

Standard Operating Procedure for SNF MOCVD

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1 Introduction

1.1 Objective

Metal-organic chemical vapor deposition (MOCVD) is one of the most important tools SNF has for growth of thin film crystalline metal alloys. Located on the second floor, MOCVD tool(aix-ccs) by Aixtron adds capabilities of being able to grow complex materials important for research at Stanford¹.

The main objectives of this document are two-fold. First, we are documenting the steps for the p-GaN/AlGaIn growth on top of already-grown n GaN/we want to provide steps needed to grow P-GaN/AlGaIn layer on top of the known recipe for GaN/c₀Al_xGa_{1-x}N/AlN buffer structure. Second, by doing so, we are also providing a tutorial for the general operation of Stanford MOCVD aix-ccs.

1.2 Background

Investigation of wide- E_g semiconductors with bandgap values of 3eV 5eV is an exciting and active field of research, for some of their interesting properties as summarized in Table 1. Amongst them, along with SiC, gallium nitride(GaN) is widely used in industry.

Property	Application
Wide Bandgap	Optoelectronics
High Breakdown Field	Power Electronics
High Electron Saturation Velocity	High Frequency Electronics
Thermal Stability	High Temperature Electronics

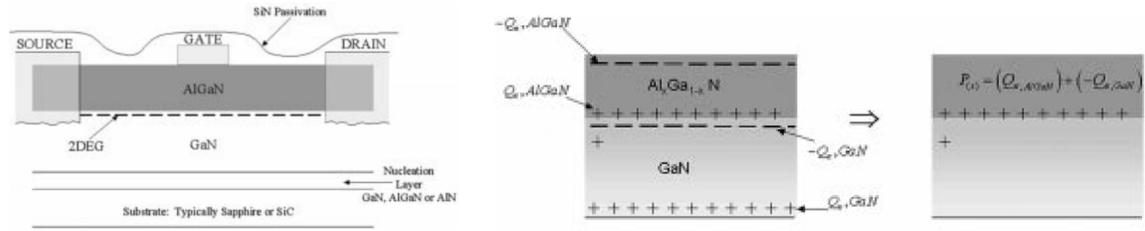
Table 1: Benefits of Wide-bandgap Materials

Amongst many possible structures using GaN, AlGaIn-GaN heterostructures shows an interesting property. As Al_xGa_{1-x}N and GaN have different lattice constants, there arises a tensile strain at the interface which causes a piezoelectric polarization. This adds to the spontaneous polarization[1]. This polarization causes a net positive charge at the AlGaIn/GaN interface, which in turn results in an electron accumulation right underneath it. We refer to this thin quantum well of electrons as in Fig.1a as **2DEG(2-dimensional electron gas)**. The GaN **HEMT(high electron mobility transistor)** makes use of this structure.

However, the AlGaIn-GaN heterojunction generates the 2DEG from its material property and hence this 2DEG exists even when there is no external bias. Thus transistors based on this structure, would conduct electric currents for zero bias and are classified as **normally-on** or **depletion-mode(d-mode)** devices. The circuits with those d-mode devices are hard to design as well as inefficient in terms of area and energy. Therefore we need to make them **normally-off** or **enhancement-mode(e-mode)** devices.

One possible way to achieve an enhancement-mode device would be using materials with proper workfunctions to perform **band engineering** and suppress the zero-bias 2DEG. P-type GaN on top of AlGaIn/GaN can function as such a material[2].

¹The complexity and dangerous involved with the tool cannot be understated. For the training and any help, please contact Dr.Xiaoping Xu (steelxu@stanford.edu), the SNF staff member in charge of this tool.



