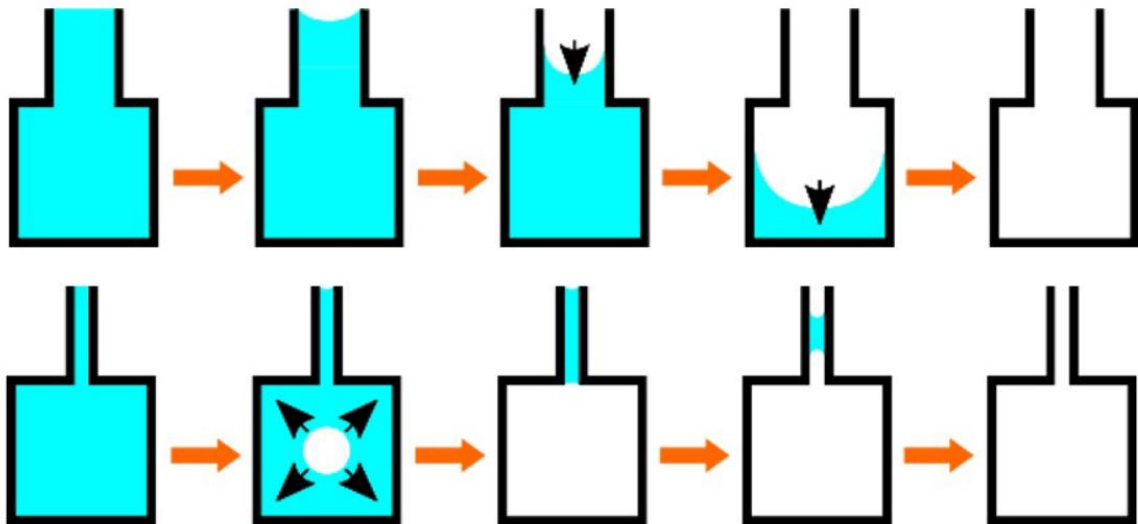


Exploring Helium Metastability Using Artificial Porous Systems



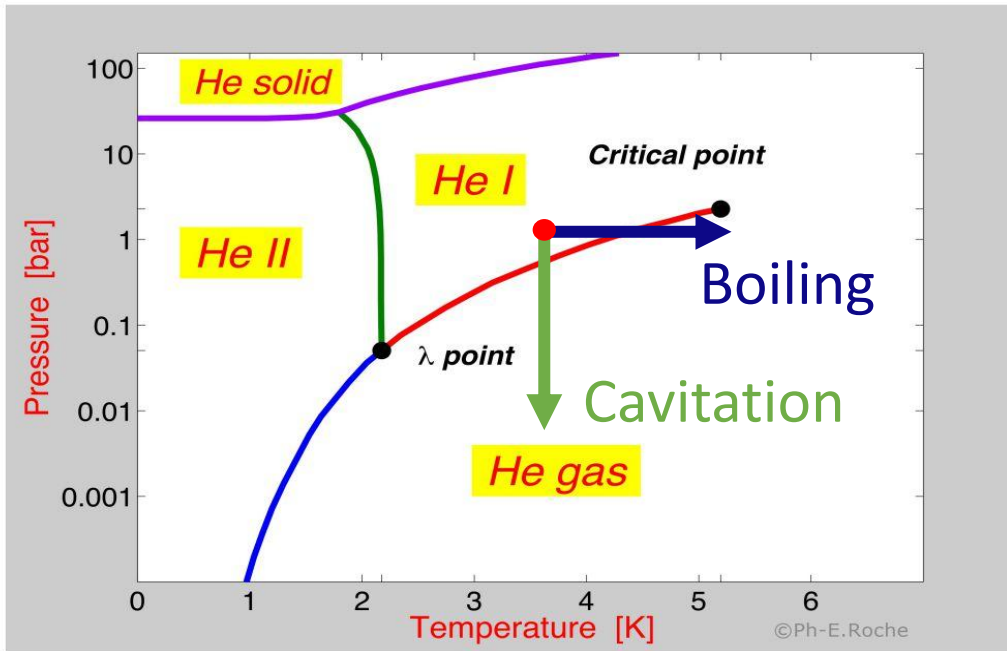
Institut Néel, Université Grenoble Alpes:

➤ [Paul COUTIN](#), Laurent Cagnon, Benoit Chabaud, Thierry Crozes, Jean-Francois Motte, Marine Schott, Panayotis Spathis, Pierre-Etienne Wolf

LPENS, Ecole Normale Supérieure, Paris

➤ Etienne Rolley, Kristina Davitt

Cavitation in liquid ^4He

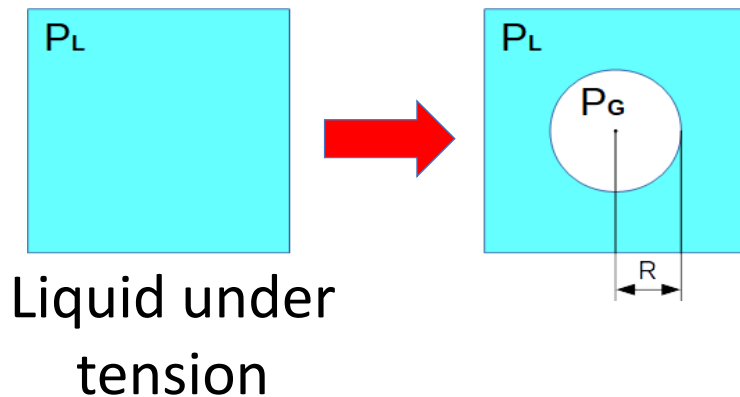


➤ Open Questions on Bubble Nucleation

- Role of surface tension σ in the energy barrier
- Dynamical effects during bubble propagation
- Finite-size effects

➤ Superfluid Phase

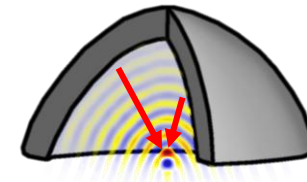
- Possible role of vortices as cavitation nucleation centers



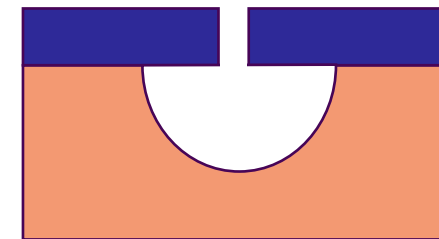
How to reach a metastable state ?

Cavitation is a stochastic process; therefore, experiments must be repeated multiple times to obtain reliable data

- Ultrasonic methods
 - Require sequential repetition of experiments
- Ink-bottle pore geometry
 - $\sim 10^7$ independent pores
 - Allowing massive parallelization of measurements

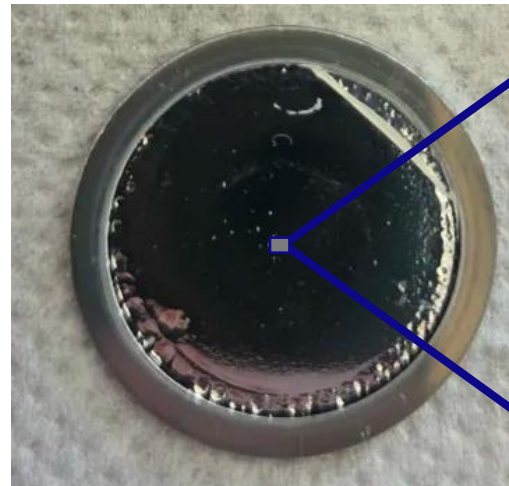
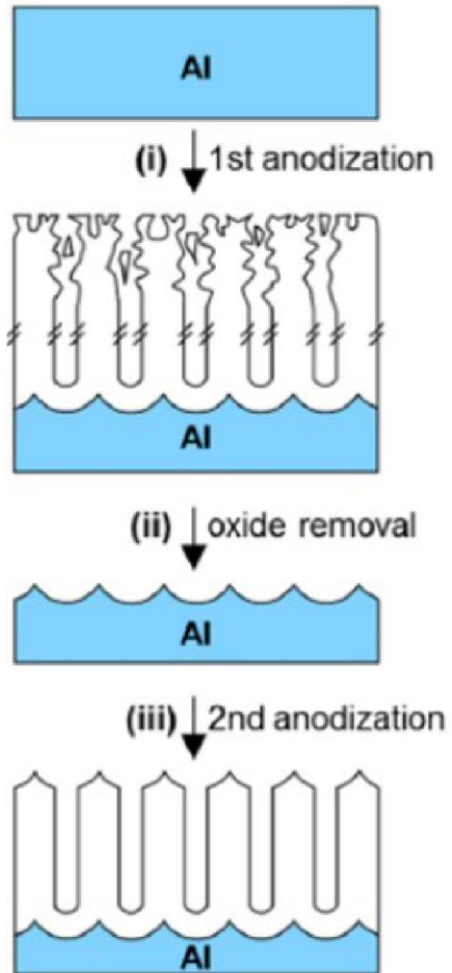


Caupin & Balibar, ENS Paris:
Dynamic Sensing

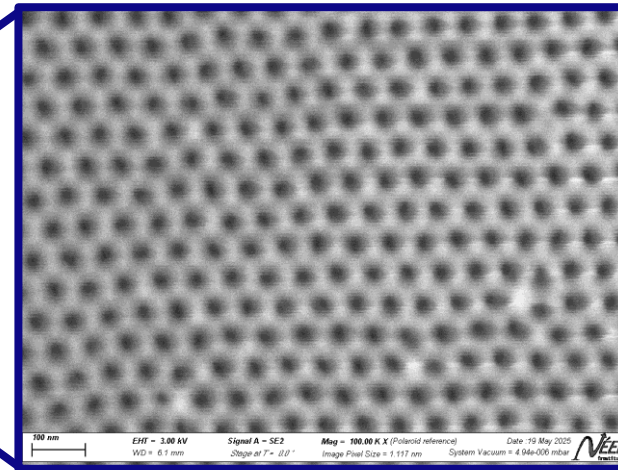


Doebele PRL 2020 & Bossert PNAS 2022:
Ink-Bottle Pores

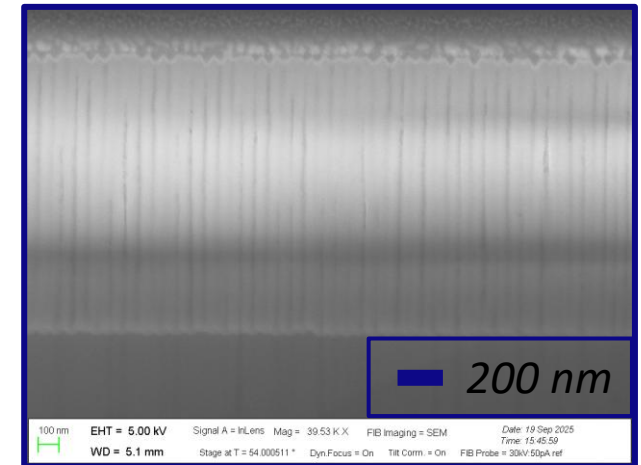
Porous Alumina Samples



Sample:
1 inch aluminium wafer

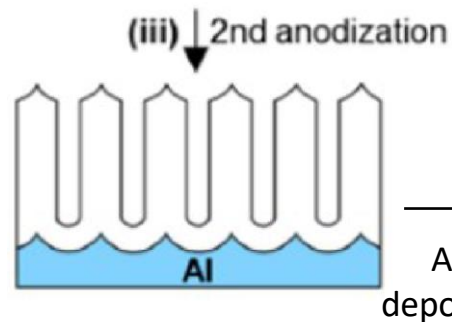
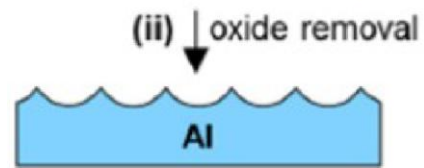
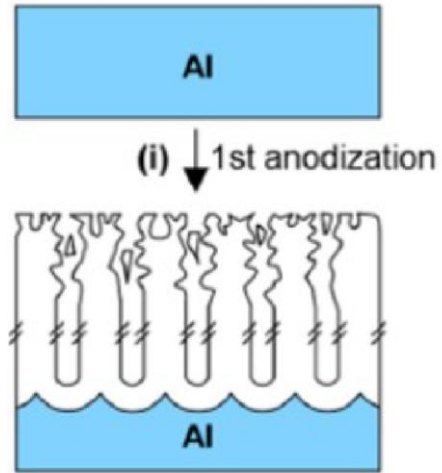


