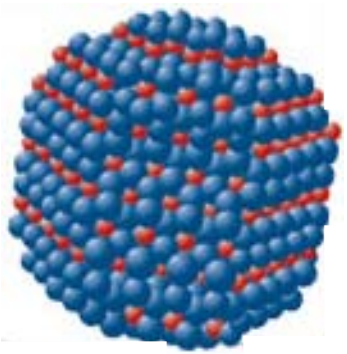
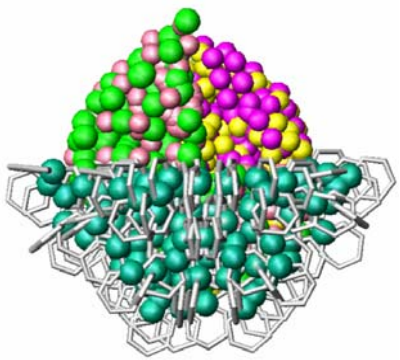
CINT Microfluidic Synthesis Discovery Platform™



Nelson S. Bell, Team Lead
Jun Liu, James A. Voigt, Tom Sounart, Julia Hsu, Murat Okandan, Jenn Hollingsworth, Ron Renzi, Melissa Petruska

CINT Discovery Workshop
January 16, 2007
Albuquerque, NM



<u>Commercialization of nanoparticle-based technologies:</u>

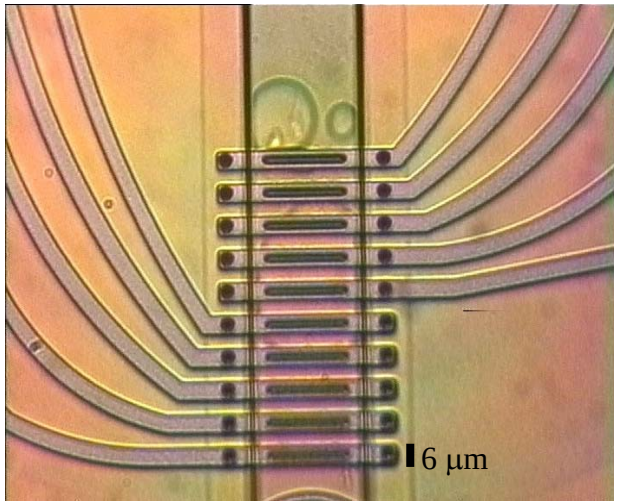
"…researchers are not focused on the requirements posed by scalable, cost-effective manufacturing. **Robust and reliable production methods are needed** to significantly expand the commercial use of nanomaterials."

"The largest barrier…is the lack of fundamental understanding…"

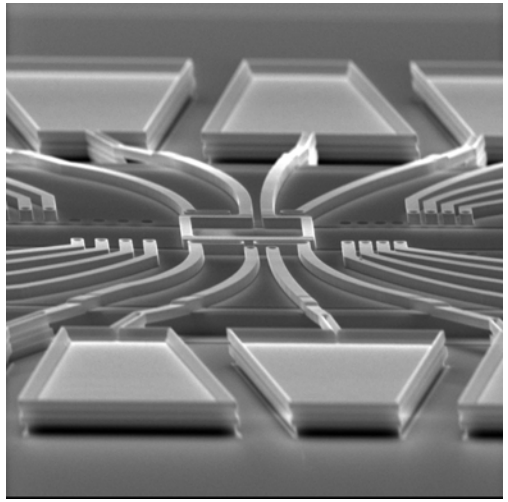
- Chemical Industry R&D Roadmap for Nanomaterials By Design (2003)

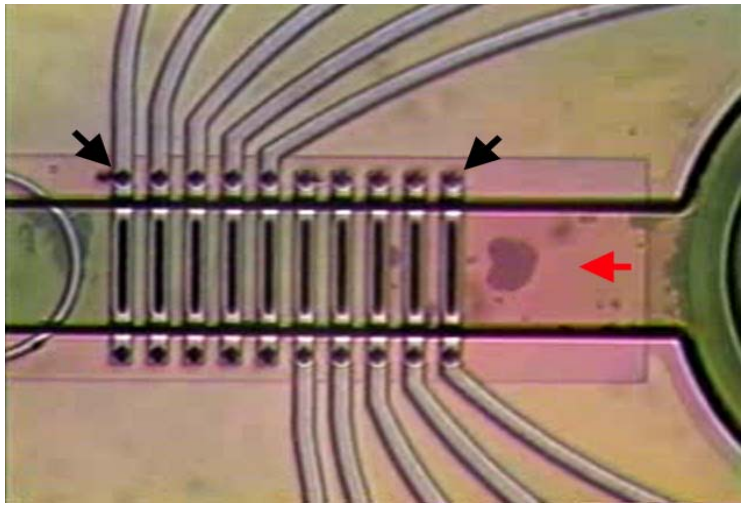
Opportunity for both science and technology

Electrochemistry/photochemistry – particle manipulation in microfluidic channels



electrolysis bubbles inside the channel





Trapping of beads by application of AC waveform (measurement by Conrad James, 1769)

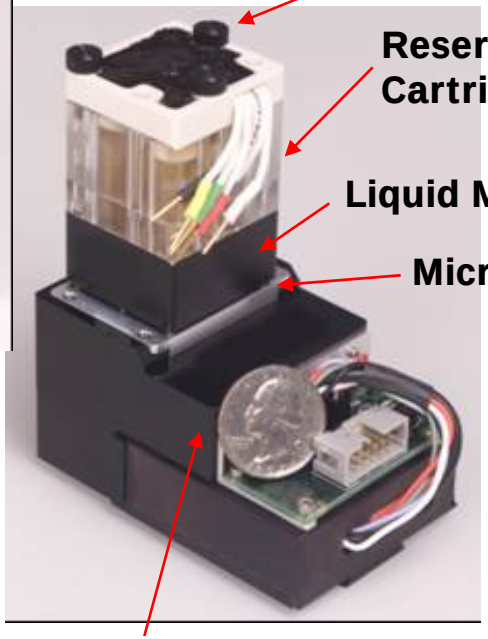
- Some of the very interesting aspects of micro-nanoscale reactions become observable/feasible at the scale of these devices.
- Reactions can be controlled by the applied field, light, temperature and flow conditions.
- Particles/streams inside the channel can be controlled by various means (dielectrophoretic trapping, magnetic fields, etc.)

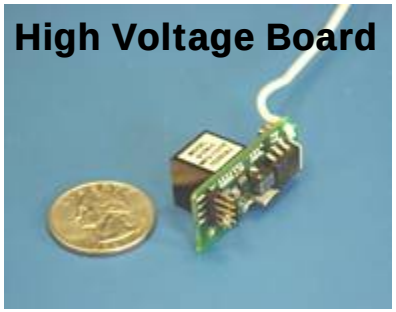
μChemLab technology enables rapid fluidic connections

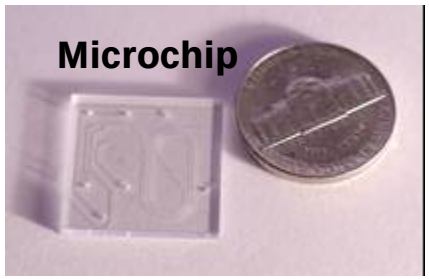
A flexible, reliable platform for routine laboratory R&D

- Hand portable
- Battery operated
- On-board data analysis

- Modular packaging
- Two analysis modules







Microfluidic Technologies

- Capillary Chip Electrophoresis
  - CGE, CZE, IEF
  - Laser-Induced Fluorescence detection
- Electrokinetic Pumping
  - EK-High Performance Liquid Chromatography (HPLC),
  - Infusion pumps
- Insulator-based Dielectrophoresis(iDEP)
  - Particle concentration
  - Sorting
- Gas Chromatography
  - SPE, GC, SAW detection
- High Pressure CapTite Capillary Fittings
  - Sub-miniature
  - Reusable

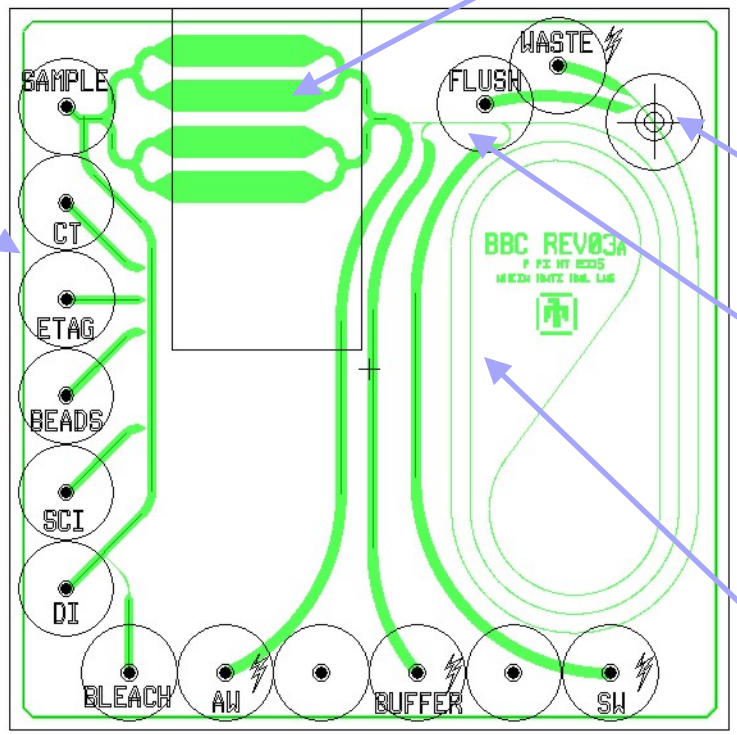
Microfluidic chip technology

- Design, Fabrication, and testing capabilities in a full range of materials:
  - Glass/Fused Silica
  - Plastic
  - Silicon
  - Ceramic

