# SK2822 MOVPE Lab Report

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***Abstract*—The system used for Metalorganic vapour-phase epitaxial (MOVPE) growth of single- or poly-crystalline III-V semiconductor thin films was demonstrated in the Electrum cleanroom laboratory. Key features of the MOVPE equipment which were elaborated upon for controlling growth sequence, include mass flow mixing of precursor gases and vent-run switching of the gas flow.**

## I. Introduction

Vapor phase Epitaxy (VPE) is a chemical deposition technique in which semiconductor epilayers are grown at a rate which is kinetically rate-limited by the lateral laminar diffusion of precursor gases onto a surface, and which is nearly independent of the reaction temperature ($T$). MOVPE is then a special class of VPE used for III-V epitaxy, in which precursors in complexion — III-precursor in methylated ($CH_3^\bullet$) complex and V-precursor in either hydride ($H^\bullet$) or $CH_3^\bullet$ complex, are made to pyrolyse and catalytically react far from equilibrium through the boundary layer on a substrate.

The equipment demonstrated in this lab was similar to Aixtron AIX 200/4.

### A. Theory

Reactor parameters governing MOVPE growth include –

*1) Total pressure ($P$):* Typically in mbar, a low total reaction pressure for a homogeneous deposition of the pyrolysed precursors on the desired surface.

*2) Gas flow rate ($Q$):* A laminar flow rate profile to ensure a constant lateral growth rate, and negligible transverse growth rate over the desired surface. Layer growth rate is then directly proportional to the precursor flow rate.

*3) Precursor partial pressure ($p$):* A V-precursor over-pressure w.r.t. III-prescursor to stablilise the desired surface from decomposing out the volatile V-precursor pyrolyte at high $T$ ($> 623K$).

### B. MOVPE Equipment

Significant components in the MOVPE system include –

*1) Gas Mixing Cabinet:* A system consisting Mass Flow Controllers (MFCs) and Electronic Pressure Controllers (EPCs) to control III- and V- precursor gas compositions and subsequent mixtures.

- For III-precursor - Composition controlled by bath temperature, bubbler EPC pressure and a subsequent MFC carrier gas flow rate through the precursor bath.
- For V-precursor - Composition controlled by vent EPC pressure and a subsequent MFC carrier gas flow rate through the precursor bath.

*2) Reaction Chamber:* A place where MOVPE growth is temperature- controlled (inductive-coupling / RF capacitive-coupling / photo-assisted), pressure- controlled (capacitive / diaphragm / ionization gauge) and flow-controlled (vent-run EPC-MFC); and at scale.

*3) Pumping and Scrubbing System:* Reduces toxic exhaust gases emanating from the reaction chamber.

Precautionary steps such as ensuring multiple sources for the same precursor and high vent-run switch frequency, result in good control over layer thickness and aid in realising sharp (atomic-layer) interfaces.

## II. Discussion

With regard to instructions in the lab manual, the following answers entail –

1) Quality of InGaAs/GaAs layer: Following the chain of observations from the article 'MOVPE_Ref', it can be concluded that the cross-hatch pattern observed on InGaAs/GaAs epilayer surface indicates an ungraded smaller lattice mismatch during the growth process – at misfit dislocation strain $< 2\%$ (alternatively at disclocation densities $< 4 \times 10^7 cm^{-2}$), and/or at In compositions of $< 0.25$.
2) $p_{\mathrm{TMIn}}$ in the reactor with $T_{bath}$ @290K:

$$p_{\mathrm{TMIn}} = \frac{P_{\mathrm{TMIn}}}{P} = \frac{P_{\mathrm{TMIn\_bubbler}}}{P_{bubbler}} \times \frac{Q_{source}}{Q}$$

$$\text{Thus, } p_{\mathrm{TMIn}} = \frac{1.34}{0.3} \times \frac{138}{15000} = \underline{0.041}$$

