Bonding of glass

- Introduction
- Field-assisted bonding with Si
- Fusion bonding
- HF-assisted bonding
- Pressure-assisted low-temperature fusion bonding
- Application : capillary electrophoresis microchip
- References

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Field-assisted thermal bonding with Si

- Or : **anodic bonding, electro-static bonding**
- To join glass (for example Corning #7740 = Pyrex) to Si
- Low process temperature (180-400 °C)
- Applied voltages : 200-1000 V
- Contacting surfaces need to be flat (roughness<1 μm) and dust-free

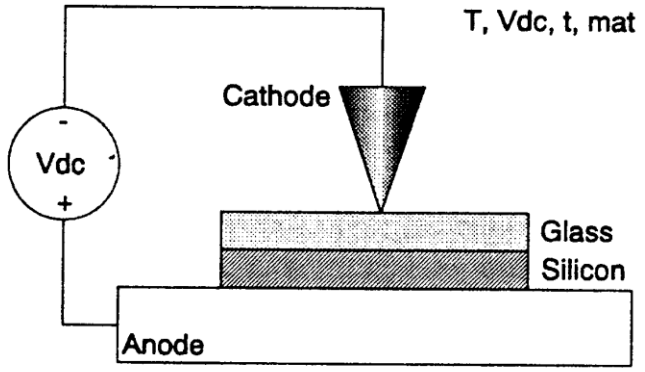


FIGURE 8.12 Principle sketch of anodic glass-to-Si bonding. Control parameters are temperature (300–400°C), bias voltage (700–1200 V), time (~2′), and materials (glasses, Si, SiO_2).

Thermal expansion coefficient of bonded materials need to be in the same range.

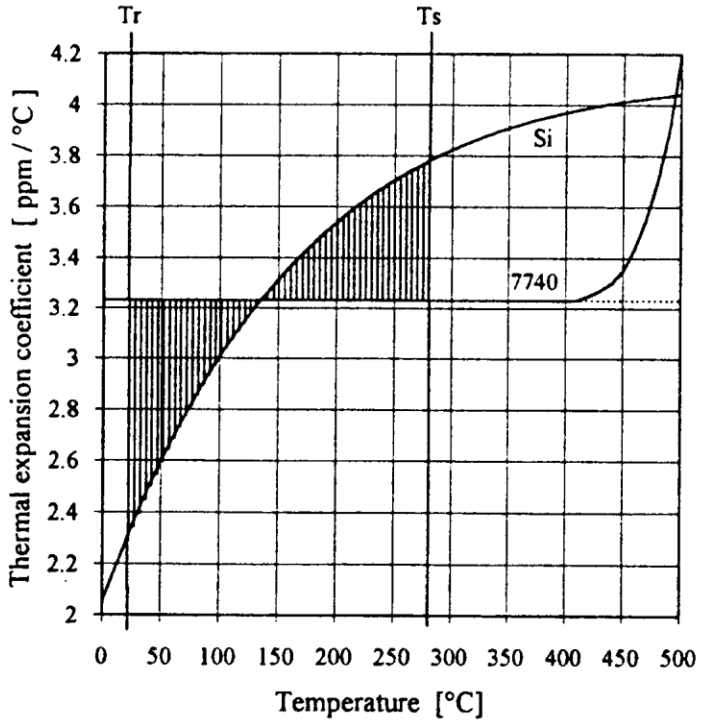


FIGURE 8.13 Thermal expansion coefficients of Si and Corning 7740 Pyrex. Tr = room temperature; Ts = seal temperature. The temperature Ts is a variable. (From Peeters, E., Process Development for 3D Silicon Microstructures with Application to Mechanical Sensor Design, Ph.D. thesis, Catholic University of Louvain, Belgium, 1994. With permission.)

Mechanism of anodic bonding

- At high T, **glass** is **conductive solid electrolyte**
- Bonding results from **the migration of Na^+** (3-5 % concentration in Pyrex) toward the cathode
- This leaves **negative space charge** (bound negative charges) in the region of the glass-Si interface.
- All voltage **is dropped over this region**, pulling glass and Si in intimate contact
- **Covalent bonds** are formed between the surface atoms of the glass and the Si.
- **Disadvantages:**
  - High electrical field
  - One-wafer process
  - Mismatch in thermal expansion

‘Improved’ anodic bonding

- **Ti-mesh electrode** is deposited over the glass wafer to give uniform high voltage over the interface. Bonding parameters: 400 °C, 600 V, 5 min.
- Deposition **of intermediate layers of SiO_2 and Al** to screen underlying Si from high electrical fields. Glass is bonded to the Al layer.
- Other possibility: **SiO_2 poly-Si bilayer**
- Bonding of two Si wafers with an **intermediate sputtered or evaporated borosilicate glass** (4-7 μm). But: glass sputtering rate is very slow.
- Bonding of **2 Si wafers, each with 1 μm SiO_2** is possible. Parameters: 900 °C, 30 V, 45 min

Thermal fusion bonding

- Based on **chemical reaction between OH-groups present at the surface** of the native or grown oxides covering the wafers
- Bond quality depends on temperature and **surface roughness (< 4 nm)**.
- **High bonding temperature (800 °C)** prevents the incorporation of active electronics before the bonding.
- Same technique can be applied for bonding of :
  - Oxidised Si – oxidised Si
  - Bare Si – bare Si
  - Oxidised Si – bare Si
  - Si – glass
  - Quartz – quartz

