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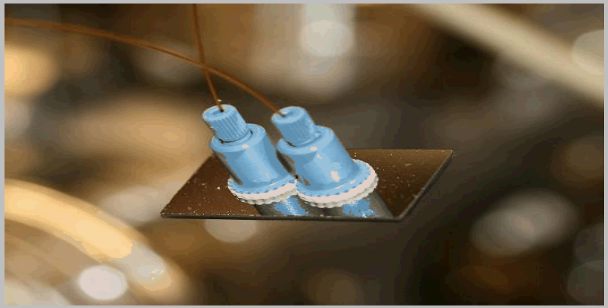

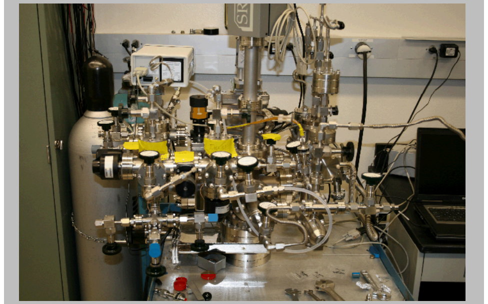

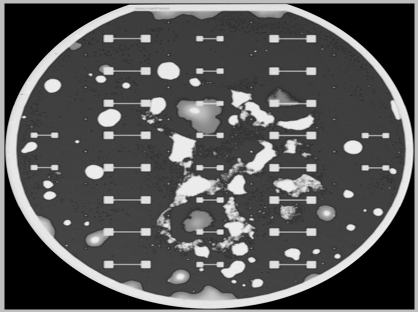

Isothermal Mass Flow Measurements

Through a Rectangular Channel Over a Wide Knudsen Range

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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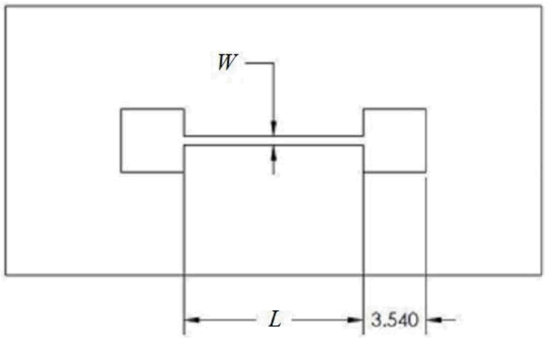
Motivation

- Lack of experimental data for flow measurements in rectangular channels at high Knudsen numbers (>50) to understand micropump and microvalve performance and to validate code and models also developed at Sandia National Labs

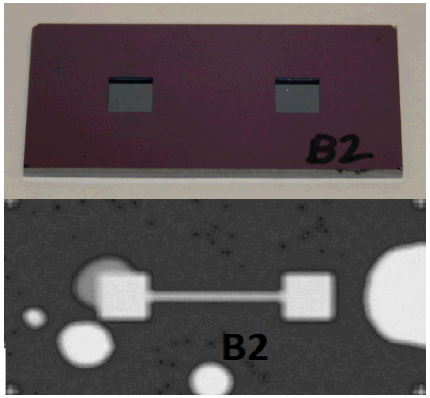
- Design channel such that Knudsen number reaches similar levels seen in micro-electro-mechanical systems(MEMS) and a chip scale vacuum micro-pump(CSVMP) at reasonable levels of vacuum (1 - 10 Pa)