$$\text{where, } P_{\mathrm{TMIn\_bubbler}} = 10^{10.52 - \frac{3014}{T}}$$

3) $Q_{eff}$ of $SiH_4$:
   a) For a double dilution source-pusher configuration

$$\frac{\Delta Q_3}{Q_3} = \frac{Q_1 \cdot \frac{\Delta Q_1}{Q_1} + Q_2 \cdot \frac{\Delta Q_2}{Q_2}}{Q_1 + Q_2} - \frac{\Delta Q_1}{Q_1}$$

$$= 0, \text{ when } \frac{\Delta Q_1}{Q_1} = \frac{\Delta Q_2}{Q_2} \in [0.05 - 0.95]$$

Thus,

i) Highest $Q_{3,eff} = 0.95{\cdot}Q_3 = 0.95{\cdot}200 = \underline{190sccm}$
ii) Lowest $Q_{3,eff} = 0.05 \cdot Q_3 = 0.05 \cdot 200 = \underline{10sccm}$

b) For a standard source-pusher configuration

$$\frac{\Delta Q_3}{Q_3} = \frac{Q_1 \cdot \frac{\Delta Q_1}{Q_1} + Q_2 \cdot \frac{\Delta Q_2}{Q_2}}{Q_1 + Q_2}$$
$$= \frac{\Delta Q_1}{Q_1} = \frac{\Delta Q_2}{Q_2} \in [0.05 - 0.95]$$

Thus,

i) Highest $Q_{3,eff} = 0.95 \cdot (0.95 \cdot Q_3) = 0.95 \cdot 0.95 \cdot 200 = \underline{180.5sccm}$
ii) Lowest $Q_{3,eff} = 0.05 \cdot (0.05 \cdot Q_3) = 0.05 \cdot 0.05 \cdot 200 = \underline{0.5sccm}$

Hence, the double dilution source-pusher configuration is more reliable than the standard one.

4) Recipe for 400nm lattice-matched InGaP on GaAs:

i Reactor $T$ ramp = $710°C$, Reactor $P$ = $100mbar$
ii $AsH_3$.line open, $AsH_3$.source = 80; "$AsH_3$ flow (vent)"
iii $AsH_3$.run, $AsH_3$.push = 420; "$AsH_3$ stabilisation flow (run)"
iv 2:00 TMGa.line open, TMGa.source = 60; "TMGa flow (vent)"
v 5:00 TMGa.run open, TMGa.push = 420; "TMGa flow (run)"
vi TMGa.run close, TMGa.line close; "Stop growth and close TMGa line"
vii 1:00 Standby; "GaAs anneal before growing InGaP"
viii $AsH_3$.run close, $AsH_3$.line close; "Close $AsH_3$ line"
ix Purge reactor gases; "Toxic gases to scrubber"
x Reactor $T$ ramp = $570°C$
xi $PH_3$.line open, $PH_3$.source = 80; "$PH_3$ flow (vent)"
xii $PH_3$.run, $PH_3$.push = 420; "$PH_3$ stabilisation flow (run)"
xiii 2:00 TMIn.line open, TMIn.source = 168.3; "TMIn flow (vent)"
xiv 2:00 TMGa.line open, TMGa.source = 80; "TMGa flow (vent)"
xv 16:03 TMIn.run open, TMIn.push = 210, TMGa.run open, TMGa.push = 210; "TMIn and TMGa flow (run)"
xvi TMIn.run close, TMIn.line close, TMGa.run close, TMGa.line close; "Stop growth and close TMIn and TMGa line"
xvii 1:00 Standby; "InGaP anneal"
xviii $PH_3$.run close, $PH_3$.line close; "Close $PH_3$ line"
xix Purge reactor gases; "Toxic gases to scrubber"
xx Reactor $T$ ramp = $28°C$, Reactor vent $P$ = $1bar$

## III. Conclusion

The MOVPE system and its constituents were studied upon, and the effect of some reaction parameters ($Q$, $p$) in these constitutents on the epilayer were numerically analysed.