Fusion bonding procedure

- Oxidised surface must undergo **hydratation**: soaking wafers in H_2O_2-H_2SO_4 mixture, diluted H_2SO_4 or boiling HNO_3. This forms hydrophilic top layer of O-H bonds.
- Additional **treatment in oxygen plasma** enhances number of OH groups at surface.
- Wafers are **rinsed in DI water** and dried
- When brought into contact, **direct bonding of the 2 wafers**
- **High temperature anneal** increases bond strength

Mechanism of fusion bonding

- Main reaction: $Si - OH + OH - Si \rightarrow H_2O + Si - O - Si$
- At anneal T<300 °C, **silanol (Si-OH) groups give rise to hydrogen bonding.**
- At 300 °C, OH-groups form water molecules, which can form water vapour voids
- Above 300 °C, water vapour disappears and **strong siloxane bond (Si-O-Si) develops**

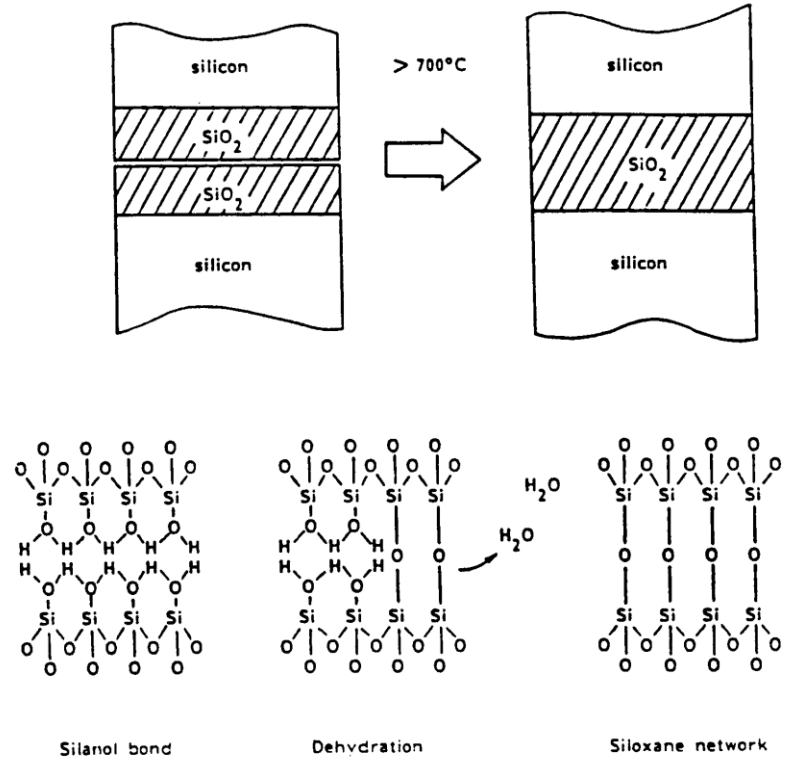


FIGURE 8.14 Schematic illustration of the silicon-to-silicon bonding by fusion and a proposed mechanism.

Intermediate thin-layer thermal glass bonding

- **LPCVD phosphosilicate glass** (PSG) (1-2 μm) can be used for Si to Si wafer bonding. Anneal at 1100 °C for 30 minutes necessary.
- Low **temperature sealing glasses 'frits'** exist.
  - Sealing temperatures: 415-650 °C.
  - Glass can be spinned, sprayed, screen-printed.
  - Preglazing to get rid of organic residues
- **Spin-on-glass (SOG)** ($Si(OH)_x$ with $2<x<4$)
  - 50 nm thick film is baked at 250 °C for 1 hr.
  - Annealing for 1 hr at 1150 °C to sinter

HF-assisted bonding

- Room temperature bonding process
- HF ($C \geq 0.1$ %) is applied in between 2 glass or SiO_2 wafers.
- Pressure is applied (~1 MPa)
- Mechanism: formation of intermediate bonding layer.
- Advantages:
  - no temperature induced stress
  - no degradation of metal leads (Au-Cr, Al,..) and IC ’s.
  - bonding strengths of 5-10 MPa

(Nakanishi et al., Proc. MEMS ’97, p. 299 (1997))

