. **In:** Indium's melting temperature is 160°C, it is maintained at 300°C. It is ramped to approx. 800°Cfor growth. The In cell was removed and replaced with a dual cell so that elemental Si could also be incorporated. On the power connector In is on the shorter (male) plug. The charge in this new In cell is very small and therefore the flux is also very low - in other words this cell is not usable for In.

iv. **Al:** Aluminum's melting point is 660°C, it is maintained at 700°C. It is ramped to about 1150°Cduring growth. Aluminum is typically only melted well into a campaign, in other words once we are sure no more show-stopping problems are likely to arise. If Al ever re-solidifies you/we have a problem; the crucible is now useless and if you re-melt aluminum the cell may be f#@k'd. The cell is very expensive so try not to let this happen.

v. **Bi:** Bismuth's melting point is 270°C, it is maintained at 300°C. It is ramped to around 600°Cfor use as a surfactant during growth and to about 425°Cfor growth of bismide samples. This temperature is varied to raise/lower the flux of Bi and increase/decrease the surfactant coverage.

vi. **N and plasma:** The rf N plasma source (fig.3) was designed by Martin Adamcyk (you can find a excellent write up about in his thesis) many years ago and it still works very well, its main feature is the ability to have it turn on as wanted (this not something that commercial set-up can do!). I've been

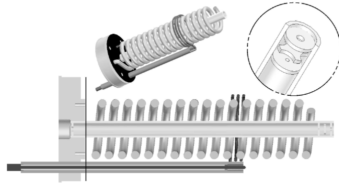


Figure 3: In-house designed and built rf plasma source for active nitrogen production. Also shown is a cut-away view and close up of the output baffle.

meaning to write up a mini-manual for this and may get around to it yet, but for the time being:

(1) flush the gas lines a minimum of three times, more if it has been some time since that previous use or some strange gas has been in the lines

(2) open the valve to the plasma source - you need to use a wrench. DO NOT torque on the set-up, you could cause a leak!

(3) slowly, very slowly begin to open the leak valve to N into the GC. Monitor both the GC pres and the parani gauge read out - the response is VERY slow to the GC! Typically you'll be working in the 10^{-6} Torr range.

(4) turn on the rf power supply and amplifier. Power supply to full, amp gain at about half.

(5) when you are ready to strike the plasma, hit the operate button on the amp. The plasma will likely not ignite right away - you need to fiddle with the rf frequency - you know it is lit when the reflected power abruptly changes. you'll want to check the window on the source to make sure it is lit - purple is cool!

(6) now tune the frequency to where you want - min reflected AND tune the amp gain for desired power forward.

(7) the plasma source will take a few minutes to settle so you will need to monitor the for/ref power for at least the first 5-10 minutes of use. We have been growing GaNAs QW's at the middling layer for GaAs growth rate calibration samples as In is no longer available

NB: after reach bake you should test the plasma to see if it fires (and re-fires), this may not happen easily. If not, set the gain to just above half and the frequency to about 162 MHz, the ref power should be high

be a local minimum here, now tune the tap position until the ref power is quite low. Try to fire the plasma again, it should work this time, try a little higher gain if not.

vii. **Dopant sources:**

(1) Si - Currently, spring 2008, the Si is loaded in the 4cc crucible of the e-science dual cell (In is loaded in the 6cc crucible). This cell was placed in the port previous used solely for In. The melting point of Si is 1410°C, i.e. it sublimes. The cell is maintained at 300°Cand ramped to approx 1100°Cduring growth. The exact temperature required will depend on growth rate and desired doping. Also note that this cell has its own shutter which needs to be opened to either Si (fully cw) or In (fully ccw) for it to work properly.

(2) CBr_4: This is only a brief over view of the operation of the source. You NEED to read through the manual, maybe twice, if you plan to use it. Currently the set-up has 3 pumps: its own ion backed by a small turbo backed by a scroll pump. All exhausted to the building exhaust. This scroll pump also backs the gas lines, if more scroll pumps become available it may be a good idea for the CBr_4 system to have it's own rough pump.

i. Works in 5 modes which are described below; see fig.vii with table. It is impossible to move between modes without passing through each intermediate mode; the modes must be accessed sequentially.

Off: Safe and prepared for downtime. Can let N2 in through valve b.

⋆ Make sure valve b is closed before going to PD mode!

PullDown: Both paths to pumps open and CBr_4 into delivery line.

Pump: Only valve **h** open for cleanliness. No gas available.

Idle: CBr_4 into pressure control circuit

Run: Stable flow of CBr_4 into growth chamber

ii. Vapour pressure of CBr_4 is 3 Torr at RT- lines at or below 1 atm at all times.

iii. CBr_4 is super nasty. Be careful. NEEDS to be exhausted properly!

iv. Never have have valves a/c and b open at the same time to avoid contaminating the CBr_4 bottle.

v. It is recommended that valve a be closed whenever the system is not in use!

Temp	I	Pwr
2240	10.00	423
1850	7.00	184

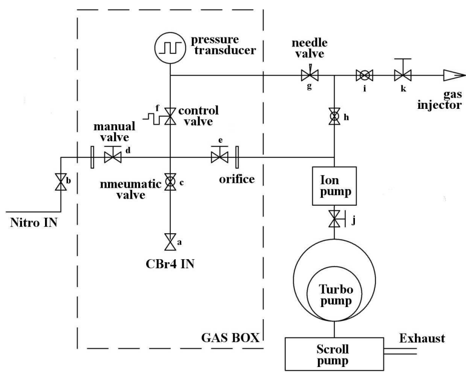
Table 1: Two points from the calibration curve forthe H cracker equipped on the MBE, a more complete calibration can be found in its manual

vi. the leak valve (g) controls the upper and lower limits of the dynamic range of pressures available to the system. Will need to adjust ONCE during testing of system.

(3) $SiBr_4$: In the summer of 2007, we added a second gas cylinder to the set-up for $SiBR_4$ (vapour pressure is 5 Torr at room temp!!!). To do this, a T-joint was used and either gas may be used depending on which cylinder valve is opened.

NB: Be careful not to contaminate one gas with the other, in other words full evacuate the system after one gas is closed off and before the other is opened (this takes at least 10 minutes)!

(4) H cracker: The same flange as is used for the CBr4 source also is used for the H cracker - in fact this piece of equipment is a H cracker with optional other inlet. The H comes from the same manifold in the rear of the system as does the N - be careful not to contaminant the tubes with the wrong gas! There is currently no power supply for the H cracker alone, we've used the same power supply as ANGLO. If you can find the manual - should be with the CBr4 manuals - there is a calibration curve for I v. Temp, Table 1 has some sample numbers.



Valve	Off	PD	Pump	Idle	Run
a	x	√	√	√	√
c	x	x	x	√	x
e?	x	√	x	x	x
h	x	√	√	√	x
i	x	x	x	x	√

Schematic of the CBr_4 gasline system. The table shows the function of particular valves under the various operation modes: √ is open, x is closed. All unlisted valve do not pertain to the normal operation of the CBr_4 source.

