Monitoring and control of binary gas mixtures from solid phase MOCVD sources using an acoustic sensor

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Improved precursor delivery: ESH perspectives

- Increasing variety of new precursors for advanced materials
  - Si VLSI (e.g., low and high K dielectrics), wide bandgap SC (GaN)
- Productivity and ESH metrics often affected
  - Low chemical stability, low vapor pressure liquid or solid sources, high toxicity …
- ESH benefits from improved precursor delivery:
  - Greater flexibility in chemical process design
    - Wider variety of precursors meet manufacturability constraints
  - Use of Advanced Process control
    - APC is key to higher yield and equipment effectiveness
    - Higher productivity minimizes ESH metrics such as materials utilization

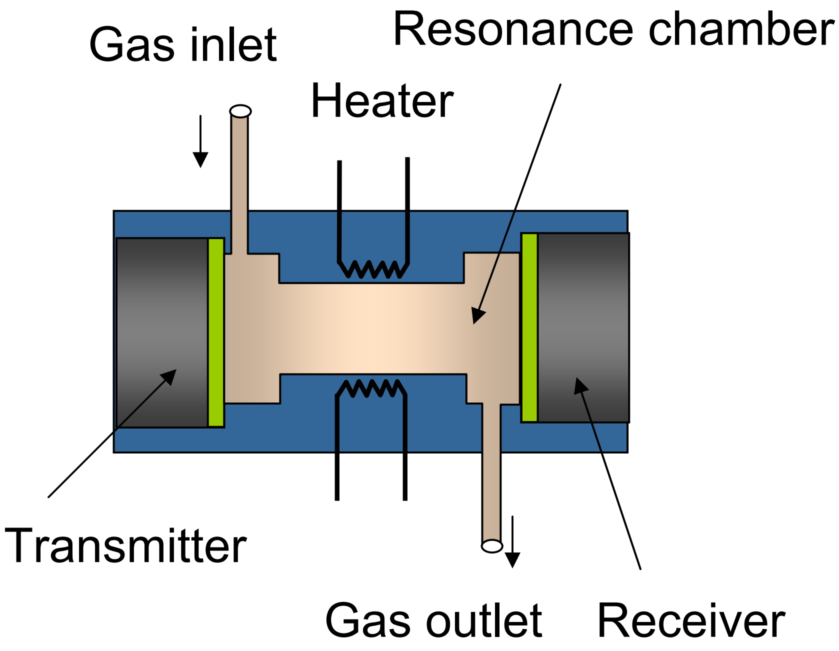
Issues with delivery of solid MOCVD precursors

- Solid MOCVD sources used in compound semiconductors
  - e.g. TMI in III/V GaN devices, Cp_2Mg for p doping
- Dosimetry issues from use of MO solid sources
  - Low vapor pressure: *TMI (1.75 Torr),* Cp_2Mg *(0.05 Torr) at* 25^oC
    - *Require heated source and feed lines*
  - Instability of metal-organic feed rate due to:
    - *Aging effects (change of crystal surface area, material redistribution, contamination)*
    - *Interaction feed 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

Inficon "Composer" acoustic transducer



Measurement of resonant frequency F

$$F = \frac{C}{2L} \quad \text{with} \quad C = \sqrt{\frac{\gamma_{avg} RT}{M_{avg}}}$$

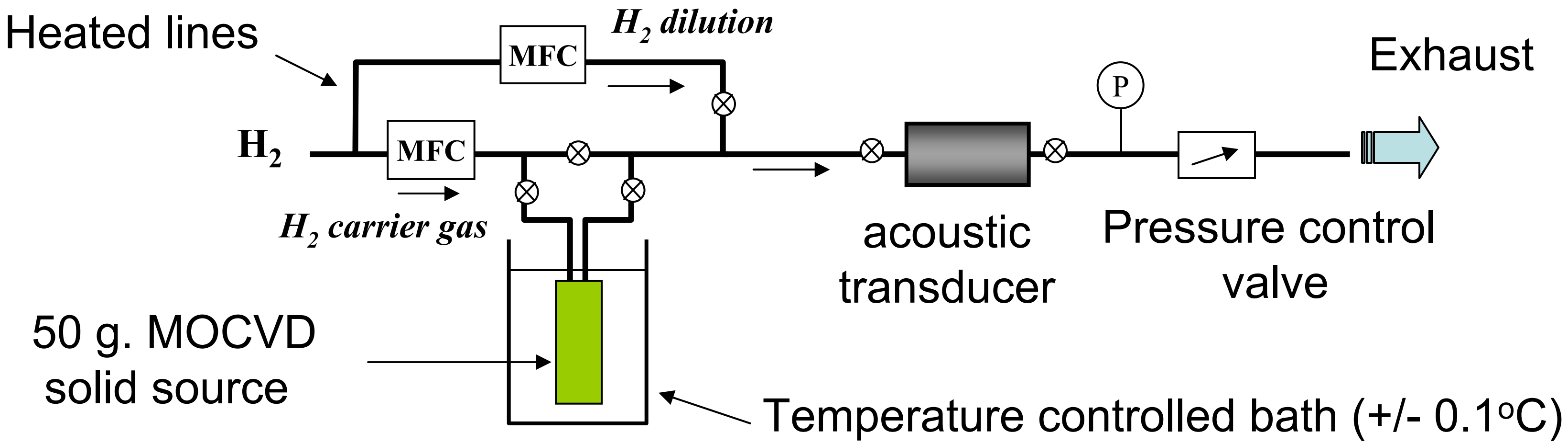
C: speed of sound, L: chamber length
T: gas temperature
γ_{avg}: average specific heat ratio
M_{avg}: average molecular weight

