

### **Microfluidic components 2021**

#### sami.franssila@aalto.fi

### Contents

- Valving \_\_\_\_\_ flow control
- Pumping \_\_\_\_\_ Tow control
- CE (capillary electrophoresis
- Microreactors

applications

### Basic geometries:straight channel

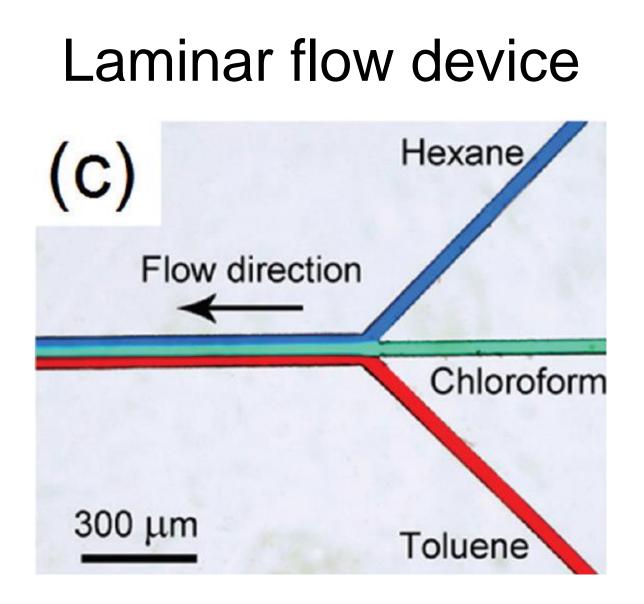
-separation channel -mixer

-microreactor

Typical channel dimensions:

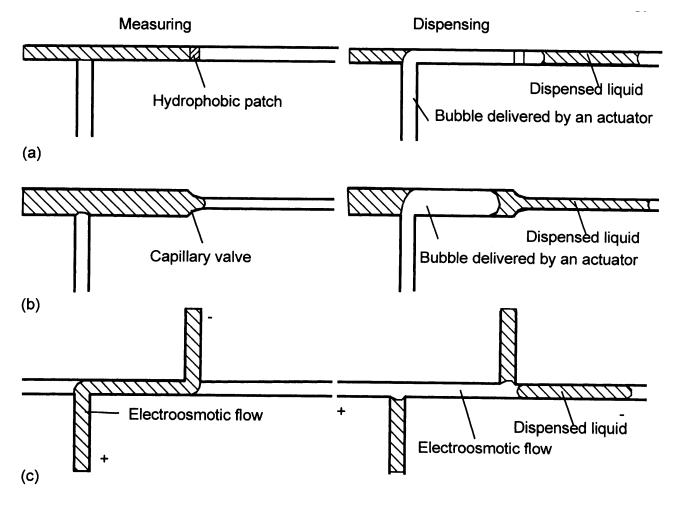
Widths 50-100 µm Depths 10-100 µm (a lot of variability !)

### Basic geometries: X,T,Y,H **Applications:** •CE injectors •mixers •filters reactors



Ren, K.; Dai, W.; Zhou, J.; Su, J.; Wu, H. Proc. Natl. Acad. Sci. U.S.A. 2011, 108, 8162–8166.

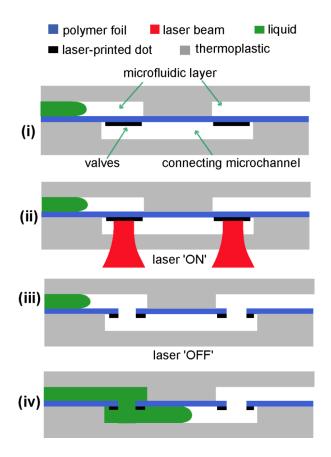
### Dispensers



Nguyen p.

### Valves

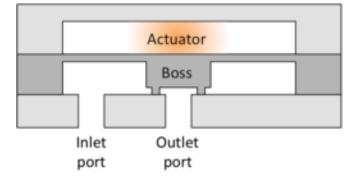
#### One shot valve



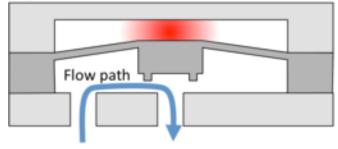
Garcia-Cordero et al: Lab on a Chip - October 2010

#### Open and close valve

**Closed valve** 



#### Open valve



Wikipedia

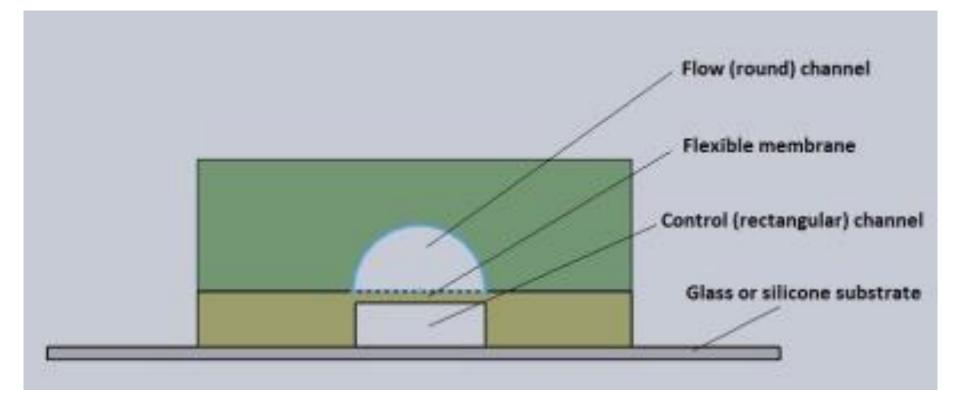
### Passive valves

### Active valves

- mechanical
- geometric
- hydrophobic

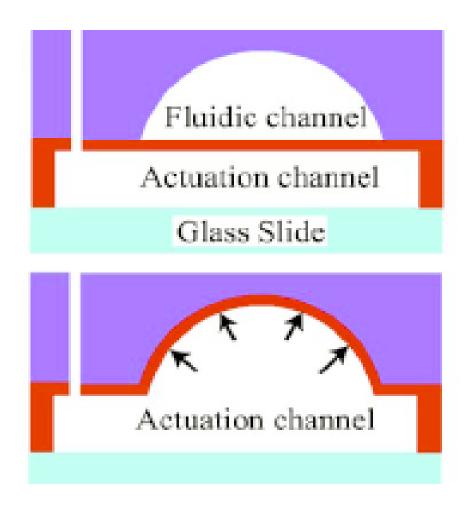
pneumatic thermopneumatic phase-change electrostatic piezoelectric thermal expansion

### Quake valve (diaphragm valve)

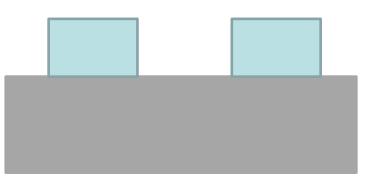


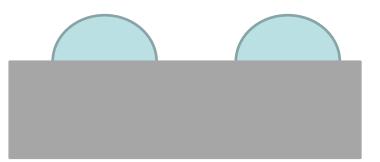
After Stephen Quake, Stanford professor and founder of Fluidigm.