- Integrative components
  - Electrodes
  - Particle packed beds
  - Polymer structures
  - Mobile monolith (valves)

- Interfacing components
  - Manifolds
  - Fittings
  - Optics

Reagent inlet manifold

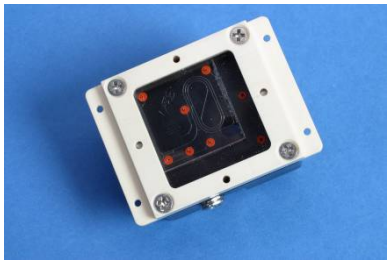


Magnetic Particle Trap

Optical Detection Point

500 nl Injection Tee

Separation Column 100 mm

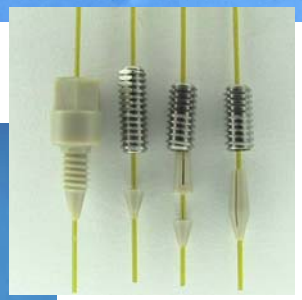


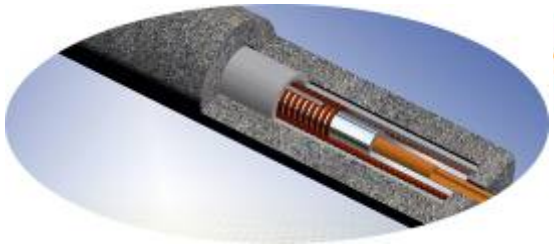
20 mm square

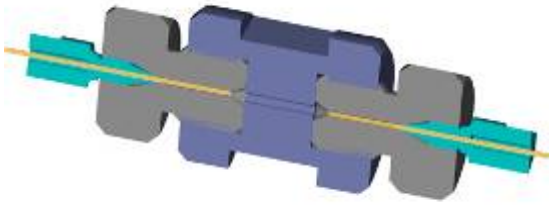
BioBriefcase Immunoassay chip

CapTite microfluidic fittings

- Capillary Ferrule Fittings
- Chip-World Manifolds and fittings
- High-pressure Valves
- Reactors
- Thermal Cell Lysers

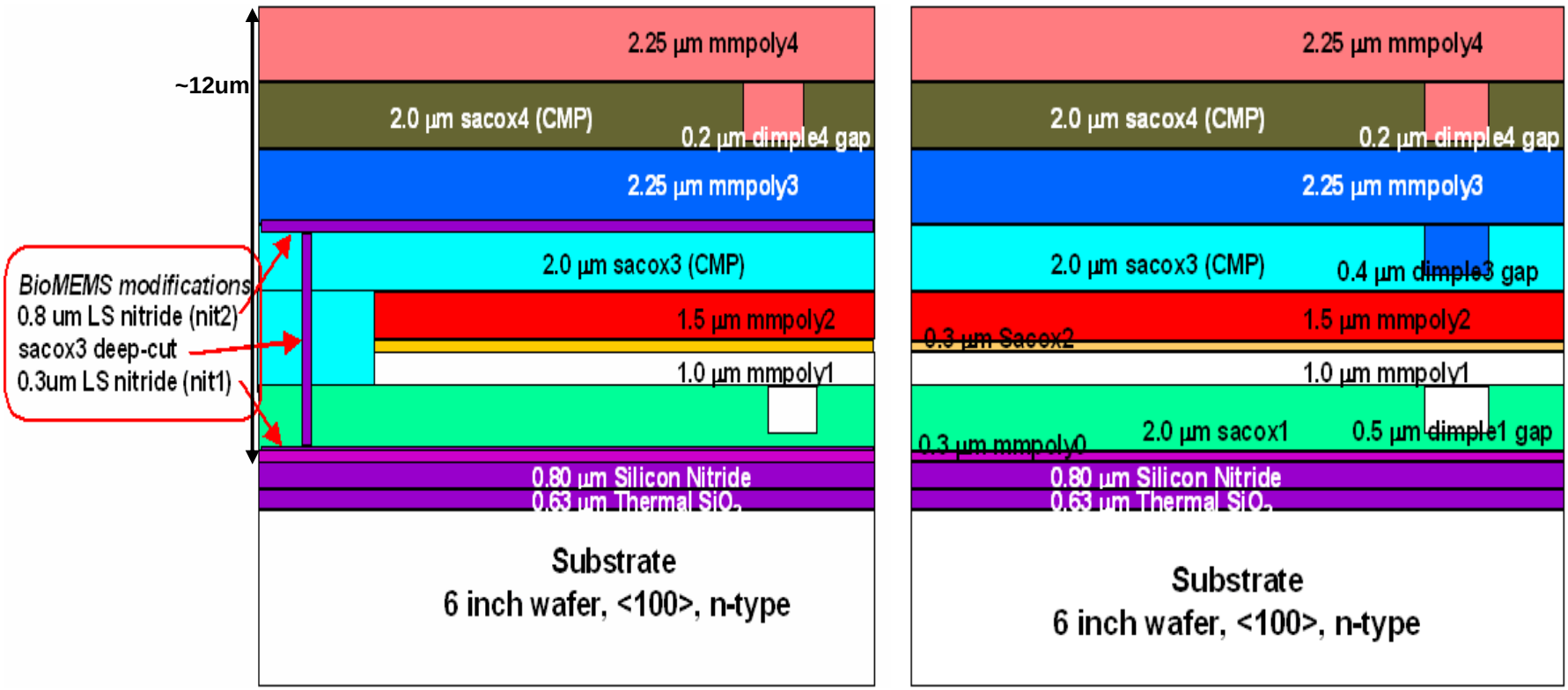






R. Renzi, 8724

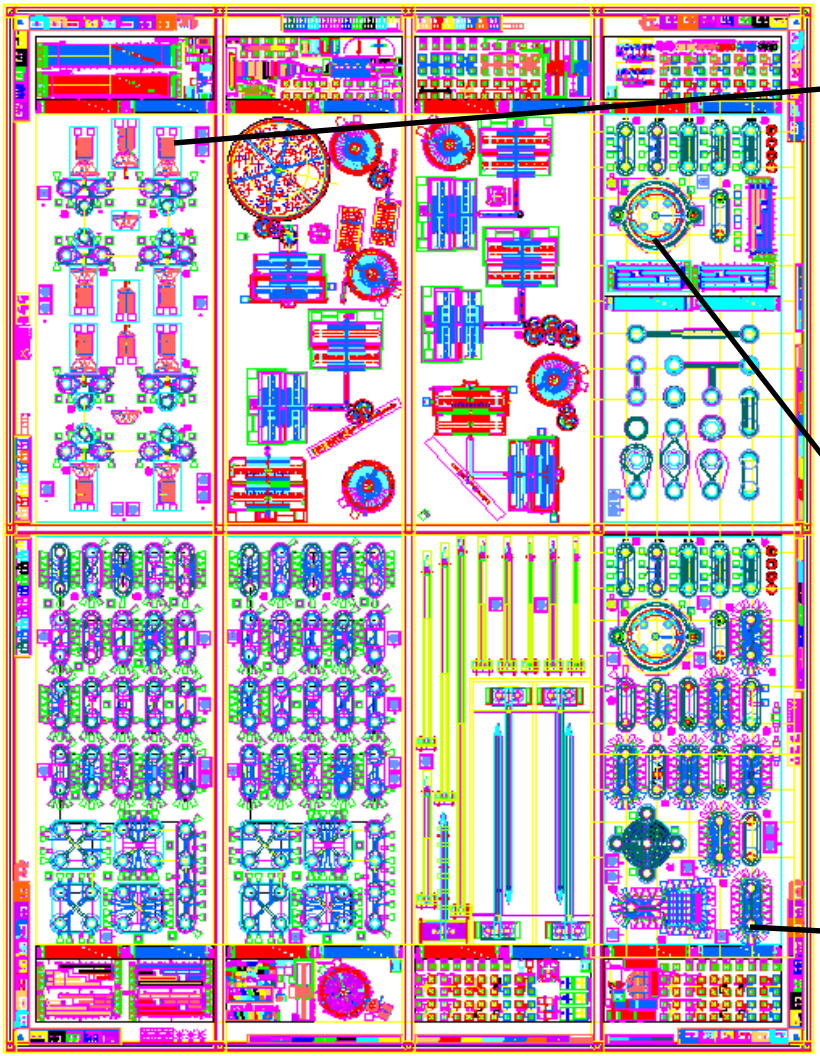
SwIFT™ and SUMMiT™

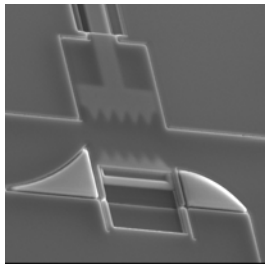


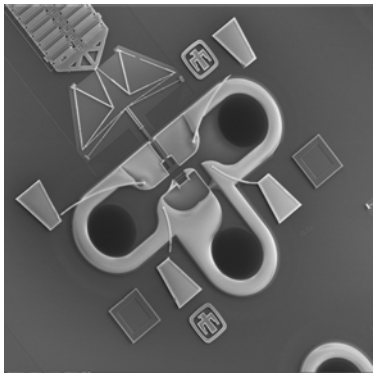
(a) (b)