Gas	Mol. weight (g/mol)	Res. Freq. (Hz)
H_2	2	4000
Cp_2Mg	154.5	440

- If binary gas mixture (precursor, carrier)
- If F_2, carrier gas resonant frequency, is known
  $\Rightarrow F/F_2 = f$ (Precursor Mole Fraction)
- High mass ratio ⇒ high sensitivity

Solid source gas delivery

Carrier gas (H_2) flown into temperature controlled sublimator to be saturated by source vapor pressure

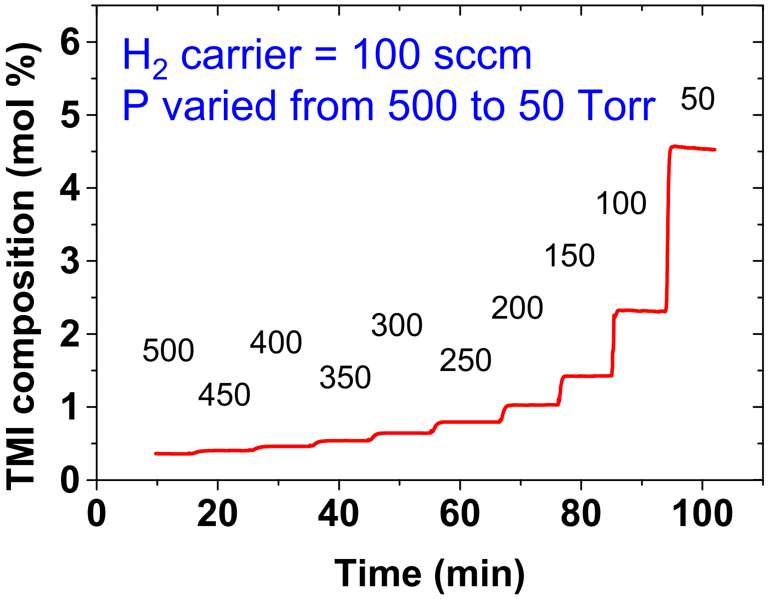


Recommended temperatures

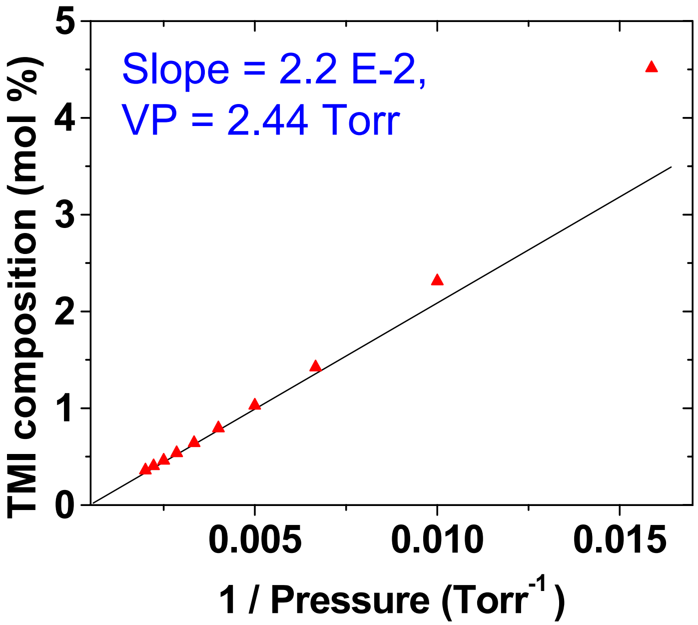
Gas	Bath T (° C)	VP (Torr)
TMI	25	2.54
Cp_2Mg	40	0.16