HF-assisted bonding

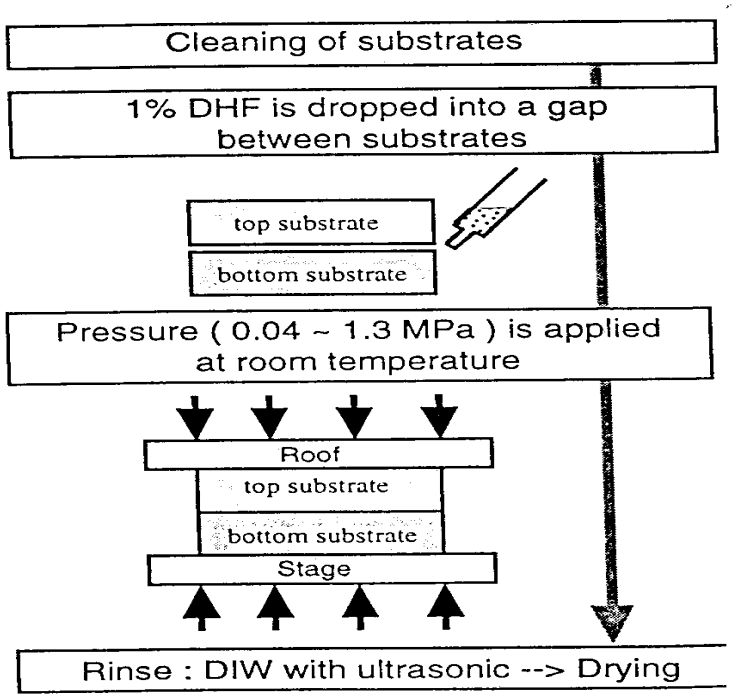
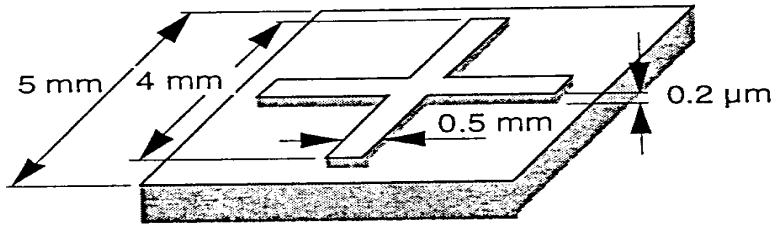


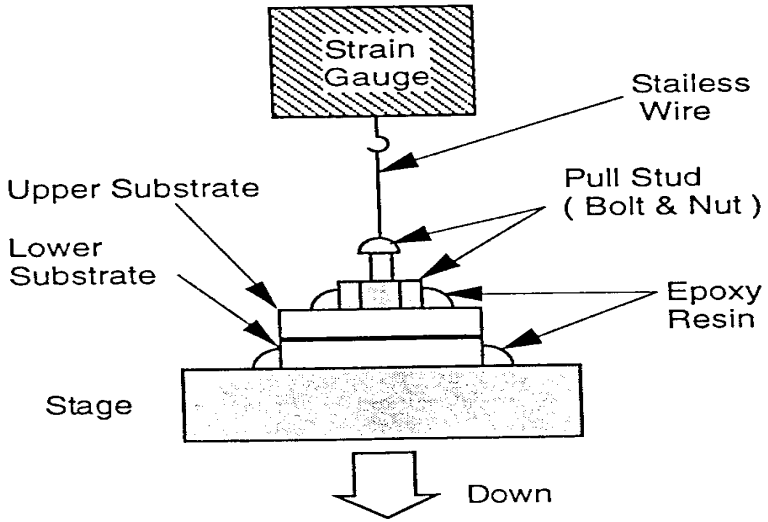
Fig.1 Procedure of HF-bonding

- Substrate cleaning: acetone, methanol, H_2SO_4+H_2O_2
- 1% HF etching to remove surface layer (5 nm)
- 1% diluted HF (DHF) is dropped in gap
- 18 hours of pressurised bonding