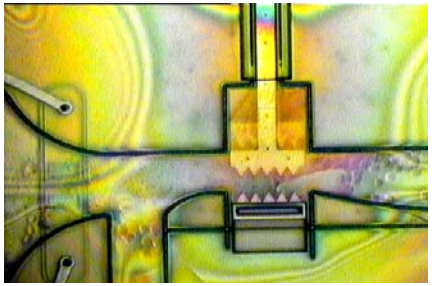
(a) SwIFT™ and (b) SUMMiT™ layers. The incorporation of the low stress silicon nitride layers allows the creation of complex microfluidic structures and enclosed cavities with optical access and provides the ability to create arbitrary fields inside these structures.

Devices in SwIFT™

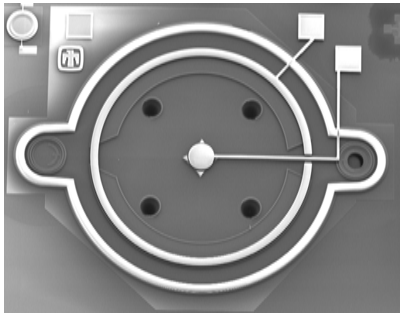




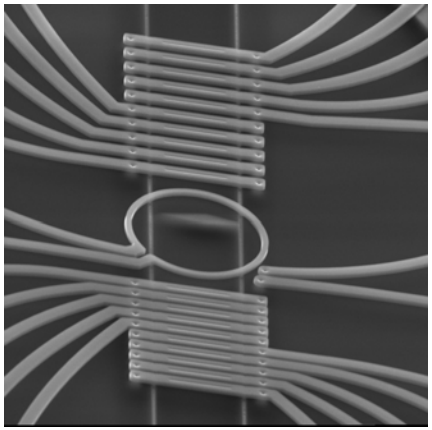




cellular manipulation

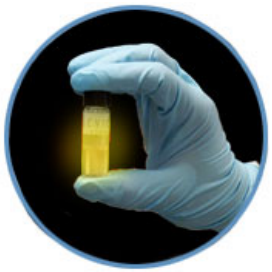


pumps



channels with electrodes

Potential Users: Who Needs It!?



"Challenges with current {*nanoparticle*} synthetic methods include the control of **defects**, **particle agglomeration**, **inhomogeneities** of composition and size distribution, and the lack of cost-effective, controllable synthetic methods amenable to **scale-up**."

- Roadmap for Nanomaterials

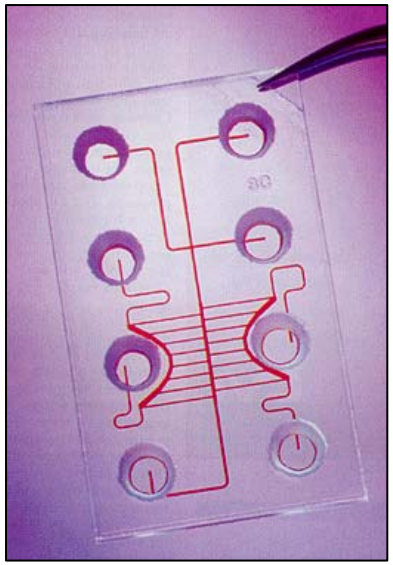
Key Science Functions...

<u>Synthetic Chemistry Kinetics</u>
- Molecular precursors
- Nanoparticles
- Catalyst Activity
- Colloidal Assembly

Overlap with other Discovery Platforms

Biological Reactions/Response

Chemical synthesis for Biomedicine

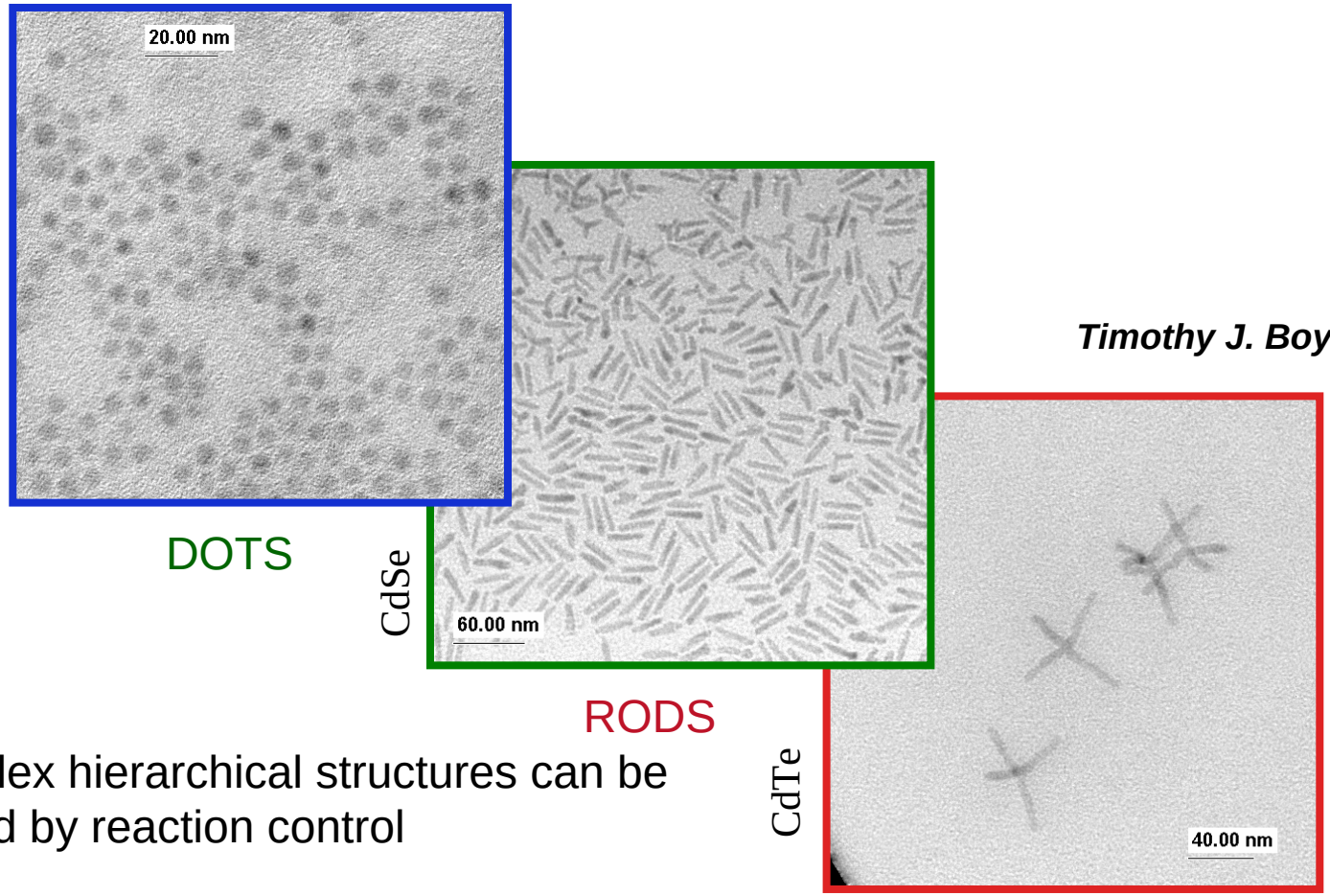


Controlled Environment
- Flow Control (laminar, electroosmosis
- Thermal Control – high heat transfer rates, low Cp
- Mass Transport Control

Reactor Control
- Fundamental Chemistry Studies
- Modeling (geometry, laminar flow)