### Why semicircular channel ?

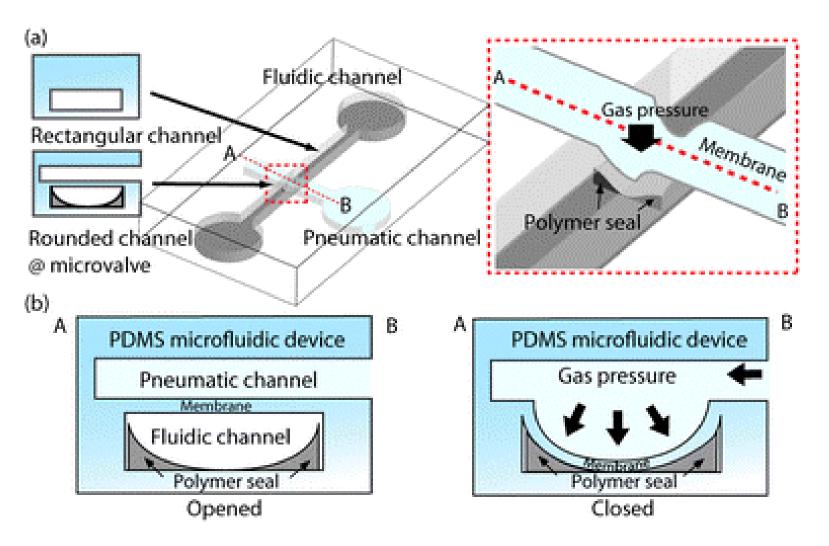


How to make semicircular channel ? Resist flow >  $T_g$ 



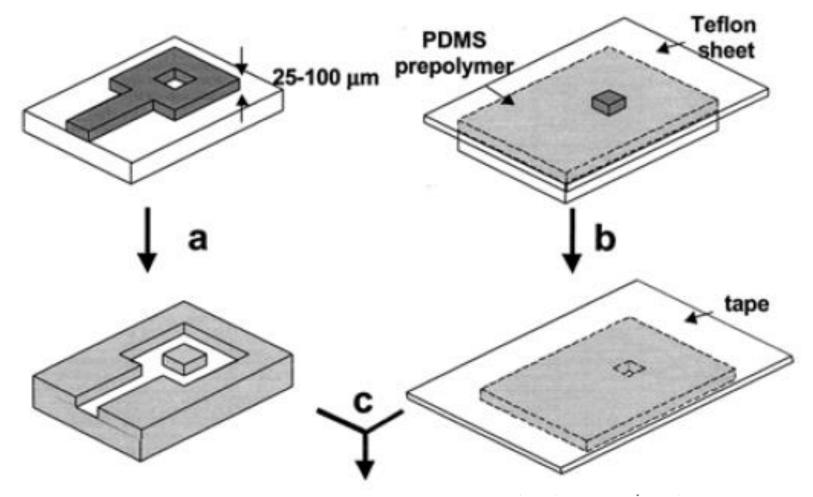


### PDMS membrane valve



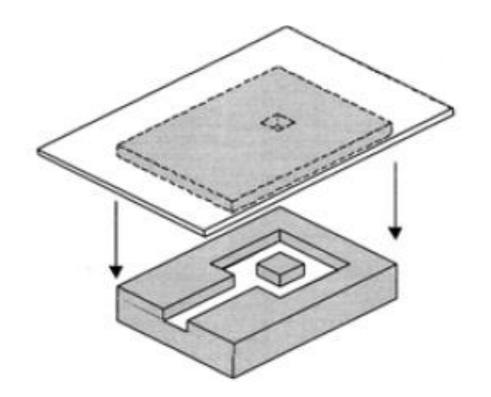
Lab Chip, 2010, 10, 2814-2817

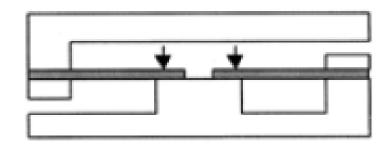
### Flap valve by PDMS molding

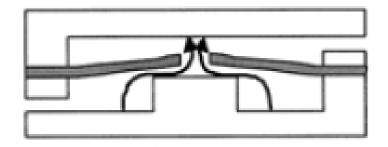


Jeon, N. L., D. T. Chiu, C. J. Wargo, H. K. Wu, I. S. Choi, J. R. Anderson, and G. M. Whitesides, 2002, Biomed. Microdevices 4, 117.

### Flap-valve (2)

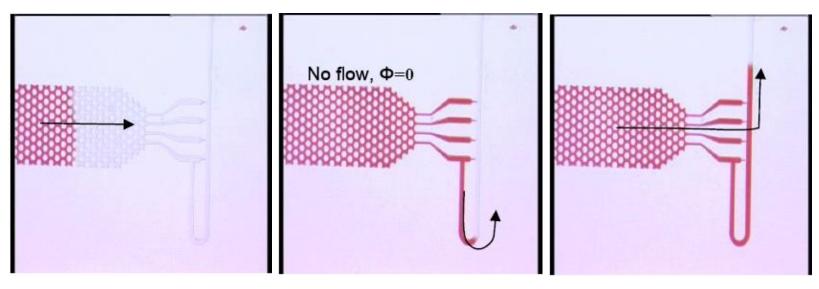






Jeon, N. L., D. T. Chiu, C. J. Wargo, H. K. Wu, I. S. Choi, J. R. Anderson, and G. M. Whitesides, 2002, Biomed. Microdevices 4, 117.

### Geometric valves



(a) (b) (c)

Capillary pump controls the capillary pressure and rate of flow. Rapid constriction of the flow channel will stop the flow. Side channel offers timing of flow.