C: Other internal components

What equipment actually falls under the heading 'other internal components' is debatable, however what is meant here is the list of items not covered in other sections that if they fail (catastrophically) you may have to repair them in order to continue the growth campaign. These items are used for preparing and moving samples, as well as controlling growth conditions at substrate among other things. There is one other 'internal' item not found here, the RHEED gun/screen, this can be found in analysis section.

i. **Wobble sticks** We have two types of wobble sticks currently on the MBE: old school bellows style and modern magnetic actuated. We have one spare magnetic one for WHEN the bellows leak - and they will eventually. The wobble sticks are used to move the sample holders: from the LL to the HTHS, from the HTHS to the GC, from the GC to the LL. The 'hooked' end mates with a hole in the sample holders, depending on the size of the hole the sample holder will be more or less stable on the wobble stick; too large and the sample holder is floppy, to tight and the sample holder is difficult to get off the wobble stick.

ii. **Trolley** This is a little train track and cart to wheel the sample holders around on. Whee! The one thing you have to be careful about is that the cart CAN NOT be in front of the loadlock when it is opening/closing; also be careful not to close the gate valve on it.

iii. **HTHS** High Temperature Heat Stage. For our gassing samples after they have been exposed air and before they go to the growth chamber. This is

now (spring 2008) set-up right of the LL. We typically bake samples out at 300°C(approx. 26%) for an hour before a growth, in an ideal world you would do this ahead of time. Previously an oven style HTHS was located just right of the GC gate valve, but it had a leak in its water lines - it is yet to be fixed. When it is fixed, it should be re-installed.

iv. **Manipulator** The manipulator holds the sample holders in the GC, it can be rotated from the load position (straight down) to the growth position (towards the cells) or away from the sources (measure flux position). The positions are indicated (poorly) on the manual control at the rear of the GC on the manipulator flange. Once samples are loaded you must close the door on the manipulator with the wobble stick - try not to damage it as you do this, the door is your friend and a HUGE pain in the a** to take out and fix.

    The manipulator also has a motor which is used to rotate the sample during growth, it is controlled by a power supply usually found right in front of the MBE. The motor is attached by set screws next to the manual position control must be removed before baking. **NB:** This motor died in 2008 and has been replaced by a stepper motor run by labview.

v. **Substrate heater** The heater in the manipulator is used to warm substrates currently (spring 2008) it is a ceramic plate with imbedded heating wire - we no longer use Ta foils here. As the power required is too much for the one power supply we have two are connecting in series, one is set to 10 V as it uses higher current and the other is then used to change the temperature, typically 20 V to 56 V. At one time we used a feedback system to maintain substrate temperature, but it was found that constant power was just as affective and simpler to use.

vi. **Shutters** Each cell is equipped with a shutter to prevent substrate exposure until you want it and they usually open and close as you need - listen for the noise. We have two kinds: solenoid and pneumatic actuated. The shutter controls can be found on the front rack and have three settings: open, shutter is open; close, shutter is closed; computer, shutter does as computer tells it to. It is recommended that the As valve be left open, all others set in the closed position until your are growing, at which point set them to computer.

vii. **RGA** - We have a SRS 200 residual gas analyser (RGA) located at one corner of the GC towards the ion pump. It is controlled by software readily available on the internet. It usually works, but may take some time to get going - typically leaving the filament on for an hour or more seems to work. A typical

scan is done at a speed of 2 from a.m.u =1 to 100, anything higher we don't really care about; the RGA is also used in leak check mode monitoring He gas in the system. Do not run the electron multiplier if the the pressure is higher than 2×10^{-7} range.

viii. **Flux gauge** This is a constant source of frustration, currently we have a Frankenstein installed - a VG flux gauge set-up with a (modified) Varian ion gauge. It does not like to start up easily; what I have found to work well, so far, is to start with a VG controller (model no. 844, we have two and one of them works and the other might as well. Also these controllers only allow one decimal place). After the gauge comes on and has been on for some time (over night, especially after a bake) then you can switch to the MG in the rack channel 2, this gives two decimal places and might even be a correct number. The board in this controller is now fried- thanks Fluxie- so we use the SenTorr, with only one decimal place. Good luck and if you are really lucky the wiring of the socket is still indicated on the set-up - if not it is something like this: starting from gap and going CCW x x F1 FC F2 G G, collector up the middle.

    NB: Only connect one filament at a time. We have had problems with the controllers blowing out one filament a then switching to the other only to blow it as well! So, be careful.

ix. **Shroud** This is the only thermal shielding inside the GC. Whenever the cells are heated above 700°Cthe shroud must be cooled. To cool the shroud turn on the phase separator (page 17) and wait about one hour.

x. **Cryo Trap** to further lower the pressure of the GC we also fill a cryo-trap located at the very top of the GC through a tube from the shroud in-take lines. This takes about five minutes and you must watch for spilling! There is also a cryo-trap in the prep chamber on a flange sort of below the LL - if you are in a hurry and want to open the LL before waiting the full 2 hours after the LL was open, then you should cool this down as well to minimise the exposure - this is NOT recommended, do not hurry it is bad, go drink a coffee.

xi. **Bakable windows** We have three windows that may be heated to remove As(?) from the window surface. They are used for LS in, LS out and one to peak through at the sample during growth, align the DRS, check sample orientation, etc.. Their controllers are on the front most rack near the growth flange, Typically we set the windows to 225°C during growth to keep them

clean, if they become quite dirty you'll need to heat them to 300°C to remove the coating; this takes a few hours to do a good job.

D: External components

This section is meant to cover all things associated with the MBE but not in the MBE or not sufficiently vital as to halt the campaign if/when they break.

i. **Phase separator:** The phase separator is a very fancy bucket that only allows liquid nitrogen in to the MBE shroud. The control unit is at the top of one of the front racks, the unit can simply be either on or off. There are 7 indicator lights, which are all self explanatory: filling, min. level low, high, overflow, alarm. The alarm indicate that the pressure in the insulated lines is too high, it should be less than 10 microns of Hg (as indicatedon the analog scale on the controller).

   NB: currently we have tricked the phase separator into believing that the pressure is low by connecting it to a broken gauge, further we now have to check another indicator to know if the pressure is low enough for the phase separator to run well - running not well means wasted LN2, a small deluge in a lab on the first floor and at least one angry email sent to Jim (we have been threatened with a cutting of the input line, this would be a disaster and also hilarious).

   NB: we have installed a heater and blower to remove the blow off form the seperator, it is operated by a switch next to the phase seperator control box. I t should be turned when the seperator is and turned off 30 minutes after the seperator is turned off.

ii. **Source temperature controllers:** You hopefully will be able to find the manual for these somewhere in the vicinity of the controllers themselves, however the manual is not very helpful. Basically, the controllers take their cues from the computers as far as set points are concerned and then regulate the amount of power the power supplies should supply to the cells. They do this based on the PiD settings, which are likely not very good. You can find full tuning procedures on the Omega website - it takes about 2.5 hrs to tune a controller.