Top view

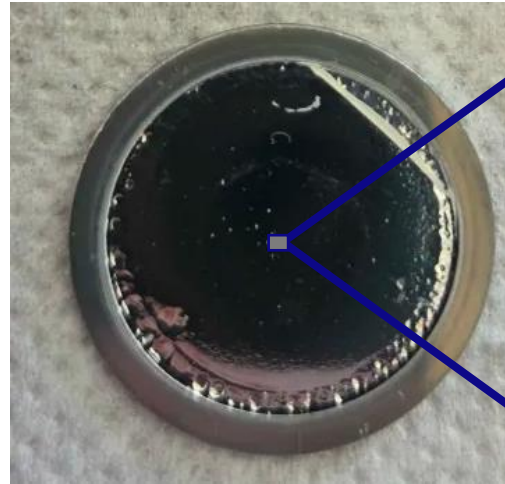


Side view

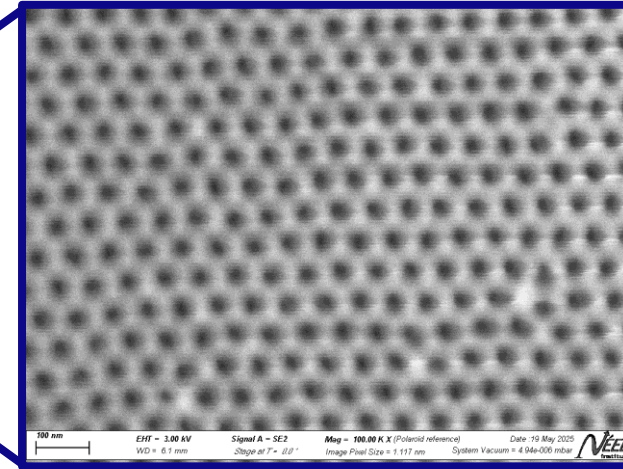
Porous Alumina Samples



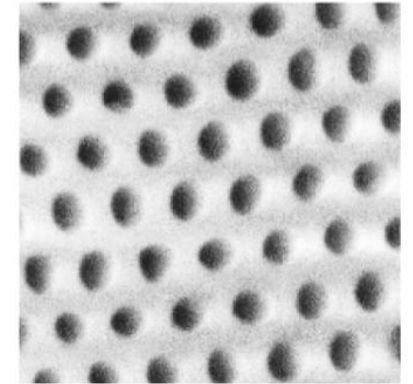
ALD
deposition



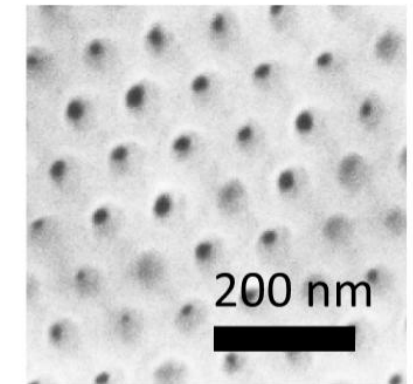
Sample:
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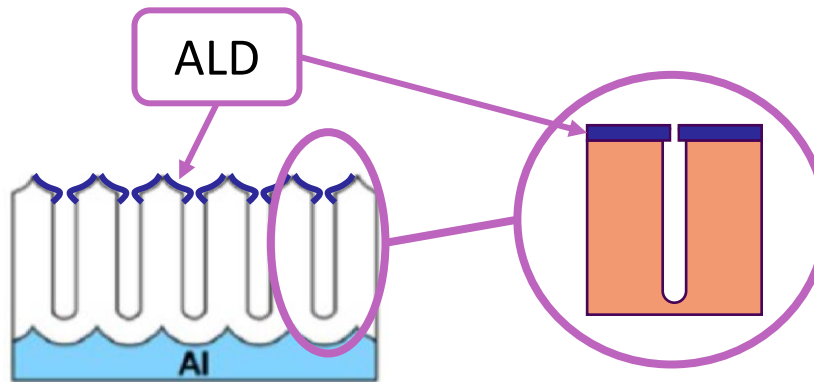
Top view



Before ALD

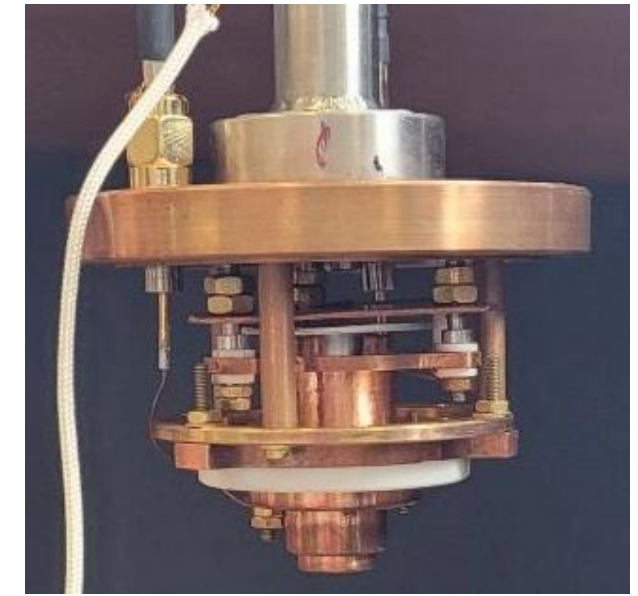
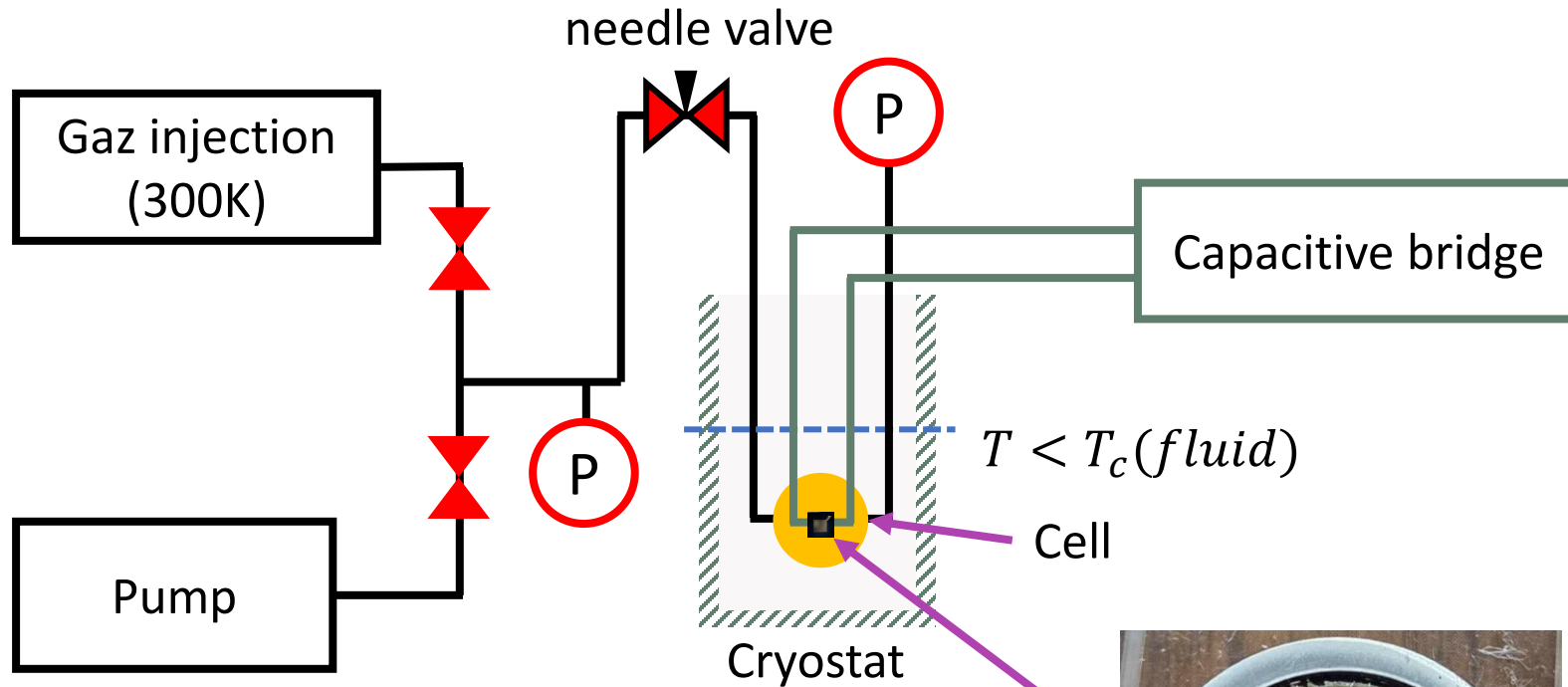