(a) 2DEG Formation Between AlGaN/GaN

(b) AlGaN-GaN Heterojunction Device

Figure 1: AlGaN-GaN High Electron Mobility Transistors [1]

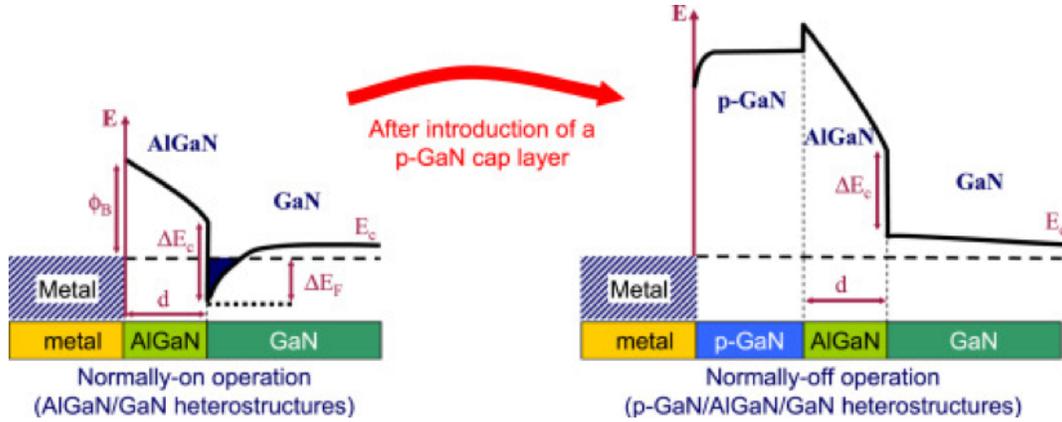


Figure 2: P-GaN cap layer renders a transistor normally-off[2].

SNF today has the ability to successfully grow two such applications: D-Mode HEMTs (normally off transistors) using AlGaN/GaN structure, and LEDs using P-doped GaN. Therefore, in this project, we merge those two recipes in order to grow P-GaN/AlGaN/GaN structure. As D-mode HEMT recipe is well-established, we leave this part of the recipe intact and provide proper modifications to the P-GaN growth from the LED recipe.

2 Pre-run Checklist

This section describes things you need to check before operating the Stanford MOCVD aix-ccs tool for safety and correct operation.

2.1 Before Entering The MOCVD Room

As there are highly toxic chemicals and vapors used in the lab, in particular arsine, you need to check the gas leak before entering the MOCVD room. When you enter the room, briefly check the hydride gas sensor located outside the entrance to the left bottom(Figure 3). All the numbers are supposed to be zero.



Figure 3: Gas sensors outside the lab door

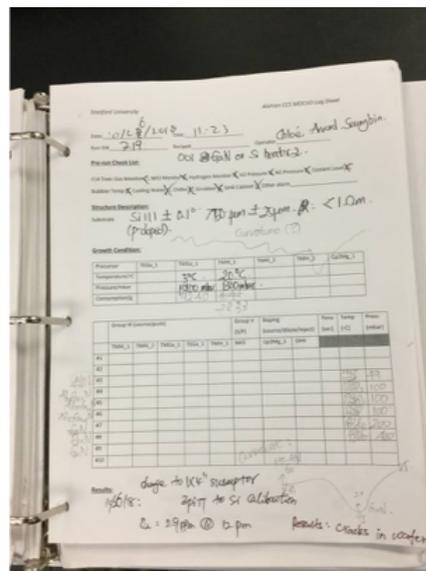


Figure 4: Log Book

If the sensors read anything above zero, do not enter the room and contact SNF staff (steelxu@stanford.edu) immediately.

2.2 Pre-run Checklist

On the desk, a log-sheet folder(Figure. 4) is present which contains the pre-run checklist that ensures all sensors and parameters are within working ranges. Follow the pre-run checklist in order and tick off the boxes if parameters are met.

2.2.1 Gas Sensors

Located next to the entrance of the MOCVD room to the left, the LEL, oxygen, ammonia and hydrogen sensors are present. If O_2 levels are below 20% or any other sensor value is above 0.1 ppm, evacuate the lab and contact the staff(Figure. 5).

2.2.2 Silane Monitoring System

Located to the left of the MOCVD tool on the wall. Ensure all systems are normal (no alarms). If alarms ring, contact the staff(Figure. 6).



Figure 5: Gas sensors for ammonia, hydrogen, etc.