   All you really want to know about these things is that they work, if they are not working it is one of a few things:

   - controller set to manual - this will automatically change back if it receives a command form the computer so be careful

- the thermocouple has a short in it - check the connector, this is where most of the problems lie. this will have the cell well overheated because the temperature being read is very incorrect
- for some reason the power supplies have tripped off, this will be obvious because the temp controllers will be trying to output max power and the cells will not be heating - make sure to set each power to ZERO before switching back on the power
- you did something silly in the growth program - how each controller is labelled may not be correct, for instance right now to heat up silicon I tell the computer to heat up indium (if I by accident tell the computer to heat up silicon it will heat up gallium 1 which is thankfully connected to nothing - this may actually be fixed now (Oct 2008))
- the PiD settings are not correct for cell it is controlling - good luck (maybe I'll put a brief procedure here, but likely not)

iii. **Growth program:** Recipes for growth are entered into the computer using the commands listed below in Table 2, with the format: *command* [source] [temperature] [time], *i.e.* ramp al 800 60:00, this will ramp aluminum to 800°Cin one hour. Temperature is entered in degrees Celsius and time is entered in minutes:seconds (or hours:minutes:seconds). The cursor must be returned to the next line before the program recognises it. Commands can be checked for syntax by the growth program.

set	make a source this temperature
ramp	increase/decrease the source to this temperature in this time
open	open source's shutter
close	close source's shutter
label	works with loop command to allow FOR loops to be entered into the recipe. This command names section to be repeated, commands entered between the label and loop command are repeated.
loop	sets number of times section is repeated: loop *name* times

Table 2: Commands for the labview MBE growth program, see table 3 for usage of above commands

iv. **DRS:** - Me or Mike to fill in

```
ramp as 400 45:00
ramp ga 950 60:00
ramp in 800 60:00
 wait 60:00
open as
wait 15:00
open ga
 wait 30:00
label QWx10
open in
wait 0:15
close in
wait 1:12
loop QWx10 10
 wait 30:00
close ga
close as

ramp as 200 45:00
ramp ga 400 60:00
ramp in 300 60:00
```

Table 3: An example growth program using most commands. The indented wait times not necessary to include, but are used here as a reminder as to when the subsequent commands can/should be executed. Note that the substrate temperature is not controlled by the program, you will need to monitor and adjust it yourself

v. **Plumbing:** All water to the MBE comes from currently the outside world and passes through two filters and onto the manifold the directs flow to the varios components needing water cooling: pumps, cells, etc. The garden hoses leading from the manifold should have indications scibblied on them as to the final destination. The line to the cells runs to another manifold which directs flow to each of the cells (except the Si/In cell) and the H_2 cracker - the swagelok pieces at this point are nasty (rust and abuse) and should be fixed up soon- maybe when we move! The water returning from the cells passes through the other half of the cells manifold on its way to a drain manifold where it rejoins the flow from the pumps to eventually be forced down the drain in the wetbench. The Si/In cell is on its own line and drain into the floor next to the MBE.

   the air 'plumbing' is a similar set-up: a manifold with many lines to and fro from it. Things that need/want air: the gaslines gate valve, the LL gatevalve, the CBr_4 source, the Bi (and Ga??) shutter and the new MBE.

vi. **ANGLO, the bake out chamber:** So named by former francophone students because of the large blockhead appearance of the chamber, it is used to bake out cells before they are loaded in to the MBE. It has been somewhat recently 'fixed-up' (by Beaton), that is there now are sufficient plumbing lines, air lines ion pump controllers (1) to operate the system. The turbo cart can used to pump the chamber to the point whence the ion pump can be turned on.

   NB: the ion pump is old but works, you just need to have faith that it will eventually settle down and not overheat. You may need to make to or three tries at this, monitoring the pressure is helpful.

Part II : Growth Procedure

1. Growing Samples

The following is a basic procedural outline for a growth using the old MBE system. It is based on a similar document written by E.C. Young and added based on my, D. Beaton, experience. It assumes a basic understanding of the components described above. Also the following does not describe in depth the recipe(s) for particular structures, but rather simply outlines the operation of the system for, during and after a growth.

1.1: Loading/Unloading Wafers

1. BEFORE you do anything to the MBE please have everything else ready, i.e. samples cleaved, sample boxes ready and labelled, etc - this will minimise the time the system is opened. Also make sure the air filtering set-up above the load lock is on

2. FIRST close the gate valve to the load lock - the switch is located on the (green) power box near the front on the rear side of the MBE

3. NEXT turn off ion gauge and THEN turn off turbo pump on rear rack.

4. WAIT a few minutes and then bleed N into load lock - you will hear the cap pop open.

5. NOW take-out and/or load wafers.

6. AFTER the turbo pump has spun down, turn off the scroll pump and re-open the gate valve. This is to prolong its lifetime

7. ONCE the sample case is back in the MBE, close off N to load lock. Now turn the scroll pump back on

8. WAIT until load lock pressure reaches 2×10^{-2}, the gauge is on first stack right hand side of indicator. THEN turn on the turbo pump

9. WAIT about 30 minutes and turn on ion gauge.

10. WAIT at least 2 hours before opening load lock to the prep chamber!!

1.2: Start up

1. Turn on the phase separator and let it run for approximately 1 hour. This will fill it with liquid nitrogen (LN2) and at the same time automatically fill most of the cryo-shroud on the MBE growth chamber.

   (i) Eventually the reservoir of Ti sublimation pump will need to be topped up with LN2 through the large flange at the top of the growth chamber. This takes approximately 5 minutes and **must be monitored**. If LN2 happens to overflow onto the flange it cause significant thermal gradients across the flange - this can cause a leak!

2. While the phase separator fills up, outgas the substrate in the high temperature heating stage (HTHS) for approximately one hour. This should be done in a two stage process to prevent the prep chamber pressure from rising too high. Most of the outgassing occurs between 100°Cand 200°C(12% power) as water is baked off, the pressure will rise dramatically during this time. After the pressure has stabilised to the low 10^{-8} range, increase the power to $15 - 20\%$, a max temperature of approximately 460°Cshould be reached. Wait until the prep chamber pressure has once again dropped below 2×10^{-8} before turning off the power.

3. **Once the growth chamber pressure in the growth chamber has dropped to about** 2×10^{-9} you can start ramping the sources you will require for growth. As will not reach 400°Cfor approximately 30 minutes, so there is no need to ramp the other sources (Ga, In, etc.) to their growth temperatures any faster than 20 or 30 minutes. Typically sources are ramped to growth temperatures in 60 minutes. Some sources will need significant time to stabilise their flux (i.e. Al needs 30 minutes). It is also necessary to ramp the As cracker up to 50% on the variac power supply shortly after the As temperature begins to rise and then to the final value of 74% when the cell reaches about 300°C.

   (i) The As valve should be opened *slightly* while the sources are ramping, this is to prolong its lifetime; it has been catching. Make sure the shutter is closed. It is believed the heating/cooling of the valve is the problem, at stable temperatures the valve should be able to move more freely. **When loading a sample into the growth chamber close the As valve**.