Thesis Overview

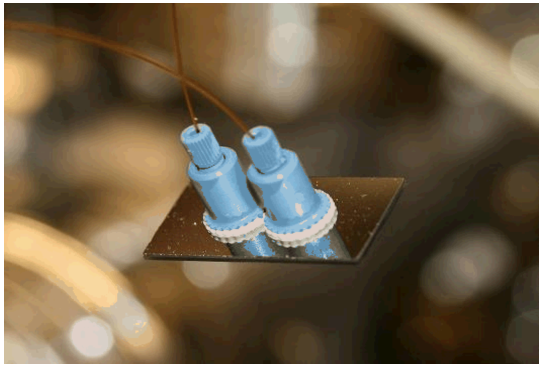
Design



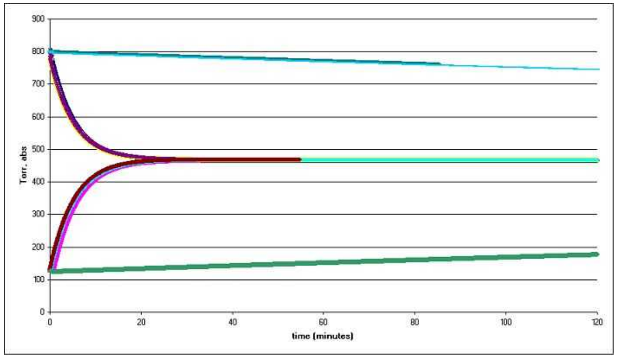
Fabrication



Mass Flow Measurements

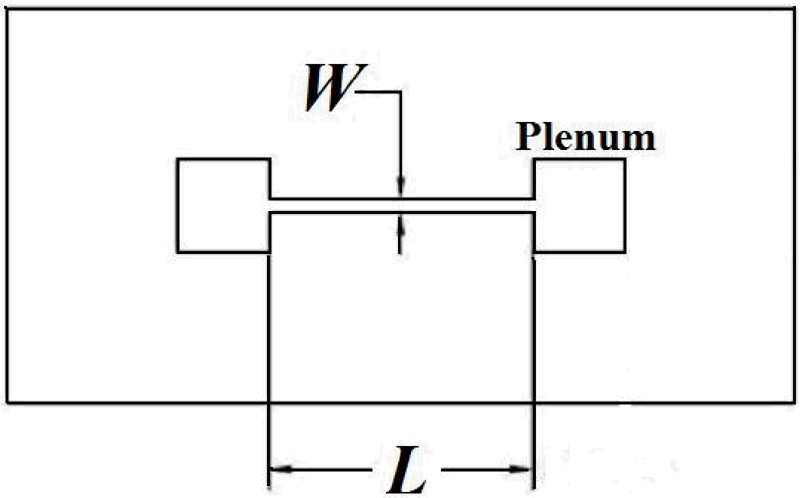


Data Analysis

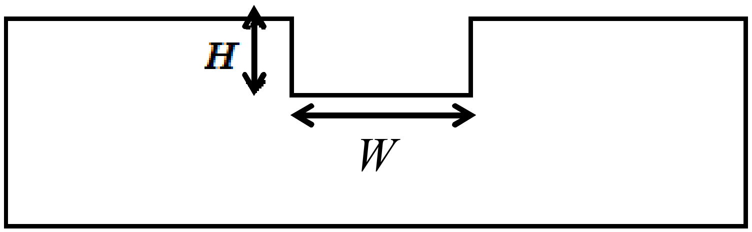


Design

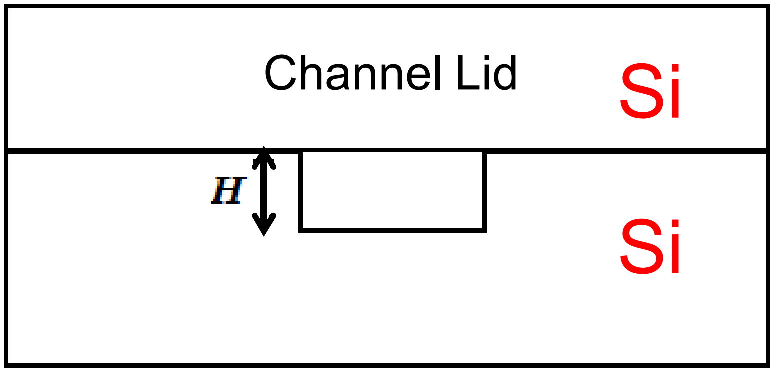
Top View



Cross-section



Direct bonding = Si surfaces



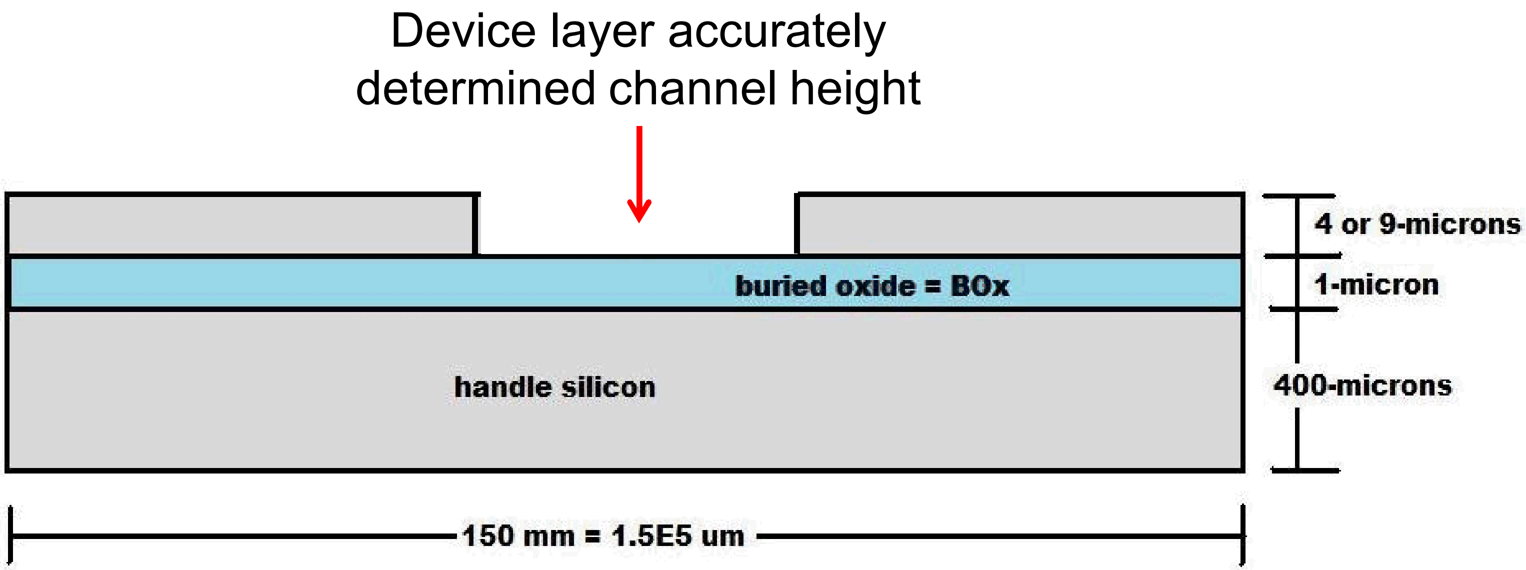
- Channel dimensions:

$L \gg H, \quad W \gg H,$

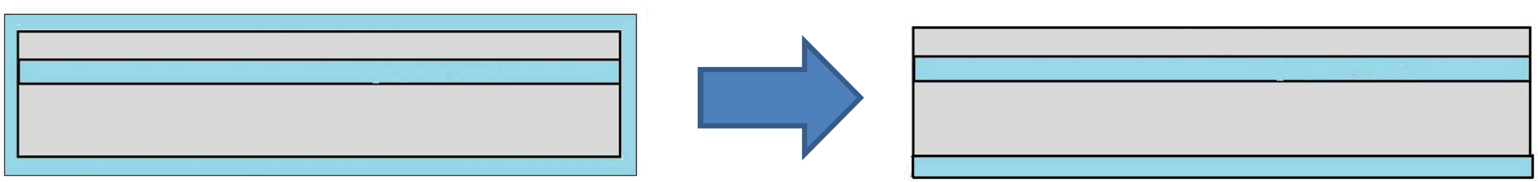
$H = 5\ \mu m, 10\ \mu m$

Plena volume > 100 × channel volume

Starting Substrate Silicon On Insulator (SOI)

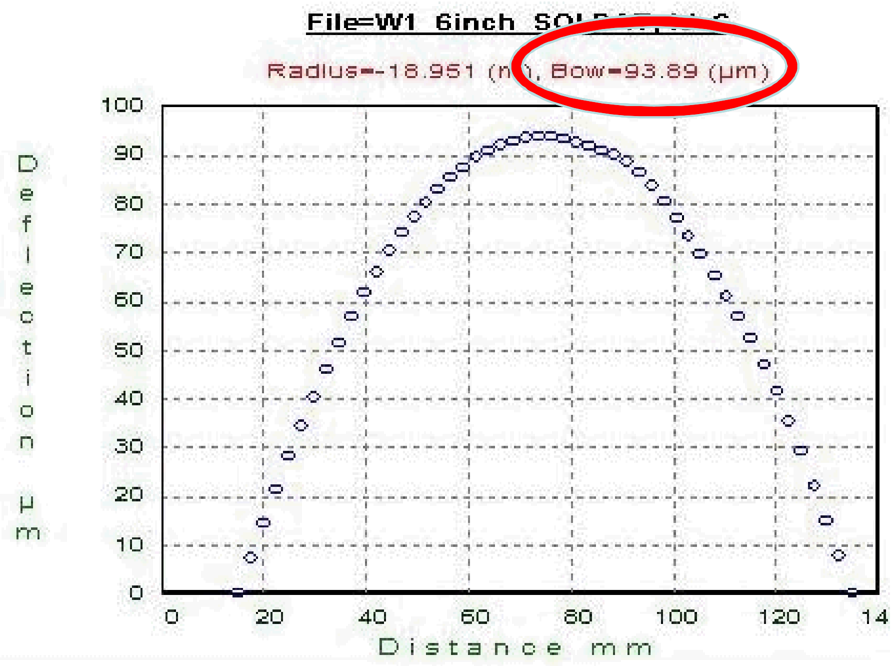


- Thermal oxide has ~270 MPa compressive stress

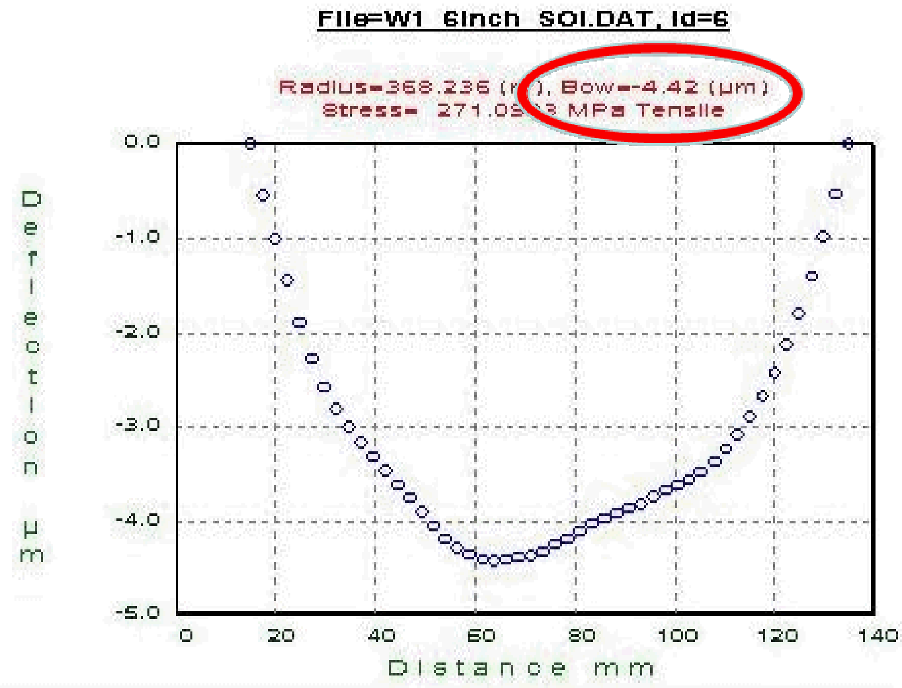


- To counteract this, 1-um thermal oxide was grown on both sides and removed from the device side