Monitoring of TMI and Cp_2Mg concentration by acoustic sensing

Effect of pressure variations



H_2 carrier = 100 sccm
P varied from 500 to 50 Torr



Slope = 2.2 E-2,
VP = 2.44 Torr

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

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

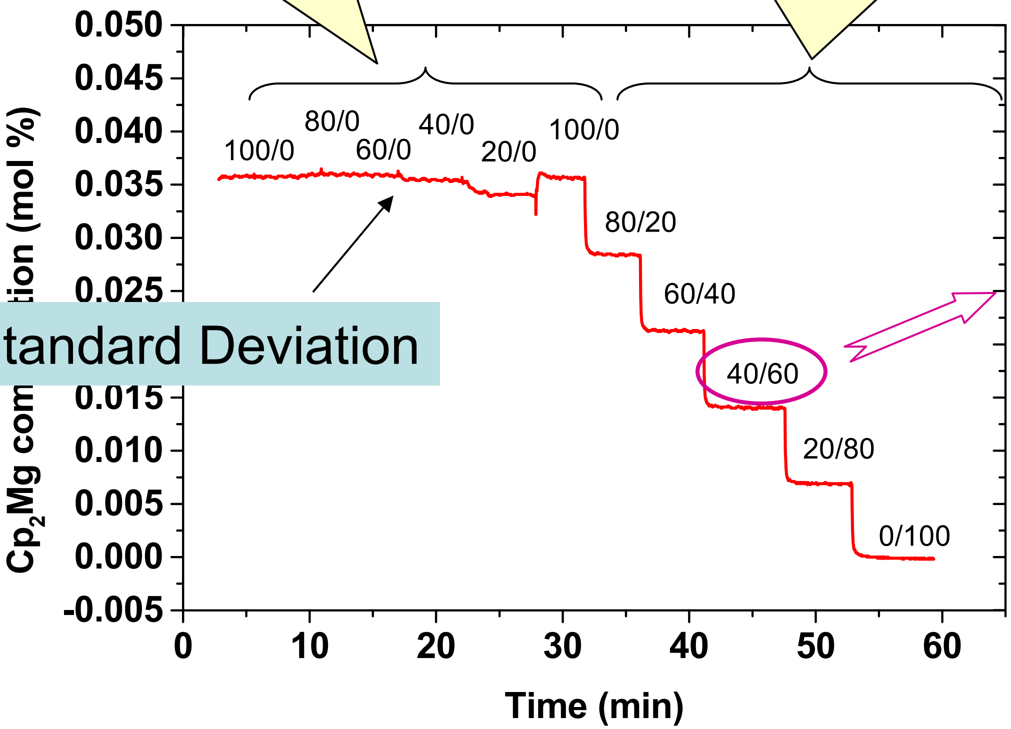
Ideal operating environment

- Requirements in reactor
  - Tune and maintain:
    - constant MO precursor concentration
    - constant gas throughput (H_2 carrier + precursor) to reactor
- Requirements in delivery system
  - Fixed pressure to minimize sensor drift (and potential low pressure failure)
  - Controllable precursor concentration to compensate for change in source vapor pressure (temperature or aging effects)

Effect of H_2 flow rates

MO composition can not be reproducibly adjusted by varying carrier gas flow

- Σ (carrier + dilution) = constant throughput
- Composition adjusted by varying H_2 dilution flow rate



3 E-5 mol% Standard Deviation

H_2 carrier flow / H_2 dilution flow (sccm)

P = 300 Torr

Control of Cp_2Mg concentration

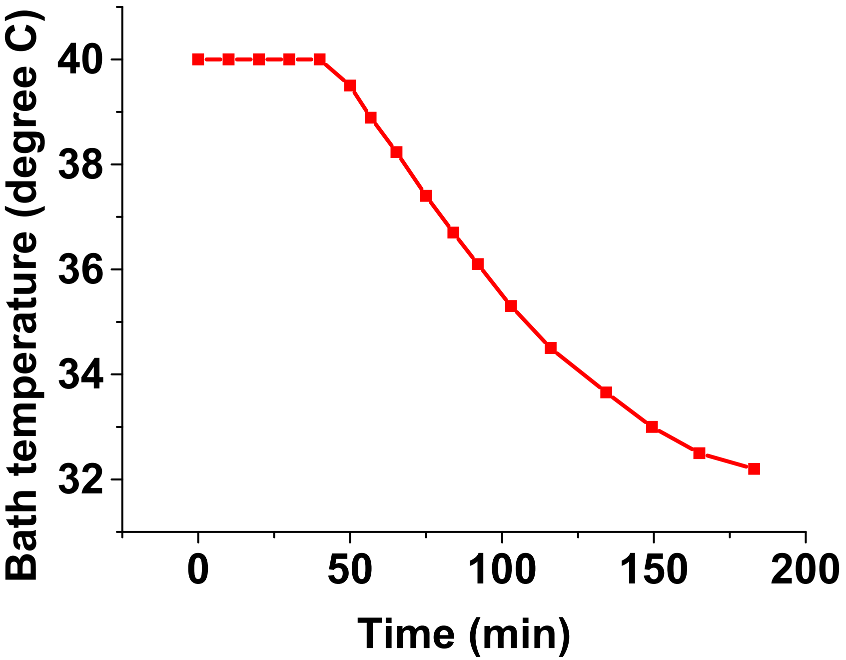
With Cp_2Mg, measurement Standard Deviation = 3 E-5 mol%