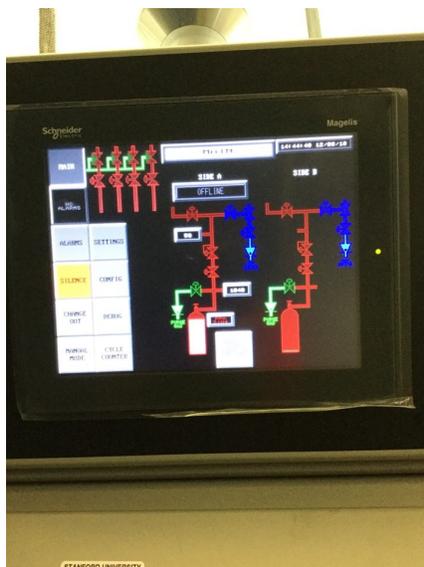


Figure 6: Silane monitoring system screen



Figure 7: Scrubber with the temperature gauge



Figure 8: Water filter pressure difference check



Figure 9: Water chiller temperature check

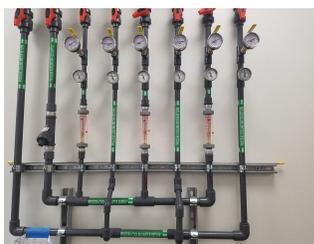


Figure 10: Cooling water pressure check

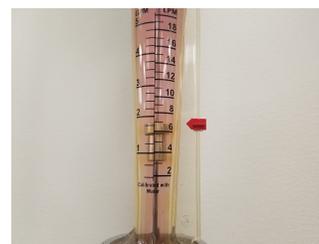


Figure 11: The gold top around the marker

2.2.3 Scrubber

Located inside the mini-room. All lights must be green and temperature range for upper heater should be near 575°C and lower heater at 500°C (Figure. 7).

2.2.4 Water Filter

Pressure gauge must be within red marker (70 to 85 psi) and differential between inlet and outlet should be under 5 psi(Figure. 8).

2.2.5 Water Chiller

The water chiller temperature should be constant at 50°C and no alarm. Fault or level LED should not be lit(Figure. 9).

2.2.6 Cooling Water Flow Check

Located at the back of the MOCVD machine, make sure gold top is at red mark(Figure. 10,11).



Figure 12: H₂ and N₂ sensors in the cabinet

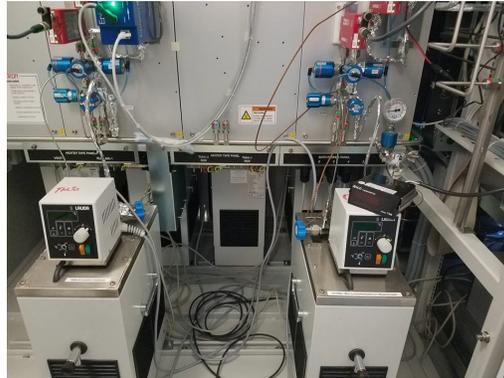


Figure 13: Bubbler thermostat to set and measure temperature



Figure 14: Bubbler water level kept 1cm below the top

2.2.7 H₂ and N₂ Sensors in the Cabinet

Located inside the front side of the cabinet next to the reactor (Figure. 8) Each of the numbers should be read 110 psi and 60 psi.

2.2.8 Bubbler

The thermostats are located in the MOCVD machine inside the metal cabinet. To power, press the green button and then orange button. The temperature can be set by pressing the down arrow button and turning the knob. It is important to set the temperature accurately as it determines the vapor pressure of the precursor. For the purposes of this recipe, we set the TMGa.1 to 5 °C and TMA1.1 to 20 °C, CP2Mg between 15 °C to 25 °C. Note, it can take up to 30 minutes for the temperature to stabilize (Figure. 13). If you are using a hot temperature of 25 °C, you can turn on the heater connected to the aluminum foil. The button for this heater is on the back side of the cabinet.

If you look behind the thermostat, you can see the bubbler water level. The water level should be kept about 1 cm below the top (Figure. 14).