HF assisted bonding



(a) Schematic of test-chip



(b) Schematic of the tensile test equipment

Fig.2 Set-up for evaluation of bond strength

Bond quality is evaluated by bond strength measurements

HF assisted bonding

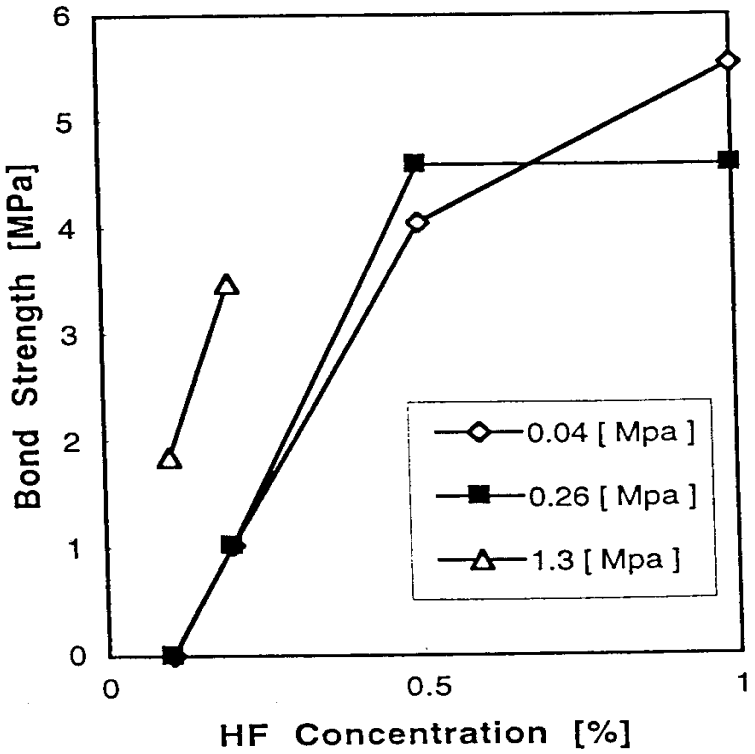


Fig.3 Relationship between HF concentration and bond strength

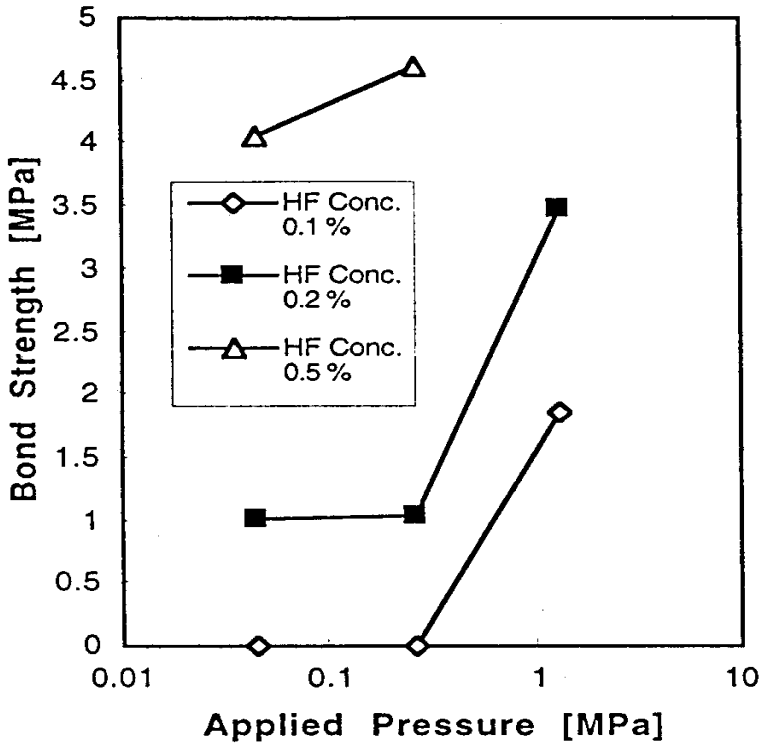


Fig.4 Relationship between applied pressure and bond strength

HF assisted bonding

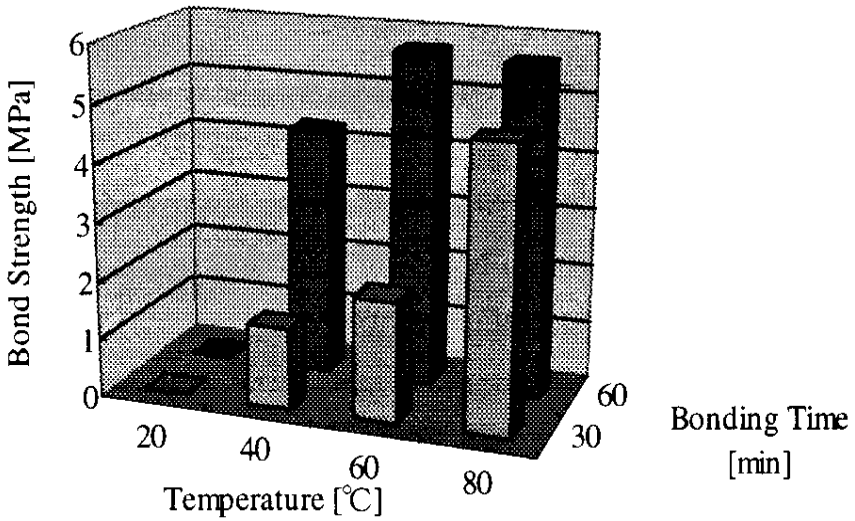


Fig.2 Relationship Between the Bond Strength and Temperature, Time for Bonding
(HF Conc. : 0.5 wt%, Applied Pressure : 1.3 MPa)

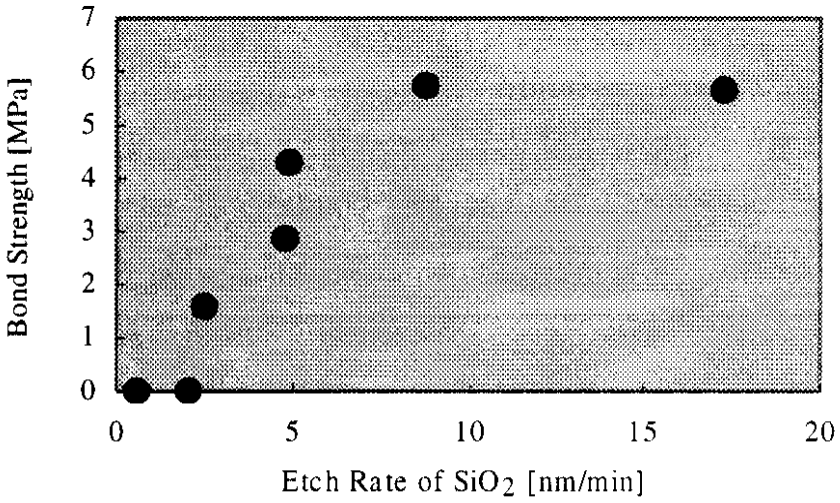


Fig.3 Relationship Between the Bond Strength and the Etch Rate of SiO_2
(Bonding Period : 1 hour, Applied Pressure : 1.3 MPa)