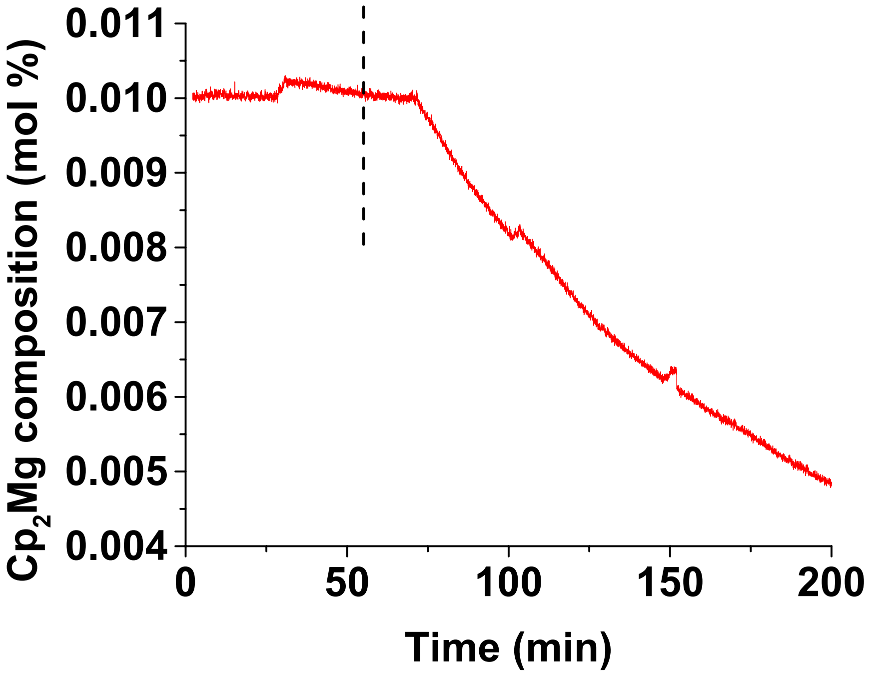
➢ accuracy better than 1 ppm with 75 / 1 mass ratio

➢ can detect Cp_2Mg concentration change resulting from 0.1 % variation in dilution flow (under 100 sccm total flow)