after 160cycles of ALD



➤ ALD deposition is required to obtain the ink-bottle shape

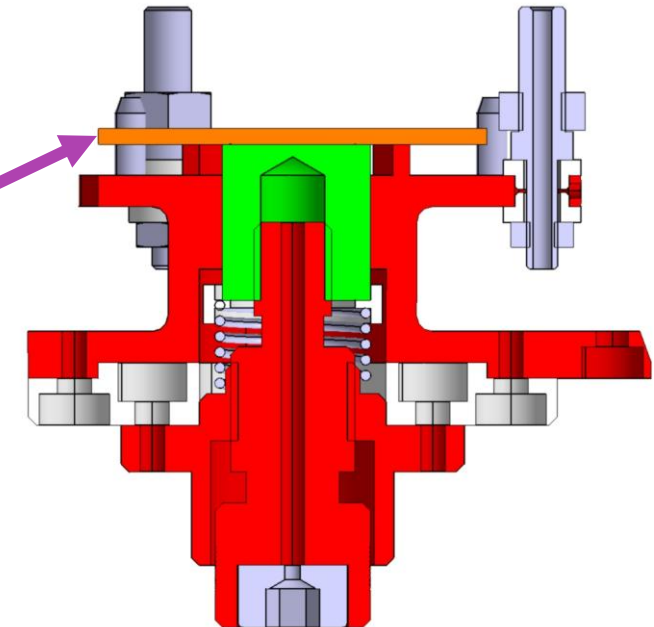
Setup for Isotherm Measurements



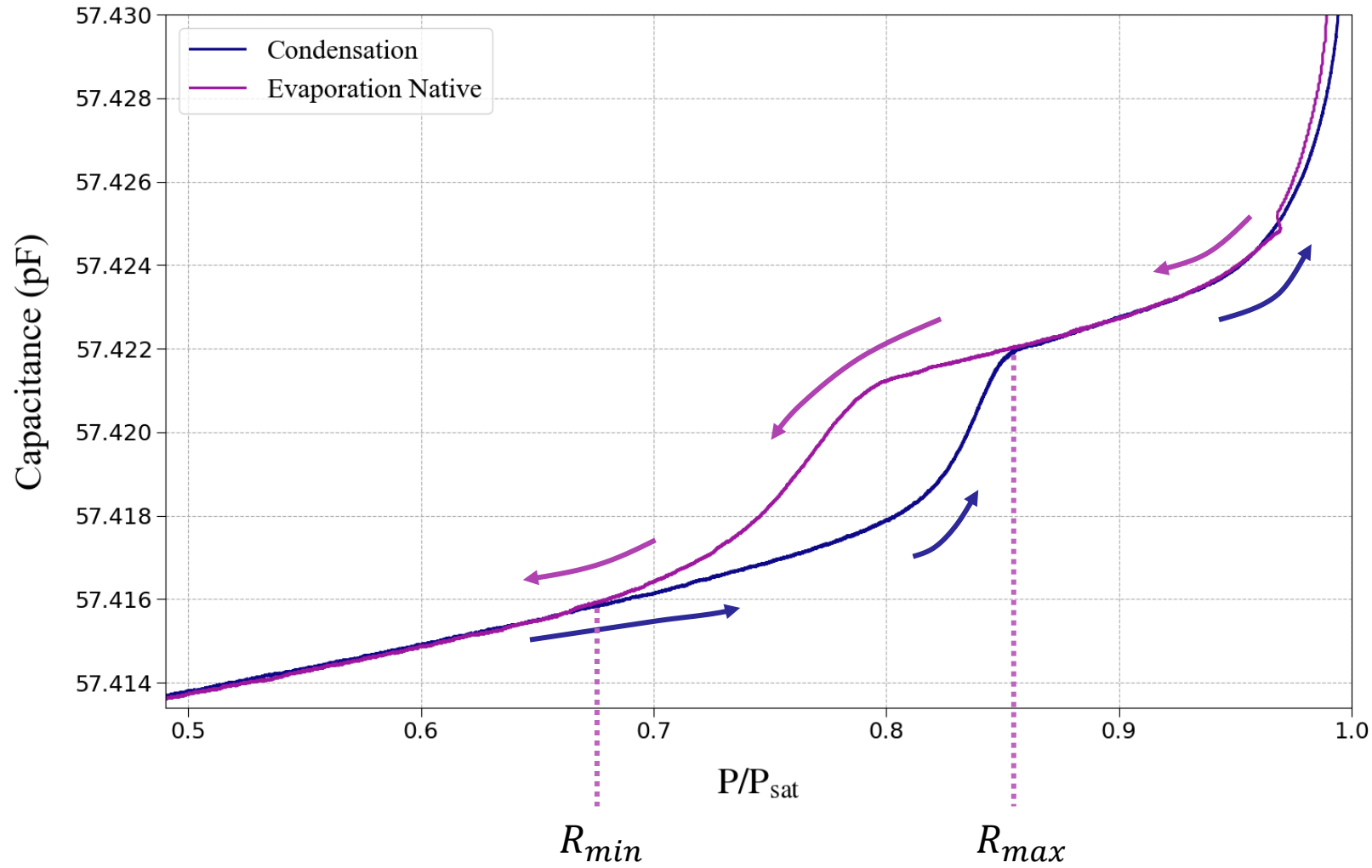
➤ Continuous Injection



Sample

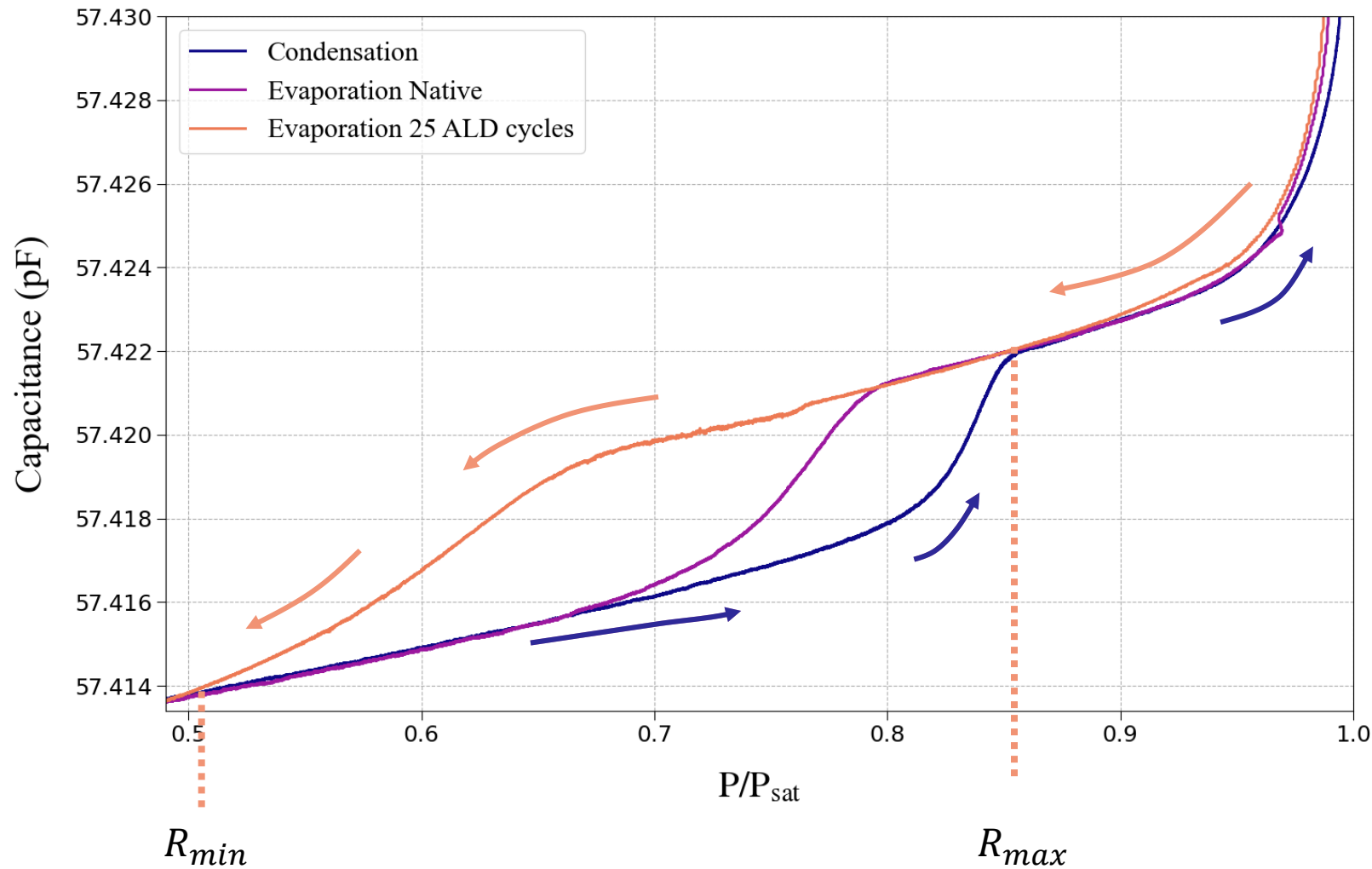


Native Porous Membranes



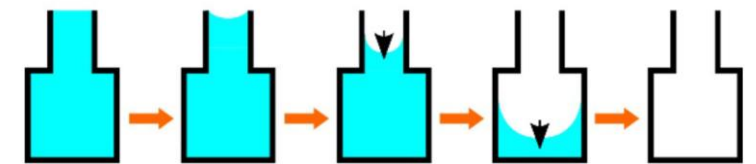
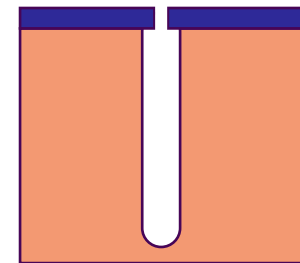
- Independent Pores
- Corrugated conical pores

Same Porous Membrane After ALD Deposition



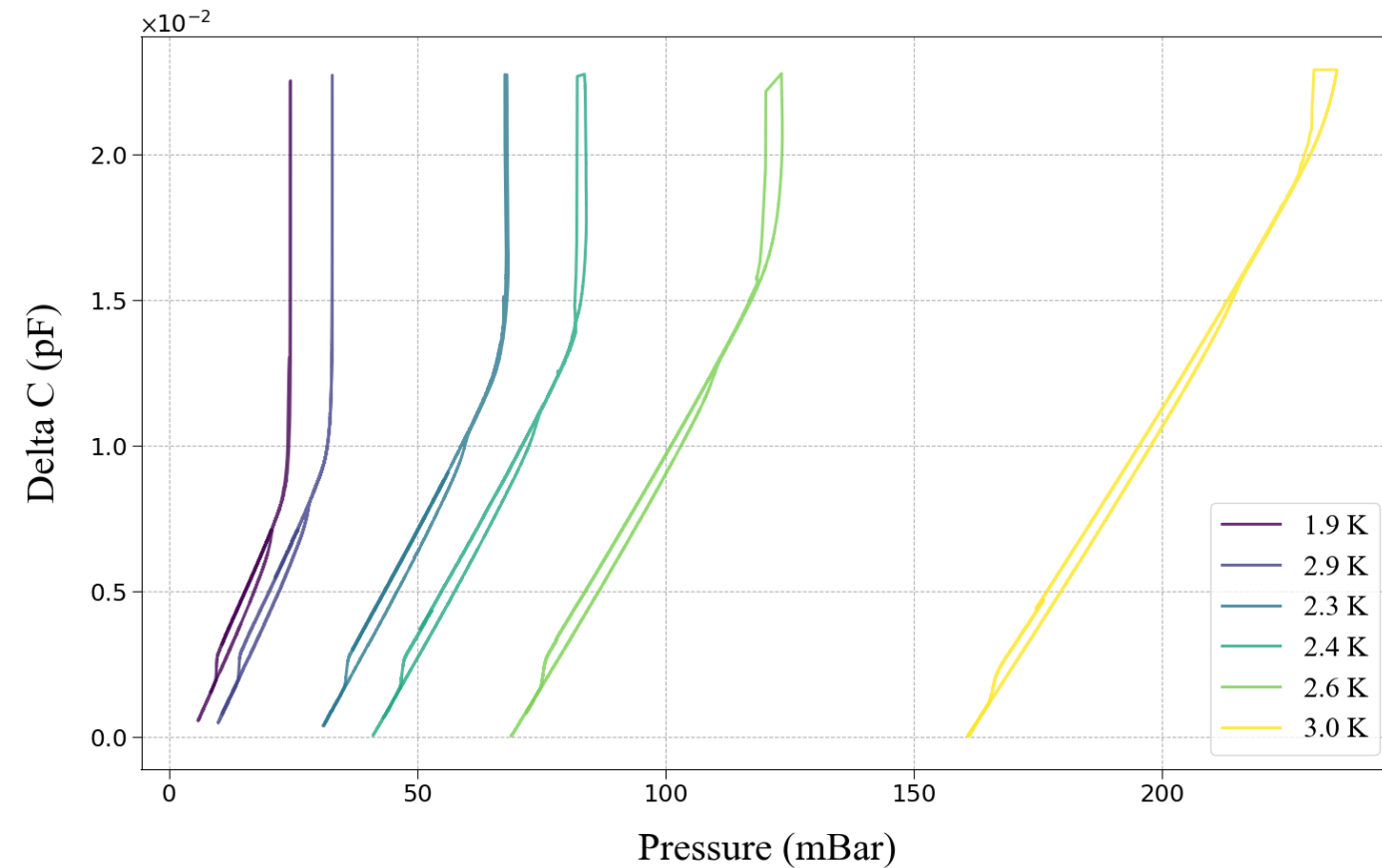
Same condensation branch,
evaporation shifts to lower
pressure:

➤ Ink bottle geometry



➤ 1 ALD cycle => 1.2Å

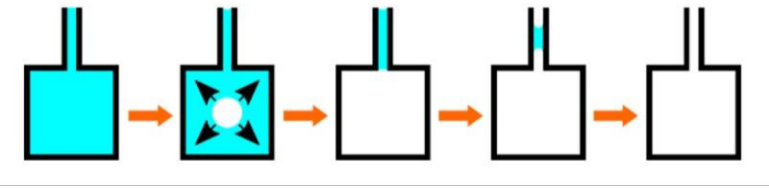
Reaching cavitation



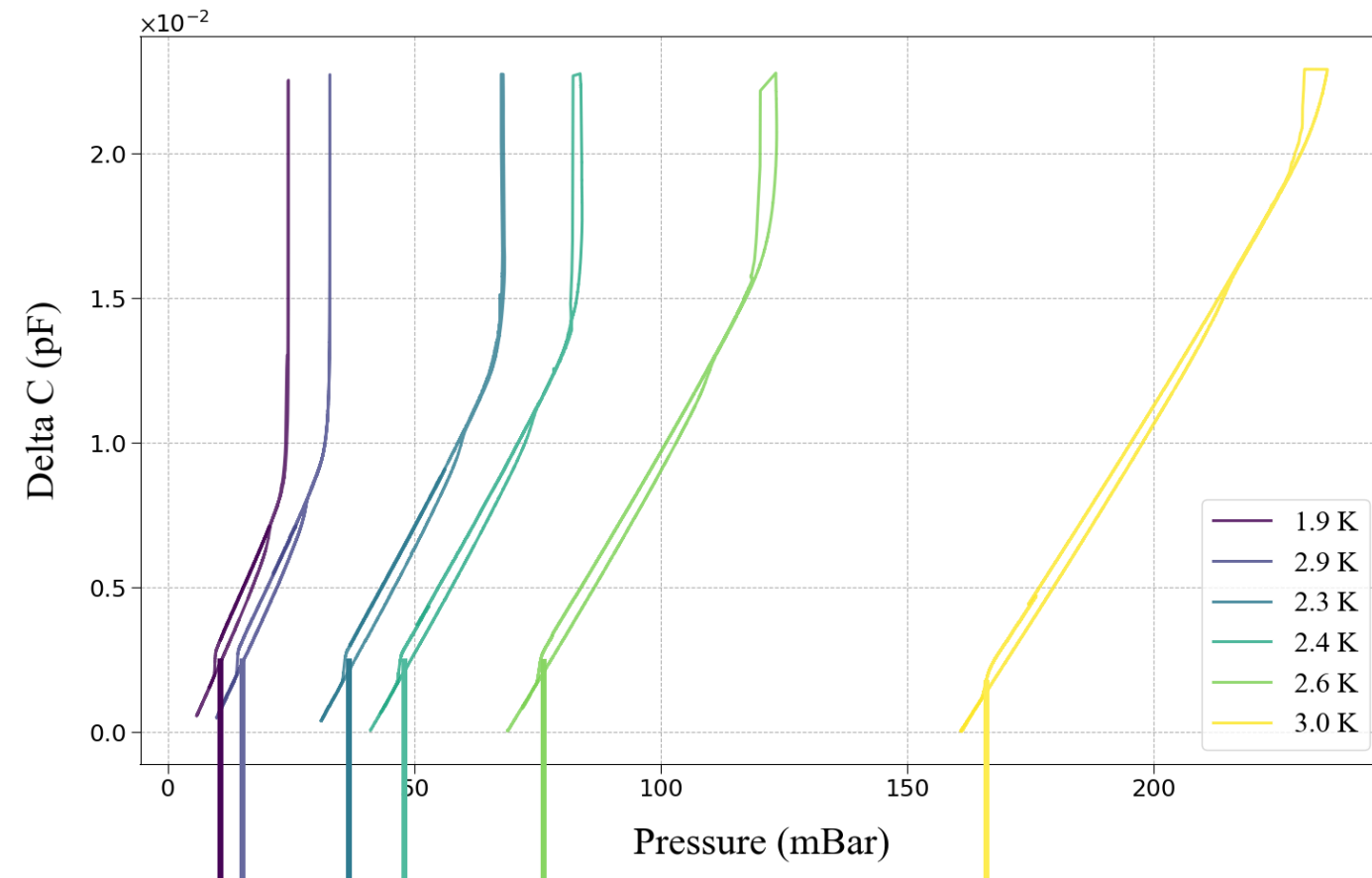
➤ Sorption isotherms at different temperatures (1.9 – 3K)