3 Loading the Sample

3.1 Moving the Sample from Outside into the Loadlock

The first step in loading the sample is using the loadlock(Figure. 15). The loadlock can be thought of as an interface between the chamber and the outside world. Purge and vent the system 3 times prior to opening the gate from the outside. When you purge, open the evacuation or purge valve and keep the vent valve closed. Then you close both of them, and open the vent valve while you keep the purge valve closed. Wait for pressure gauge to left most reading for pump and again to the right for vent. After you finish the 3 cycles, both valves should be closed.

After that, open the outer door, load your sample into the loadlock and close the outer door. Purge and vent the loadlock 3 times again. Do not seal the sample box when loading into load lock, otherwise it might implode during pumping.

3.2 Moving the Sample from the Loadlock to the Chamber

Use your glovebox gloves(Figure. 16) to put your hands inside the chamber. Of course, you should be wearing the latex gloves before you put your hands into the black main gloves. While your hands are going inside the chamber, you should use the low pressure pedal(Figure. 17) so that the pressure inside does not change drastically. Remember, when you are taking your hands out, the high pressure pedal will need to be used. When your hands are in, you can wear another pair of latex gloves inside the chamber, as in Figure. 18. Therefore, you are wearing 3 layers of gloves (latex-glovebox-latex) in total. Then, you can simply open the loadlock door inside the chamber, and take your sample out.

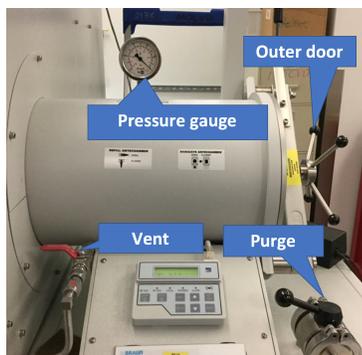


Figure 15: Loadlock



Figure 16: Glovebox gloves



Figure 17: Glovebox pedals



Figure 18: Latex gloves on glovebox gloves on latex gloves



Figure 19: Pushing the showerhead unlock button



Figure 20: Susceptor



Figure 21: Vacuum holder

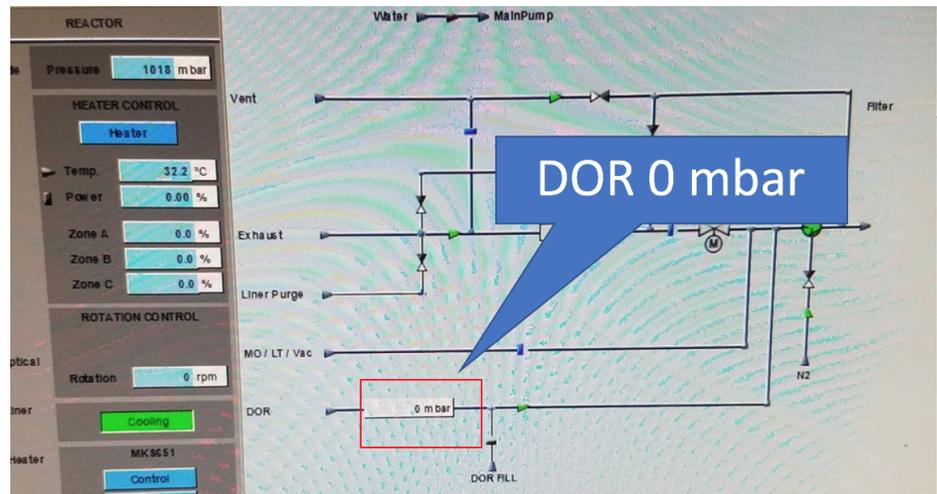


Figure 22: DOR pressure check

3.3 Placing the Sample from the Susceptor of the Reactor

The reactor should be open so that you can place your sample on the susceptor. Push the yellow showerhead unlock button(Figure.19) in order to open the reactor. While your left hand is pushing the button, your right hand will lift up the black handle of the reactor. When open, your reactor will look like Figure. 20. In Figure. 20, you can see 3 susceptor spots on which samples can be placed on. One of them is used for the sample, and the other two are filled with dummy wafers. You can tell the one from the color difference. In the figure, my left hand is pointing at the correct susceptor. Also, my right hand is pointing at a groove on the other side of the right susceptor.