⇒ Excellent prognosis for real-time control of MO feed rate

Effect of temperature drift in open loop configuration

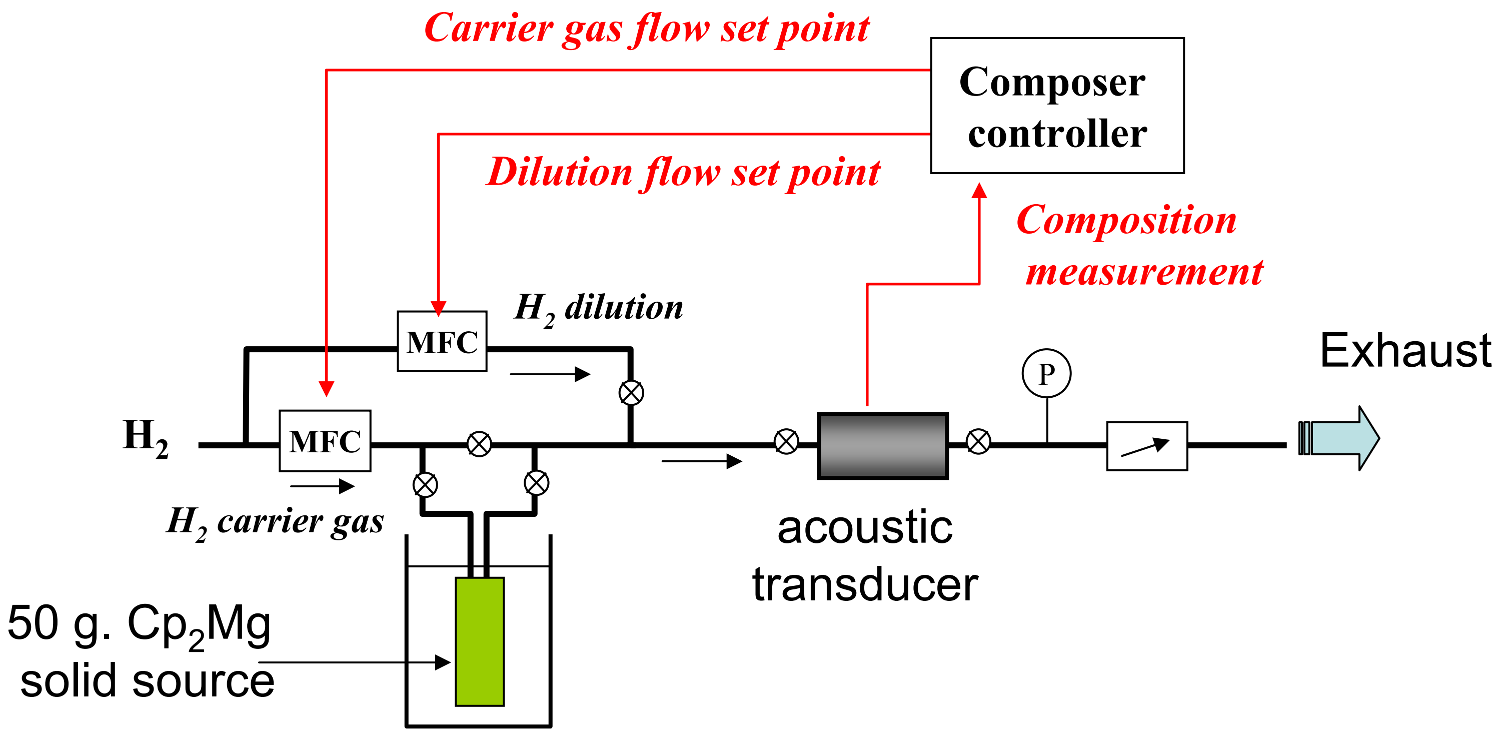




- Cp_2Mg bath temperature varied from 40° to 32°C
  - -Vapor pressure down from 0.16 to 0.08 Torr
  - - "Simulates" aging effects

- Open loop configuration:
  - dilution flow = 100 sccm
  - sublimator flow = 50 sccm
- Cp_2Mg composition down from 0.01 to 0.005 mol%