Microfluidic Synthesis - Motivation

Phase stability and morphology of nanoparticles is thermally and compositionally controlled

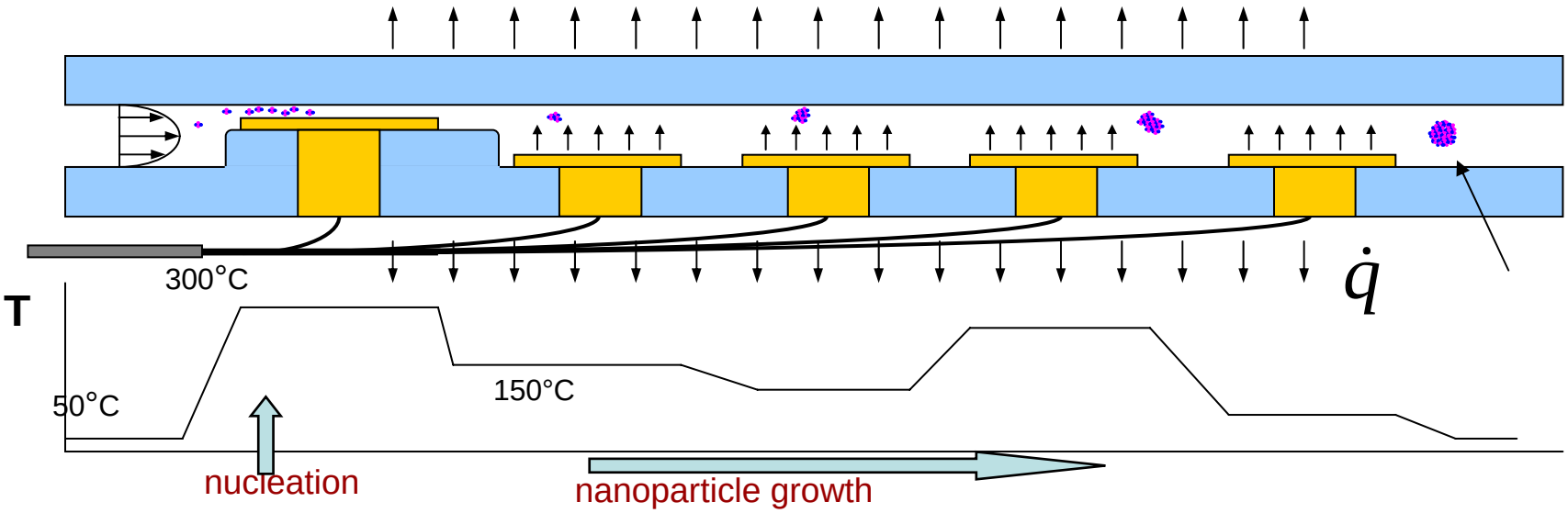


Timothy J. Boyle, Sandia

Complex hierarchical structures can be formed by reaction control

Microfluidic Synthesis Discovery Platform:

Understanding the Kinetics of Nanomaterial Synthesis

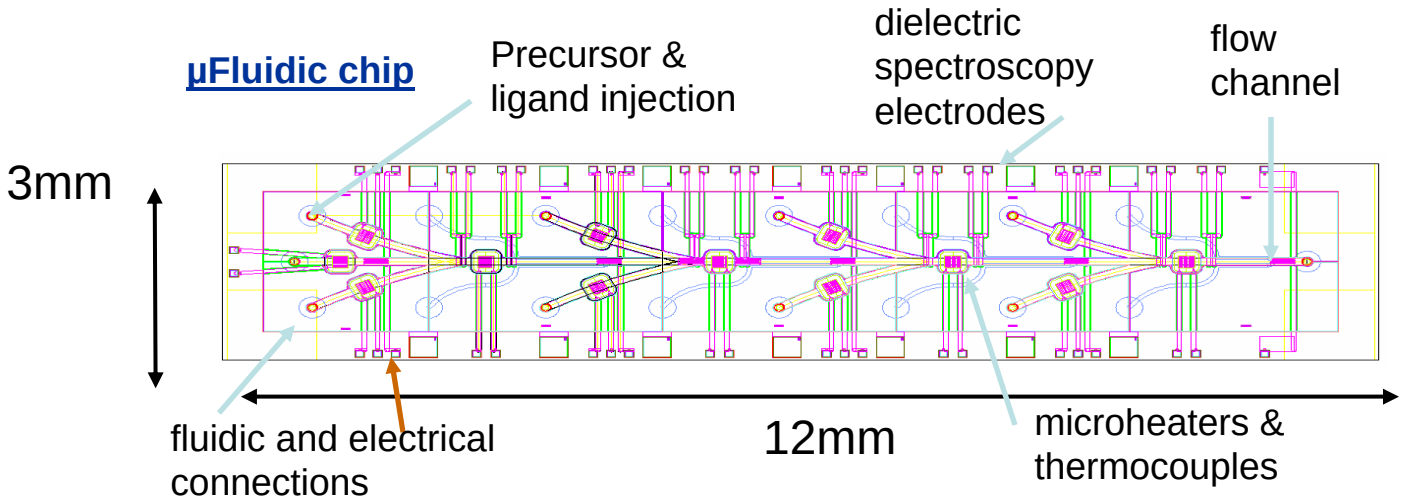


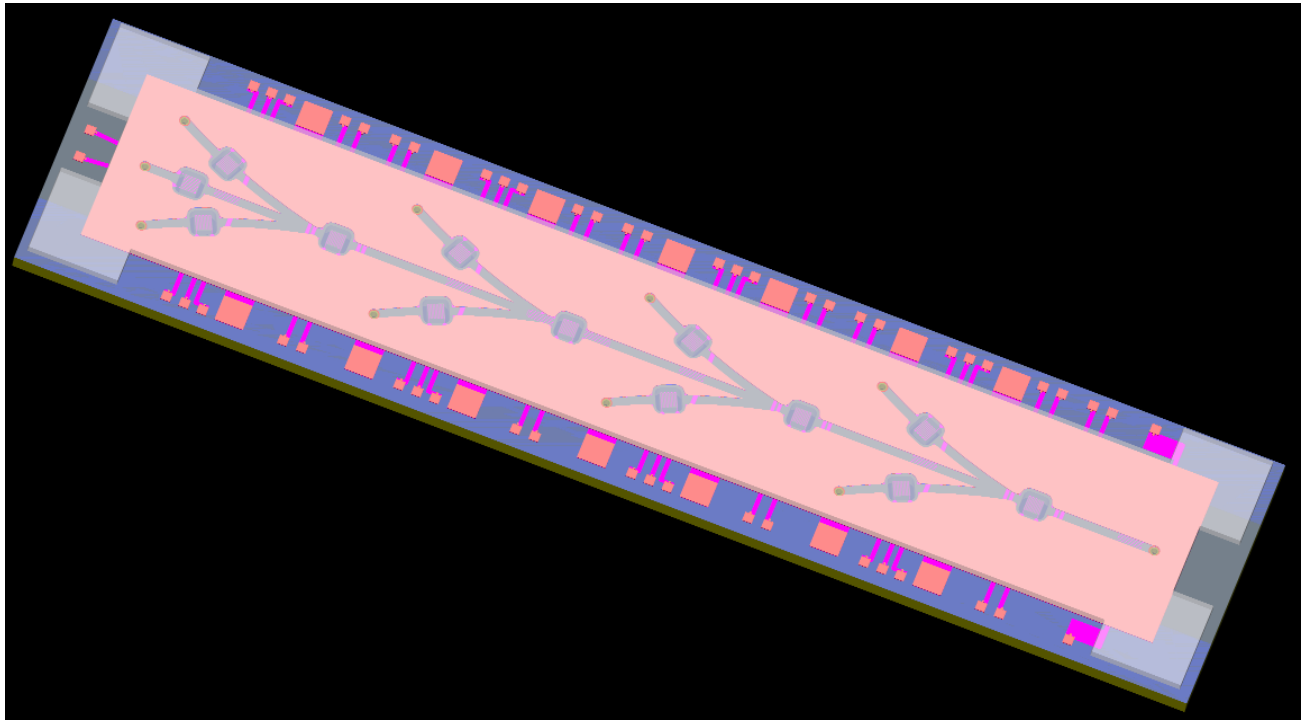
Development of a basic understanding is needed for future "continuous flow" reactors.
Control of thermal profile allows fundamental studies of synthesis reactions.

- What are the values of the thermodynamic parameters that control nanoparticle synthesis? (enthalpy, entropy, surface energy)
- What is the mechanism of nanoparticle nucleation and growth? (i.e. diffusion, surface reaction, self-assembly)
- What is the mechanism of the formation of shell layers around a core nanoparticle?
- How does the surfactant composition relate to the quantum confinement properties of nanoparticles?