   NB: this has become less critical in the past years - I think we may have recently taken the entire As cell assembly out and clean it. If the valve

does once again begin to ctach,then the above note should be returned to.

4. The outgassed sample can be loaded into the growth chamber while the sources are ramping, **if and only if** the pressure in the prep chamber is **less than** 2×10^{-8} Torr. After you have loaded the sample and closed the manipulator door don't forget to retract the trolley back into the prep chamber and shut the growth chamber gate valve. If it is taking a long time time to get the sample to sit properly (> 1 min.) in the manipulator, retract the trolley and close the gate valve before continuing. This is to keep nasty As out of the prep chamber. Remember that the load lock gate valve and the growth chamber gate valves should never open at the same time.

   (i) Manoeuvring the sample exactly into place takes some practice. Do not drop the sample holder or you will be banished from the MBE forever!

5. Rotate the manipulator into growth position and start to heat to the substrate. There are two power supplies for the substrate heater connected in series. Adjust the upper power supply to 10 V and the lower to 20 V for a total of 30 V; the upper power supply should be maintained at 10V and the lower power supply used to adjust the power (the latter works at lower currents). This should be sufficient power to ramp the substrate to approximately 200°C, this will take awhile.

   (i) The total amount of power that needs to be sent to the substrate heater to reach 600°Cfor the thermal oxide removal depends on the sample holder! More recently cleaned holders will need less power. This also holds for all other power supply voltages and corresponding substrate temperatures quoted below.

6. While waiting for the sources/sample to heat up:

   (i) Begin to set up for optical temperature measurement, see note on setting up the DRS on page 18; You should by this time be able to obtain a temperature measurement in the labview software.

   (ii) The RHEED gun can also be turned on at this time. Follow the instructions on the power supply for the ramping up of the voltage and current - voltage first then the current, both done slowly! If you look at the substrate with the RHEED gun at this point you should see a fuzzy image

with no features (except the specular reflection) due to the amorphous nature of the native oxide.

7. Before increasing the substrate heating power further open the As shutter and open the As valve fully to prevent As desorption from the surface. It is now safe to increase the substrate temperature for oxide removal.

   (i) Once the substrate temperature reaches 580°Cthe native oxide on the surface of the GaAs wafer will begin to desorb. To ensure all the oxide is removed, make sure the substrate temp reaches above 600°Cand stays there for at least 10 minutes. You can check the progression of the oxide desorption by looking at the RHEED and rotating the substrate. The RHEED pattern should be spotty due to the 3-dimensional surface features (pits!) that are an artifact of the desorption process.

1.3: Growth

1. some notes here on the operation of other components (DRS, RHEED, etc) during/for the growth stage

2. Ramp the substrate down to approximately $550 - 580$°Cand begin growing the buffer layer by opening the Ga shutter. The RHEED should quickly become **streaky** as the surface smooths and it should show the usual 4×2 reconstruction when the sample is rotated - this is observed as a *tripling* of the streaky lines. A good practice is to grow the buffer for 15 minutes or longer (≥ 150 nm), buffer growth times of 30 minutes are typical.

3. a few more notes here on things to watch for and monitor etc

4. During growth the sample may be rotated, periods of 5 s are typical - depends on what/why you are growing. This is not necessary during growth of the buffer layer, but is recommended.

1.4: Shut down

1. **Immediately** ramp down the substrate temperature, close all shutters and the As valve, return the manipulator to the load position.

2. Let the pressure in the growth chamber drop to 2×10^{-8} before opening the gate valve to remove the sample. Don't drop it!

3. If your are done for the day ramp down the cells; As needs at least one hour to cool and the cracker should be cooled slower than the cell.

4. At the end of the day, make sure all sources are at their rest temperatures, turn off the phase separator and empty the remains of the LN2 into the small blue dewar, turn off all of the bandgap thermometer electronics and the RHEED gun - **ramp down as indicated on hardware**. It is also a good idea to set all the shutters to the closed rather than the computer controlled setting.

1.5: Epi-layer considerations

There are several things you will need to keep in mind when everyou a re growing an epi-layer - hopefully, as you become more familiar with growing samples these items should become second nature to you. Also please note that the more novel samples (new materials and heterostructure) will require more consideration than your more routine films.

Starting from standard GaAs growth conditions, which are rather less specific than some other materials, we will first need to consider what range of parameters are available to us, what changes we need to make in order to grow the desired film and how quickly we can reach the new conditions. You may also need to keep in mind whether or not this may effect previously grown layers. Typically all films start at a substrate temperature of 580°Cjust after the oxide has been removed by opening the Ga shutter, where the Ga temperature is set such that the growth rate is approx. 1 μm/hr (currently 950°C, fall 2008). This is followed by at least 15 minutes of simple GaAs buffer growth, this is to smooth the substrate after after the violent removal of oxide (rms roughness 5 nm $\rightarrow$ 0.3 nm). At this point growths conditions can be modified to grow other films, GaAs grows very well up to 650°C, down to 300°Cover a wide range of growth rates 0.1 $\rightarrow$ 2 μ/hr and III:V ratios (1 $\rightarrow$ 10+).

Growth rate This entirely controlled for the arrival of group III atom (Ga, Al, In) as we grow under group V rich conditions (mostly). In the interest of full disclosure we set the flux of Ga by the desired growth rate - approx. 1 μ/hr for standard GaAs growth. For bismide films it is necessary to slow the growth rate down to $\frac{1}{10}$ of this - though the people at NREL have had success at 2 μ/hr.

As overpressure As stated above we work under conditions of high As flux/overpressure, typical values of the III:V ratio range from 2 to 10. Stoichiometric levels of As

overpressure can be observed as a change in the RHEED pattern -2×4 going to 1×1 - this is general considered as a combination of 2×4 and 4×2 (Adamcyk thesis). Again for the growth of bismide films this is radically different than standard and near stoichiometric levels are used.

Alloy composition This can be more or less well approximated by comparing the fluxes of the two (or more) elements - this assumes sticking coefficients of 1 for each of the elemnts and also that there exist no competition between them (definitely true of group III's, they are the rate limiting element). But lets work it out: I want 30% Al in an AlGaAs film, so I want the flux of Al to be $\frac{1}{3}$ that of Ga; (using old as number b/c we haven't melted Al in some time)at T$_{Ga}$ = 950°Cwe have a flux of 1.8×10^{-7}, so the required Al flux if 0.6×10^{-7} and the corresponding temperature is about 1170°C. Do remember that the addition of 30% more group III elements will increase the growth rate an equivalent amount.