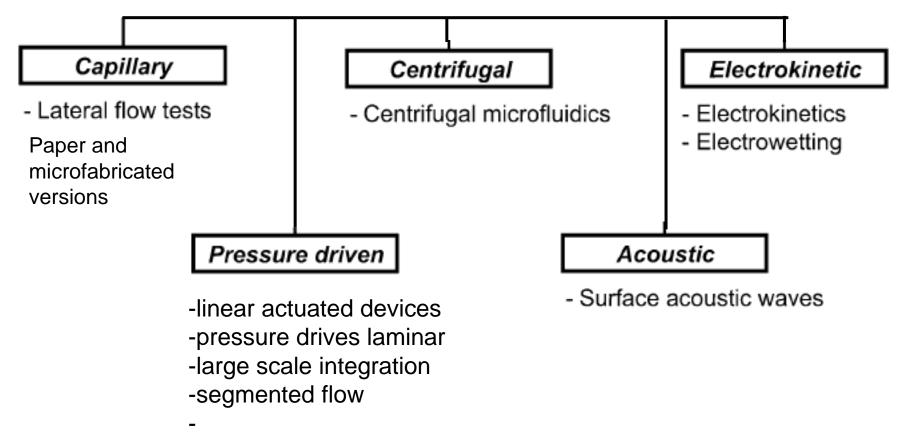
### Pumps

- bubble pumps
- membrane pumps
- diffuser pumps
- rotary pumps
- electrohydrodynamic
- electro-osmotic
- ultrasonic pumps
- vacuum pumps

Criteria:

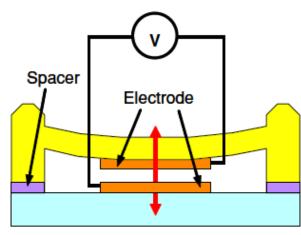
- size, miniaturizationpotentialpower consumptionflow rate
- -reliability

### Pumping operating principles

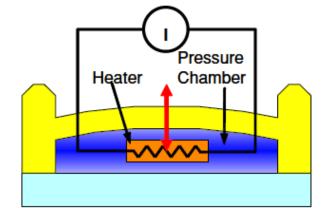


Chem. Soc. Rev., 2010, 39, 1153

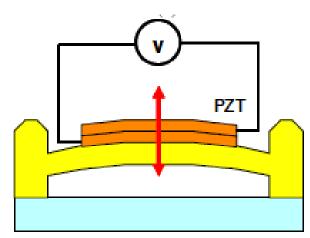
### **Diaphragm pumps: actuation**

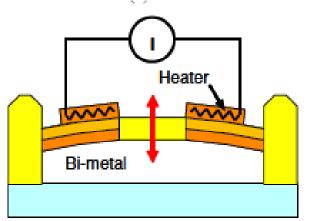


electrostatic



Phase change

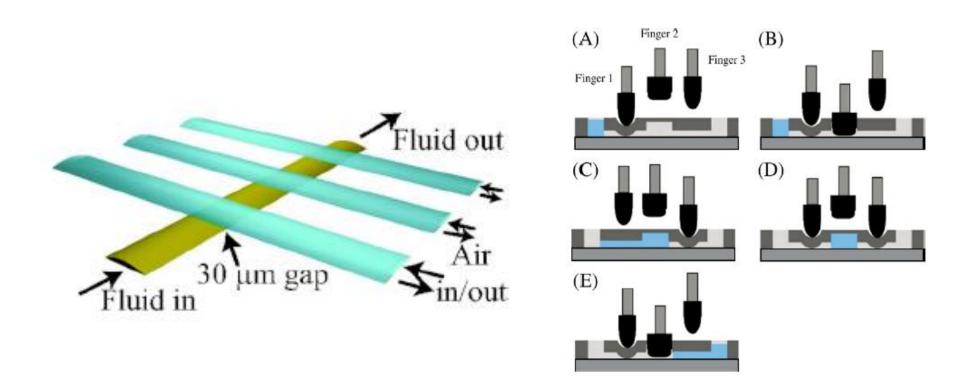




piezoelectric

Thermal expansion

## Peristaltic pump = 3 valves in series



Unger, M. A., H. P. Chou, T. Thorsen, A. Scherer, and S. R. Quake, 2000, Science 288, 113.

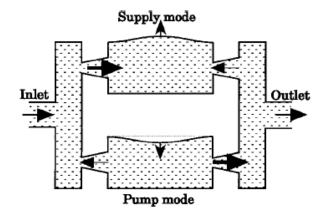
Pilarski PM, Adamia S, Backhouse CJ. An adaptable microvalving system for on-chip polymerase chain reactions. J Immunol Methods 2005;305:48–58.

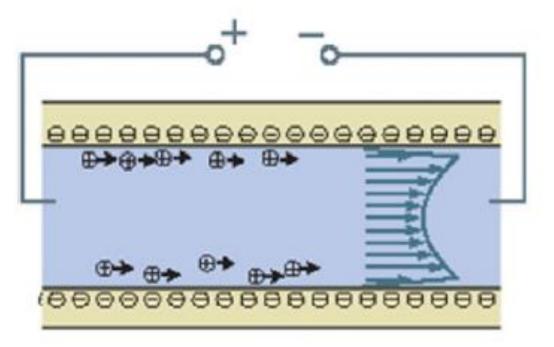
### Pumps without moving parts

Flow is from higher Laplace pressure to lower.

Surface tension driven pump J. Micromech. Microeng. 18 (2008) 087002 (5pp)

ro



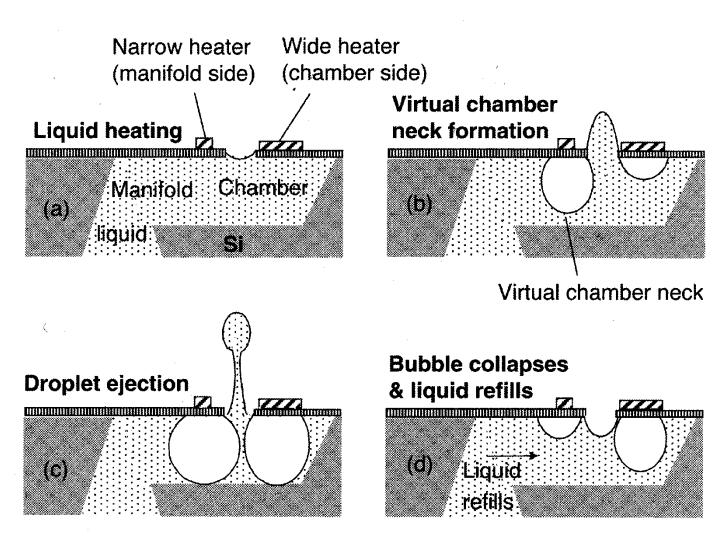


Electro-osmotic pump

J K Luo $^{1,2},$  Y Q Fu $^{2,3},$  Y Li $^4,$  X Y Du $^2,$  A J Flewitt $^2,$  A J Walton  $^4$  and W I Milne $^2$ 