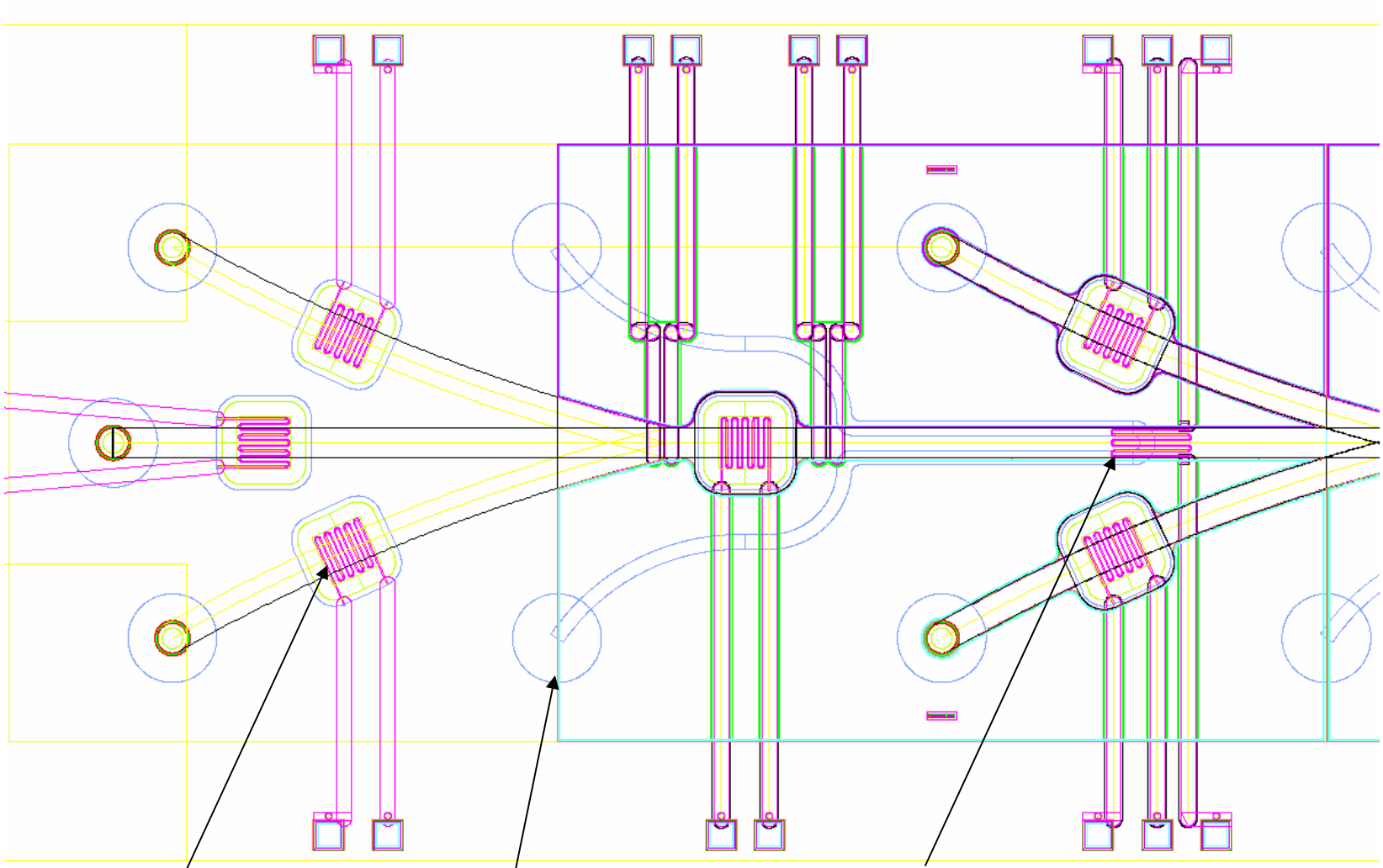
- Microfluidic chip will be integrated within a carrier platform
- A SWIFT-light process will be used to develop the heater and sensor elements.
- Users include synthetic chemists, quantum dot material synthesis.

Microfluidic Synthesis Platform - Design





Microfluidic Platform – Entry Stage

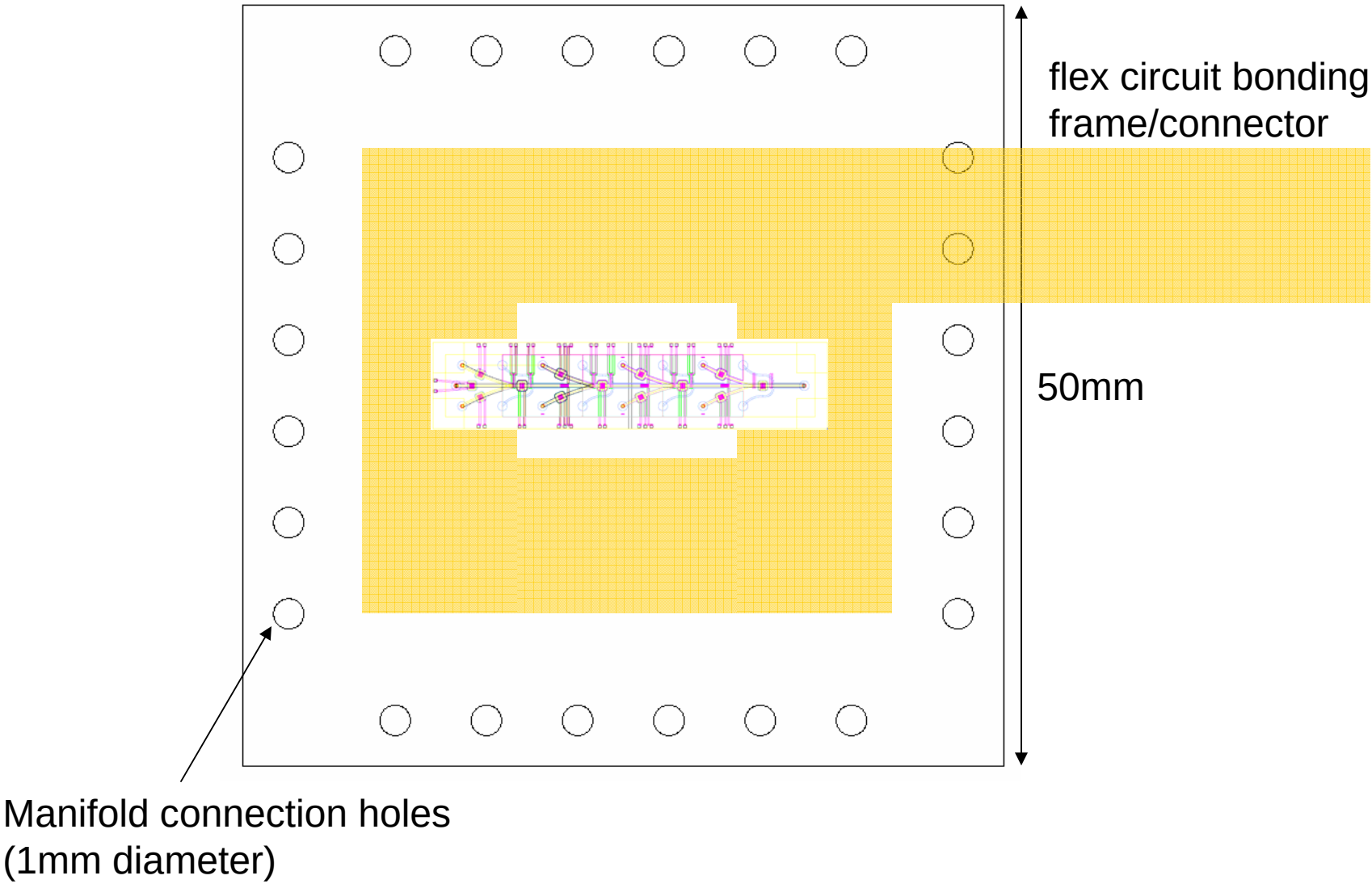


isolated resistors for high temp.