Oxide Eliminates Bow



Before oxide



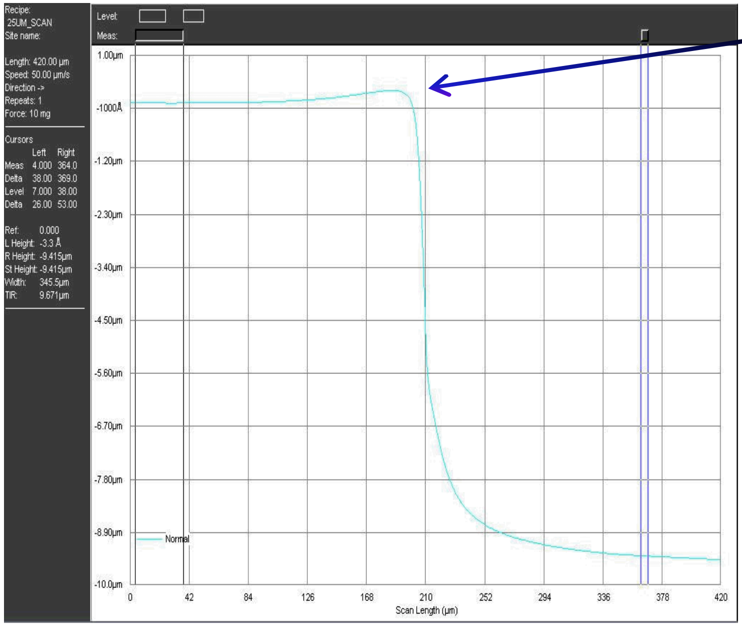
After oxide

W2 5-micron device SOI from device side

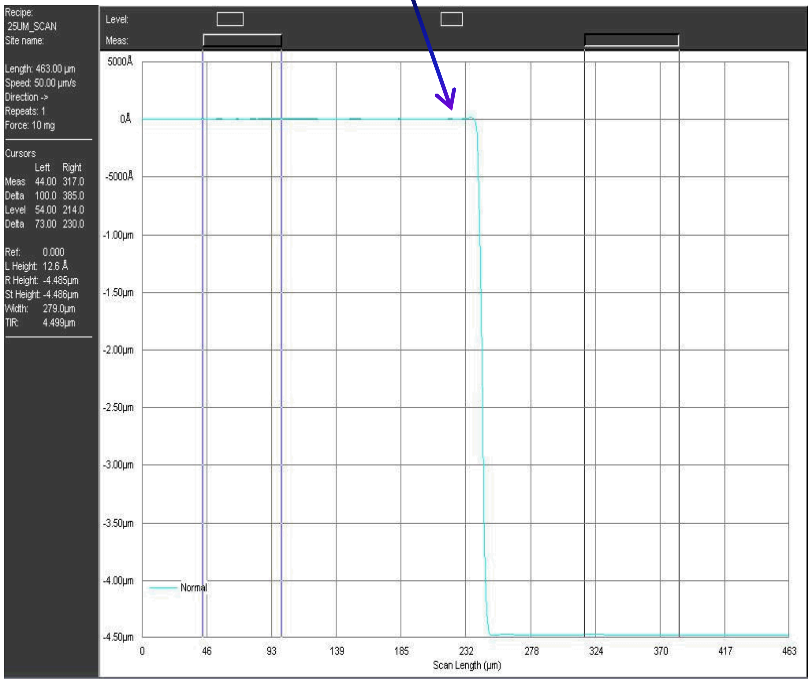
Wafer bow was measured using a 670-nm laser to scan across the wafer at room temperature

SOI Edge Bead Removal (EBR)

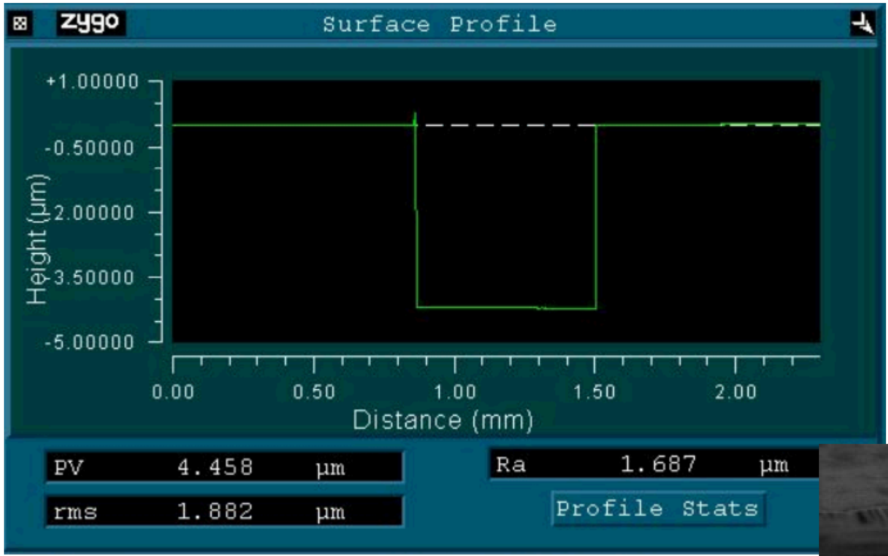
0.25 μm hump can hinder bonding around edge



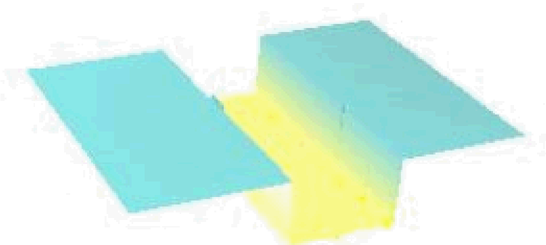
Edge bead removed



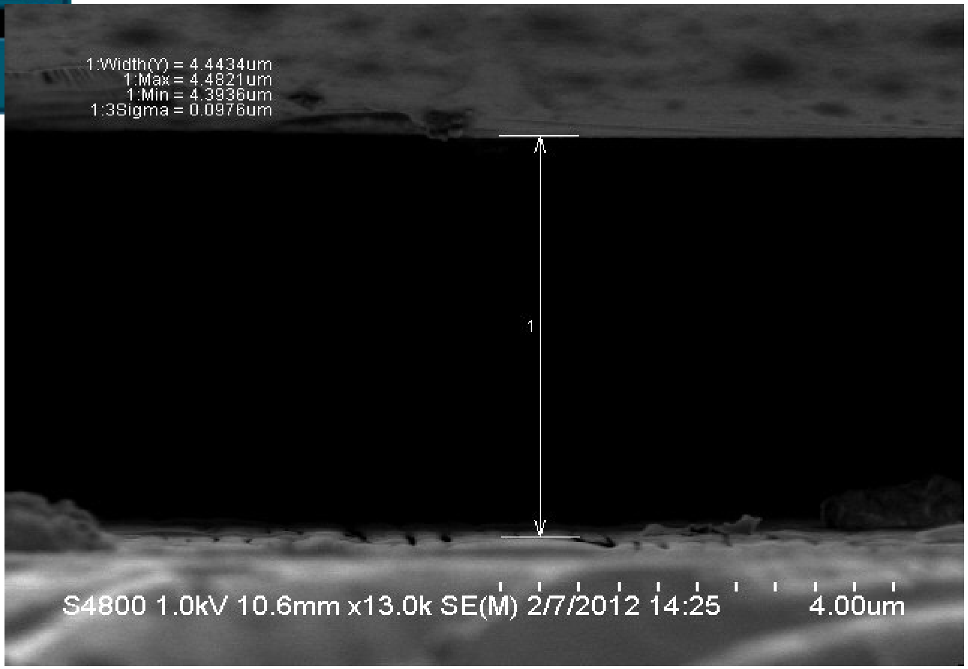
Channel Characterization



White Light Interferometry



- Manufacturer Specifications: 4 $\mu m \pm 0.5\ \mu m$ and 9 $\mu m \pm 0.5\ \mu m$
- Buried Oxide Specifications: 1 $\mu m \pm 5\%$
- EBR and Channels etched using deep reactive-ion etching (DRIE)