Nozzle-diffuser pump, Olsson, Stemme 1997

### Thermal ink jet

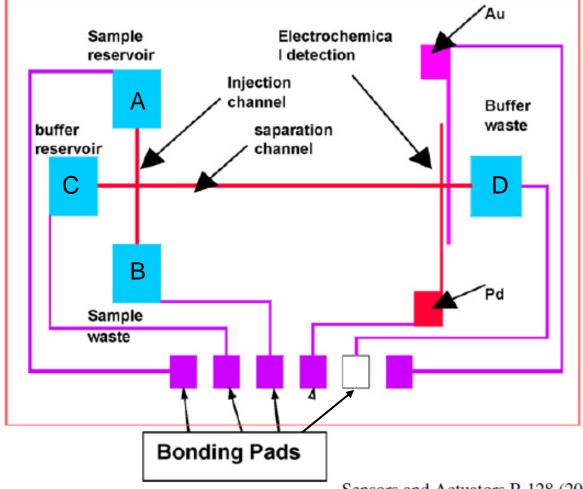


**MEMS Handbook** 

# Chip Example: Electrophoresis chip with electrochemical detection

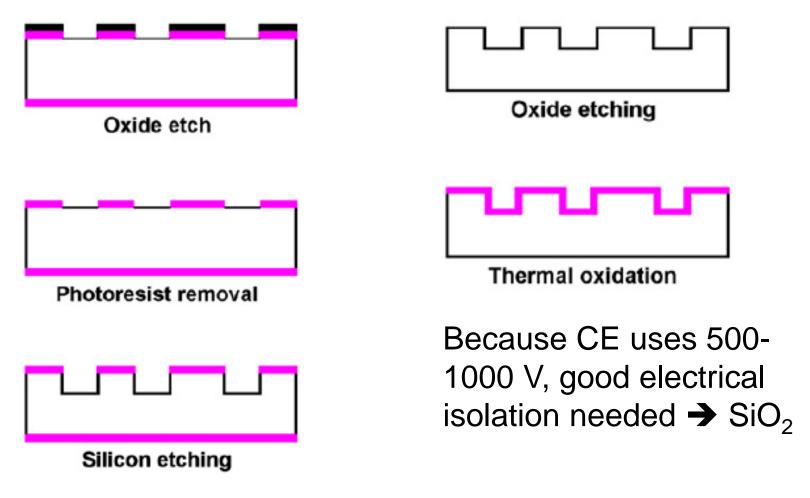
Injection: apply voltage to A and B.

Separation: apply voltage to C and D.

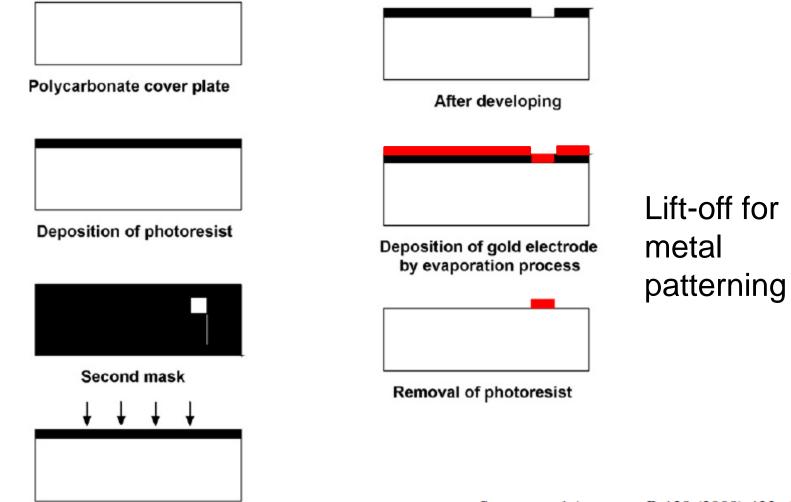


Sensors and Actuators B 128 (2008) 422-426

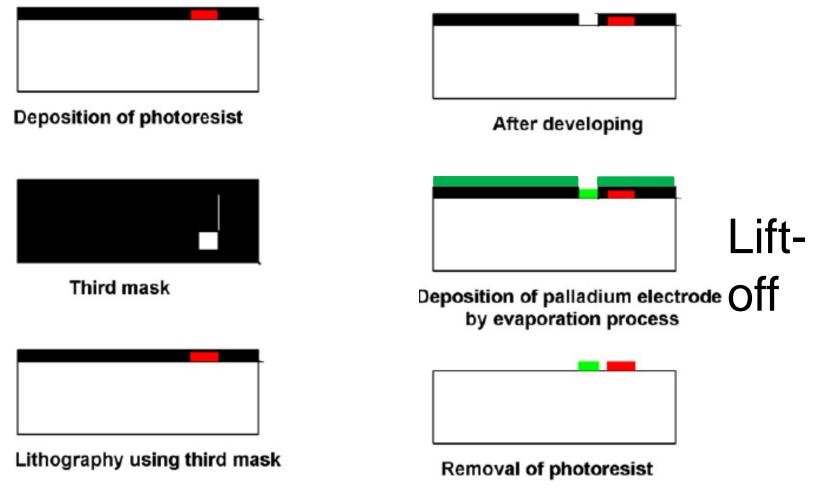
### CE chip fabrication (2): channels in silicon



### CE chip fabrication (3): Au-electrode on polycarbonate



### CE chip fabrication (4): Pd-electrode on polycarbonate

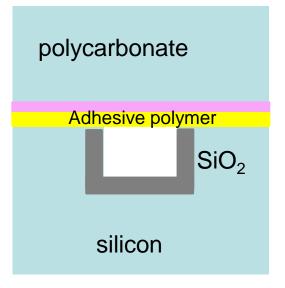


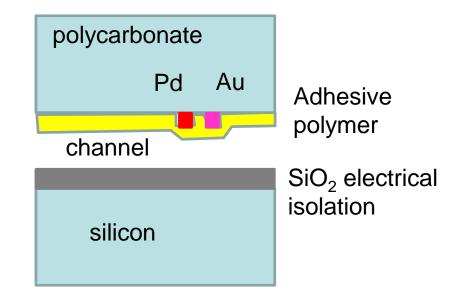
Sensors and Actuators B 128 (2008) 422-426