[Doping concentration] This could be done as above except the fluxes involved with the dopant sources is typically to low to measure at useful temperatures. So what do we do... we work out how many atoms reach the film surface based on these formulae:

$$\phi = \frac{3.51 \times 10^{22} P}{\sqrt{mT}} \tag{1a}$$

$$flux = \phi A \tag{1b}$$

where P flux in Torr (BEP), m is the atomic mass, T is the source temperature in Kelvin and A is the substrate area in cm^{-2}. ϕ is a measure of the impingement of atoms at the surface and has units of cm^{-2} s^{-1}. The final doping concentration will be given by $\frac{flux_A}{flux_B}$×density of B atoms

1.6: Sample recording

It is of the utmost importance that you maintain excellent records of your growths, both for yourself and others in the lab, as well as those who will come after you. We have traditionally done this with paper - you will find somewhere in the lab copies of almost every samples grown in our reactor overthe past 12+ years. Also, each grower has kept his/her own records of their own growths, typically complete with results from measurements thereof, in a spreadsheet file (I also recommend keeping your own paper copies in a separate folder). Recently I have created a web based approach, a database, for to keep track of my samples. You are required to keep papers copies and it is recommended that you also have a secondary record for your

own purposes This database is far easier to search through. Below you will find a short description of both record keeping methods.

Paper

The paper version of the growth log - a sort of fill in the blanks document (/mbelab/growth_sheet.pdf + .gri file) should be found somewhere near the mbe computer. It includes blanks for run #, date, grower, substrate info, epi-layer(s) info, and notes. **You should be filling this in as you grow**, more extensive notes will always prove useful. Completed forms are to be put into the growth log binder, and again I suggest keeping your own paper copy.

DataBase

The database is more or less a computerised version of the paper form - though it also for easy, quick searching of the samples. You'll need a password to access it and I'll have to remember how to give you one. The URL is:

https://turing.phas.ubc.ca/ dbeaton/database/input_1a.html
OR *update.html* OR *query.html* OR *analy.html*

There is also a help page (I think) that you can link to from these sites. The files are kept in my turing public directory and should eventually be moved to the server or mbelab.

It should be (hopefully) easy to use. I will show you or someone will if I have shown them.

Part III : Analysis tools

1. RHEED

The RHEED set-up is used to probe the sample in-situ. The start up procedure is written on the power supply, be careful to follow these instructions (they are re-iterated below in the growth procedure). The electron gun is on one side of the growth flange and a phosphorescent window is on the other side. They are positioned such that the reflected beam pattern appears in the window. This window has a shutter which is used to block the electron beam when you are not interested in seeing the beam pattern. The direction of the beam can be moved using the controls attached to the power supply. Always have beam blanking off when you are viewing the pattern and try to only use the x or y positioning controls to move the beam around - unless you understand what you are doing (i.e have read the manual) DON'T touch anything except X and Y. *You don't want to look in the MBE windows while the RHEED gun is on without beam blanking, unless you would like some stray X-rays in your eyes.*

NB: to view the straight through beam use : X=3.7, Y=5.8

NB: 1.7 amps and above GREATLY REDUCED FILAMENT LIFETIME, according to manual. Never exceed 1.5. You may increase the brightness by slightly increasing the filament current, then ALWAYS monitor the emission current.

NB: Baking the RHEED gun: Look over baking in the RHEED manual first. Remove the can covering the ceramic HV feedthroughs and wrap with several layers of Al foil. Fix a thermocouple to the gun and wrap the whole instrument with some layers of foil. Wrap with heating tape (2 seem to work OK) way so that the heat will be evenly distributed. Cover with several more layers of foil. Heat to 180-220C for a day or 2. DO NOT EXCEED 250C as this will damage the deflection coils. Do not exceed a heating rate of 60C/hour.

NB: We have currently been trying to make our own RHEED screens rather than sending them away for re-coated ($600 for one, $200 for each subsequent one). We have not been successful (yet???), it is likely a huge waste of time and really, unless you are doing a RHEED project the RHEED is only a nicety to let you know that the oxide is off (bright points) and grow has started (gets streaky).

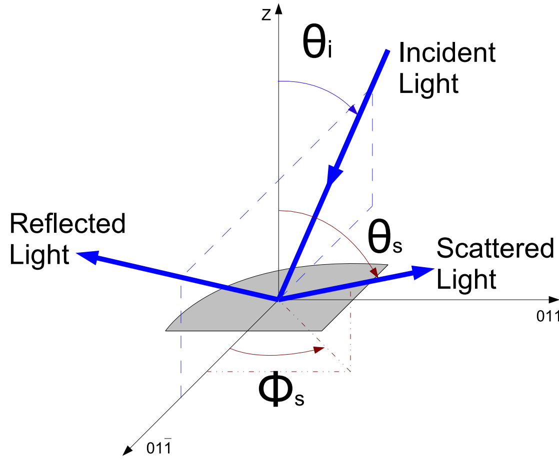


Figure 4: Configuration of the incident, reflected and scattered beams with respect to the surface normal of the substrate in the light scattering setup.

2. Light scattering

The fact that the light scattering apparatus is able to observe changes on the substrate accurately is an amazing technical feat. The substrate is nearly atomically flat, even after the oxide is removed and will scatter marginal light in directions other than specular. Elements in the MBE such as the nearby substrate holder or the in-chamber optics are rough and will scatter light isotropically. The scattered light from the growth chamber has a signal orders of magnitude greater then light scattered from the substrate. Only carefully alignment of optical beam paths to reduce these effect allows the light scattering apparatus to work. What you are measuring physically is the PSD at an angle depend spatial frequency. Consider the geometric arrangement of the incident optical beam, the reflected optical beam, a beam of scattered non-specular light and the substrate as presented in Figure 4, then the light will scatter as:

$$\frac{dP}{P_o d\Omega} = \frac{16\pi^2}{\lambda^4} \cos\theta_i \cos^2\theta_s Q(\theta_i, \theta_s, \phi_s) PSD_{2D}(\vec{q}) \tag{2}$$

Where dP is the fractional power measured in solid angle $d\Omega$, and P_o is the initial power of the light incident to the surface, λ is the optical wavelength and θ_i; θ_s and ϕ_s are the angles of the incident beam and detected scattered light with respect to the surface normal. $PSD_{2D}(\vec{q})$ is the power spectral density at the spatial frequency

λ (nm)	θ_i (deg)	θ_s (deg)	ϕ_s (deg)	$\lvert\vec{q}\rvert$ (μm^{-1})
632.8	55	-25	0	12.3
514.5	55	-25	0	15.2
457.9	55	-25	0	17.0
244.0	55	-25	0	32.0
244.0	55	-65	22.5	43.5

$\vec{q}$. Note that if P_o could be accurately measured, the PSD could be measured absolutely, but practically P_o is impractical to measure inside the MBE. For light scattered off a rough surface the spatial frequency represents the momentum transfer that must occur on the surface, thus $\vec{q}$ is determined by the angles and wavelength (see Whitwick PhD thesis).

The light scattering apparatus is limited by the positions of the available optical ports. The angles of optical ports and the wavelength used determine this spatial frequency which be calculated from Equation **??**. The table below gives a summary of the MBE growth chamber ports and the resulting spatial frequencies.