Closed loop concentration control



Carrier gas flow set point

Composer controller

Dilution flow set point

Composition measurement

H_2 dilution

Exhaust

H_2

H_2 carrier gas

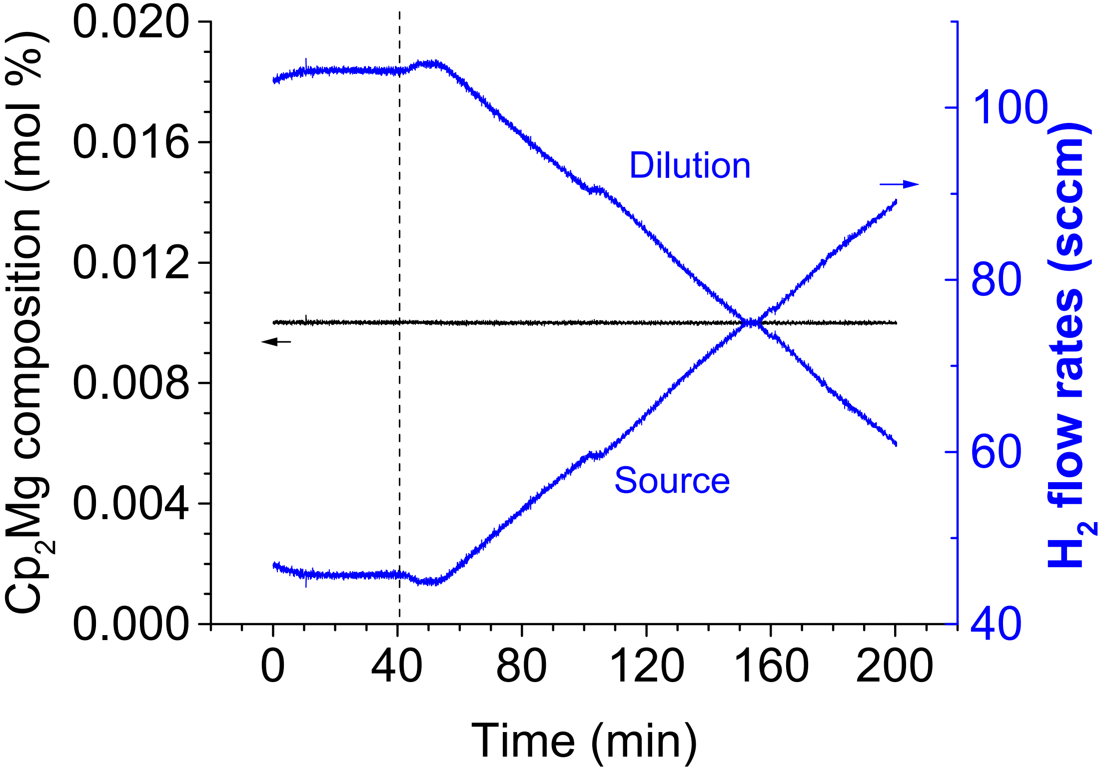
acoustic transducer

50 g. Cp_2Mg solid source

H_2 dilution and carrier flows corrected to keep composition on target

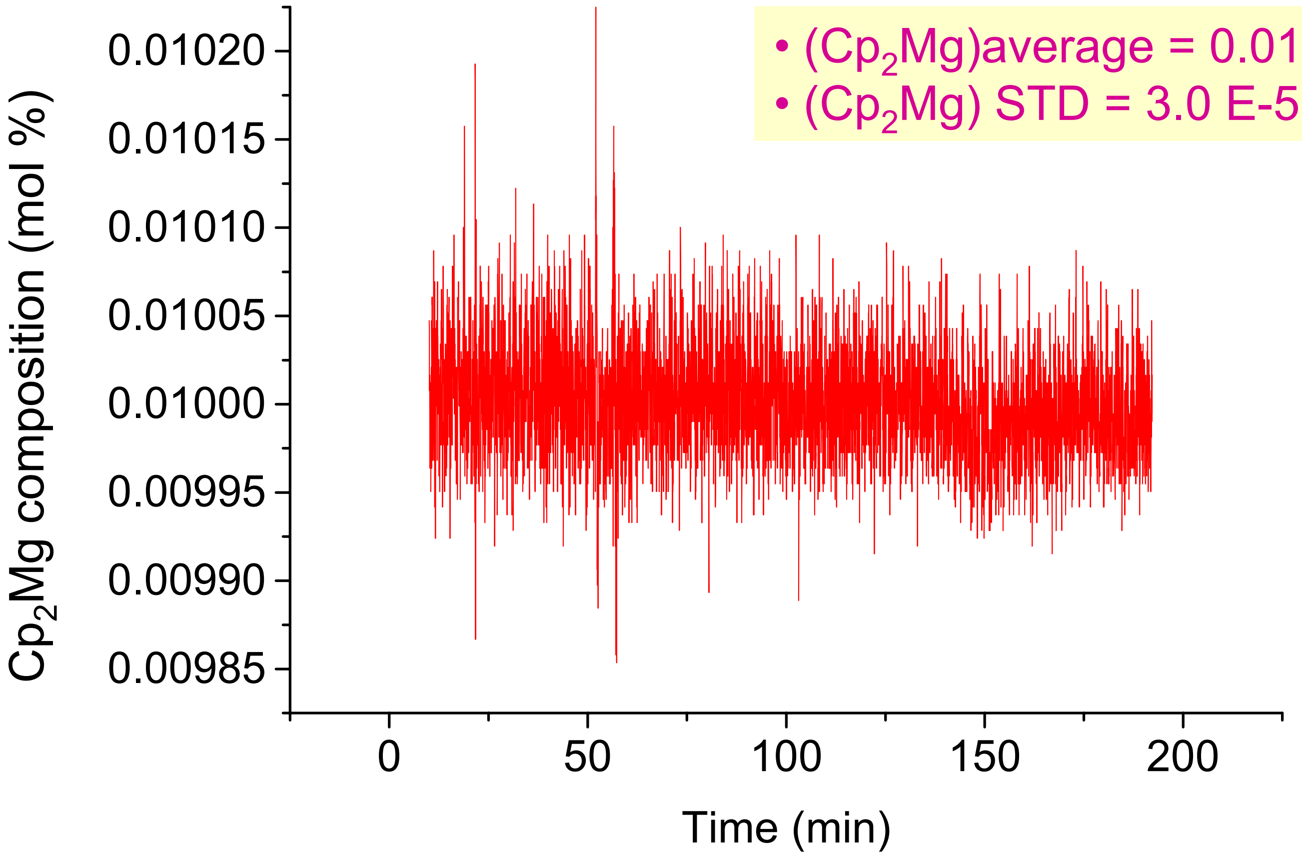
- Proportional Integral Derivative close loop control
- Primary control variables adjusted every second

Effect of temperature drift on composition in closed loop control



- Source temperature varied from 40 to 32^{o}C
- Σ (H_2 flows) = 150 sccm, P = 300 Torr
- Cp_2Mg composition target = 0.01 mol% (0.3 umol/min)