### Adhesive bonding of Si and PC

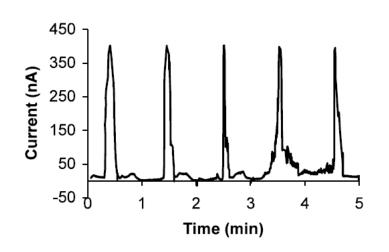
Cross section across the channel

Cross-section along the channel





### **CE** separations



repetition  $\begin{pmatrix} 100 \\ 800 \\ 500 \\ 200 \\ -100 \\ 0 \\ 50 \\ 100 \\ 0 \\ 50 \\ 100 \\ 50 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 100 \\ 10$ 

Electropherogram for multiple injections of 10 mM hydrogen peroxide. Conditions: running buffer, phosphate buffer (10 mM, pH 7.4); separation potential, /400 V; injection potential, /400 V; detection potential, /700 mV

Electropherogram for sample solution containing 50 mM hydrogen peroxide (a), ascorbic acid (b) and uric acid (c), respectively. Conditions: two injections in the same run, other conditions were the same as those in Fig. 3.

### Retraction

Sensors and Actuators B 128 (2008) 422-426

"The publisher regrets that the results presented in Figs. 6–8 are copied without permission or reference from an article that has already been published in Bios 19 (2003) 149–153 (DOI of the original article: doi:10.1016/S0956-5663(03)00178-7; PII of the original article: S0956-5663(03)00178-7)."

In reality, practically everything is copied from:

Ralph Wilke & Stephanus Büttgenbach:

A micromachined capillary electrophoresis chip with fully integrated electrodes for separation and electrochemical detection,

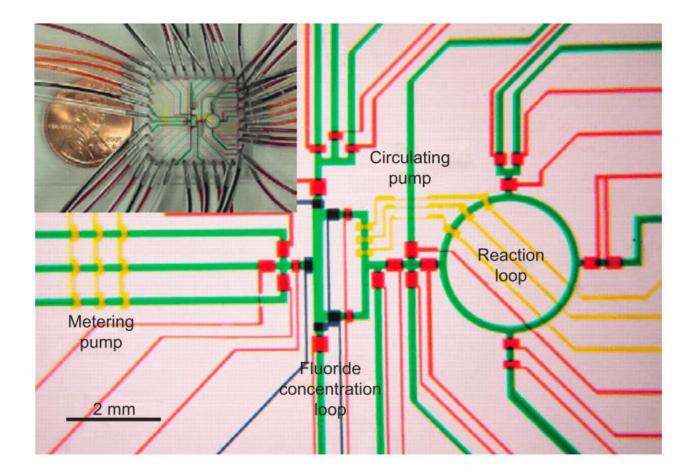
Biosensors and Bioelectronics, Volume 19, Issue 3, 2003, Pages 149-153

### Original vs. copy

To overcome this difficulties we used a bonding procedure with an amorphous fluorocarbon polymer named CYTOP<sup>®</sup>. CYTOP<sup>®</sup> is a registered trademark of Asashi Glass Co. (Tokyo, JP) and was purchased from Sigma-Aldrich (Milwaukee, WI) as poly(1,1,2,4,4,5,5,6,7,7-decafluoro-3-oxa-1,6heptadiene), silane modified, 9 wt.% solution in perfluorotributylamine. Both the cover plate and the microchannels were dehydrated at 90 °C for at least 30 min on a hotplate. The cover plate was cooled to room temperature and subsequently spin-coated with CYTOP<sup>®</sup>. During a softbake at 90 °C for 30 min the solvent was driven out. The two substrates were then aligned and bonded at 160 °C for at least 2 h with weights on top.

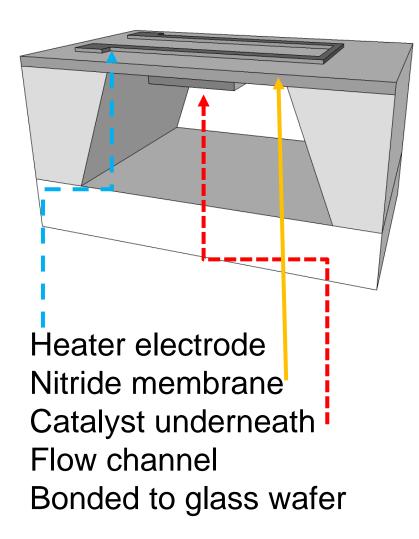
To overcome this difficulty we used a bonding procedure with an amorphous fluorocarbon polymer named CY-TOP®, CY-TOP<sup>®</sup> is a registered trademark of Asoshi Aldrich as poly(1,1,2,4,4,5,5,6,7,7-decafluoro-3-ora-1,6-heptadiene), silane modified, and 9 wt% solution in perfluorotributylamine. Both the cover plate and micro-channels were dehydrated at 90 °C for 30 min on the hot plate. The cover plate was cooled to room temperature and subsequently spin coated with CY-TOP®. During a soft bake at 90 °C for 30 min and the solvent was driven out. The substrates were then aligned and bonded at 160 °C for 2 h with weights on top.

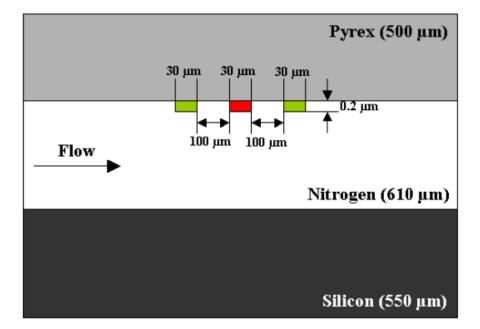
#### Microreactors



C.-C. Lee *et al.*, "Multistep synthesis of a radiolabeled imaging probe using integrated microfluidics.," *Science*, 2005.