Pressure-assisted low-temperature fusion bonding

Low temperature bonding process (100-200 °C)
Cleaning of the substrates (no HF)
Pressure is applied (~10 MPa)
Mechanism:

$$Si-OH+OH-Si \rightarrow Si-O-Si+H_2O$$

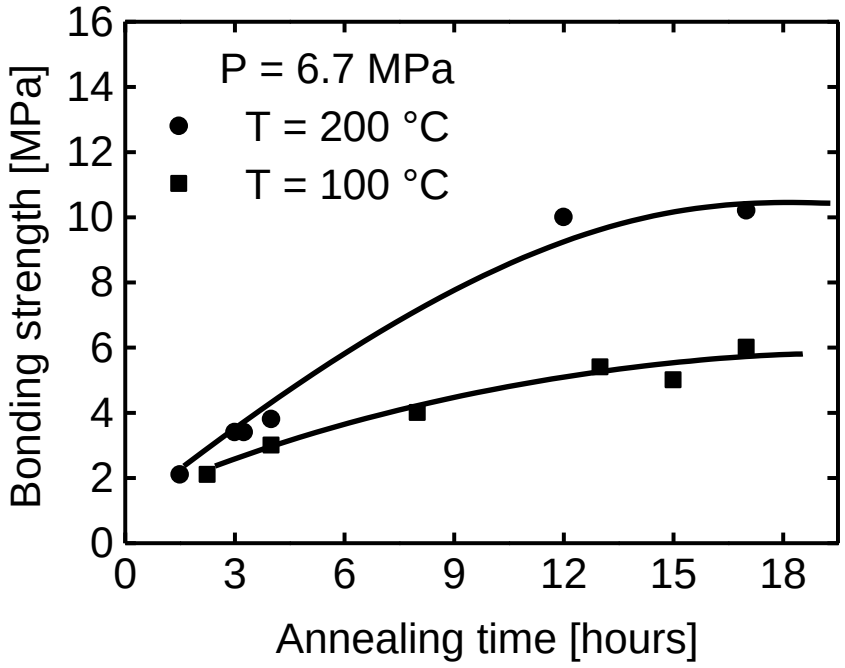
Advantages:

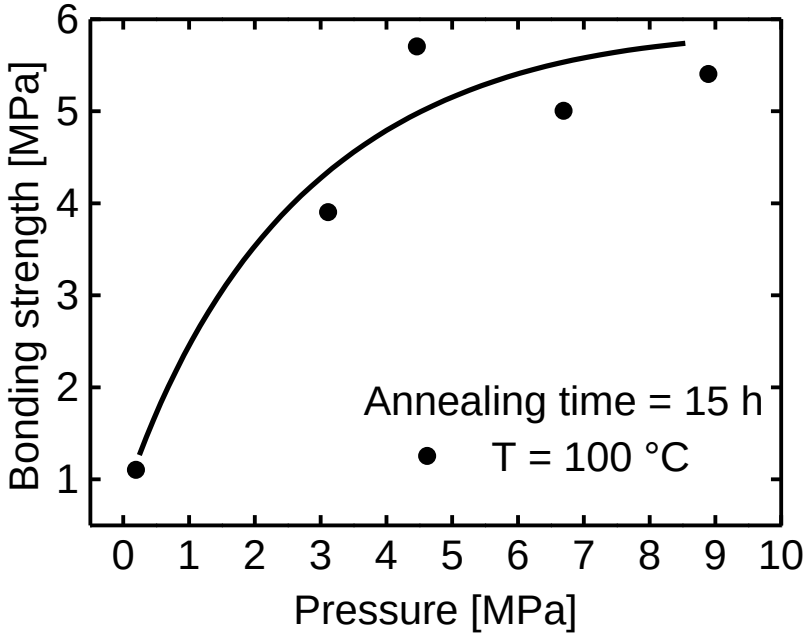
- no temperature induced stress
- no degradation of metal leads (Au-Cr, Al,..) and IC 's.
- bonding strengths of 5-10 MPa
- no HF

(Sayah et al., Sens & Act. A (2000))

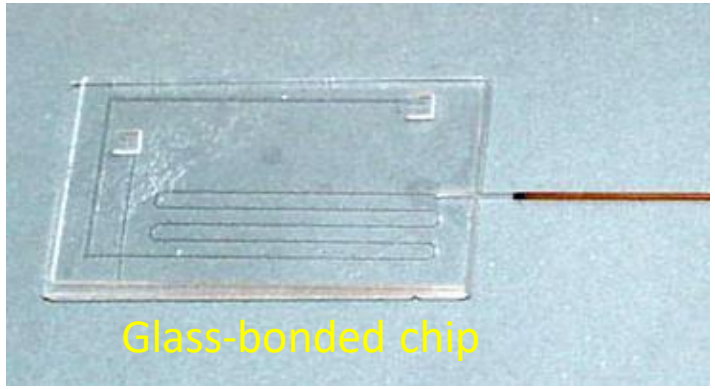
Pressure-assisted low-temperature fusion bonding