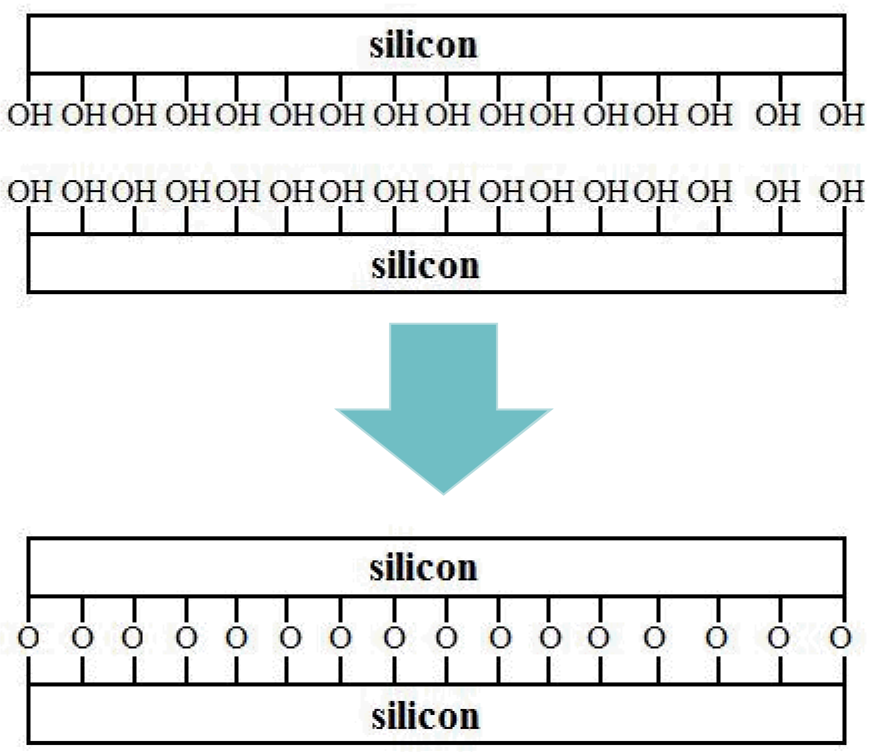


Nominal 5 μm channel measurements ranged from 4.44 μm to 4.66 μm

Channel Lid Bonding

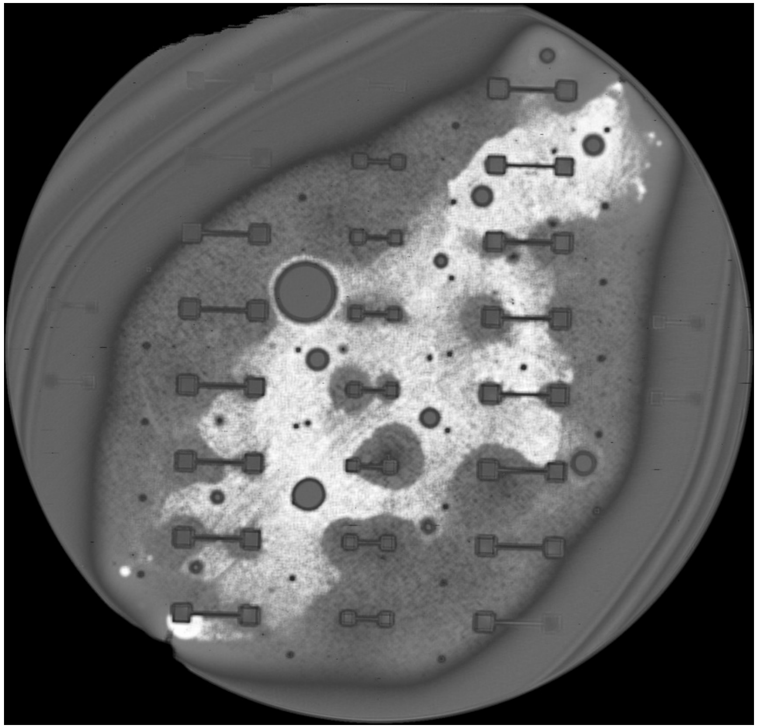
O_2 Plasma Activated Direct Bonding

- Dilute HF dip, then SC1 (H_2O:H_2O_2:NH_4OH) and O_2 plasma surface activation create free dangling bonds consisting of silanol
- Simply bringing the wafers into contact forms siloxane releasing H_2 and H_2O in the process

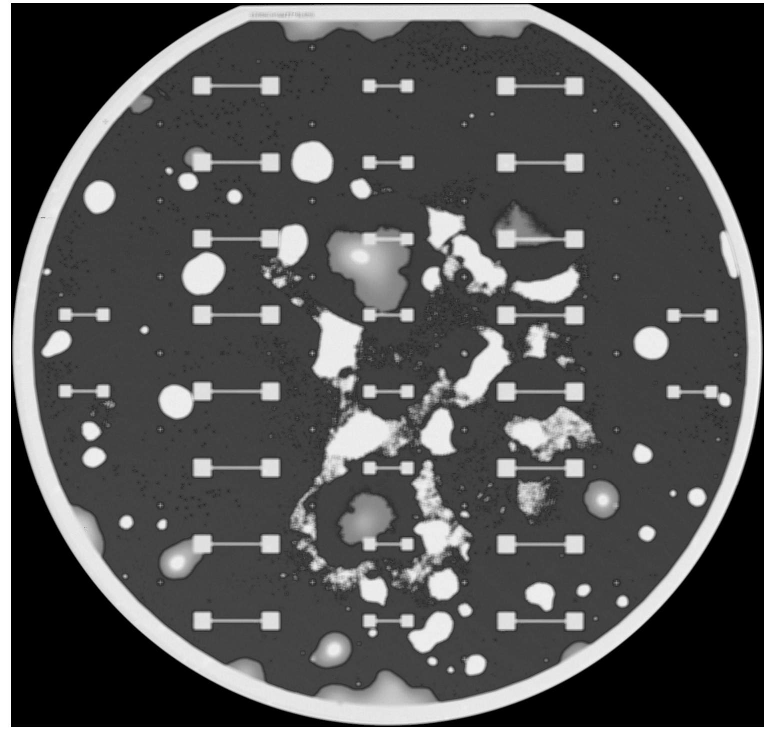


Lid Bond Inspection

using C-mode scanning acoustic microscopy (CSAM)



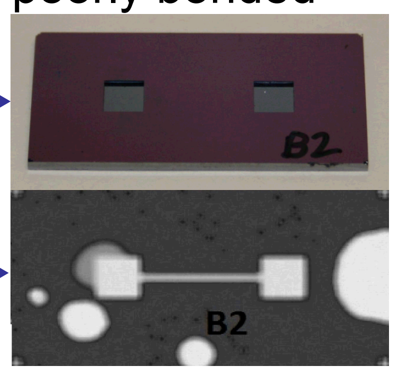
CSAM Large wafer bow, poorly bonded



CSAM after compensative oxide shows vast majority of wafer bonded

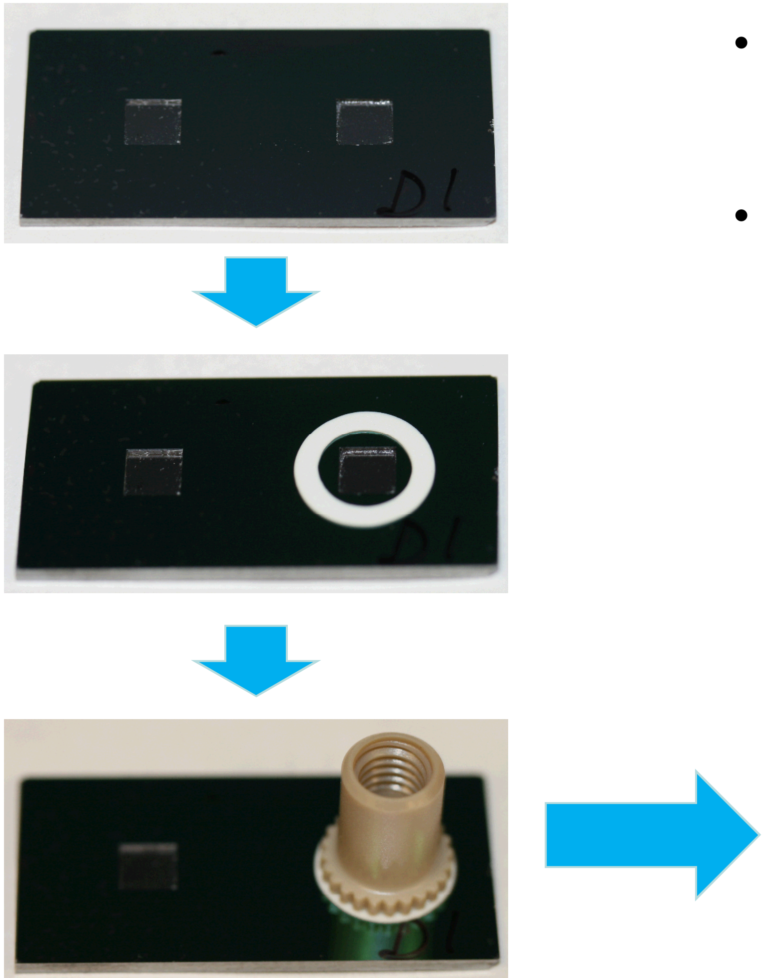
Photograph →

CSAM →



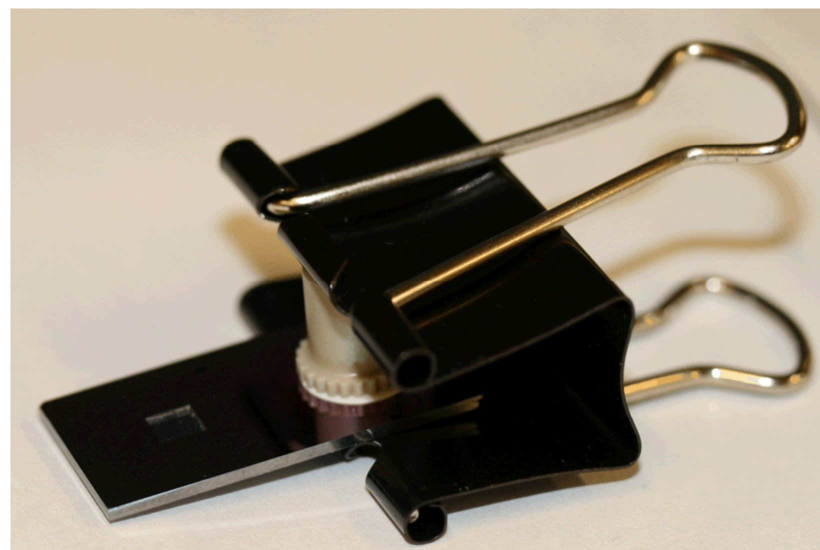
Individual channels labeled for quality assurance