➤ Evaporation proceeds through cavitation

➤ $R_{\text{bottleneck}} \approx 1\text{nm}$



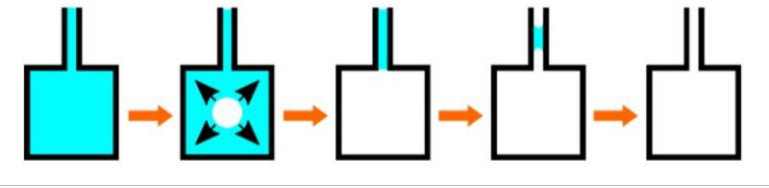
Reaching cavitation



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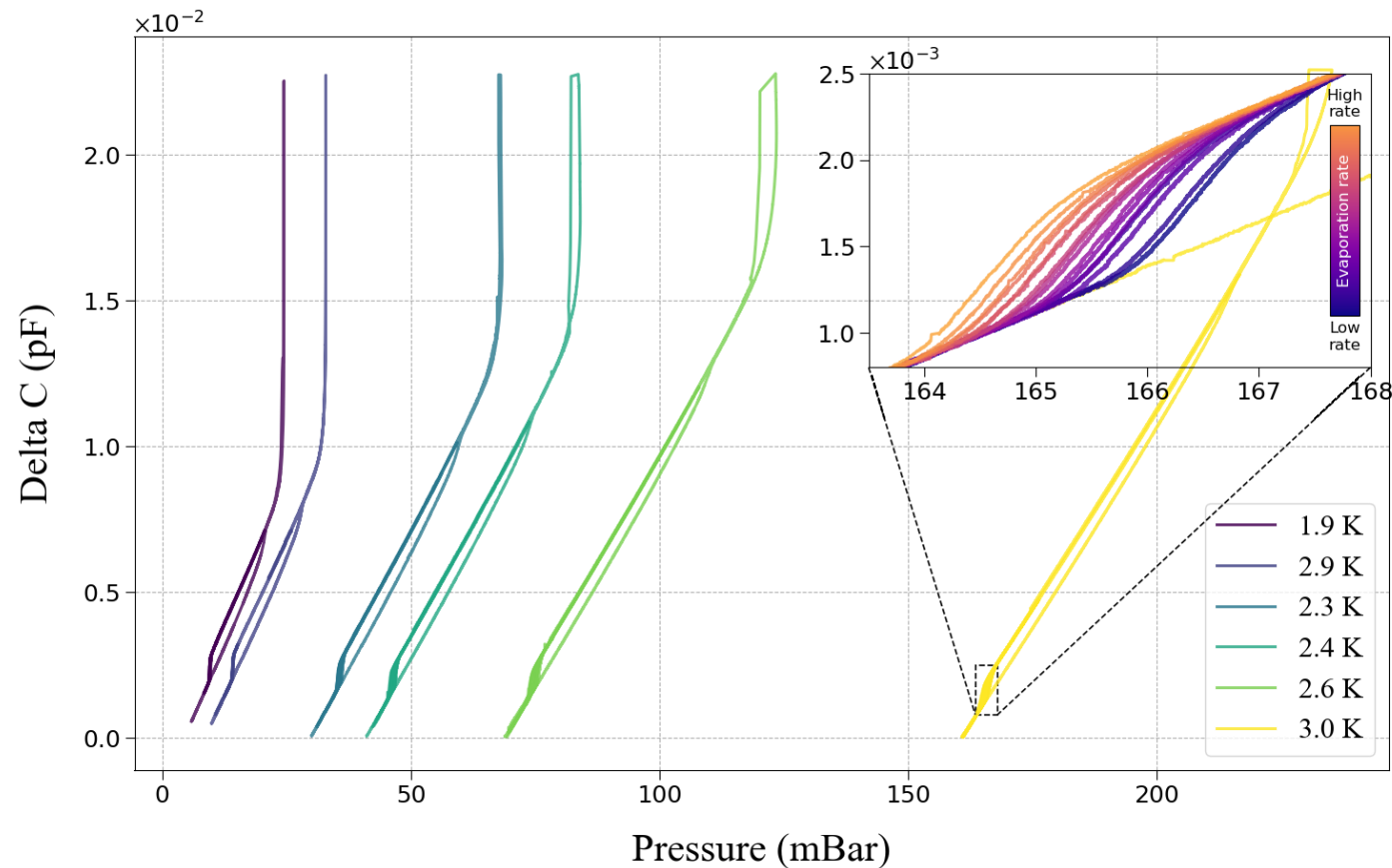
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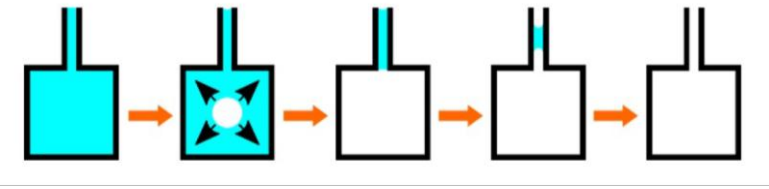


➤ Extraction of the cavitation threshold $P_{cav}(T)$

Reaching cavitation

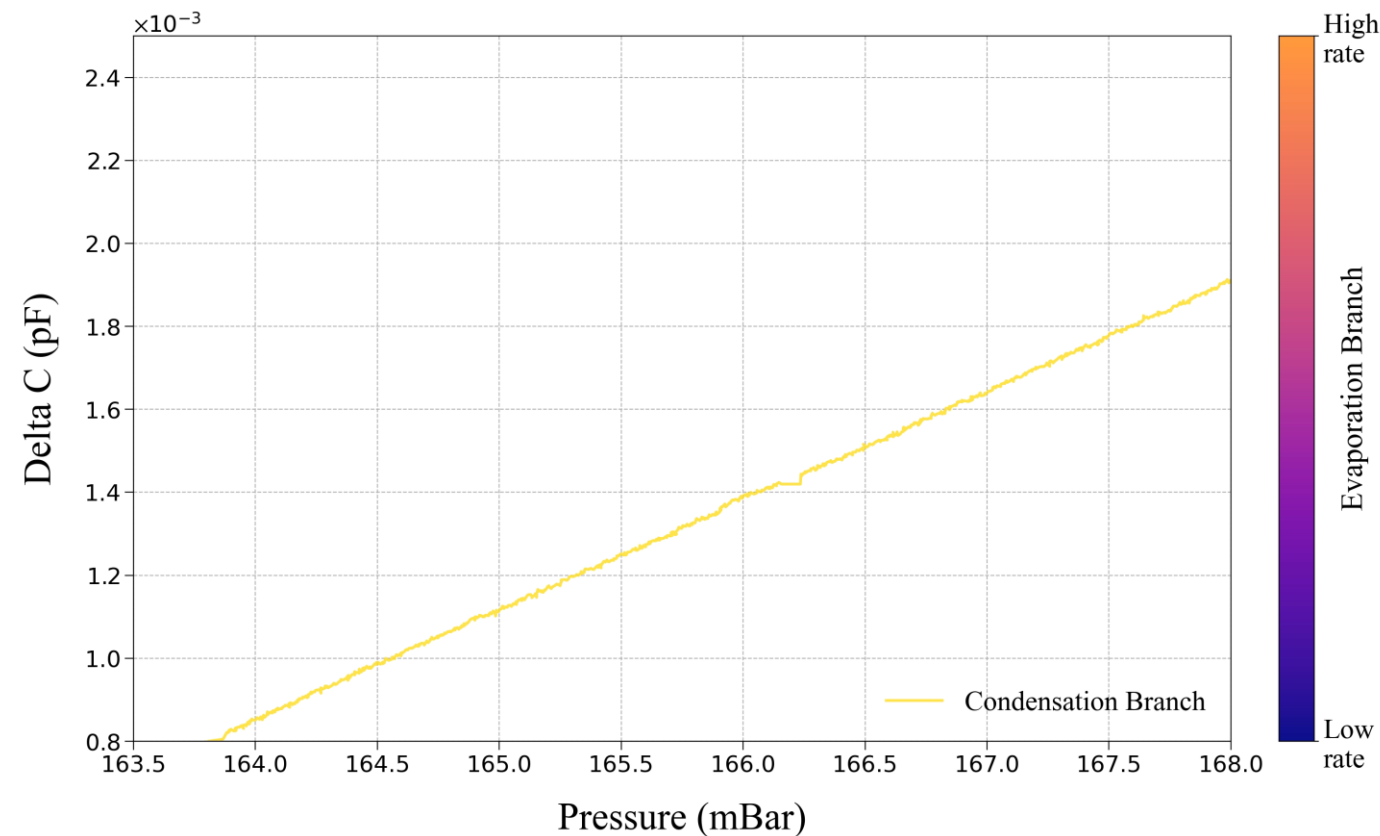


- Sorption isotherms at different temperatures (1.9 – 3K)
- Evaporation proceeds through cavitation
 - $R_{\text{bottleneck}} \approx 1\text{nm}$
- Extraction of the cavitation threshold $P_{\text{cav}}(T)$
- The same isotherm is measured at different pressure sweep rates (dP/dt)



Cavitation Analysis Through Controlled Pressure Rates

$$\text{Rate } A = -dP_L/dt$$



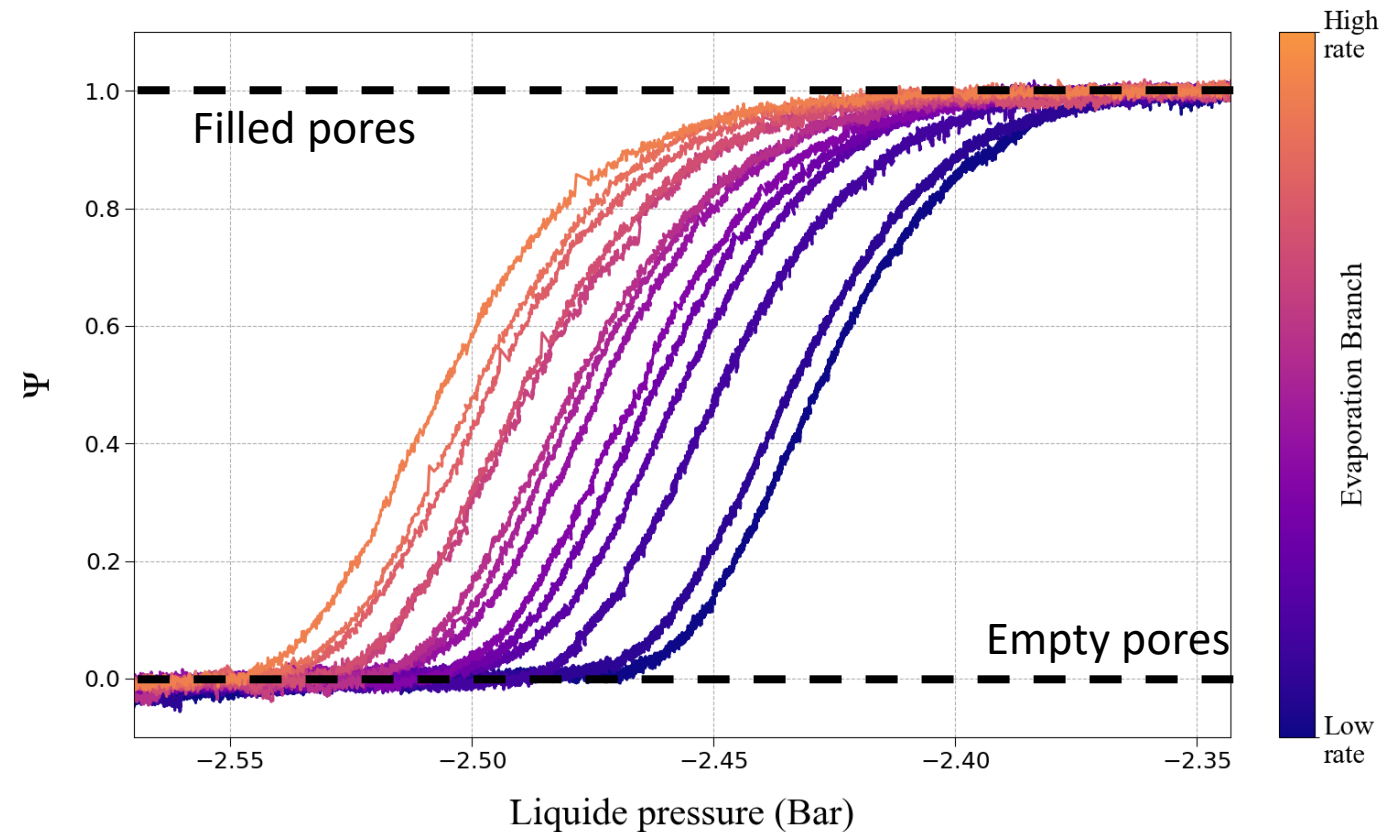
ASSUMING the capacitance is proportional to the mass of adsorbed fluid:

- Normalize the data to obtain the fraction ψ of filled pores as a function of P
- Subtract the condensation branch
- Convert the gaseous pressure to liquid pressure with $\mu_g = \mu_L$

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By varying the pressure sweep A , we determine for each isotherm the experimental barrier energy E_B :

$$E_B = k_B T \ln \left(\frac{J_0 V_{\text{exp}} \tau_{\text{exp}}}{\ln 2} \right)$$

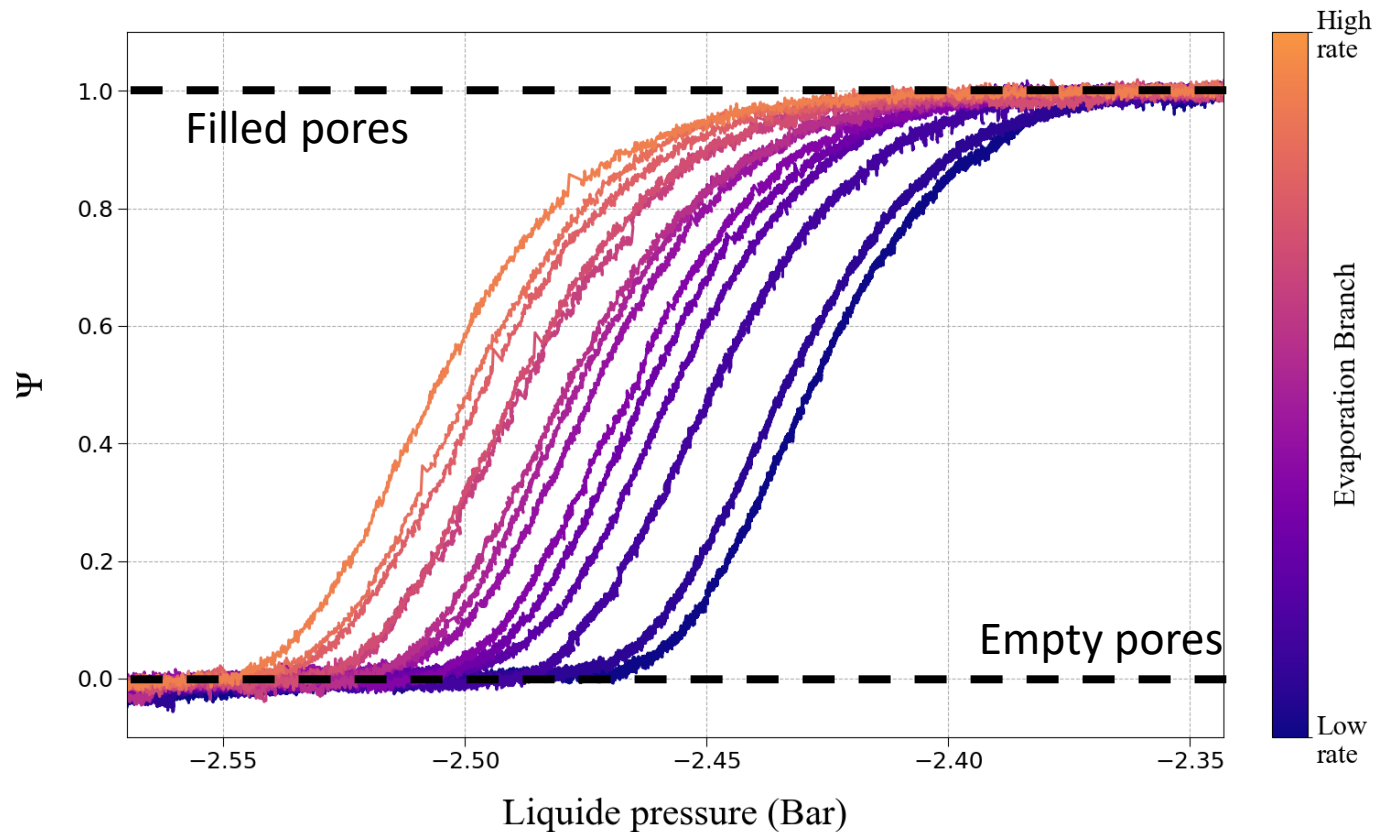
where:

J_0 : prefactor assumed constant over the full sweep-rate range

V_{exp} : fixed by the geometry of the porous sample

τ_{exp} : controlled through the pressure sweep rate during cavitation

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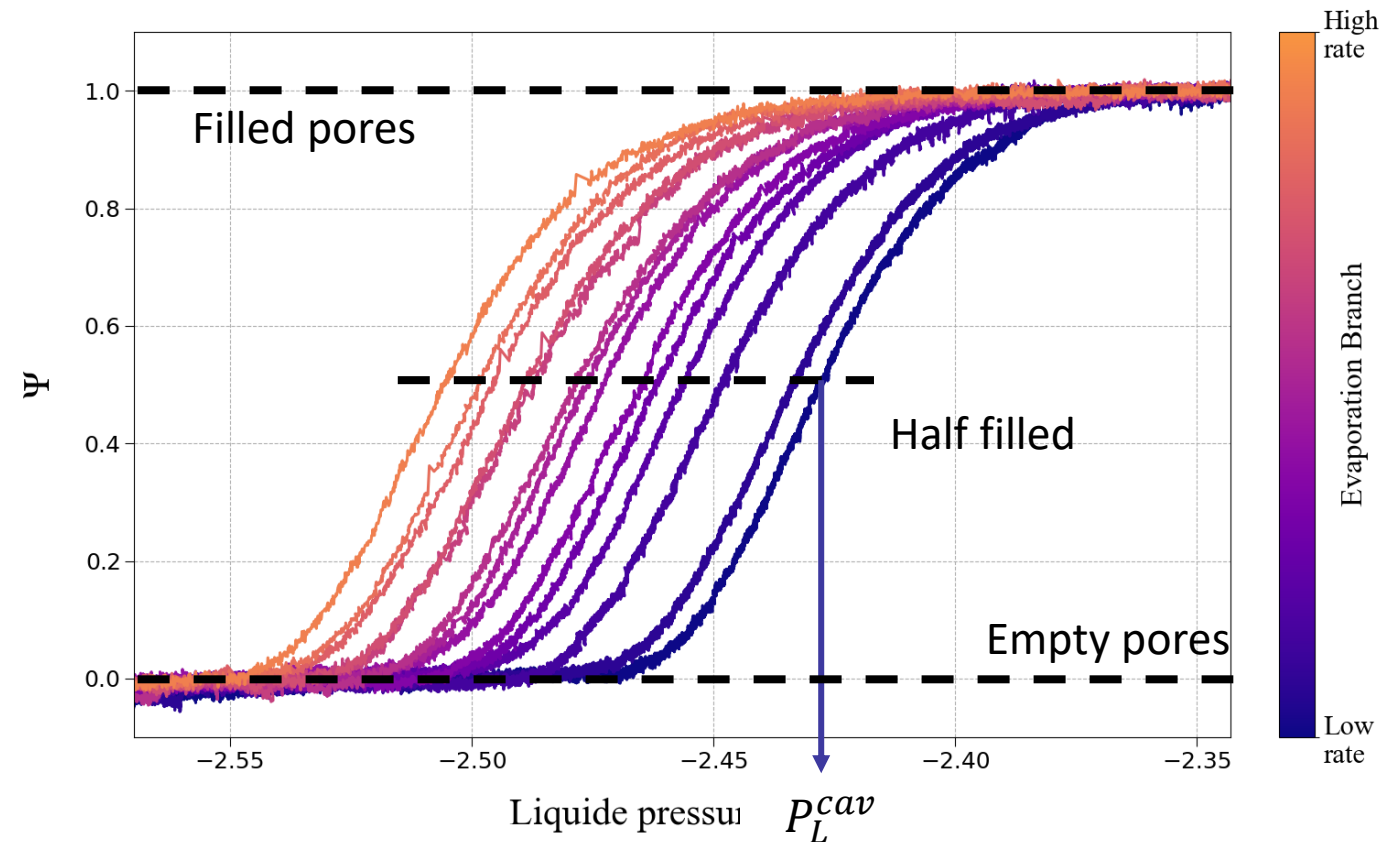
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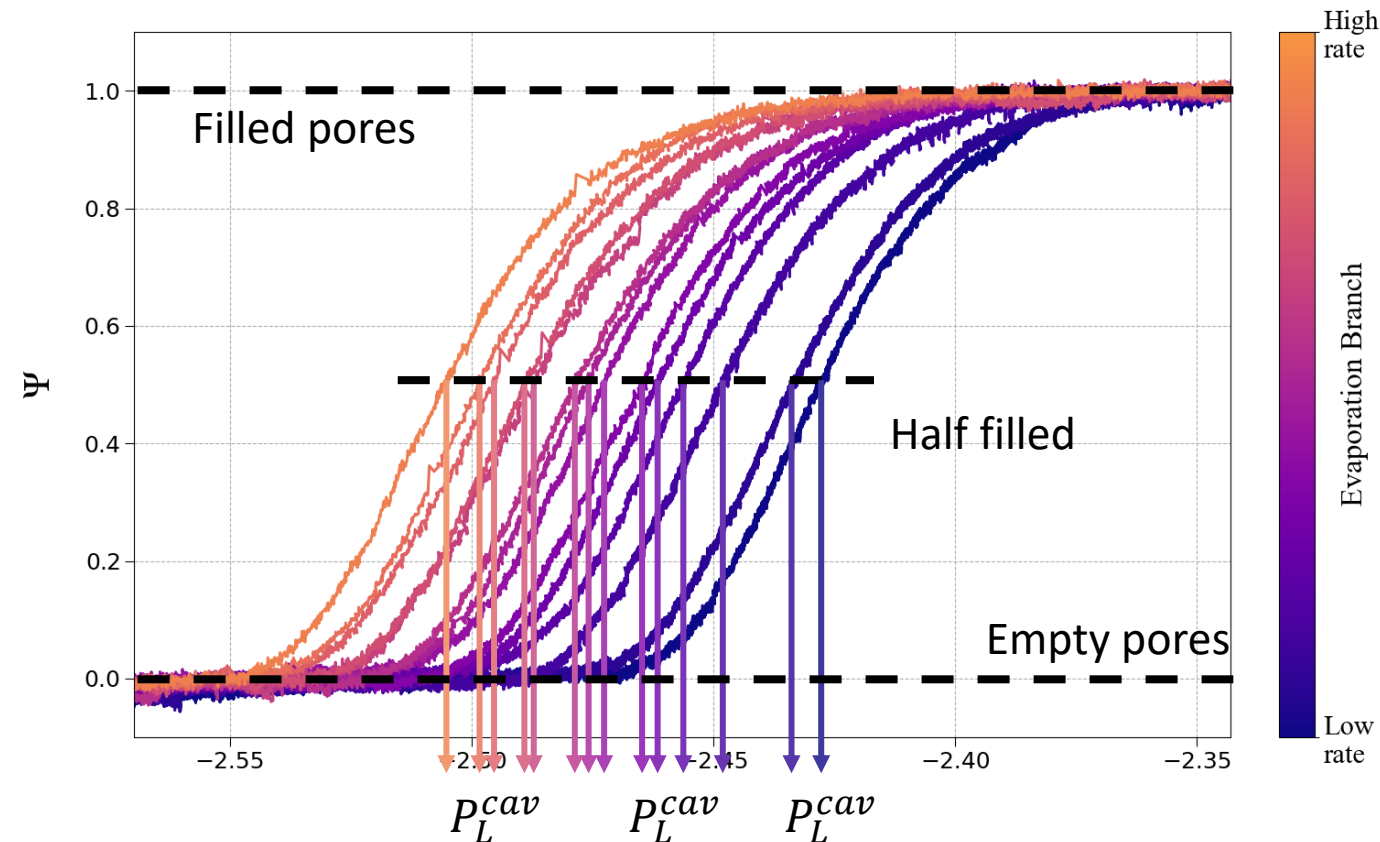
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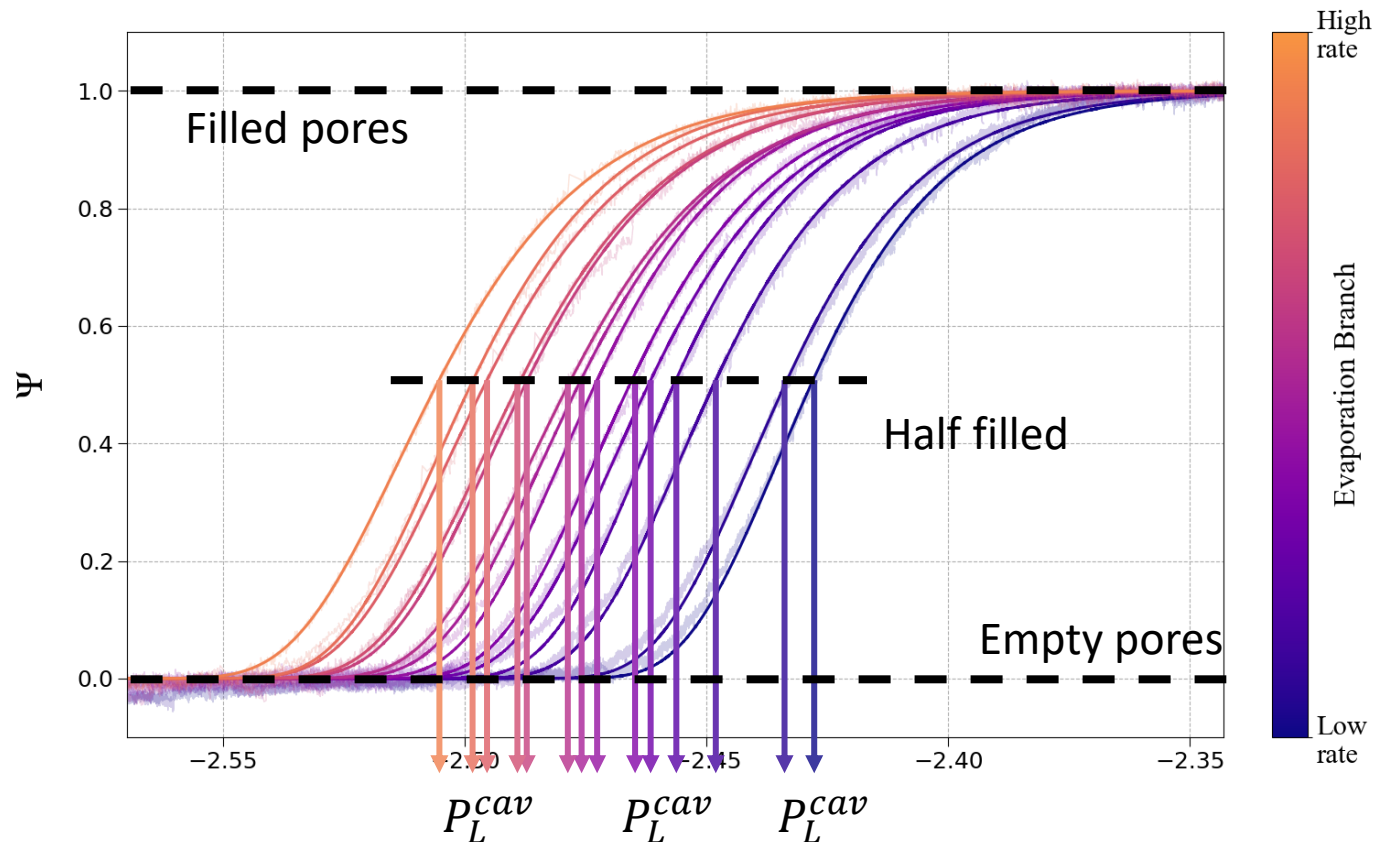
τ_{exp} : controlled through the pressure sweep rate during cavitation