### Simple microreactor

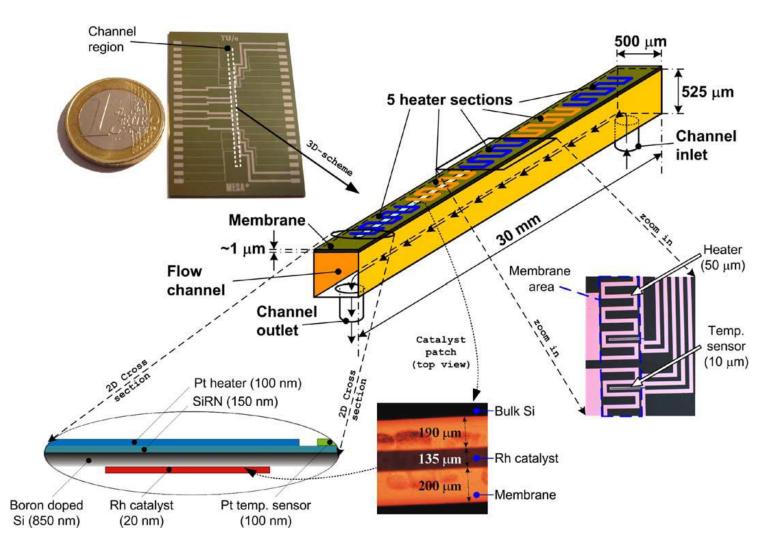




Microreactor dimensions Shin & Besser,

J. Micromech. Microeng. 16 (2006) 731-741

### Linear microreactor



R.M. Tiggelaar et al. / Sensors and Actuators A 119 (2005) 196–205

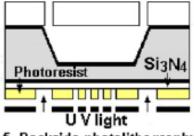
# Microreactor with platinum heaters and glass cover



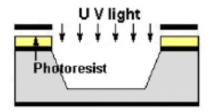
1. Thermal oxidation



3. Inside thermal oxidation and silicon-glass anode bonding



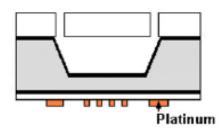
5. Backside photolithography of heaters & sensors



2. Front side photolithography and KOH etching



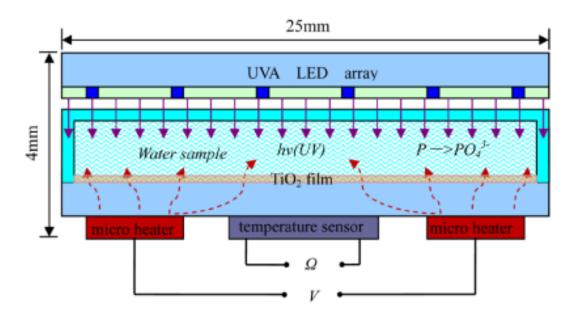
4. Depositing Si3N4



6. Depositing Ti/Pt and lift-off photoresist

Transparent glass for detection; silicon spreads heat well for temperature uniformity

### TiO<sub>2</sub> photocatalytic reactor



"Thermally assisted photocatalytic microfluidic chip and its application to the digestion of total phosphorus (TP) in freshwater" Heating only locally → power consumption reduced.

UV activation  $\rightarrow$  lower temperature suffices.

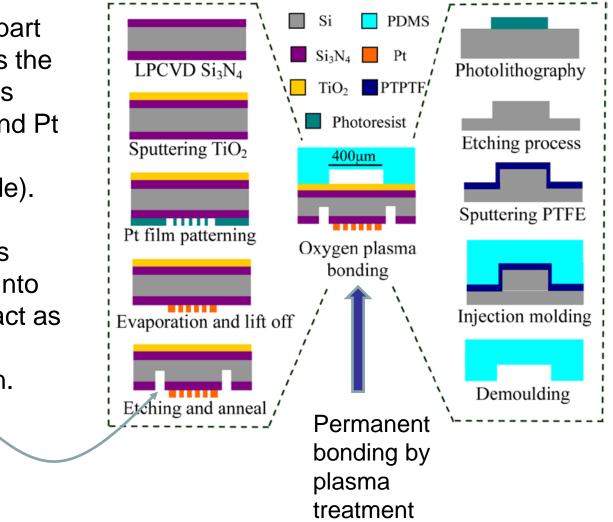
 $TiO_2$  photocatalytic  $\rightarrow$  no oxidant consumption

Integrated electrodes can monitor phosphorous removal.

### TiO<sub>2</sub> reactor fabrication

Silicon part contains the thin films (TiO2 and Pt heater electrode).

Grooves etched into silicon act as thermal isolation.

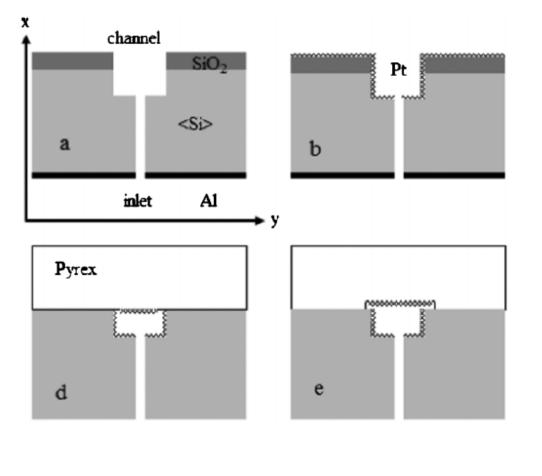


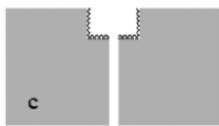
PTFE (Teflon) acts as an anti-adhesive layer, making release from mould easier.

PDMS transparency in UV is essential.

> Tong et al: J. Micromech. Microeng. 25 (2015) 025006

### Catalytic microreactor

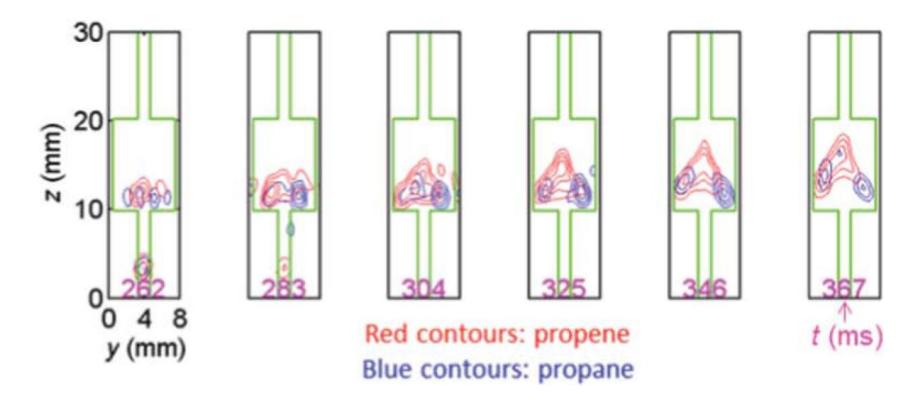




Having all four walls of the reactor covered by a catalyst requires aligned bonding.