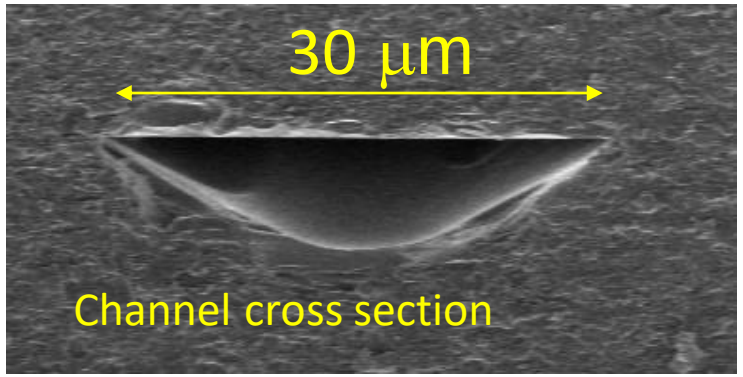
Measurement of bonding strength

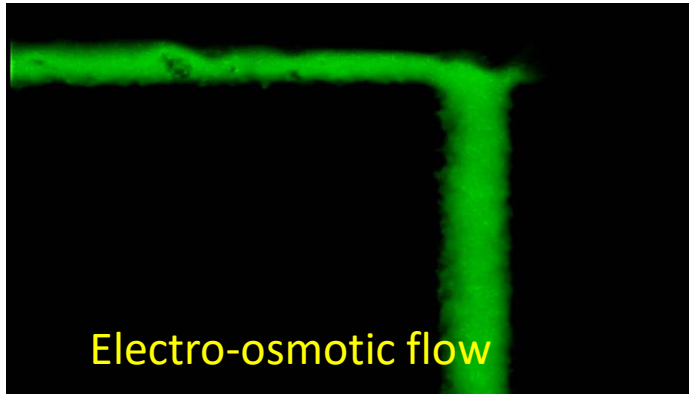




Capillary electrophoresis chip





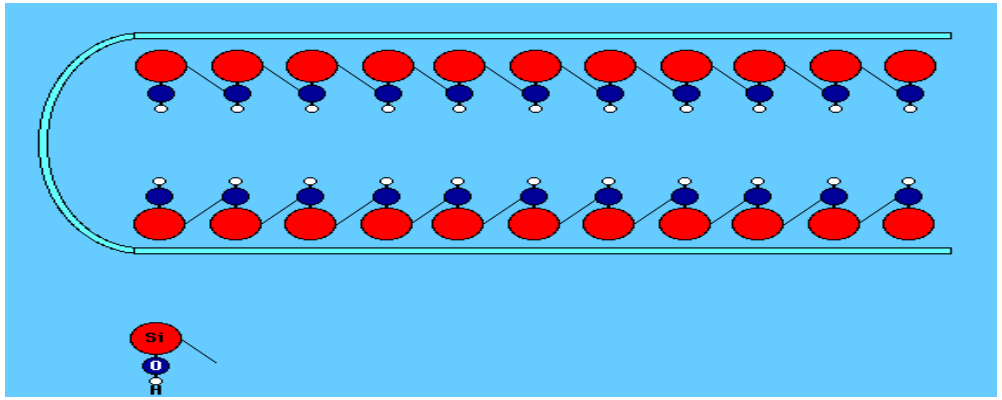


Separation principle

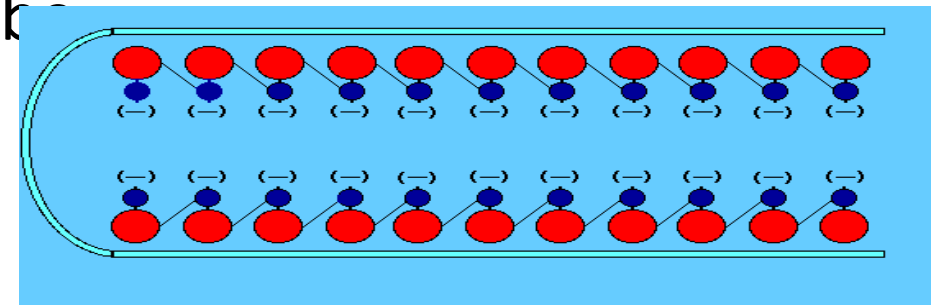
- Electrophoresis : different transport velocity of molecules in a liquid in an electric field, based on the **different charge/mass ratio** of the various biomolecules.
- This principle can be translated to the capillary / microchip format:
  - **Capillary** : hollow quartz tube (inner Ø 25-100 μm, length 0.1-1 m)
  - **Microchip** : microchannel realised in glass or quartz wafers by microstructuring and bonding techniques

Electro-osmotic flow (EOF) (i)

- Capillary is made of fused silica (SiO_2)

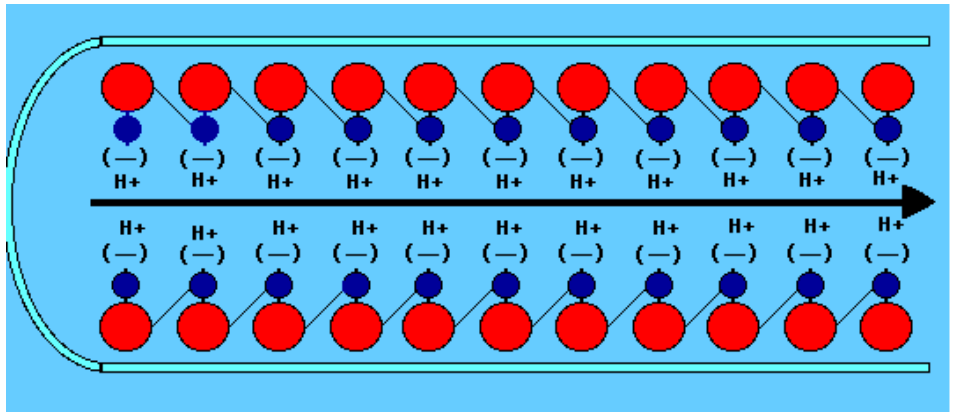


- Filling the channel with buffer solution disociates hydroxyl groups, leaving negative charge on inside of tub