For all rates, the pore distribution is fitted by:

$$\ln \psi = \ln(1/2) e^{-\alpha(P_L - P_L^{\text{cav}})}$$

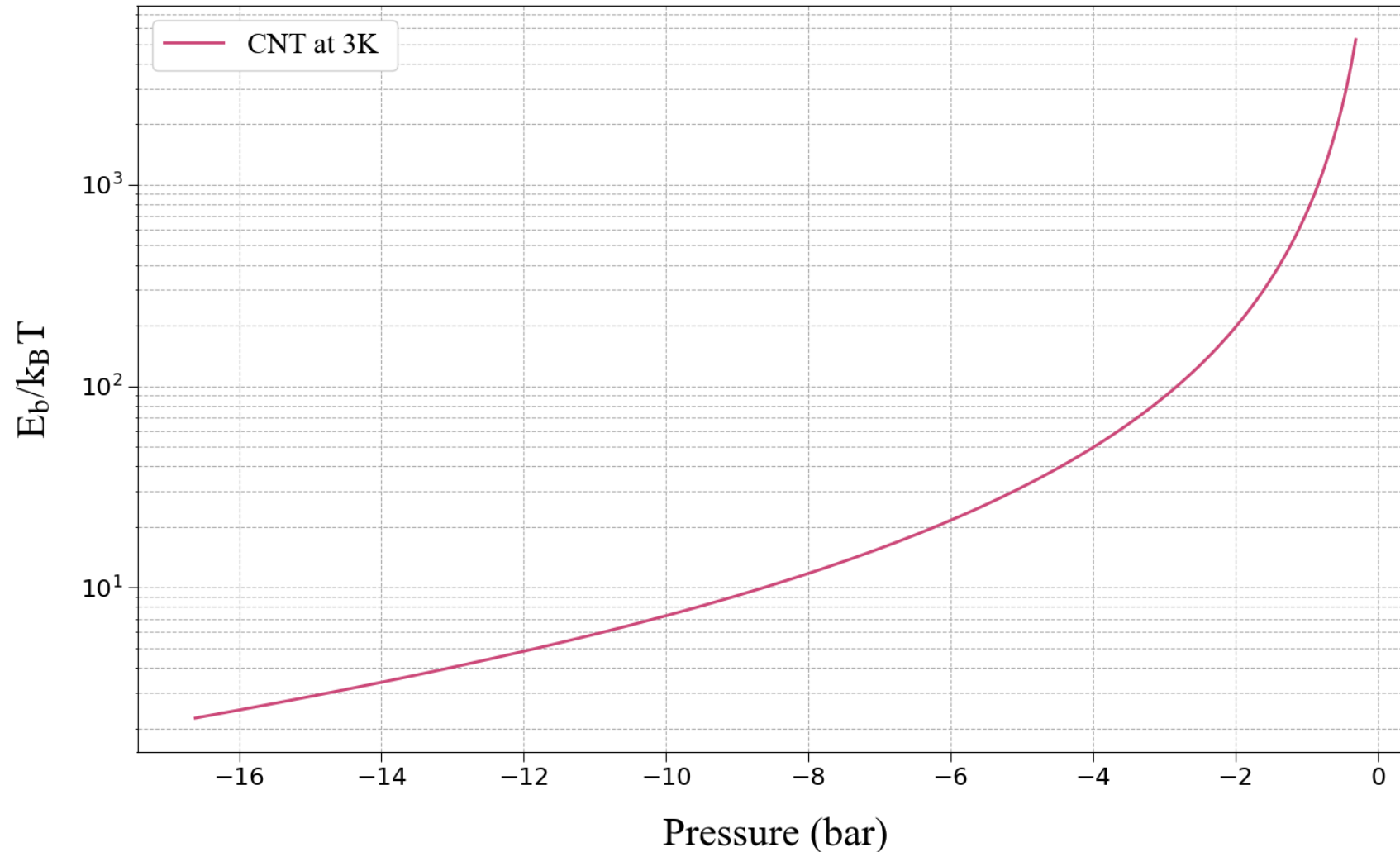
With $\alpha = -\frac{d(\ln A)}{dP_L^*} = \frac{1}{k_B T} \frac{dE_b}{dP_L}$

Rate $A = -dP_L/dt$

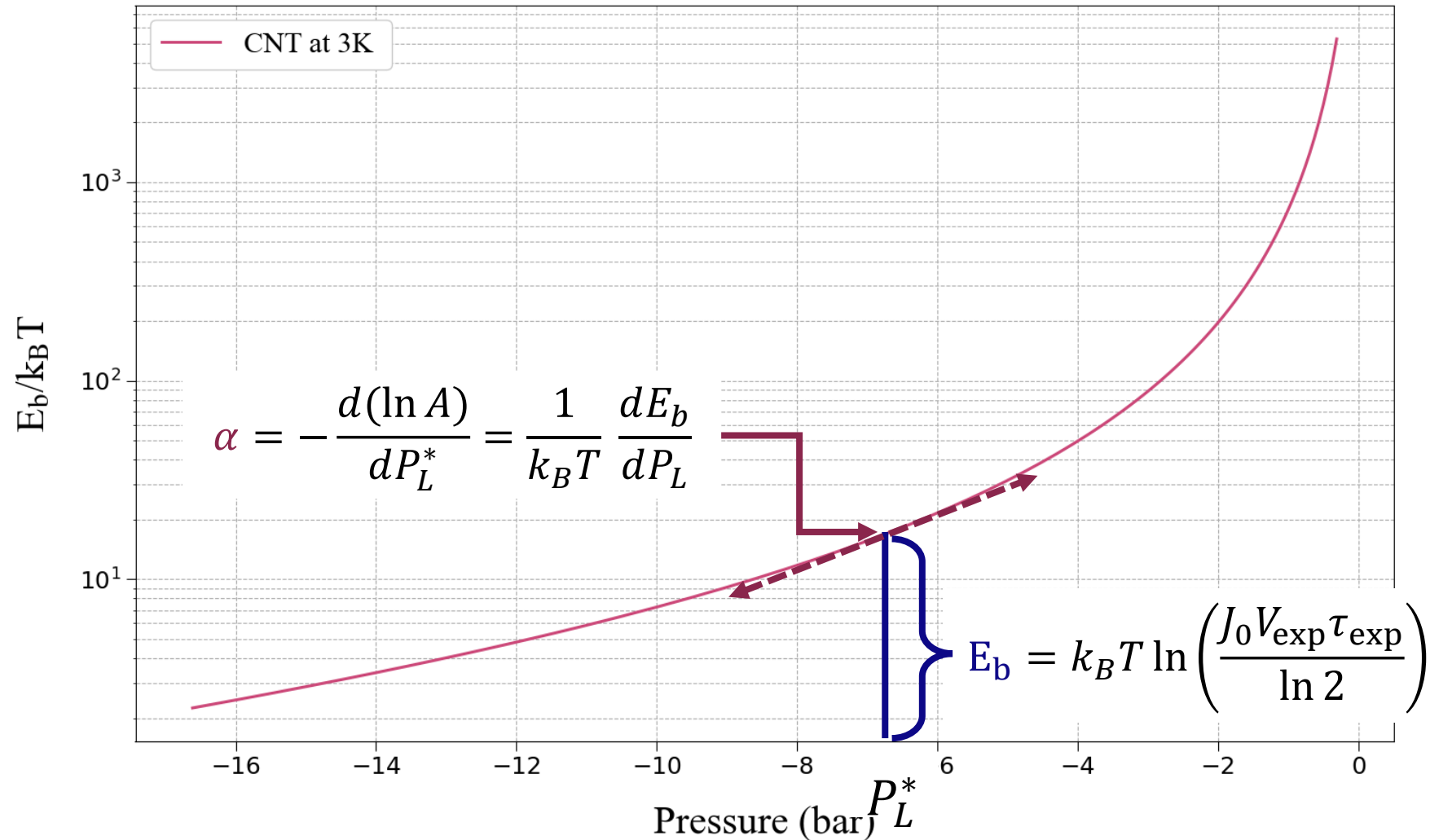


➤ The same isotherm is measured at different pressure sweep rates (dP/dt)