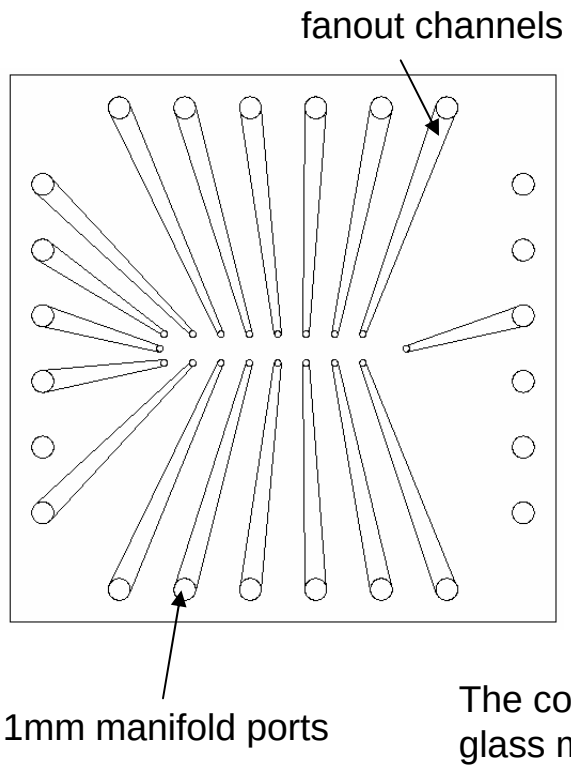
channels for temp. control in silicon bulk

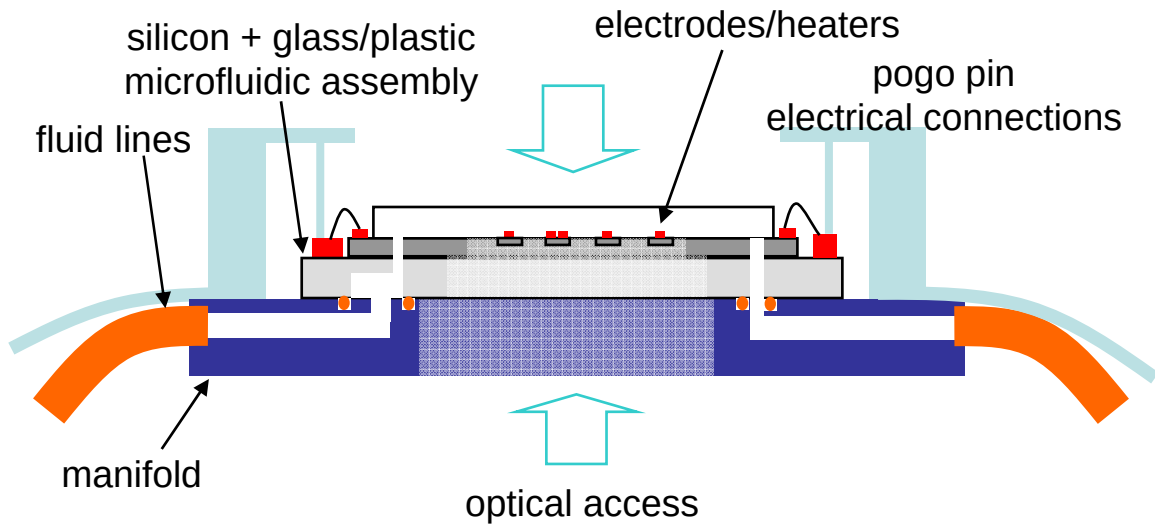
non-isolated resistors for temp. measurement/control

Microfluidic Platform

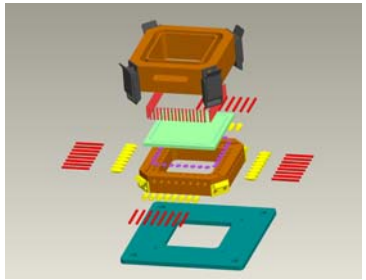


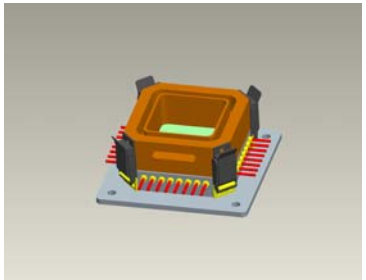
Microfluidic Platform – fanout and manifold connections





The cover and fan out glass must be anodically bonded to the Microfluidic Synthesis DP.





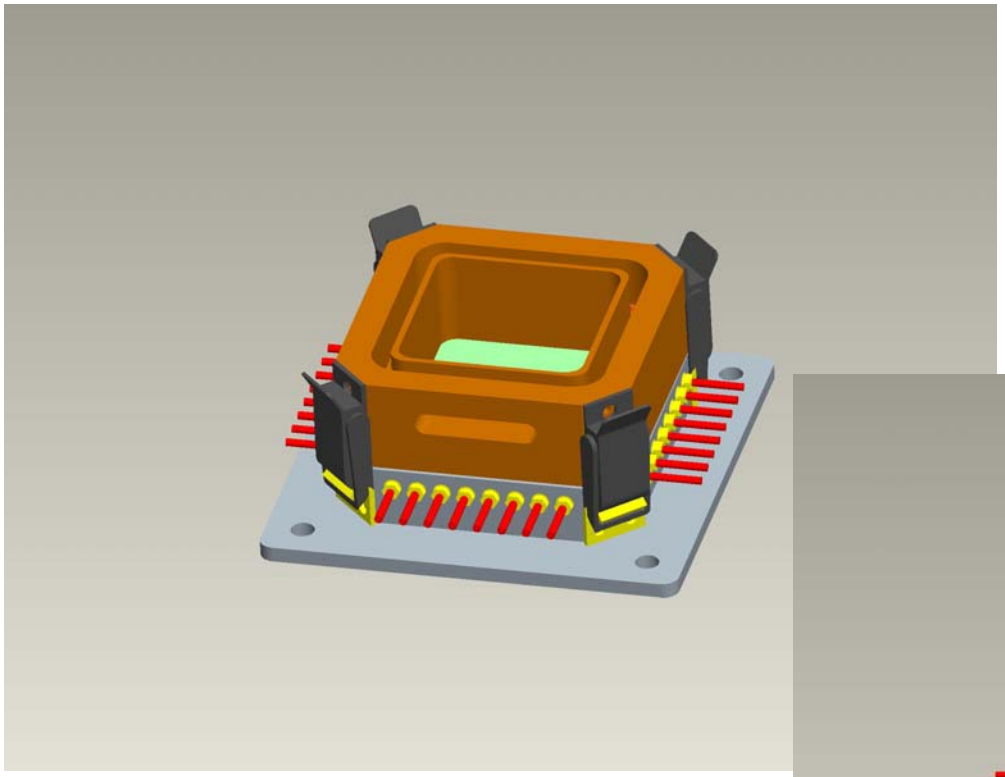
Integration package

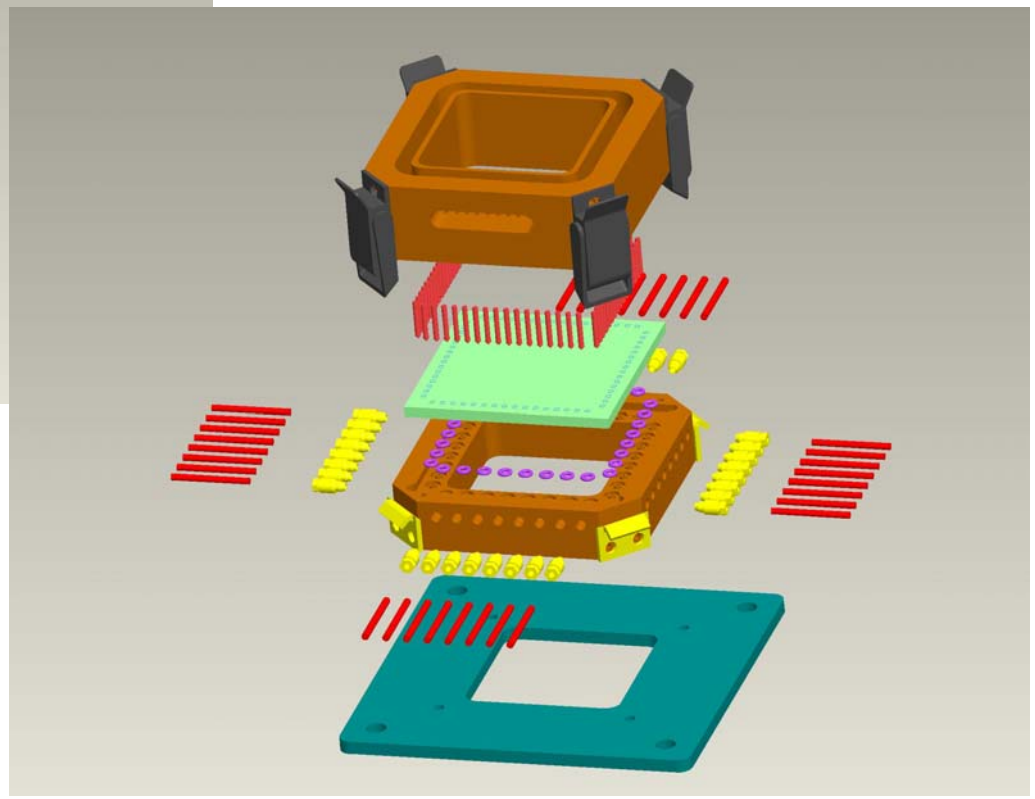
- 64 pogo-pin connections utilizing one common interface connector
- 32 fluidic connections
- 1.5 inch square top/bottom access