Three separate light sources are available. A beam of coherent UV light of wavelength 244.0 nm. The UV light was generated by frequency doubling 488.0 nm light from a 2 W Spectra Physics Argon Ion which contained a Beamlok Etalon for increased stability. The frequency doubler was a Spectra Wavetrian equipped with a LBO non-linear optical crystal. This setup is capable of generating $15 - 100$ mW of UV light; typically 40 mW was used. The optical beam was coupled to a 10 m UV transmitting fibre optic cable, losses for UV light transmitted are large (0.35 dB/m) and this light source has only 2 mW incident to the GaAs sample. The second light source used was a tunable Ar ion laser with optics for the laser lines of: 457.9 nm, 465.8 nm, 476.5 nm, 488.0 nm, 496.5 nm, 501.7 nm and 514.5 nm; the 457.9 nm laser line was most often used. For visible light the absorption in the fibre cable is less and the power incident to the sample is greater, typically ~ 30 mW. A third possible light source is a He-Ne laser (632.8 nm) which can be mounted directly onto a MBE optical viewport.

The incident beam is put into the growth chamber through a converted non-optical shutter port. A mirror is mounted within the shutter port to direct the port's line of sight to the substrate. The detector assembly is mounted on source port. The specular beam was directed out of the growth chamber through another converted shutter port and blocked to reduce the overall background intensity.

Light Scattering Alignment

- Put semi-reflecting sample into growth chamber at the load position; GaAs with Ga or Bi droplets works well. You need to see the reflected beam and the spot on the sample
- By eye, align sample to the [110] or the $[1\bar{1}0]$ directions, in the optical viewports there are objects 0° or 90° to the LS optical plane.
- Move the position and direction of the fibre collimating lens until the beam in on the sample. This is a two person job. The converted shutter ports have a narrow window of acceptance.
- Move the position and direction of the fibre collimating lens until the reflected beam leaves the MBE chamber while keeping the beam spot on the sample. Rotating the sample helps.
- Fine tune the position of the beam on the sample until the beam is centred, while checking that the beam leaves the chamber. Rotate the sample.
- Place the input iris apperature in the beam path such that it blocks the extra blue line that surrounds the laser beam, also mount the optical chopper and detector assemble with the photomultiplier tube on the MBE.
- In the detector assembly find the optical plane from the focusing lens, place the iris apperature in this plane and centre in on the bright beam spot.
- Check that the detector has the proper line filter.
- Mount the photomultiplier tube and power at $0.4 - 1.1$ kV.
- Connect the chopper/lock-in amplifer.
- Move the detector until till maximum signal is seen. You will need to lower photomultiplier tube voltage and attenuate the optical beam to do this. Mark this position well as you will need remove and then reattach the photomultiplier tube later.

Light Scattering Measurement

- Check beam blocks on the MBE, i.e. at the input and reflected beam ports

- Activate laser, the Ar^{+} laser take 30 minutes to warm up and become stable. Very important for the Frequency doubler. Block the beam until it is stable
- By eye, align sample to the [110] or the [$1\bar{1}0$] directions in the optical viewports there are object 0° or 90° to the LS optical plane. May need to remove the detector for this. (Or with the RHEED and stepper align to RHEED and rotate sample by 22.5^o (I think)). Note that there is a wobble to the sample holder in the MBE so you will want to align the sample first.
- Loose laser beam into chamber, check that the beam spot is not on the sample holder and that the reflected beam is leaving the MBE chamber. If not, adjust the sample holder tilt until desire result is reached. If if not try rotating the sample 180°. If if if not, you realign the light scattering apparatus.
- re-Attach detector assemble
- In the detector assemble open the iris apperature and place a round thin piece of paper on the iris apperature. You should see a image of the sample, with the sample being a shadow in the middle. You may need to turn off the lights and activate the DRS lamp to see a good image. Centre the iris apperature by adjusting how the detector assemble sits on the MBE. Close the iris apperature until you can see no light, then close it 30% more.
- Place photomultiplier tube on detector assemble in prealigned position
- Power photomultiplier tube with $0.7 - 1.1$ kV. The higher the voltage the better, but may overload the detector.
- Activate optical chopper and measure light scattering signal with lock-in amplifer.
- Align laser to optical fibre to optimise power to sample (optional).

3. Photoluminescence

Currently there are two set-ups for PL - one designed and built by R. Lewis for use with the cryostat for both PL and EL measurements and another built by Xainfeng Lu. The first- superior both in use and signal strength - now has a prism added to allow for EL (see below) and PL measurements to be taken on the same cold sample in the cryostat. I will not go into the details about the design here because it is not necessary and hopefully you will not have to concern yourself with it, but below you will read about how to use the set-up.

DO NOT take this set-up apart, DO NOT adjust the components thereof.

The set up was designed to work in coincidence with the cryostat and as such has the sample lens focal length set accordingly (approx 6cm), it can also be used apart from the cryostat in a vertical arrangement. Placed betwen the two lenses is the prism which redirects the laser beam to the sample, the prism is unnecessary for use in the vertical arrangement. The fibre currently being used has a diameter of 500 μm and a numerical aperture of... something - anywho Ryan thought about all this and now I/you don't have to. To align the set up [roughly], simply remove the fibre and look at the sample (laser off!) or X - it will appear as a real image at the fibre connector. You need to trick your eye into focusing here. To align the set-up [finely] adjust the 3-axis stage slowly and carefully until a maximum is reached. I recommend using a reference sample for all PL measurements (r1477), this allows for consistency and comparison of samples over time.

PL measurements in the lab are typically performed with the 523 nm green diode pumped frequency doubled YLF laser, and unfortunately often at personal settings of input power (500 mW) and rep. rate (400 Hz). We also have a CW HeNe laser, and the larger Ar ion lasers laser. The oxide people most often use the 6 W diode 808 nm diode laser and their own set-up. EL measurements are performed much the same way, now the samples are electrically pumped not optical pumped - you'll need a working LED (or something better) for these measurements

Both set-ups use the same spectrometer and WinSpec32 software for data collection, both are more-or-less straight forward to use - cool the detector, set the integration time, collect a background, collect data, convert to ASCII, plot data, sit back and admire data. If the 'noise' appears, you'll need to saturate the detector (and restart the program?) to make it go away.

EL and LI

Electroluminescence (EL) is a similar measurement to PL except that in this case you are excited carriers electrically not optically and possibly measuring the ouptput intensity as a fuunction of pump current (LI). The optical cryostat has been wired for up to 8 contacts, i.e. 7 diodes with a common ground- even if you are not planning on cooling your EL/IL sample it is still easiest to load the sample onto the crostat sample holder. Again the optics set-up (same as for PL) is designed to work with the cryostat and alignment is as is described above.

4. XRD

The X-ray diffractometer is currently Ampel Rm. 443. This system is far simpler to use that the old Rigaku set-up, has a higher resolution and is altogether better in my opinion. Only its use will be covered here, for a complete guide to its use please refer to its operation manual. If you want information on operating the previous XRD system... tough, and it is in pieces in the AMPEL basement. The new system is highly automated and therefore quite easy to use, if not so easy to get the hang of. You simply need to load the sample properly (its position relative the beam is obviously very important) and run an analysis algorithm from the computer. There is also the option of running a manual scan. However for most scans there should exist pre-written batch programs (thank Mario and Nikolaj). These batch programs are made up several smaller, pre-written programs that ramp up, optimise scan parameters, perform the scan, save the data, and ramp down to idle.