Estimating the experimental Barrier Energy and Local Slope



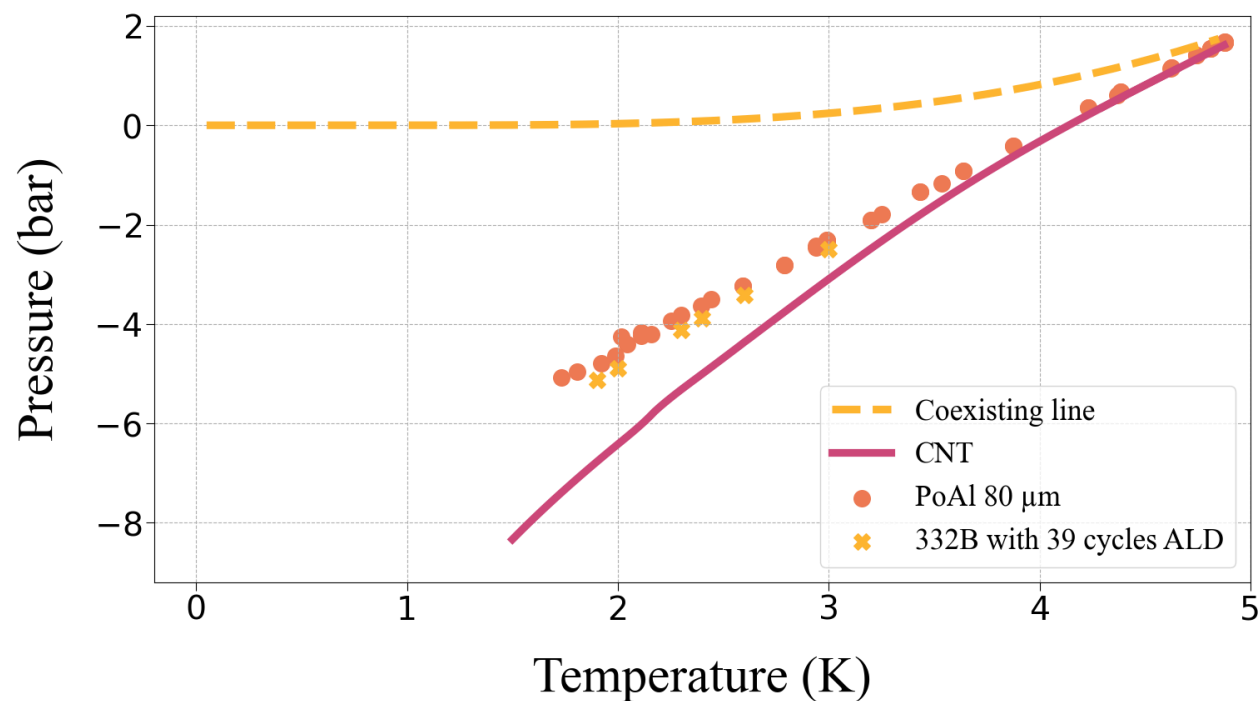
Estimating the experimental Barrier Energy and Local Slope



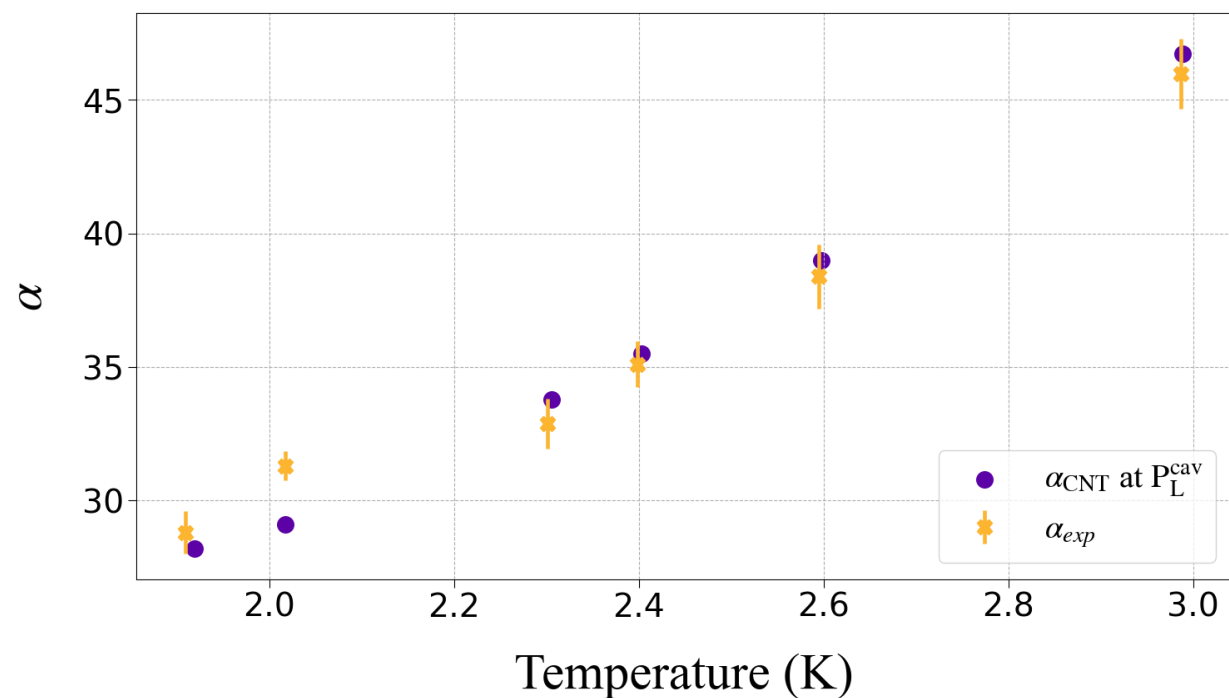
Comparison with theory

CNT (+ σ bulk) not valid below 4K

$E_b < E_b^{\text{CNT}}$ below 4K



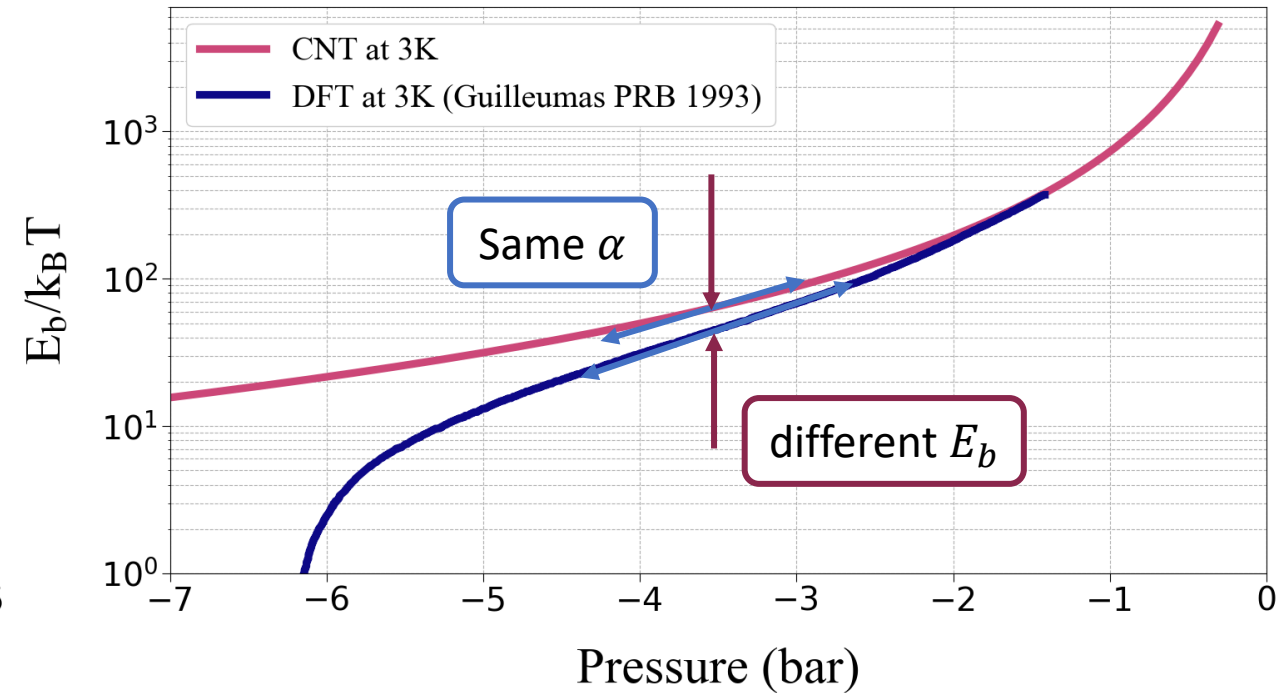
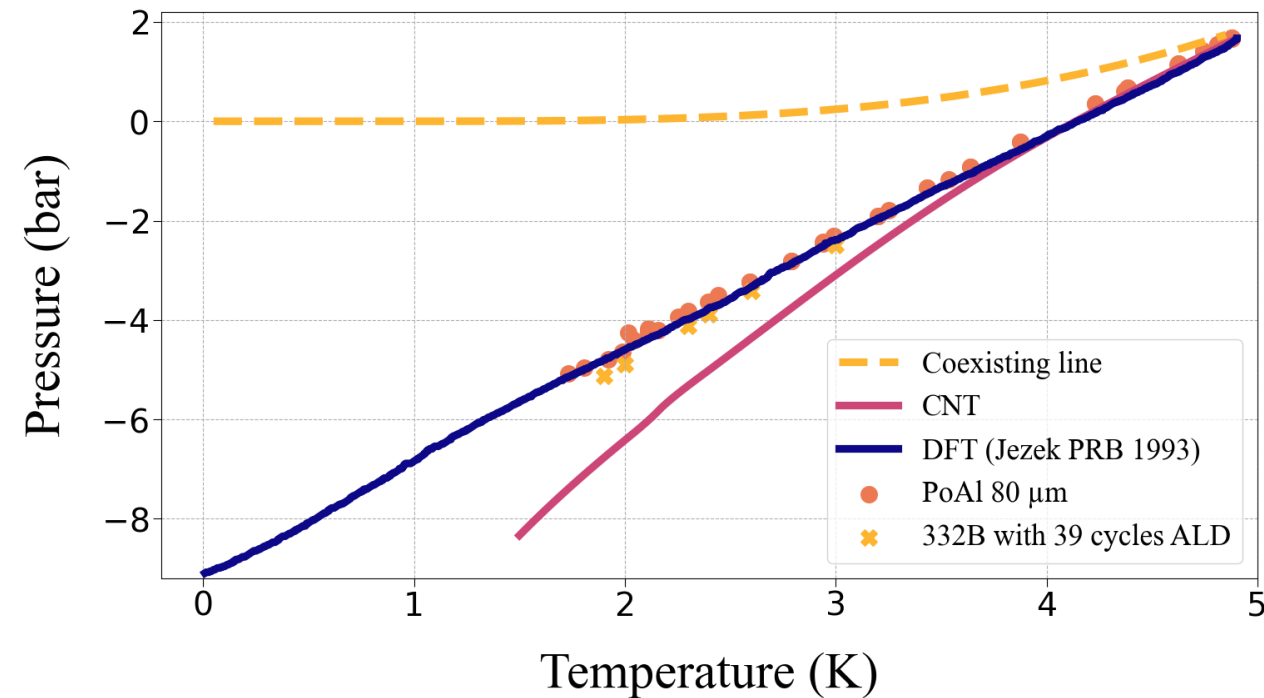
$\alpha \sim \alpha^{\text{CNT}}$