- O-ring seals to 500 psi
- Dowel pin alignment
- 4-place draw-latch compression

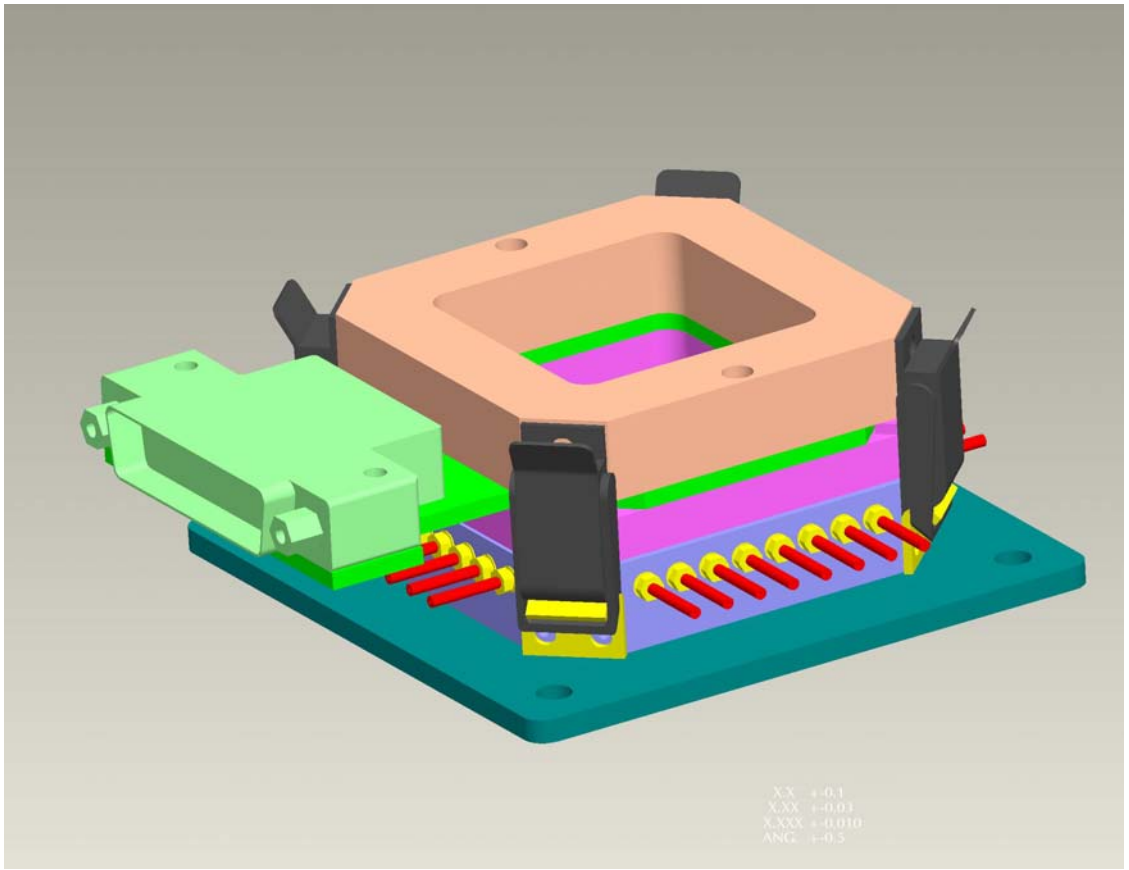
- Circuit board connections soldered directly to pogo-pins
- MDM connector for reliable
- assembly and disassembly

Microfluidic Platform (manifold – Ron Renzi, SNL/CA)





CINT Discovery Manifold with Integrated Connector



Specifications:

- 64 pogo-pin connections utilizing one common interface connector
- 32 fluidic connections
- 1.5 inch square top/bottom access
- O-ring seals to 500 psi
- Dowel pin alignment
- 4-place draw-latch compression
- Circuit board connections soldered directly to pogo-pins
- MDM connector for reliable assembly and disassembly

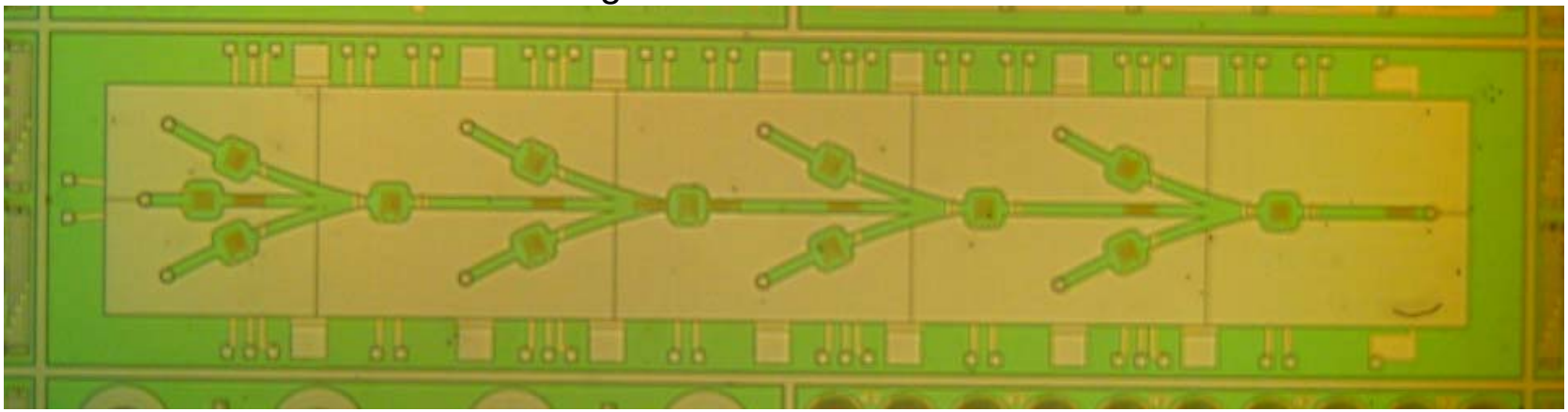
Approximate Size: 2.5” x 3.5” x 1.25”

R.F.Renzi 11/08/05

Microfluidic Platform –Manifold and Manufactured Network

The microfluidic Discovery Platform™ is nearing completion and removal, and the manifold for use of the platform is complete. It is estimated that the assembly can be completed in early October.

Microfluidic Network before dicing and release from the wafer.