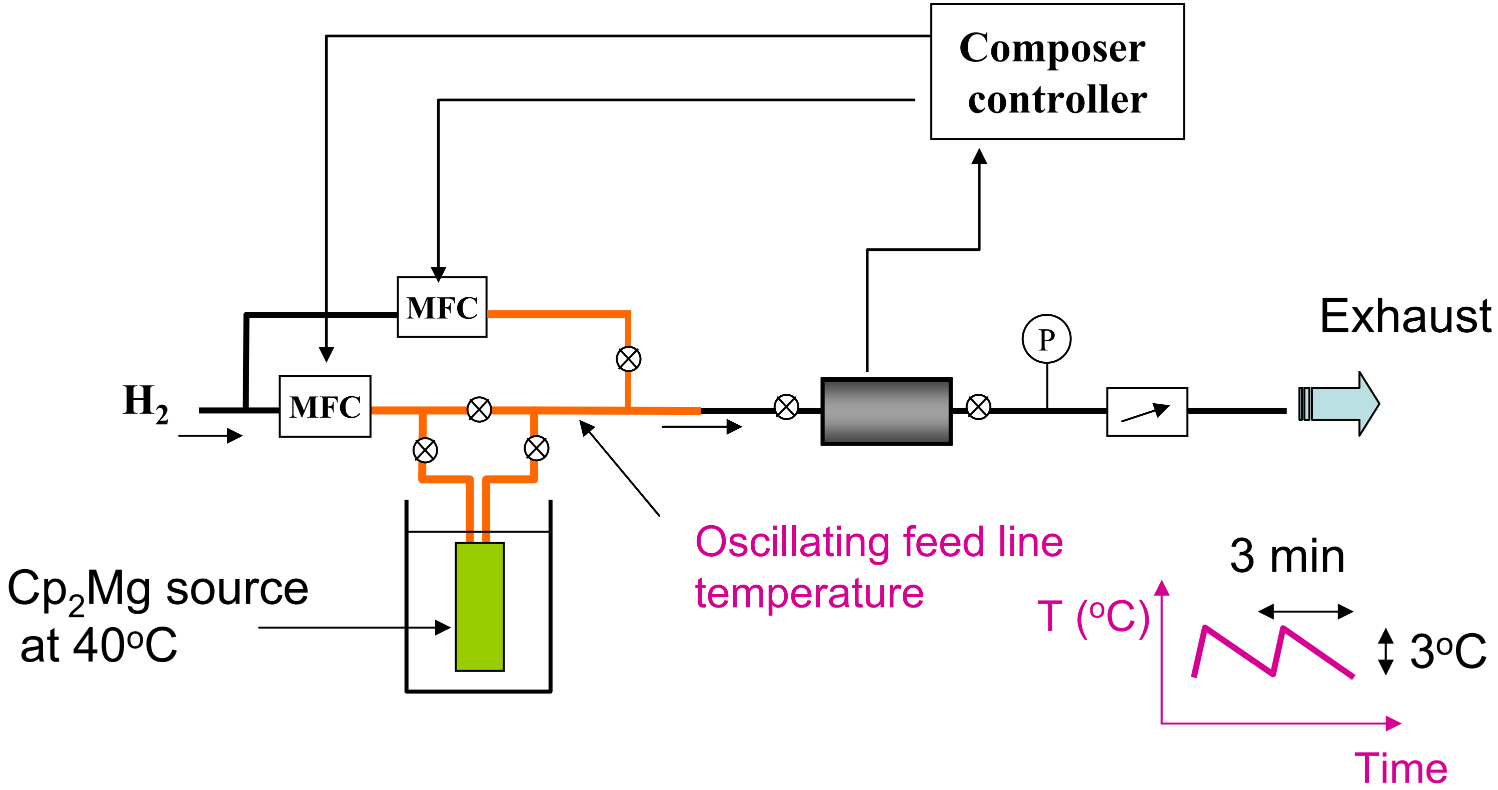
Closed loop control performance



- (Cp_2Mg)average = 0.010009 mol %
- (Cp_2Mg) STD = 3.0 E-5 mol %

Cp_2Mg composition controlled within a 1 % range despite variation of the source vapor pressure from 0.16 to 0.08 Torr.

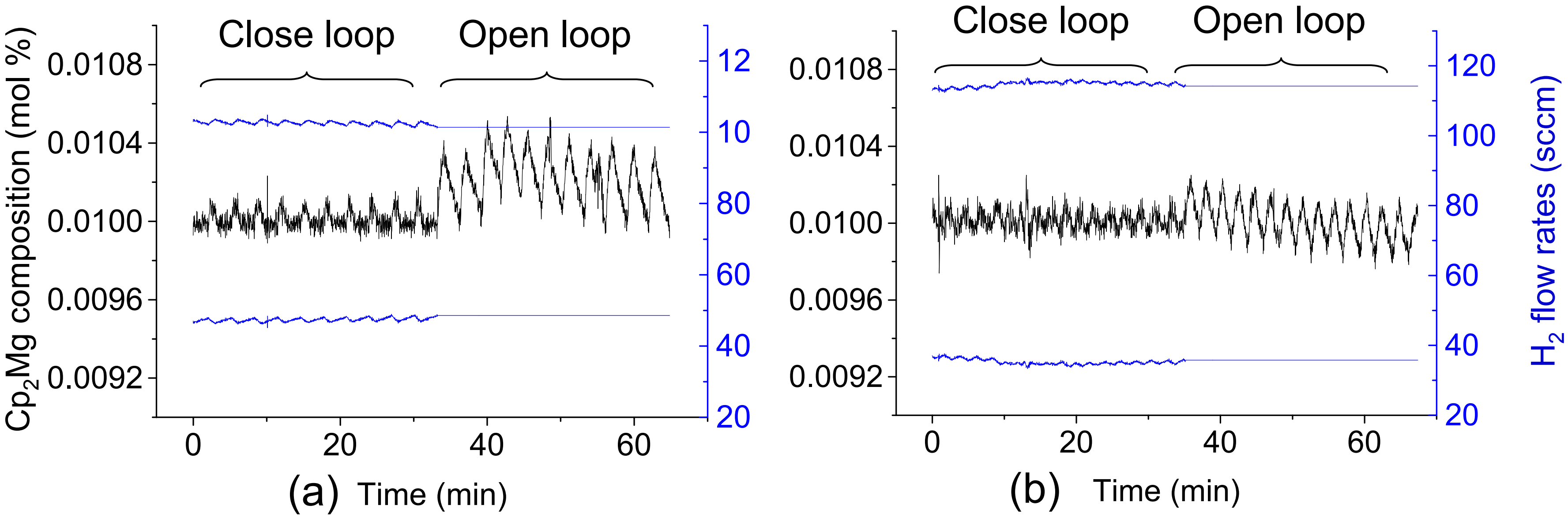
Closed loop control in presence of short term disturbances