Fitting Assembly

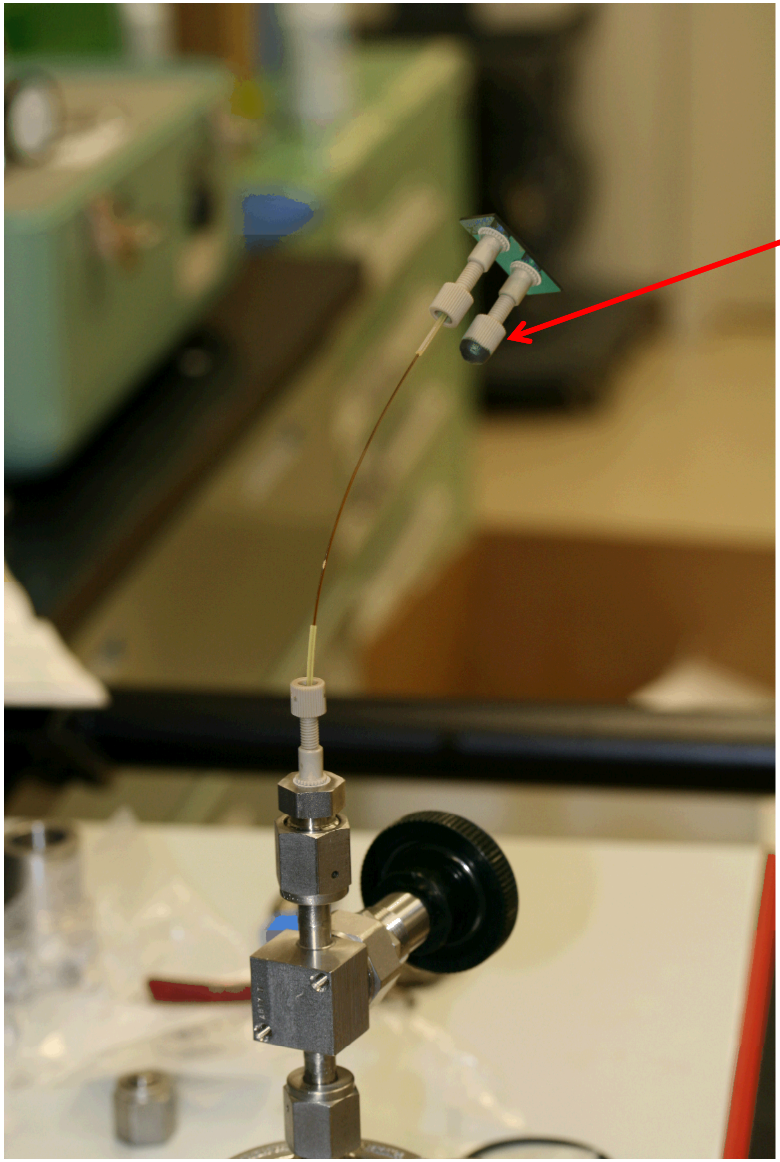


- Align epoxy ring and fitting provided with Upchurch part# N-133

- Fasten with provided spring loaded clamp and bake assembly in oven at 170 ºC for 1 hour to cure epoxy