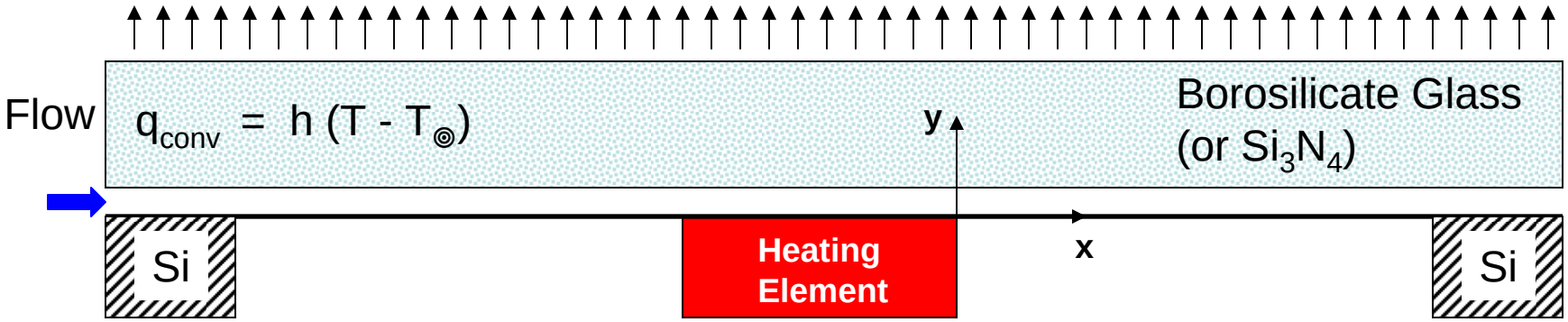


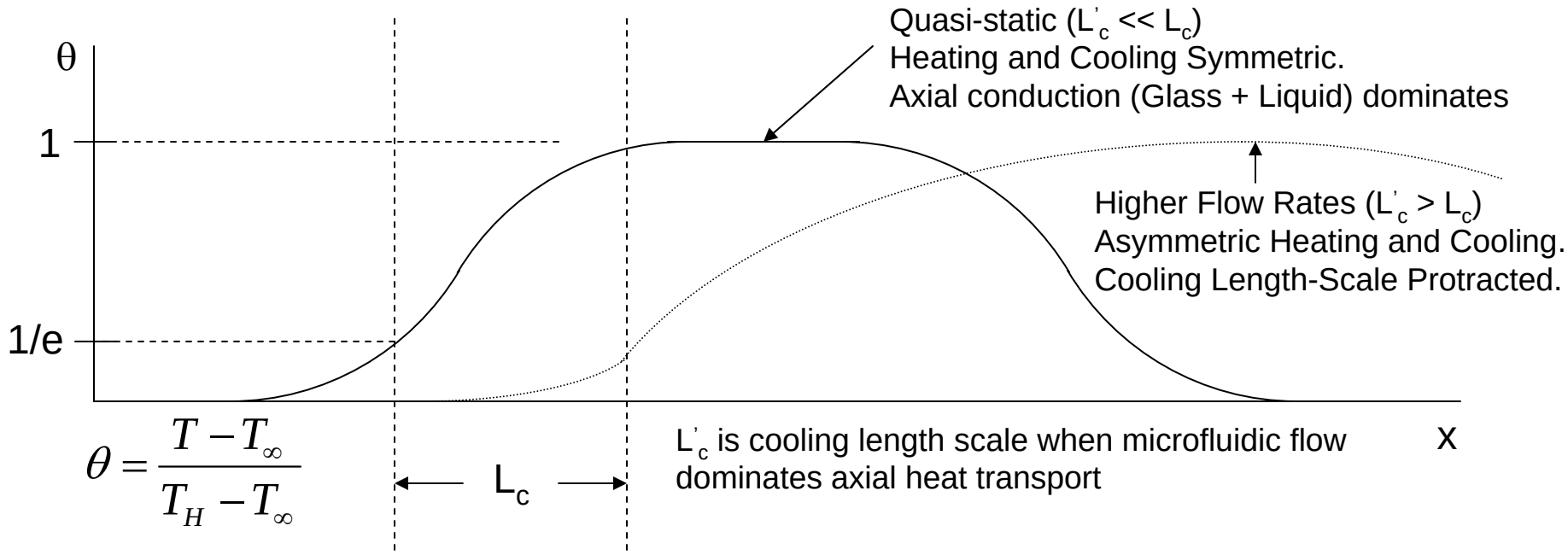
Timeline

(end of August)

- Bosch etch of wafers — next week
- Release — 2 weeks
- Fan out parts — ready
- Manifold — ready
- Anodic Bonding — 1-2 weeks
- Summary: 4-5 parts should be ready in approximately one month
- Control and electronics development – Bill Smith
- Thermal Modeling – Justin Micak

Axial Temperature Profile Modeling





$$\theta = \frac{T - T_\infty}{T_H - T_\infty}$$

Axial Temperature Profile

Globally Air-Cooled Chip (Fan)

Bi << 1 – Convection-Limited Cooling; T = T(x) in chip
(Convection is rate-limiting step for transverse heat transport)
Best Case – High-speed turbulent fan flow over chip:

Bi ~ 10^{-2} – 10^{-1} for glass lid
Bi ~ 10^{-4} for nitride lid

<u>No-Flow or Quasi-Static Temperature Decay From Heat Element</u>
(Conduction-Dominated Axial Heat Transport)

$$\theta = e^{-x/L_c} \qquad L_c = \sqrt{\frac{kd_g}{2h}}$$

$k \equiv$ Thermal Conductivity
$d_g \equiv$ Depth (thickness) of Glass
$h \equiv$ Convection Coefficient

<u>Dynamic Solution: Effect of Microchannel Flow</u>
(Convection-Dominated Axial Heat Transport)

$$\theta = e^{-x/L_c^{'}}$$

$$\theta = \phi\left[1 - e^{-x/L_c^{'}}\right] \qquad L_c^{'} = \frac{\rho C_p v d_{ch}}{2h}$$

$\rho \equiv$ Fluid Density
$C_p \equiv$ Fluid Heat Capacity
$v \equiv$ Mean Fluid Speed
$d_{ch} \equiv$ Channel Depth
$\phi \equiv$ Scaled Heating Power

Axial Temperature Profile

Liquid-Cooled Chip

Bi >> 1 – Conduction-Limited Cooling; T = T(x,y) in chip
(Conduction is rate-limiting step for transverse heat transport)

- Minimum Axial Temperature Gradient
- Feasible with Liquid-Cooled Chip such as by cooling channels in a 2-Layer Glass Lid

$$L_c \sim d_g$$

Also important (particularly for creating a *general* tool):

- Independent Control of Heating and Cooling Profiles
- Independent Control of Temperature Profile at Each Heating Element
- Cooling of Bulk Si to Prevent Overheating Chip

Axial Temperature Profile

Bottom Line

Minimization of temperature gradient with minimum fluid residence time in gradient when flow rate is set such that: $L_c \sim L'_c$

Decay Length:

Air-Cooled

L_c ~ 1 mm for glass lid (*Unacceptable and Unfeasible*)

L_c ~ 100 µm for nitride lid

Liquid-Cooled

L_c ~ 200 µm for glass lid

Fluid Residence Time in Temperature Gradient:

Nitride

t_{rg} ~ 40 ms

Liquid-Cooled Glass

t_{rg} ~ 8 ms

- Must have liquid cooling for glass lid.
- Integrated liquid cooling provides best temperature control
- Nitride Lid is another option:
  - Need mixing design for confluence
  - Shorter channels (~1 mm?)
  - Less control of temperature profile and longer heat/cool res. time
  - How do we keep from heating entire chip unless T cooled down to background temperature between each heating element?

Technical Challenges

	Fall 2006	Winter 2006	Spring 2007	Summer 2007	Fall 2007
Initial Design	✓				
Heat and Mass Transfer Modeling					
Design Refinement					
Alpha Platform					
Internal Testing					
Literature Prep.					
User Testing					
Masks	✓				
Production					

Estimated Cost per Final Device

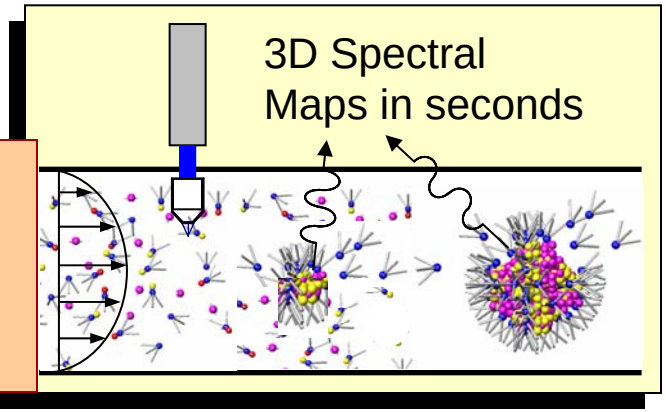
- Our SAMPLES program offers module fabrication for $10K/module. There are approximately 50 good die sites or reticle fields on each wafer. The $10K buys you (two wafers worth of devices) or 100 unreleased modules.
- The cost for release etch for outside customers would be $2000 for 100 modules.
- The fluidic access would require that the wafers be lapped and polished ($180/wafer) and then Bosch etched ($1300/wafer).

Total processing cost of 100 released modules ~ $15000 or $150/device.

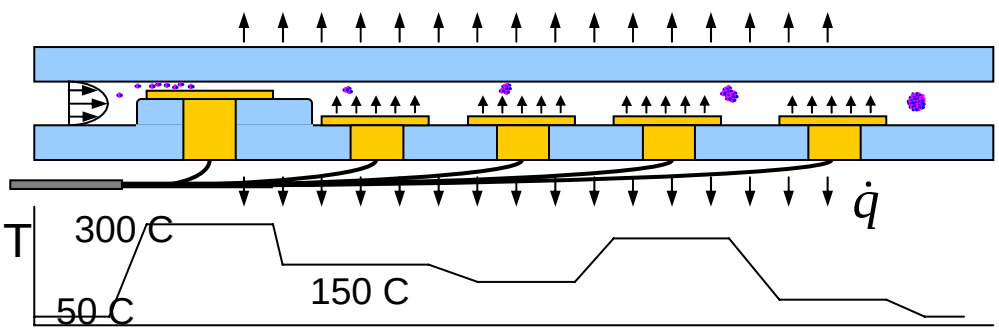
Future Directions

Improved Optical Diagnostics:

3D Hyperspectral Scanner
- Sub-μm resolution
- Single-dot sensitive
- 3D scanning



- Advanced Microfabrication: local temperature control, mixing, etc.



- Other Chemistries and Materials

Issues to Address

- How complex should prototypes be?
  - Highly complex microfluidic systems can be prepared by microlithography, with integrated heaters, electrodes, and sensor packages.
  - Degree of mixing, temperature control
- How do we monitor the reaction *in situ*/kinetically?
  - Spatially resolved monitoring can be used to study NC nucleation and growth processes.
  - Calorimetry, optics
- What timescales are involved for basic understanding?
  - Rapid monitoring of chemical changes is needed for process modeling and optimization.
- What problem(s) should the first generation DP™ address?
  - Q-dot synthesis, assembly, chemical assembly, catalysis....

Users Interests

- materials
- systems
- applications

Types of Studies

- “magic number” synthesis
- optical effects during synthesis
- reaction kinetics
- growth and dissolution
- heteronucleation of shell structures
- shape control
- non-congruency in growth
- ligand effects