Temperature-based Control of Liquid Precursor Delivery for ALD Processes

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Motivation:

- Seek better control of the ALD/CVD process through better understanding of the evaporation / sublimation process.
- Develop a device and a method to compensate for the effects of evaporative cooling of the precursor to increase yield.

Temperature, Vapor Pressure, Energy (Heat) Flow, Transients:

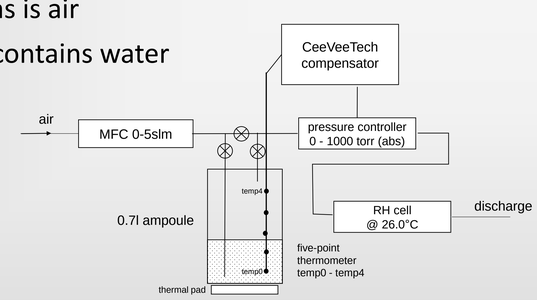
- the evaporative energy (heat) loss increases with the carrier gas flow (bubbler) or the shot frequency (vapor draw).
- the temperature drop increases with the energy loss.
- vapor pressure and delivery to the ALD process decreases with temperature drop.

Precursor	change in vapor pressure per degree C
water	5.0%
TMAl	5.6%
PDMATa	7.2%

- after a flow or a shot frequency change, the precursor transitions to a different temperature until evaporative energy loss is in balance with heat flow from the environment.

Apparatus:

- thermal environment of the bubbler: still air at room temperature
- commercial RH sensor in 26.0C temperature cell
- carrier gas is air
- bubbler contains water

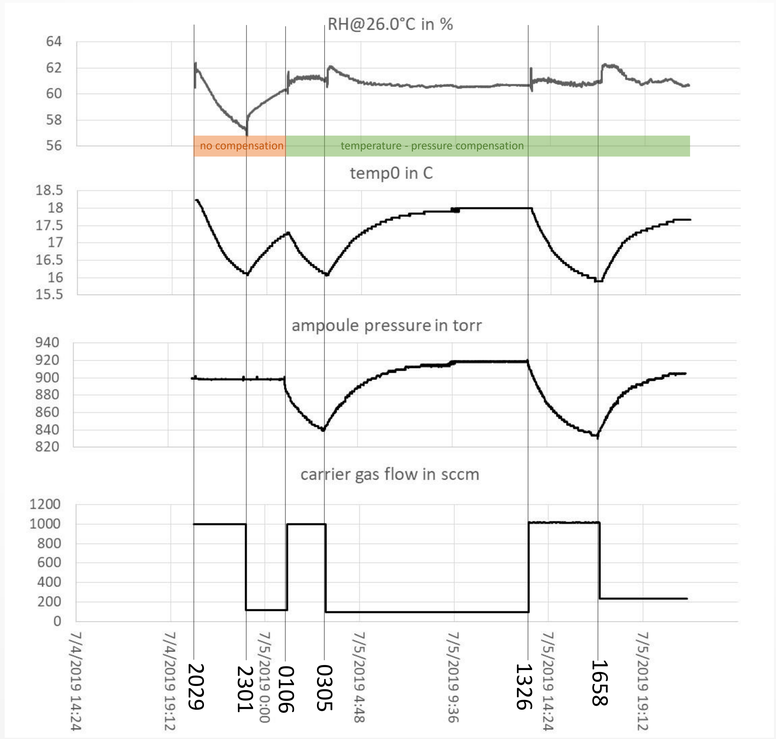


Temperature - Pressure compensation:

The output concentration c = (vapor pressure)/(total pressure) should be proportional to the vapor pressure at bubbler temperature unless there are other factors "masking" the equilibrium vapor pressure.

When the temperature decreases due to evaporative cooling the output concentration can be maintained by lowering the ampoule pressure.

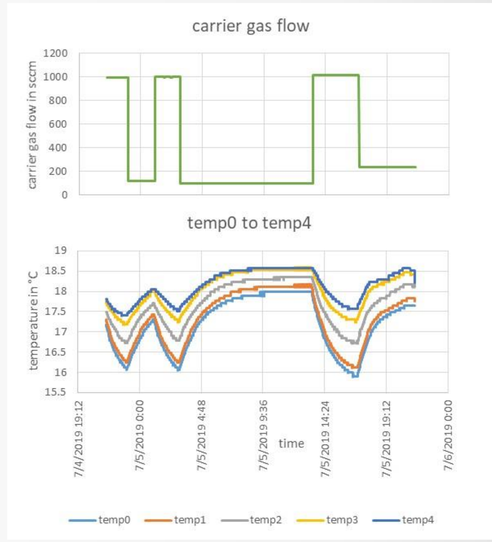
Efficacy of Temperature-Pressure Compensation:



- The ampoule contains 125ml water. Level at about temp1.
- The vapor pressure of water at 26.0C is 25.5 torr
- equilibrium vapor pressure[torr] = 10^(A - B/T[K]):
  water (NIST database): A = 9.210, B= 2298.9
  full compensation is observed when using: A = 7.41, B = 1690.7
- cuts from high flow to low flow, RH increases by about 1% before adjusting to "normal" within about one hour.

Temperature Layering:

- temperature layers observed in head space
- temperature in the liquid is uniform due to agitation from bubbling



Summary:

- Temperature-Pressure Compensation is effective to compensate for loss of precursor.
- Temperature strata develop in the head space and can be used for liquid level indication
- The temperature is uniform in the liquid.