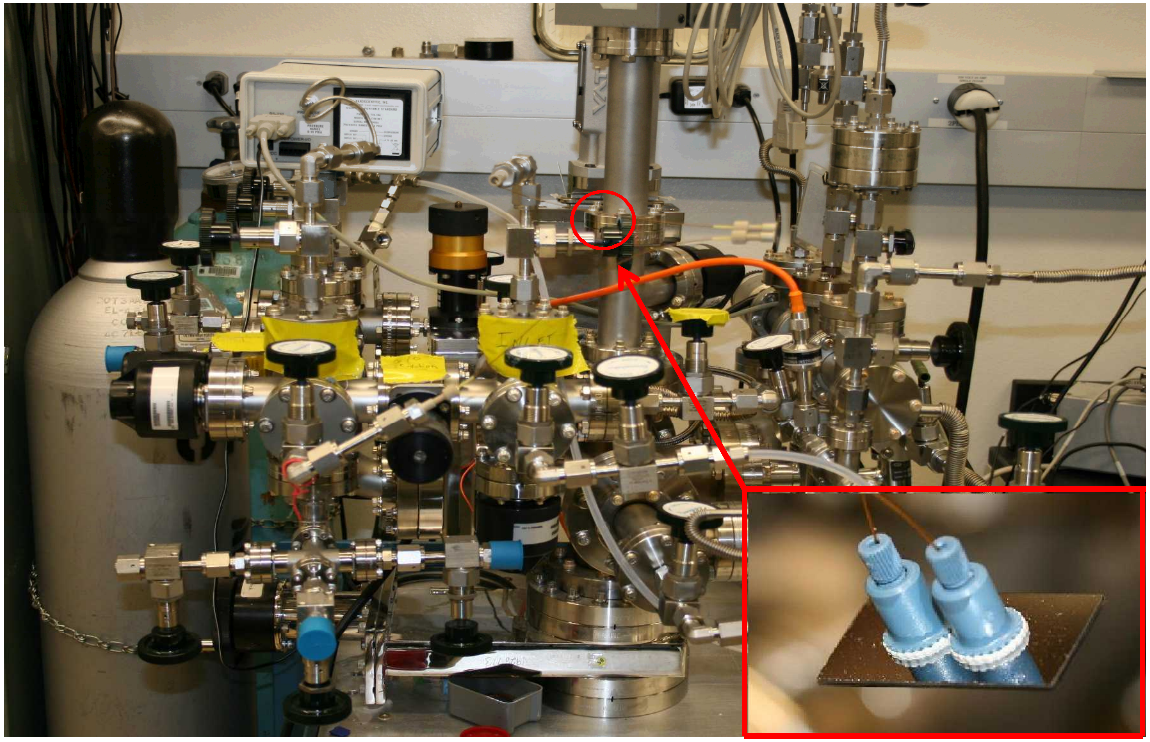
Leak Testing



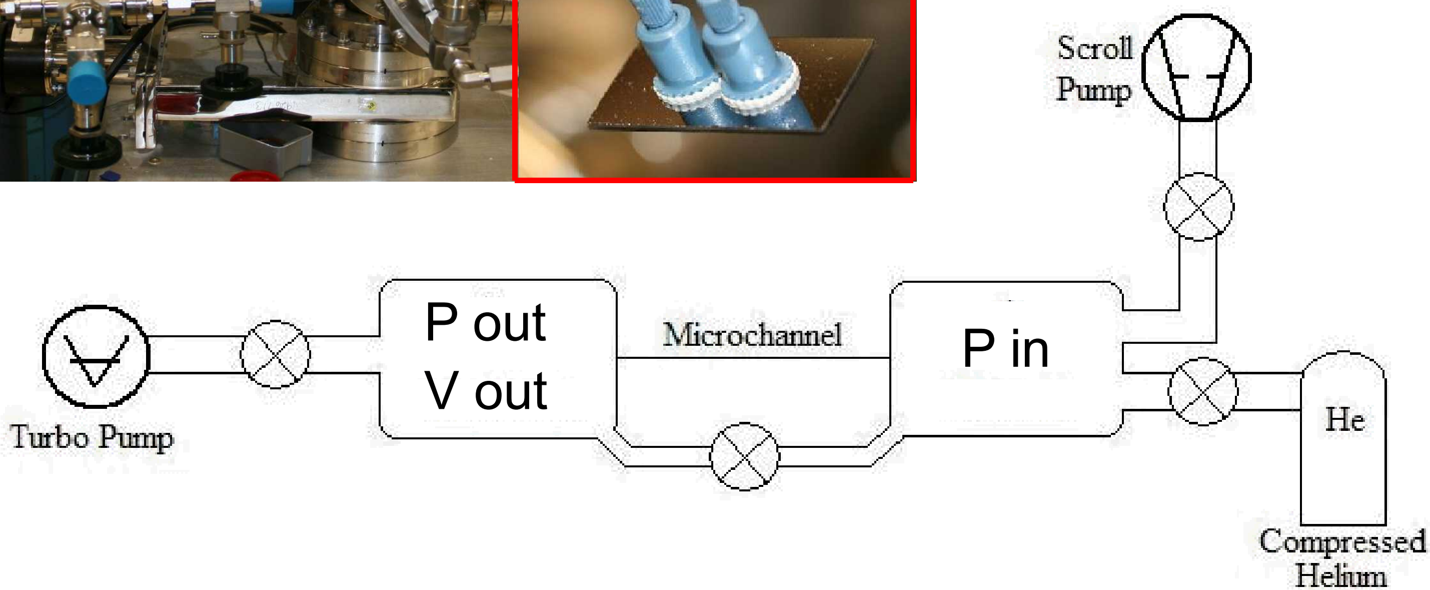
Plug one end of channel and pump for He leak test

- Channel-fitting assembly leak tested down to 10^{-9}(atm)cc/s = $(1.786 \times 10^{-15}$ kg/s) using quadrupole mass spectrometer
- 1% of the lowest measured mass flow rate

Measurement System



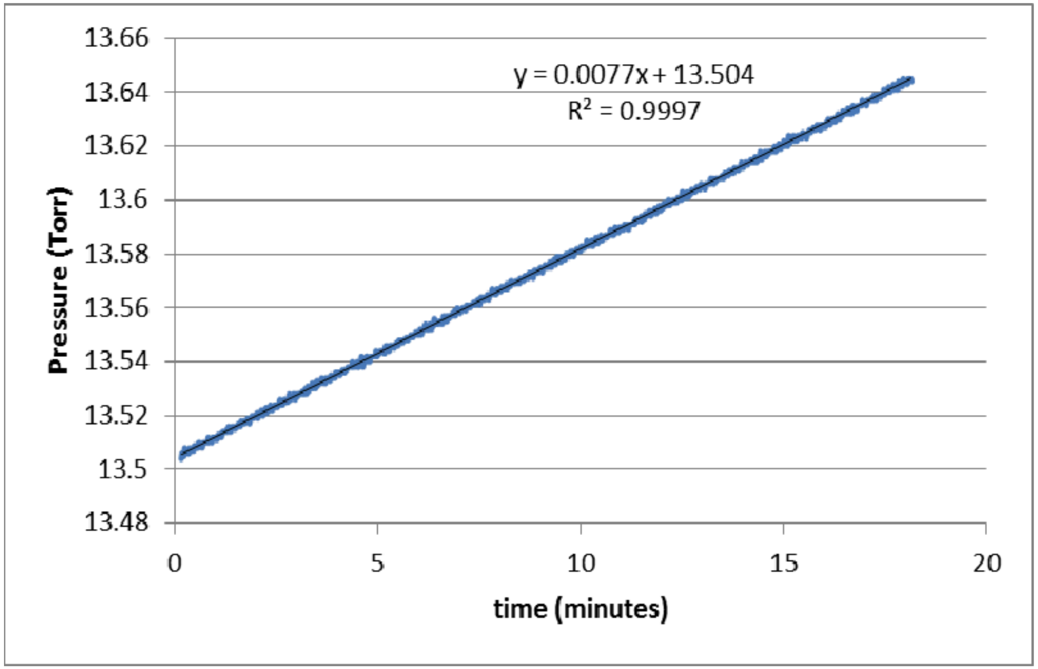
- Manually controlled valves used to set starting pressures for each test
- Pressures used for measurements range from 250 kPa to 2.63 Pa



Flow Measurements

$$\frac{P_{in}}{P_{out}} \approx 5\,,$$ for all experiments coinciding with (*Ewart*, 2007)

Raw data measured from the outlet tank



Mass Flow Rate calculated using the Ideal Gas Law:

$$\frac{dm}{dt} = \frac{V_{out}}{RT}\frac{dP_{out}}{dt}$$

$$V_{out} = 412.5\text{cc} \pm 4\text{cc}$$

$$T = 296.0\text{ K} \pm 0.5\text{ K}$$

Gas: Helium for all measurements

Closed-Form Equation

based on Direct Simulation Monte Carlo (DSMC) models for all regimes and any rectangular channel(Gallis and Torczynski, 2012)

$$\dot{M} = \dot{M}_c\left(1 + \frac{6p_\lambda}{p_m}\varpi[p_1,p_2]\right)$$

$$\varpi[p_1,p_2] = \frac{2-\alpha}{\alpha}\left\{(1+b_1\alpha) + \left(\varepsilon b_0 - (1+b_1\alpha)\right)\frac{b_2 p_\lambda}{p_{1-}p_2}\ln\left[\frac{p_1 + b_2 p_\lambda}{p_2 + b_2 p_\lambda}\right]\right\}$$

$$p_m = \frac{p_1 + p_2}{2} \qquad p_\lambda = \frac{p\lambda}{H}$$

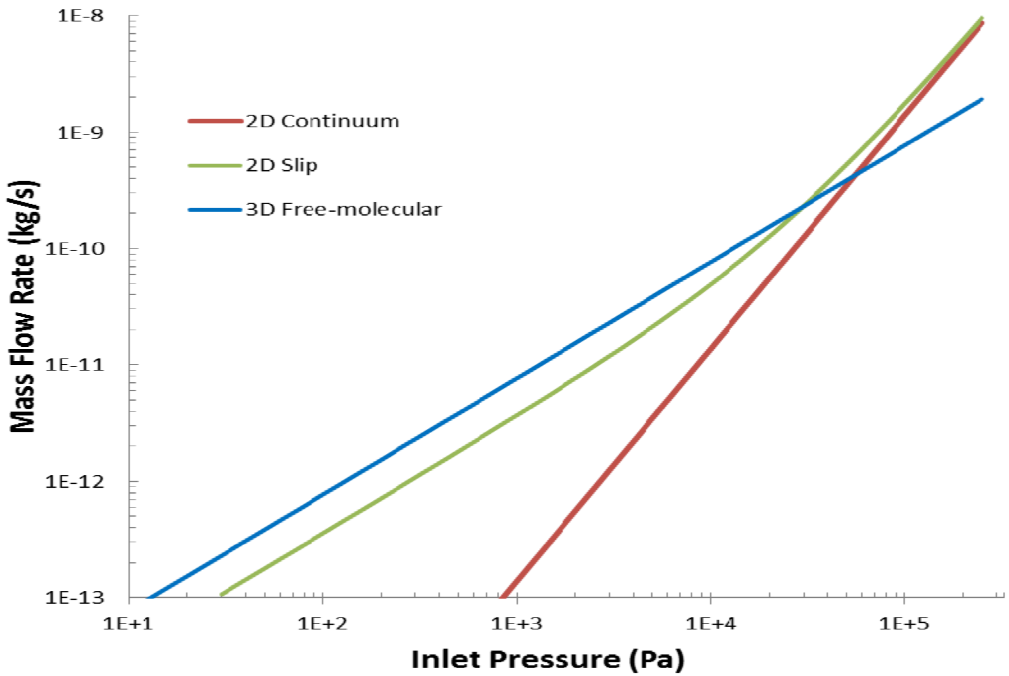
Parameters:

- b_0 gives the correct free-molecular limit
- $b_0 = 3.26224$ for channel with 9.67 μm height and 3.76031 for channel with 4.5 μm height