Carefully load sample using vacuum holder provided inside glove box (black sample suction device, Figure. 21). Note that this suction device's vacuum is always on and only when the button is pressed is the vacuum released. Handle the wafer from the backside when loading into reactor gate. If pieces, consult Xiaoqing to change susceptor to 3 × 2 inches. Load the sample into center of the susceptor or reactor bed. Slowly close the reactor lid, while holding the black handle up. You might need to hold the reactor top and bottom together tightly for sealing. If properly done, you will see the DOR pressure 0 mbar from the screen(Figure. 22).

4 Running Your Recipe

Now is the main part, in which you perform MOCVD to grow layers. After all these steps, it is likely that your precursors are set to the right temperature. Go to the bubblers(Figure. 14), and open the valves for the TMGa₁ and TMAI₁ by turning the knob counter-clockwise until fully open and then quarter turn clockwise. Careful not to over extend the opening as it can easily damage the valve and cause the valve to leak.

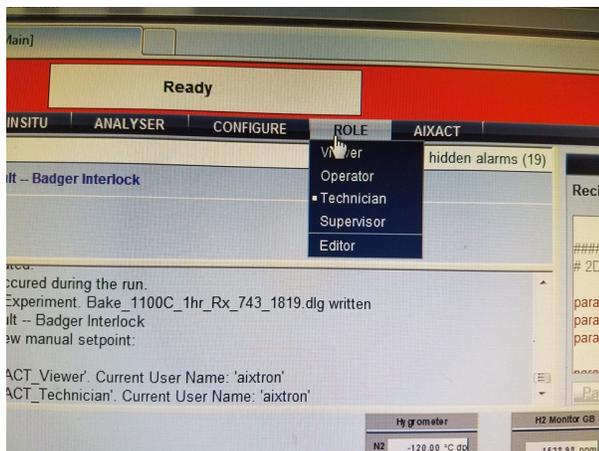


Figure 23: Change your role to 'technician'

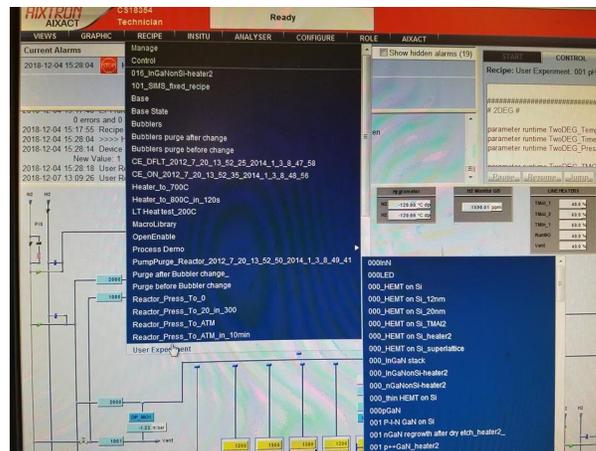


Figure 24: Loading your recipe

Then there come the software-level steps you need to take. You should enable the tool from the badger, so that you can run the recipe. Then, you change your role to 'technician' as in Figure. 23. Now, you will be able to pick your recipe as in Figure. 24. Go to recipe-user experiment, and scroll down to find your recipe. When it comes to writing down the recipe, you should talk to the staff.

Press the start button to begin the recipe. The second screen on the right monitor will startup after the temperature of 400 °C has been reached. On this screen, monitor the temperatures, reflection curve, and curvature for each step. Record the conditions in the log-sheet. Record the true temperature and set temperature (generally not the same, where true temperature is lower). Observe film reflection and curvature evolution by analyzing in-situ Epi-TT and Epi-curve monitors throughout the process for each step. On the left screen, you can see the gas flow of all the precursors, for each step, you need to record the gas flow rates and pressures, and write them down in the log sheet. Waiting for several hours of the process can be tedious, so you had better prepare something to read whilst the growth.