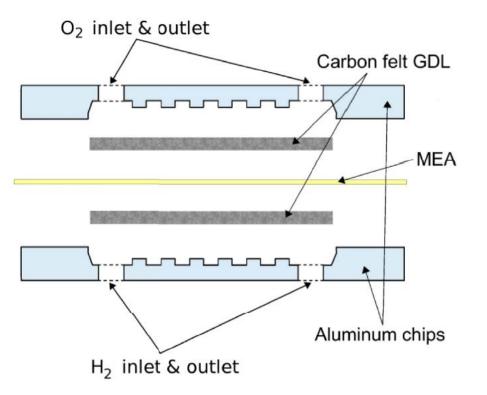
Cover wafer with catalyst cannot be directly bonded, because it is not smooth.

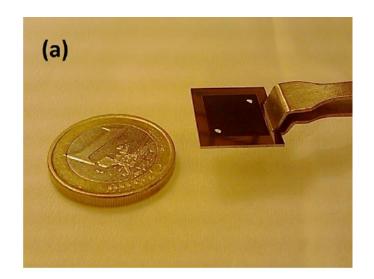
# Hydrogenation of propene into propane



Flow patterns detected by NMR with 10 ms time resolution.

## Micro fuel cell



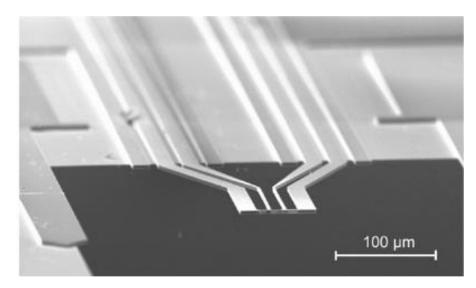


Anode and cathode are microfabricated, But many manual assembly steps are needed to put together a functioning fuel cell.

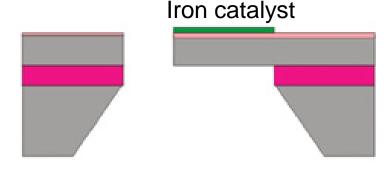
Borghei, Franssila et al: Energy 65 (2014) 612e620

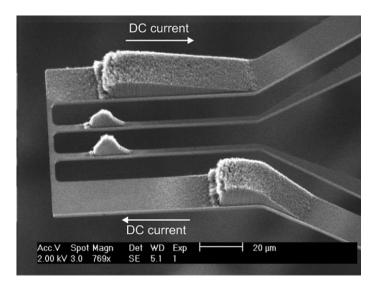
Scotti, Franssila... JMEMS 2014

#### A microreaction spot



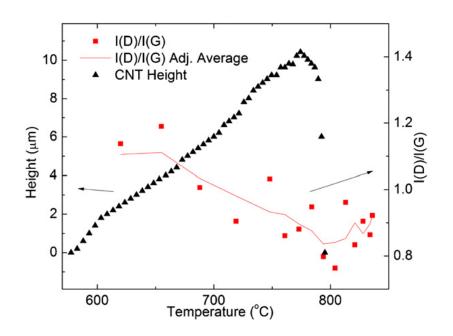
Locally heating the reaction spot only, in this case to high temperature of ca. 800°C for CNT synthesis.

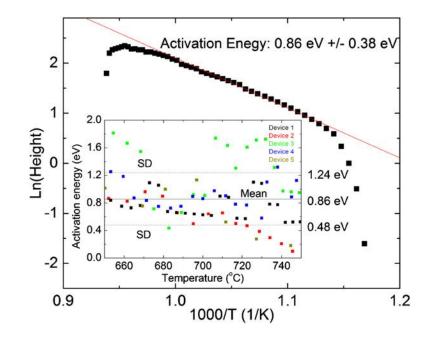




Daniel S Engstrøm<sup>1</sup>, Nalin L Rupesinghe<sup>2</sup>, Kenneth B K Teo<sup>2</sup>, J.Micromech.Microeng. 2011 William I Milne<sup>3</sup> and Peter Bøgild<sup>1</sup>

#### **CNT** results





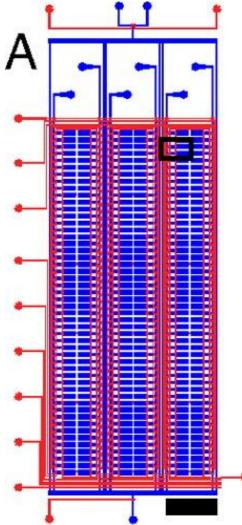
CNT height as a function of growth temperature.

Raman of  $I_D/I_G$  ratio

# Activation energy of CNT growth

Daniel S Engstrøm<sup>1</sup>, Nalin L Rupesinghe<sup>2</sup>, Kenneth B K Teo<sup>2</sup>, William I Milne<sup>3</sup> and Peter Bøgild<sup>1</sup> J.Micromech.Microeng. 2011

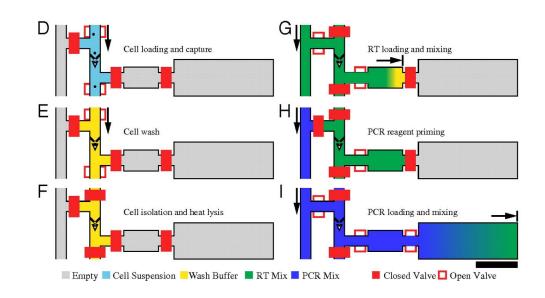
## Combining reactors with valves



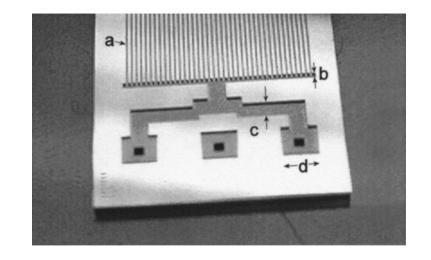
Adam White et al: PNAS August 23, 2011 108 (34) 13999

Six independent sampleloading lanes, each containing 50 cellprocessing units → 300 parallel PCR reactions. 1,000 measurements on a device with an area of one square inch.

(*D*) A single-cell suspension is injected into the device. (*E*) Cell traps isolate single cells from the fluid stream and permit washing of cells to remove extracellular RNA. (*F*) Actuation of pneumatic valves results in single-cell isolation prior to heat lysis. (*G*) Injection of reagent (green) for RT reaction (10 nL). (*H*) Reagent injection line is flushed with subsequent reagent (blue) for PCR. (*I*) Reagent for qPCR (blue) is combined with RT product in 50 nL qPCR chamber.