Limiting Forms of Model

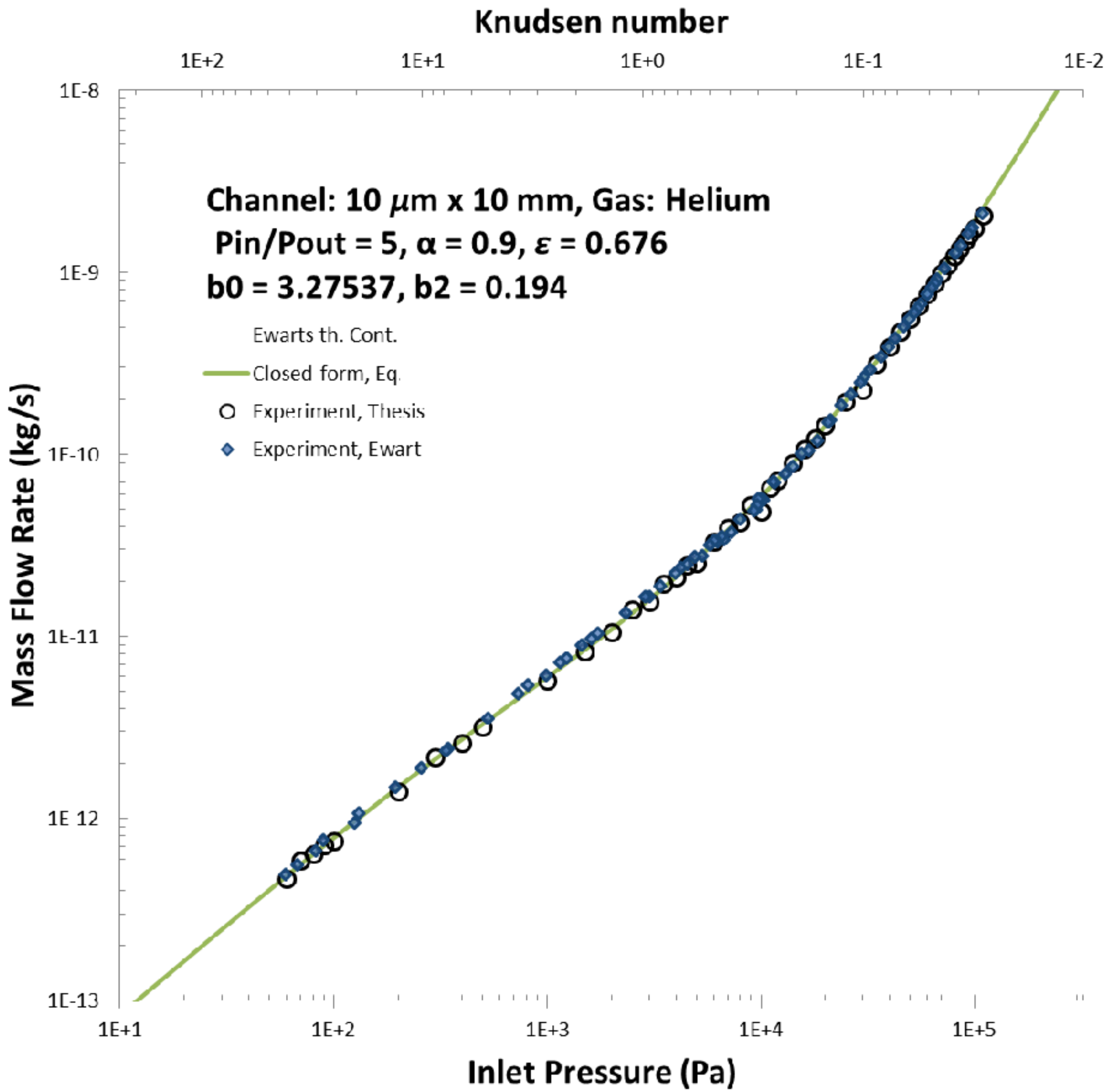
Flow characteristics depend on the Knudsen number, $Kn_m = \frac{\lambda_m}{H}$

- $0 < Kn < 0.001$, **continuum** regime $\quad \dot{M}_c = \frac{2WH^3 p_m (p_1 - p_2)}{3\pi\mu c^2 L}$
- $0.001 < Kn < 0.1$, **slip** flow $\quad \dot{M}_s = \dot{M}_c \left(1 + \frac{6p_\lambda}{p_m}\varpi_s\right), \varpi_s = \frac{2-\alpha}{\alpha}(1 + b_1\alpha).$
- $Kn > 10$, **free-molecular** $\quad \dot{M}_F = \dot{M}_c \left(\frac{6p_\lambda}{p_m}\varpi_F\right), \varpi_F = \frac{2-\alpha}{\alpha}\varepsilon b_0$

