Monitoring and Control of Binary Gas Mixtures from Solid Phase MOCVD Sources Using an Acoustic Sensor

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Issues with delivery of solid MOCVD precursors

- Solid MOCVD sources used in compound semiconductors
  - e.g. TM in III/V GaAs devices, Cp2Mg for p-doping
- Dosimetry issues from use of MO solid sources
  - Low vapor pressure: TM (1.75 Torr), Cp2Mg (0.05 Torr) at 25°C
    - Require heated source and feed lines
  - Instability of metal-organic feed rate due to:
    - Aging effects (change of crystal surface area, material redistribution, contamination) ⇒ Vapor/Pressure variation
  - Interaction fixed line / MO vapor ⇒ condensation
  - Incomplete saturation at high flows

- Reproducibility issues affect device performance
- Only small fraction of the source is used before being replaced

⇒ Need for real-time monitoring and control of the MO precursor concentration

**Effect of pressure variations**

- $P > 150$ Torr, composition measurements vary accordingly with $VP/P$
- $P < 50$ Torr, measurement failure due to insufficient transfer of acoustic energy
- Between 50 and 150 Torr, higher concentration achievable but sensor response becomes non-linear vs. $1/P$

- Varying pressure is not recommended to adjust composition due to effects of pressure change on acoustic measurements

**Effect of $H_2$ flow rates**

- MO composition can not be reproducibly adjusted by varying carrier gas flow
- If binary gas mixture (precursor, carrier)
  - $F_2$, carrier gas resonant frequency, is known
    - High mass ratio ⇒ high sensitivity

- Control of Cp2Mg concentration
  - Requires in reactor
    - Tuning and maintain constant MO precursor concentration with a constant throughput (H2 carrier + dilution) to reactor
  - Requirements in delivery system
    - Fixed pressure to minimize acoustic sensor drift and controllable precursor concentration to compensate for change in source vapor pressure (temperature or aging effects)

Effect of temperature drift in open loop configuration

- $Cp2Mg$ bath temperature varied from 40° to 32°C
  - Vapor pressure down from 0.16 to 0.08 Torr ⇒ "stimulates" aging effects

- Open loop configuration:
  - dilution flow = 50 sccm
  - $Cp2Mg$ composition down from 0.01 to 0.005 mol%

Closed loop control performance

- Source temperature varied from 40° to 32°C
  - $I_2$ (flows) = 150 sccm, $P$ = 300 Torr
  - $Cp2Mg$ composition target = 0.01 mol% (0.3 umol/min)

Conclusions

- Acoustic sensing provides very accurate measurements of metal organic concentration obtained from low VP solid source
  - Measure trace amount below 1 ppm
  - Use of resonant cavity provides good energy transfer and allows concentration measurement down to 50 Torr in sublimator
  - Interesting to obtain higher concentrations

- Use of closed loop control with acoustic sensing enables stable delivery from low vapor pressure MOCVD solid sources
  - Precursor composition control within 1% even at low precursor concentration, e.g., controlling $Cp2Mg$ to 1% variability at 0.01 mol% concentration in carrier gas
  - Compensate long term drifts due to source aging as well as short term drift due to source variability