Comparison with theory

CNT (+ σ bulk) not valid below 4K

➤ We have a better fit with the DFT



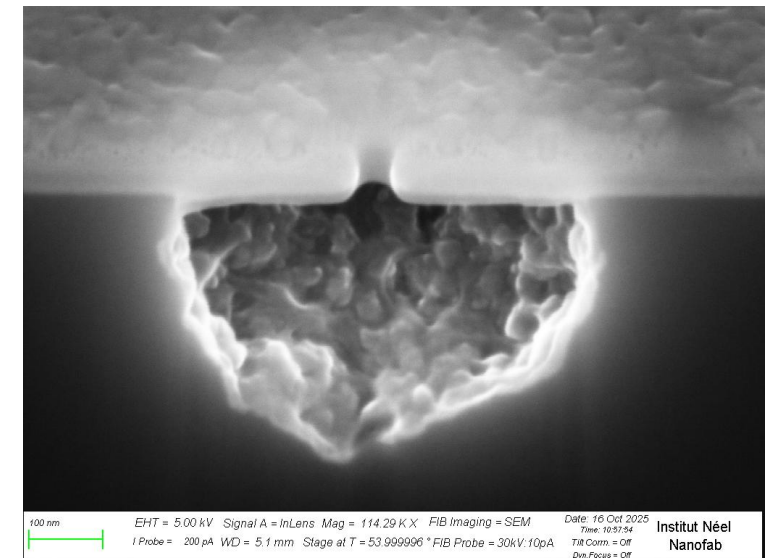
Conclusions and Perspectives

➤ Conclusions

- CNT with bulk surface tension does not describe the low-temperature regime
- Experimental measurements agree with DFT predictions
- No observable signature of vortex-assisted nucleation

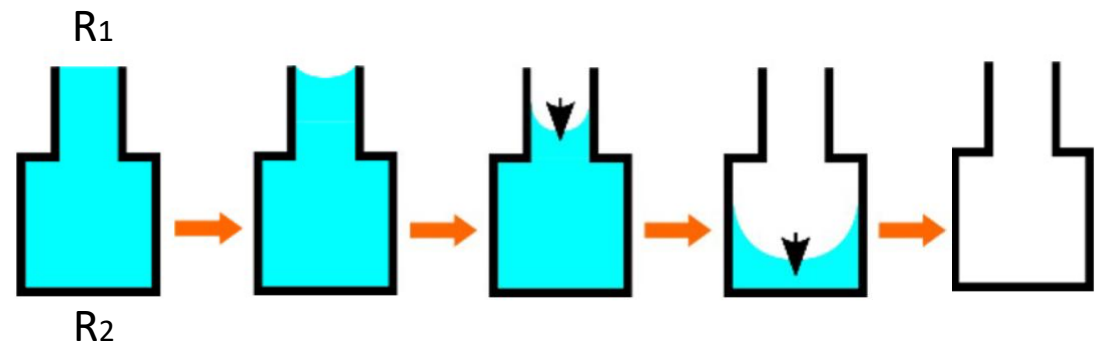
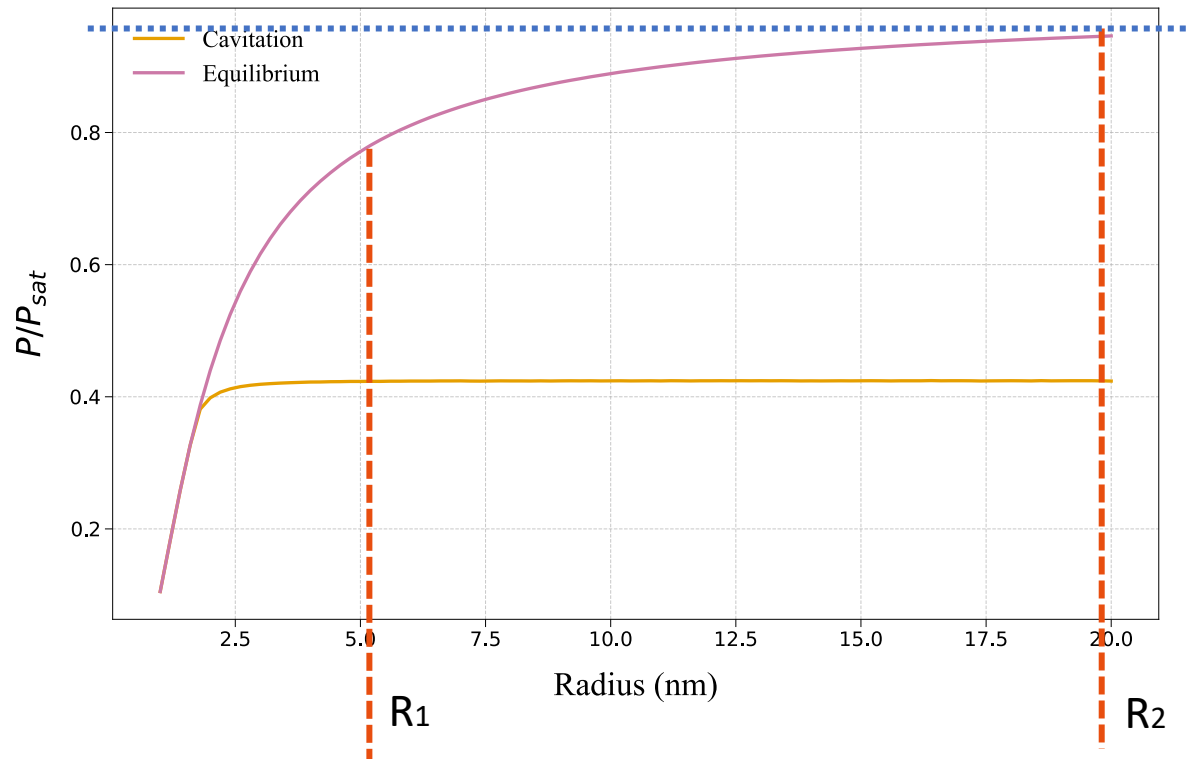
➤ Perspectives

- Study of cavitation closer to the critical point
- Enhanced investigation of confinement effects
- New porous system under investigation



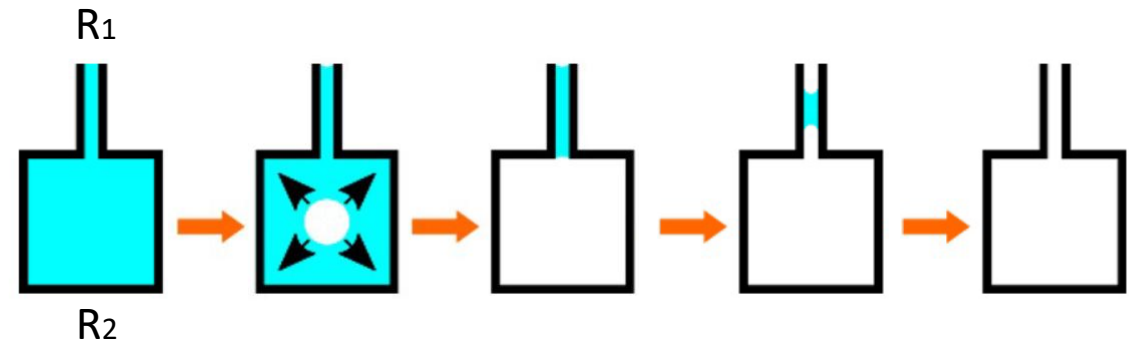
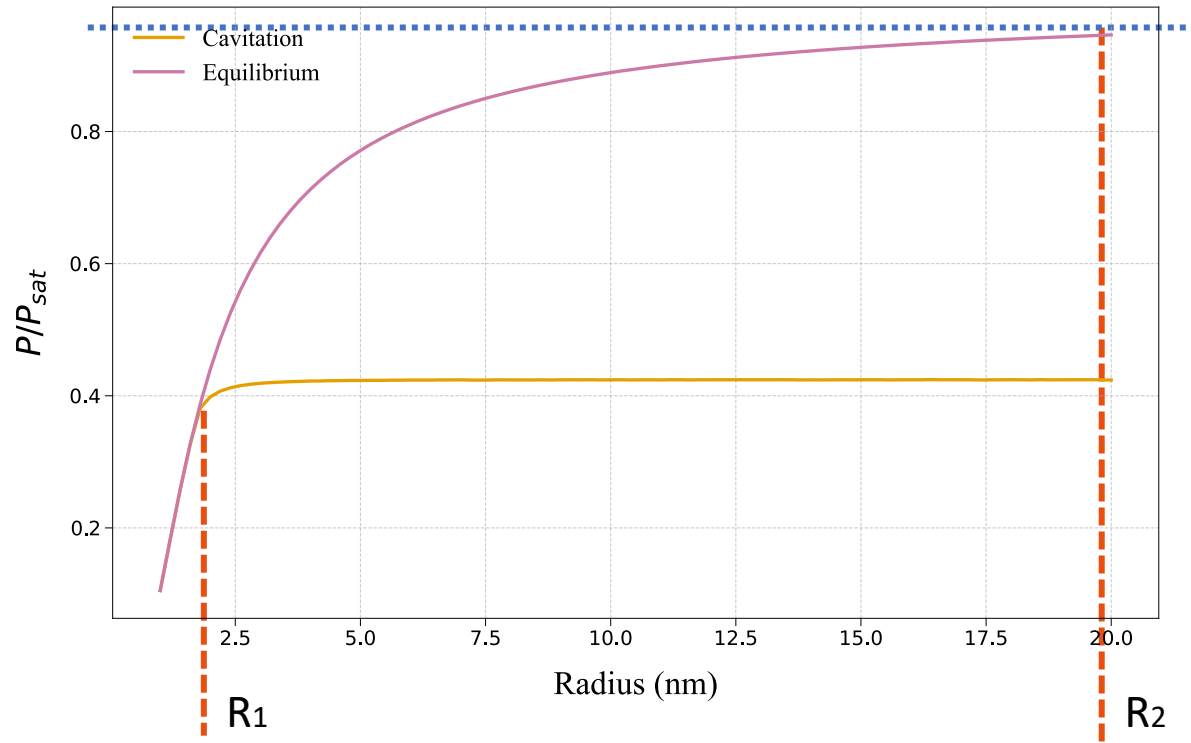
Reaching Cavitation: Using Ink-Bottle Shaped Pores

Normalized Pressure as a Function of Radius

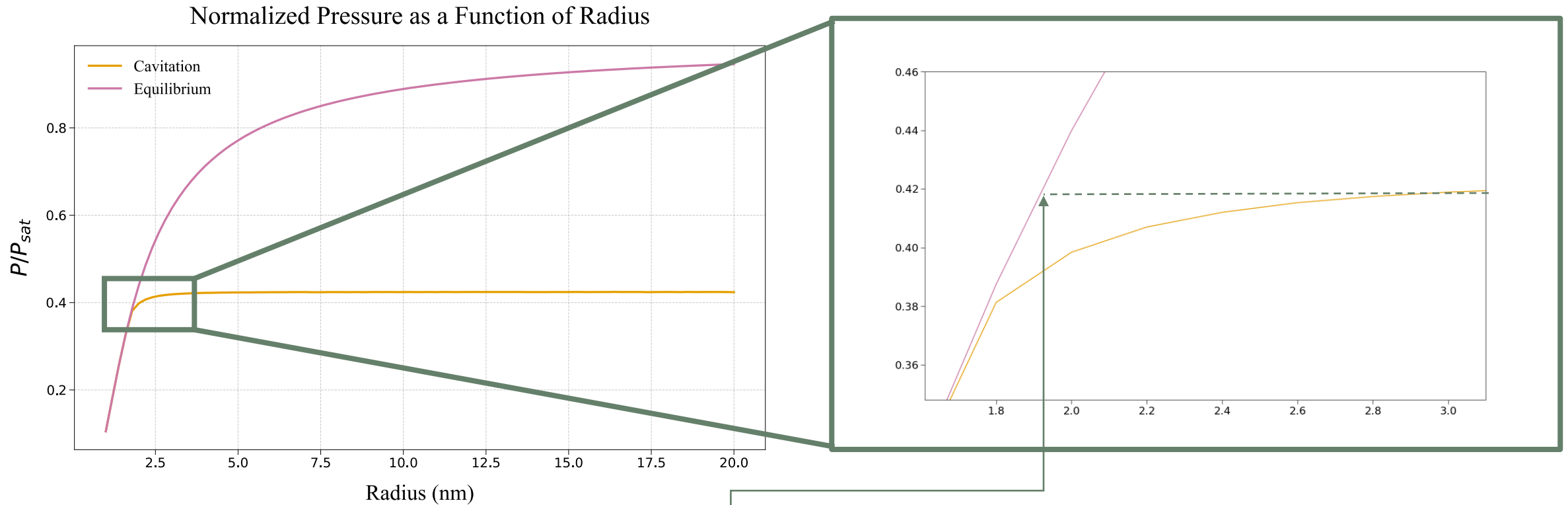


Reaching Cavitation: Using Ink-Bottle Shaped Pores

Normalized Pressure as a Function of Radius



Reaching Cavitation: Using Ink-Bottle Shaped Pores



Minimum Radius to
Observe Cavitation :
 $R \approx 2 \text{ nm}$

- We use an ALD deposition to close the constrictions and obtain this radius value