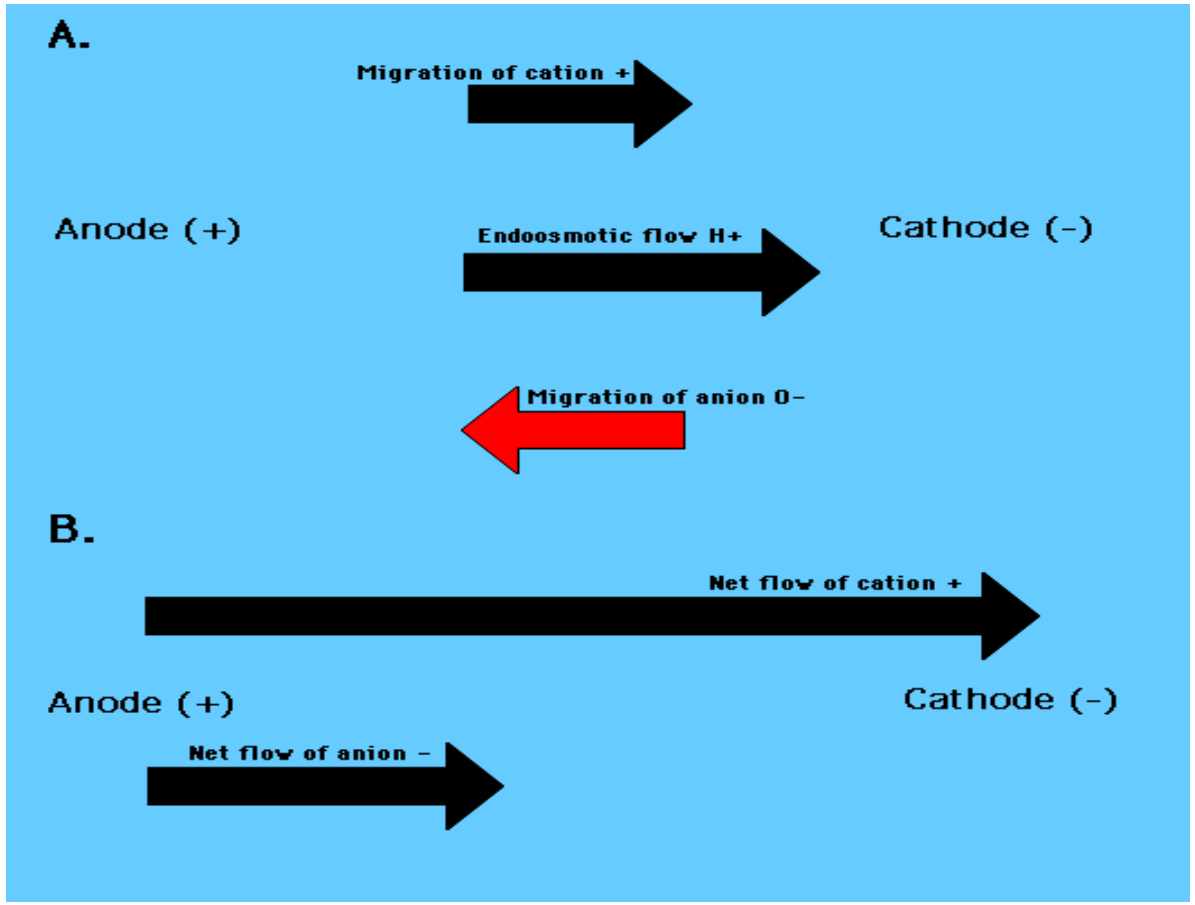
Electro-osmotic flow (EOF) (ii)

- Applying a voltage across the capillary causes hydrogen ion flow, resulting in EOF or bulk flow



- Turning off the voltage stops the EOF
- EOF mobility adds to electrophoretic mobilities of anions (-) and cations (+)