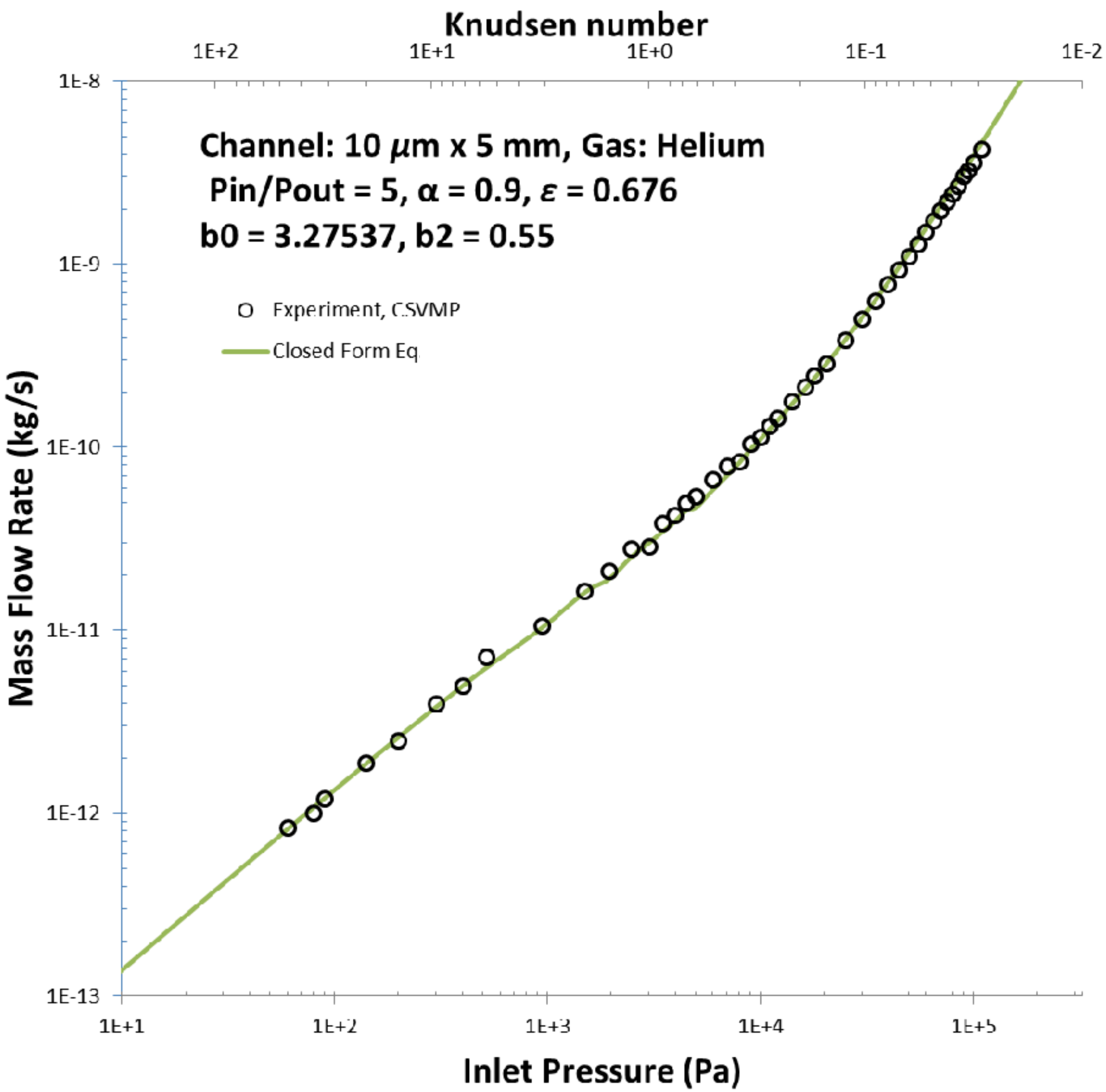


Channel: 9.67 μm X 10 mm

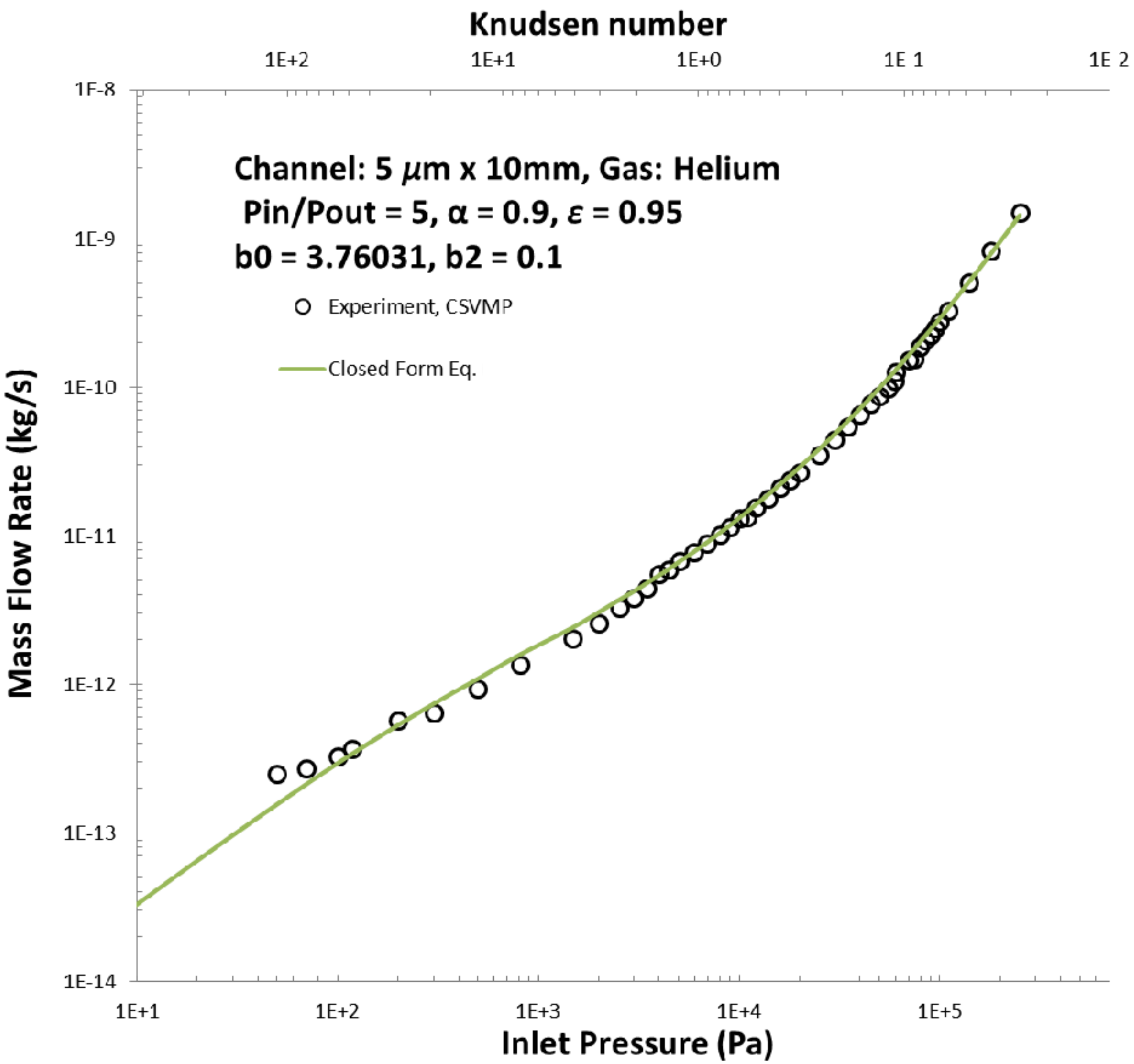
- Channel similar to Ewart et al.(2007)
- matches the closed-form equation



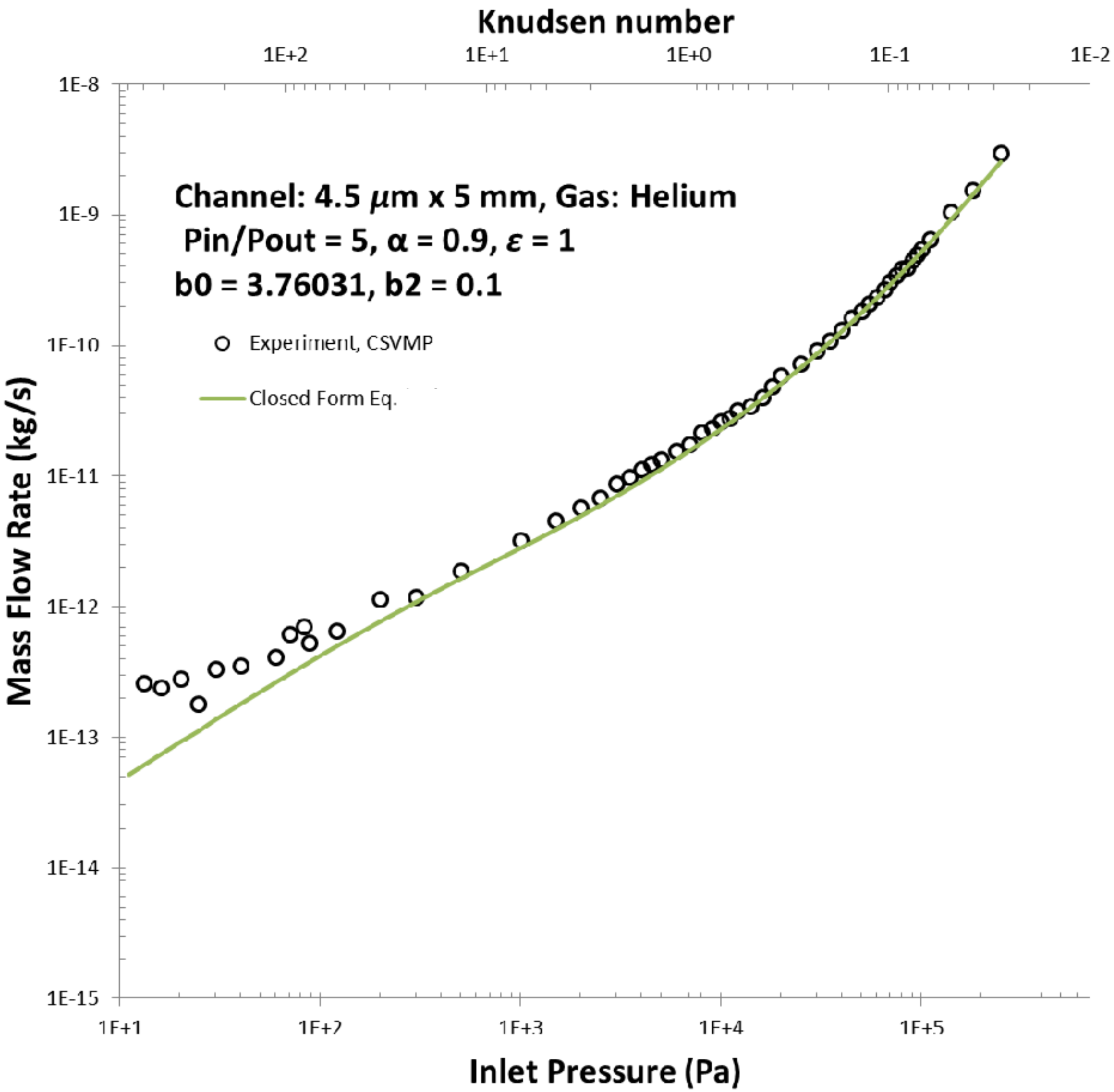
Channel: 9.67 μm X 5 mm



Channel: 4.5 μm X 10 mm



Channel: 4.5 μm X 5 mm



Calculated Error

$$\frac{\Delta Q_m}{Q_m} = \frac{\Delta V}{V} + \frac{\Delta T}{T} + \frac{\Delta a}{a}$$

- $\Delta T/T$, accounts for the non-isothermal effects evaluated at ±2% (Graur, Méolans et al. 2005).
- $\Delta V/V$ is the uncertainty of the volume measurement, ±2%.
- $\Delta a/a$ is the error on the coefficient *a* in (5) and is evaluated at ±0.5% at the higher pressures, but increases to ±1% at the lower pressures.
- Combining these gives us a maximum percent error of ±5% at anytime.

Conclusions

- Isothermal helium mass flow for a very wide Knudsen range from 0.001 to **550**, well into the free-molecular range
- Flows measured were on the order of 10^{-13} kgs^{-1} (**300 years** to accumulate **1 gram** of helium).
- Data set valuable for validating model and further predicting free-molecular flow for engineering applications MEMS such as micropumps, microvalves.