5 Post-growth Procedures

After the process is done, you can open the reactor, take out the samples, and close the reactor door carefully. This unloading step is pretty much the same as the loading step, the only difference is that you are taking the sample out.

Then with the sample out of the reactor but still inside the chamber, use the loadlock to take the sample out of the tool. The 3-pump-3-purge rule still applies here. Put your sample in the loadlock, perform the pump and purge cycles to remove any toxic gas inside the loadlock. Then you can open the loadlock from outside to take your sample out. After that, you can do the pump and purge cycles again, to remove any external contamination inside the loadlock.

Then you run the baking recipe to clean the reactor. Go to recipe, from user experiment option you will find the recipe named **Bake_1100C**. Run the recipe, whilst keeping the 2 dummy wafers still in. After this recipe is completely run, then you can disable the tool from the badger. Right after you disable it, the alarm will ring. Do not freak out, just click the *Mute the Alarm Buzzer* button in the main screen.



Figure 25: Gas flow dispenser

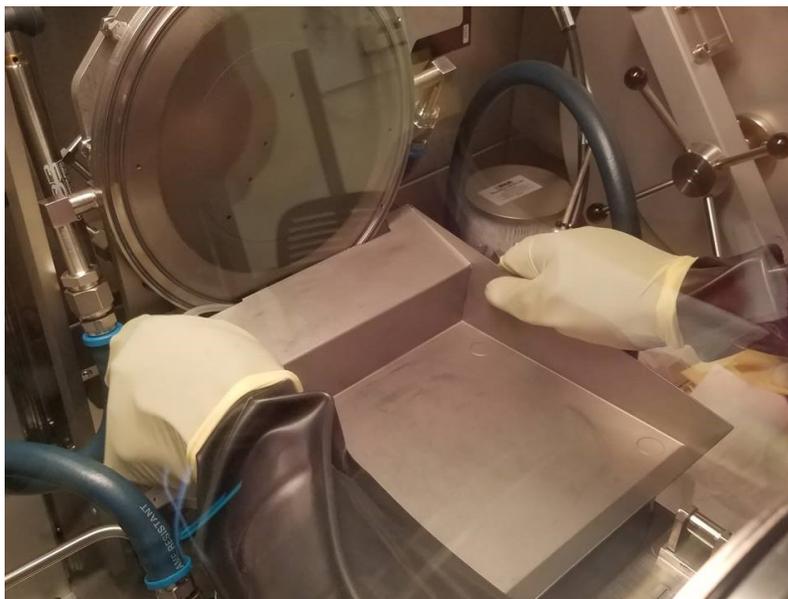


Figure 26: Metal dirt holder



Figure 27: Vacuum brush



Figure 28: Vacuum button



Figure 29: Cleaning

Now you are left with the last step: cleaning. After baking, the chemical leftovers are burnt and ready to be removed. You need to clean anything physically left on the showerhead. First, you open the reactor as before. Then, inside the chamber, you will find a part called the **gas flow dispenser**(Figure. 25). Between the showerhead and the susceptor, you can see the holes in which we need to fit the part into. Fix the part to the reactor using the black bolt at the center of the part. On top of it, you can put the **metal dirt holder**(Figure. 26), which practically functions as a dustpan while we are cleaning the showerhead. Then hold the vacuum brush as in Figure 27, turn on using the button(Figure 28). Sweep the shower head multiple time until you do not see any change in the color of the showerhead. Do it vertically, horizontally, and in other directions as well(Figure 29). After you are done, turn off the vacuum, place the metal dirt holder and the gas flow dispenser back, and close the chamber. You are done.

References

- [1] U. K. Mishra, P. Parikh, and Y.-F. Wu, "Algan/gan hems-an overview of device operation and applications," *Proceedings of the IEEE*, vol. 90, pp. 1022–1031, June 2002.
- [2] C. S. Suh, A. Chini, Y. Fu, C. Poblenz, J. S. Speck, and U. K. Mishra, "p-gan/algan/gan enhancement-mode hems," in *2006 64th Device Research Conference*, pp. 163–164, June 2006.