#### Microreactor pros and cons





Small volume good if expensive and/or dangerous chemicals

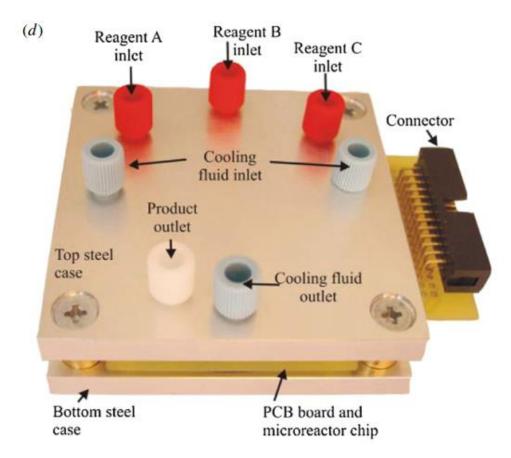
Fast reactions because small diffusion distances

Large surface area (either positive or negative effect)

Good temperature control and fast ramp rates

Good flow control because of laminar flow

## **Reactor packaging**

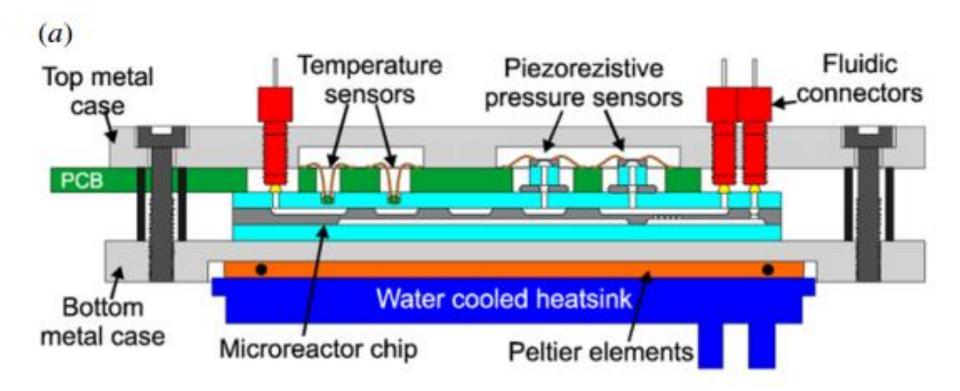


Fluidic inlets/outlets for reactants, products, waste

Fluidic inlets for cooling fluids, broth, oxygen

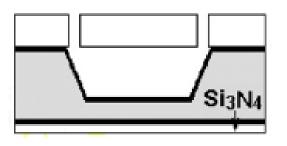
Electrical connections to driving electrodes and detector electrodes

## The complete system



J. Micromech. Microeng. 23 (2013) 035014

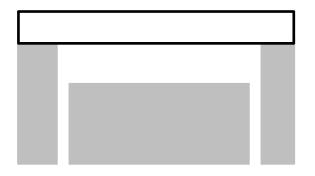
#### Inlet/outlet holes



Sandblasting in glass

Punching in PDMS

active channel

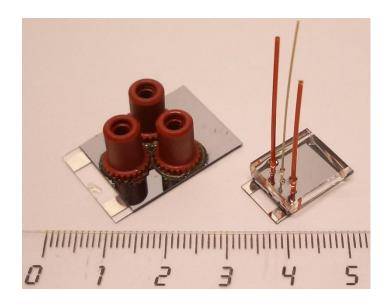




DRIE/plasma etching in silicon

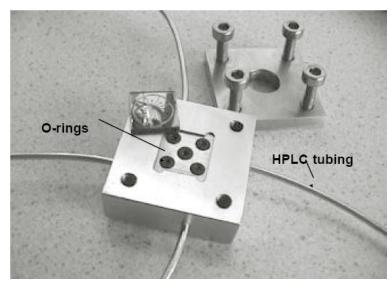
Drilling in copper

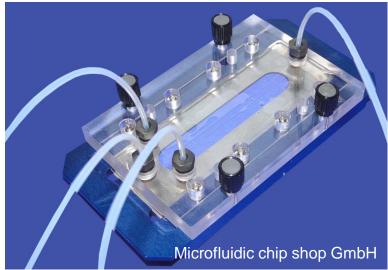
#### Fluidic connectors



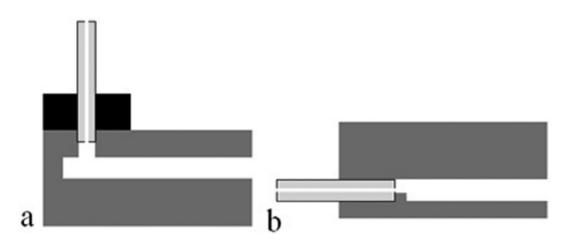
Ville Saarela, TKK

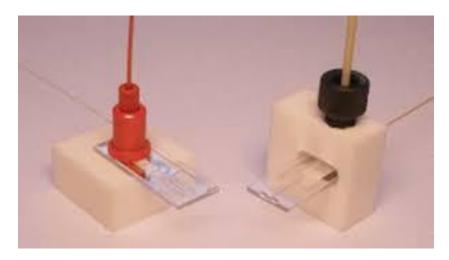
Micro-macro conversion is often difficult, because of size differences → dead volume issues





#### In-plane vs, out-of-plane



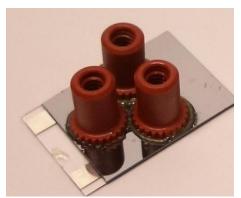


Liquid connection is from the side; and gas connection from top.

## Interconnect consideration

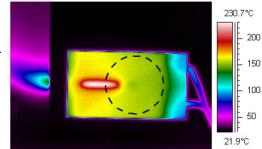
 Is the microfluidic device re-usable or disposable?
In other words: how frequently do world-to-chip connections need to be broken and re-made?

- the amount of chip area used by the interconnections
- pressure tolerance
- temperature tolerance
- automation vs. manual labor
- price

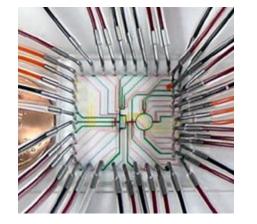


Ville Saarela, Aalto

Very large area taken up by connectors Ville Saarela, Aalto



Chip running so hot that gluing is not an option



Cumbersome manual connections

https://sites.google.com/site/van damlab/research/microfluidicinterfaces

#### Take home messages

Integrated systems are often desired, but difficult.

Silicon and glass for high-T and high pressure systems.

Some of the components remain macroscopic, e.g. pump or detector.

Integrated valves enable systems that start to resemble microelectronic circuits.

Microreactors benefit from miniaturization in multiple ways.

Interconnects often non-micromachined, and cumbersome.