1. The very first thing you'll need to do is book the machine.

2. Then you need to prepare and mount your sample in some way. I aleways remove the out edge of the grown on wafer to avoid the pin marks visible in the cover - material in this region is obviously poor. I then cleave a 7 mm wide piece from the remaining $\frac{1}{4}$-wafer (7 mm is what fits in to the finger mask for the e-beam). I use the piece which includes the edge of the wafer for XRD. I use either double sided tape or the sticky tabs fro the AFM to adhere my XRD sample to one-third of a glass slide (you can easily scratch and cleave the slides up for this purpose and this size fits back into the 1.5" sample holders when completed) with an appropriate label.

3. X'pert program: This is the program that keeps track of users (id: MBELAB, pw:mbelab) and their projects; currently the group has one project- bulk GaNAs. From here you open X'pert data collector using the icon on the task bar. This part of the software runs the XRD. Programs are opened and saved from here; currently there is no way to erase programs. We have simply been changing save file names and saving over old programs. The control also pops up, with this you can move the sample holder for loading, scanning, and unloading.

4. Sample loading: Once the table is flat (Psi=90°)) a sample can be loaded. Samples are placed on a 2" Si wafer held at the corners by scotch tape. Try not to cover to much of the sample when doing this as not to mess up too much of the sample. The Si wafer is then loaded on the XRD table and

held in place by three spring loaded clips - one of the clips should be located on the flat edge of the wafer for stability. And only one should be moved in and out when loading and unloading samples, this will make it easier to position ($\hat{x}$ and $\hat{y}$) the sample such that the center of the sample is in the center of the table. The $\hat{z}$ (in-out) position of the sample surface needs to be checked relative to reference: a caliper attachment is used for this purpose. The working parameters for the sample lateral position are indicated on the caliper. Be careful not to break the sample when doing this - the finger of the caliper is spring loaded and needs to be gently released onto the sample.

5. Currently no generally accepted nomenclatures used for the naming of algorithms, though program names tend to reflect their scan parameters in a somewhat convoluted manner. The typical program name goes as follows: material[peak] optimised parameters steptime beampath (only indicate lower) scantime, i.e. GaAs[004] 1deg Om/2th 2s/step 1h.

6. Data from the XRD needs to exported to one of the Windows^{TM} machines to be fitted. This can easy or very difficult depending on the sample. Data imported to the program is fitted using a guess of the growth parameters entered in the model table. The play/FF button runs the fitting program - hopefully it gets to something reasonable. If not you'll need to make another, better guess of the structure and/or parameters thereof.

    (i) We use a program called RADS Mercury to fit to our samples (requires .x00 files, need to convert X'pert file in viewer program) it takes a given model heterostructure and then adjusts parameters (thickness, Bi content, etc.) to fit the XRD data. It does a pretty good job, mostly. mostly we are concerned with the film content and thickness, these are measured by the distance from the [004] GaAs peak to the split-off peak and the spacing of the Pendellosung fringes respectively - if the program is not behaving very nicely or is having a hard time fitting your data, it is best to simply guess (based on growth log and your own common sense) and check the result.

5. Hall and Resistivity

Previously (2001-02) E. Strohm performed Hall and resistivity measurements on GaNAs samples using the large electromagent, where low temperature

(77 K) measurements were performed in the same way with the addition of LN@ at the sample (kept dry by creating a overpressure of nitrogen in the immediate vicinity). With the purchase the optical cryostat, the set-up has been modified to allow for samples to be used in the cryostat - this required using permanent magnets which are less convenient for several reasons, namely DC operation and an inability to perform $+\hat{z}$ and $-\hat{z}$ more-or-less simultaneously.

The current set-up has samples mounted on copper plates with GE varnished paper added to electrically insulate the sample and contact pads from the copper. Samples are typically 7 mm squares with small ohmic contacts at the corners. Samples are connected to the current source and voltmeter through the cryostat wiring box and Hall/res switiching box; both the current source and voltmemter are controlled by computer. The Hall/res switching box simplifies (less likely to make errors) the changing of the source (+I, -I) and measure (+V, -V) contacts. There is a very nice write up on the Hall/res procedure you can find at the NIST website under *Hall Effect Measurements*.

Ohmic contacts

In order to properly conduct measurements of the electronic properties of the samples you will need to (in most cases) have ohmic contacts - using the e-beam these are easily achieved (they much more easily achieved for higher doping concentrations), see table below. The n-type ohmic contacts need to be anneal at 410 °Cfor 2 minutes and the p-type contacts need to be anneal for 30 s at 450C.

doping	contact	thicknesses, nm
n-GaAs	Ni/AuGe/Au	25/100/150
p-GaAs	Ti/Pt/Au	50/100/200

Table 4: Ohmic contacts for doped GaAs. Higher doping will allow for 'more' ohmic (less resistive contacts). The top most layer of Au need only be sufficiently thick for wiring bonding. There are references for these in a paper the MBE08 proceedings paper by R.B. Lewis

In other cases you may require Schottky type contacts, Cr/Au (25/100 nm) seems to work well for both n- and p-type GaAs - contacts of this type that are large in size will effectively be ohmic.

IV

Seperate from the above measurements IV/resistivity measurement necessary for qauntifying the mobility, you may want/need to simply measure the IV of a sample in order to find its doping concentration. This is performed with a finger mask; 5 'fingers' of Cr/Au, no need for ohmic contacts in this case, are evaporated onto the sample and only 4 are necessary to perform the measurement. The outer most fingers (of your chosen set of four) will be used for current in/out (+I, -I) and the inner to for measuring the voltage drop across the space between two fingers (+V, -V). There exists a simple probing station that allows for quick easy measurements, equipped with Au-plated positionable probes.

Others/elsewhere

RBS, SIMS, ARPES, TEM, RTS, DLTS, PDS, PRS, Auger is a partial list of measurements we can and may have done on our samples in the past in collaboration with persons who have the expertise and equipment to do the measurements (some of these people are fairly close by in AMPEL, at UBC or SFU).

Part IV : You, your MBE and a PhD

So You're Going to do a Ph.D. or post -doc in our group... you have now tied your life to the MBE chamber, at least for the duration of your degree/posting. In what follows is a number of things you can do to help keep that time as short as possible, though you can expect that with even routine maintenance that your degree will run 12 months longer than most.

1. Maintaining your MBE: Daily

It is a VERY good idea to check on the MBE system each day when you arrive and once just before you leave. This will allow you to spot (see, hear, or smell) problems as soon as possible and keep the system up and running better, longer and smoother. Here is a brief list of things to have a peak at (the order is more or less as they can be found as you wander around the MBE):

> computer, GC pressure, Prep chamber pressure, Ion pumps, cryopump temperature, phase separator, LL foreline pressure, cell temperatures and power outputs, As cracker variac setting, samples loaded/positions, LL pressure, LL turbo, gaslines foreline pressure, gas lines pressure, gaslines turbo, water filters, CBr4 pressure and temperature, As valve.