Set On/Off heating elements to generate 3°C temperature oscillations in feed line

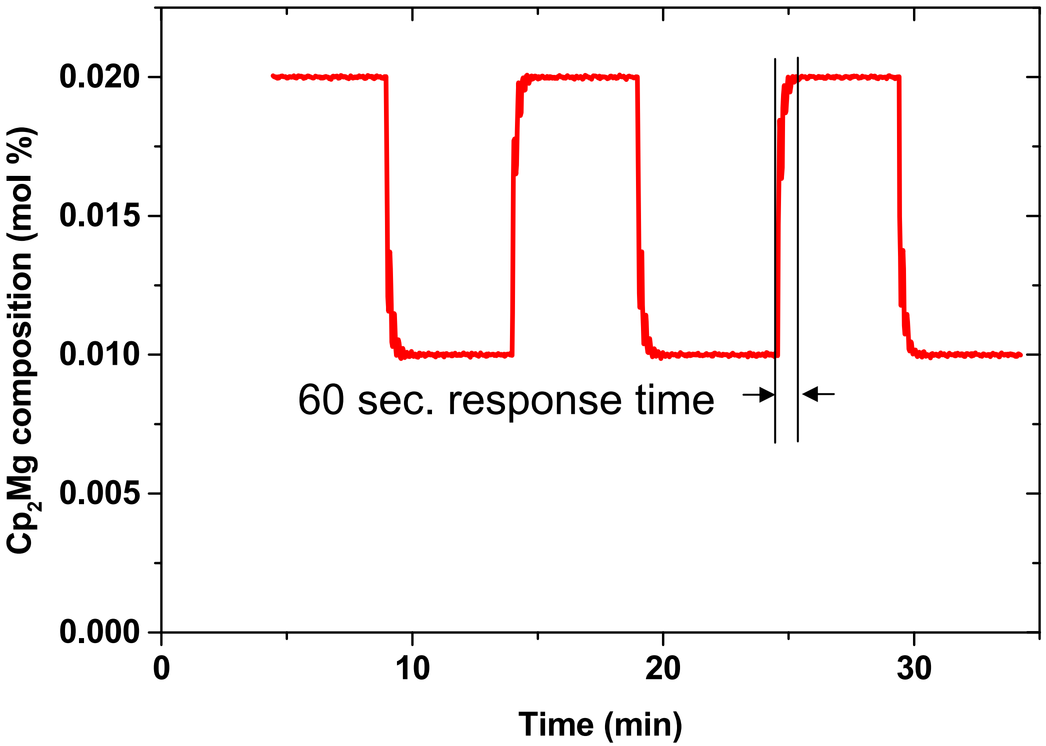
Cp_2Mg concentration control in presence of disturbances

T(source) = 40°C; T(Feed line) = 50 +/-1.5°C in (a); 60 +/-1.5°C in (b)



- Feedback control results in significant reduction of composition variations in presence of disturbances
- Higher feed line temperature minimizes MO condensation

Composition profiling



Use of closed loop control allows reproducible composition profiling with 1 min. response time

Conclusions

- Acoustic sensing provides very accurate measurements of metal organic concentration obtained from low VP solid source
- Use of closed loop control with acoustic sensing enables stable delivery of low vapor pressure MOCVD solid sources
  - Control of the composition within 1% even at low precursor concentration (e.g., 0.01 mol % with Cp_2Mg)
  - Compensate long term drifts due to source aging as well as short term drift due to source variability
- Use of APC on reactant delivery system could significantly increase the tool productivity and reduce the precursor utilization.

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