Of these listed item it is most important to check the pressure(s), the cells and the water filters. The pressures should be as follows (or better):

Prep	2×10^{-10} Torr
GC	2×10^{-8} Torr
LL	1×10^{-8} Torr

If you discover the MBE not in this state IMMEDIATELY try to discover why the pressure is so high (if it is lower, either the gauge is busted - problem, or the MBE gods are smiling on you - not a problem). If you have been properly trained, you will know what are accepted temperatures for each of the cells (listed above in the source section), and furthermore once you are growing on a regular basis then you also be able to recognise if the temperature controllers are trying to drive too much power to a particular cell.

The cells, cryo-pump, etc are water cooled AND the **pressure in the water lines has to be monitored**, it should be **greater than** 30 **psi**. They tend to

clog up quickly, as the water in the building is terribly dirty. The filters are located behind the MBE. There are two filters set up in parallel this is to avoid having to shut the water off to the pump as one filter is being switched. Do try not to make a mess when switching them. We typically get our filters from physics stores, but they also be purchased, slightly cheaper, at filters.com who delivers them to AMPEL directly.

- **NB:** some day we will have finally set-up our closed circuit water cooling system and the water filter problem will cease to be.

2. Maintaining your MBE: Monthly

In order to continue to grow high quality films it is a good idea to regularly check certain parameters, i.e. growth rate and BEP's. For this reason I proposed the following schedule for the old MBE:

First Monday of every month :

Flux measurements of all frequently used sources: Ga, As, Bi, etc, These measurements consist of noting the GC pressure and flux gauge pressure with the shutter open, as well as the flux gauge pressure with the shutter closed (record the minimum value). The actual flux will be the difference between the latter two.

Every other first Tuesday of the month :

Growth rate calibration sample, grow a quantum well (InGaAs 6-10s, with In at 800 C, or GaNAs QW) with EXACTLY 1 hour or EXACTLY 30 minutes of GaAs on top. It is a good idea to calibrate all Ga temperatures normally in use; currently 950 C and 850 C, approximately 1 μm/hr and 1 nm/min respectively. The growth rate can more-or-less be constantly monitored when fringes are observed for the top most epi-layer.

Flux Measurement Procedure

In order to have consistent measurements of the BEP's of the cells the following procedure should be followed: however please note that until a non-Frankenstein gauge and set-up is incorporated consistent results will remain allusive and measurements from different dates may or may not give comparable values. Frequent measurements- more often than once per month- is the easiest method to maintain

a consistent understanding of the fluxes. Any time you can anticipate a vent, especially one where the flux gauge will be removed, it is a VERY good idea to do a full flux measurement of all active cells for comparison post-vent and bake.

1. The position of the flux gauge should remain constant - note the marks on the current set-up
2. Cells that are used at lower temperatures should be measured first, i.e. Bi then Ga, this will keep a lower background pressure longer. As should be measured last or separately.
3. For Knudsen cells, simply opening and closing the shutter will give a BG and flux measurement. The GC pressure should also be recorded. Remember to allow sufficient time for the cell the settle on a given temperature before doing a flux measurement.
4. more detailed measurements should be made in areas of interest, i.e. near 950°Cfor Gallium
5. For Arsenic both 400°Cand 350°Cshould be measured for various valve settings. Less settings can be measured for 400°Cand again more detail is required in the area of interest for bismide growth at 350°C. It is best to record the GC and flux pressure before opening the valve to the to-be-measured position and THEN record the flux pressure after a few moments after opening and THEN again record the flux pressure some short time after re-closing. At least 5 minutes should be give between measurements.
6. Further to the above AS valve position for As:Ga ratio = 1 should be found, again for both T_{Ga} = 950°Cand 850°C. The RHEED pattern will change from 2×4 to 1×1 at this point (see Adamcyk PhD thesis).

3. Maintaining your MBE: Venting and Baking

Please note that venting and baking the MBE can go very smoothly, this requires 2 things: your preparedness and nothing tragic happening. Before you have decided to open the MBE up to fix something, you should evaluate the system entirely: is there enough Ga (or Bi, As, etc), is anything almost broken, do we have all the replacements parts we need, things like this. Make a list and assign tasks.

The next page has a check list of things to check after you have vented, but you put on the bake box.

Baking check list

Things to remove/disconnect:

- ☐ the cooling needs to be shut off and the waterlines need to blown out
- ☐ the LN2 lines need to be disconnected
- ☐ the manipulator motor needs to be removed
- ☐ the Ga and Bi shutter actuators need to be removed
- ☐ the RHEED needs to be disconnected
- ☐ the LL ion gauge needs to be turned off and disconnected
- ☐ the cooling lines to the H cracker needs to be removed
- ☐ the micrometer forthe As valve needs to be removed
- ☐ the N plasma power cable needs to be disconnected
- ☐ the power lines to the heated windows needs to be disconnected
- ☐ the RGA needs to be removed
- ☐ remove the sample cartridge
- ☐ the LL cover needs to be replaced with a 8" blank
- ☐ set the laminar flow curtain out of the way

Chamber bake set-up:

- ○ the ion pump gatevalve(s) should be partially (half) closed
- ○ the gate valve between the growth and prep chamber should be left partially open
- ○ the load lock should be left partially open
- ○ the cryopump gatevalve should be plugged into the wall, not its interlock
- ○ the rack power interlock needs to be shut off and the bake interlock turned on
- ○ cells should be heated to approx 300 °C
- ○ the As cracker needs to have approx 6 V
- ○ a Mo blank should be loaded in to the manipulator and placed in the growth position
- ○ every window needs to covered in tinfoil

Now put on Bake Box

Troubleshooting

This section still needs work, so for the time being your on you own - Good Luck.

if the cryopump clunks - shut it off, disconnect the lines to the pump, it needs new clean gas.

Useful contacts

FOR SAMPLES:

Wafer tech - www.wafertech.co.uk Robert Whiteside, rwhiteside@wafertech.co.uk

Tel +44 (0)1908 210444, don't forget 8hr time diff Fax +44 (0)1908 210443

NB: easiest to phone in order after receiving quote and pay by credit card, need to call approx 1am PST, samples arrived within 3-4 days!!

FOR OTHER

www.filters.com for water filters sales@filters.com 800 815 4583 614 274 9069

part no. GX50-9-7/8-02, cost: 2 cases of 40 ($2.70ea) $216.00. NB: cheaper than physics stores.

**

www.entegris.com for sample holders webrequest@entegris.com: 800 394 4083 or 800 394 4084

1.5in tray H22-15-0615 and cover H22-151-0615 6 piece sq tray H20-550860-1415 and cover H20-02-1216

**

PRAXAIR acct. #41013 604 255 6007 604 527 0710 - in Delta for speciallty gases, i.e. UHP He

**

for the small connector, so as not to have to go to the elec shop anymore

female (SLW-150-01-G-S) @digikey SAM1089-50-ND male (TSW-150-05-G-S) @digikey SAM1013-50-ND

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