Effective mobilities



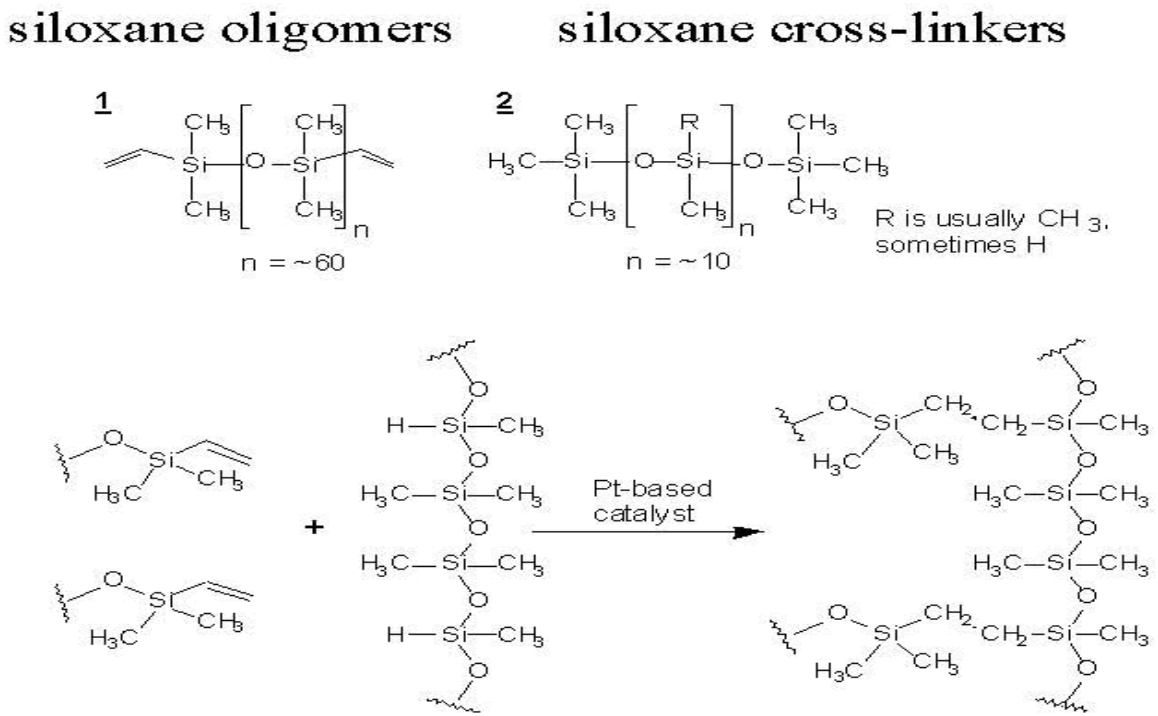
- Straight, not parabolic, pumping profile.

Electrophoresis and electrochromatography

- Separation using EOF- pumping
  - in a hollow quartz capillary : **Capillary electrophoresis (CE)**
  - in a microchannel on a glass or quartz chip : **Microchip electrophoresis**
- Separation can be based on the different diffusion of biomolecules in a **stationary phase** within a capillary or microchannel ⇒
  - **High Performance Liquid Chromatography (HPLC),** when liquid transported by mechanical pumping
  - **Capillary electro-chromatography (CEC)**, when liquid transported by EOF
  - **microchip CEC (μCEC)**, when having CEC in channel on microchip

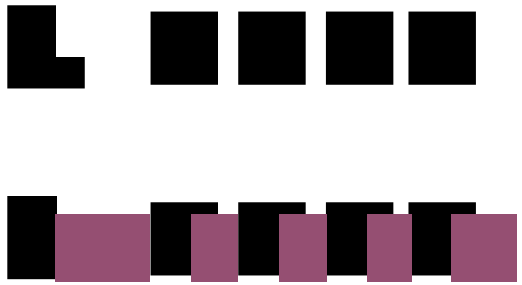
Connectors to the chip (i)

- **PDMS (Polydimethylsiloxane) reservoirs on top of the chip**
  - PDMS : moulded elastomer (silicone)
  - Contains base and curing agent (containing Pt-based catalyst)



Connectors to the chip (ii)

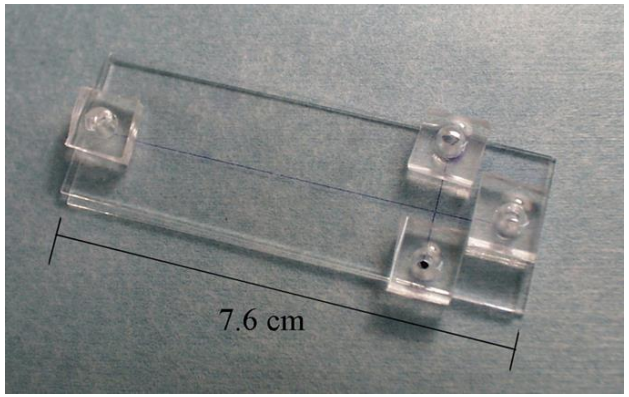
- **PDMS (Polydimethylsiloxane) reservoirs on top of the chip**
  - Three-dimensional cross-linked structure upon mixing (10:1)



Metal mould

Casted PDMS structure

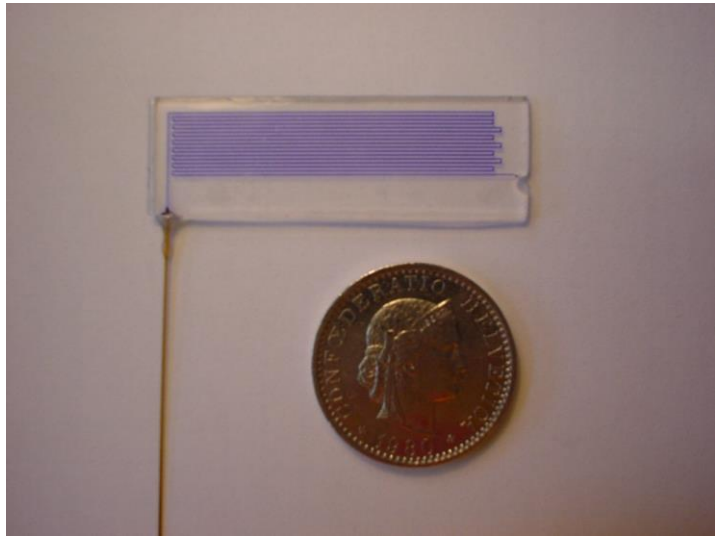
  - Adhesion promotor



Connectors to the chip (iii)

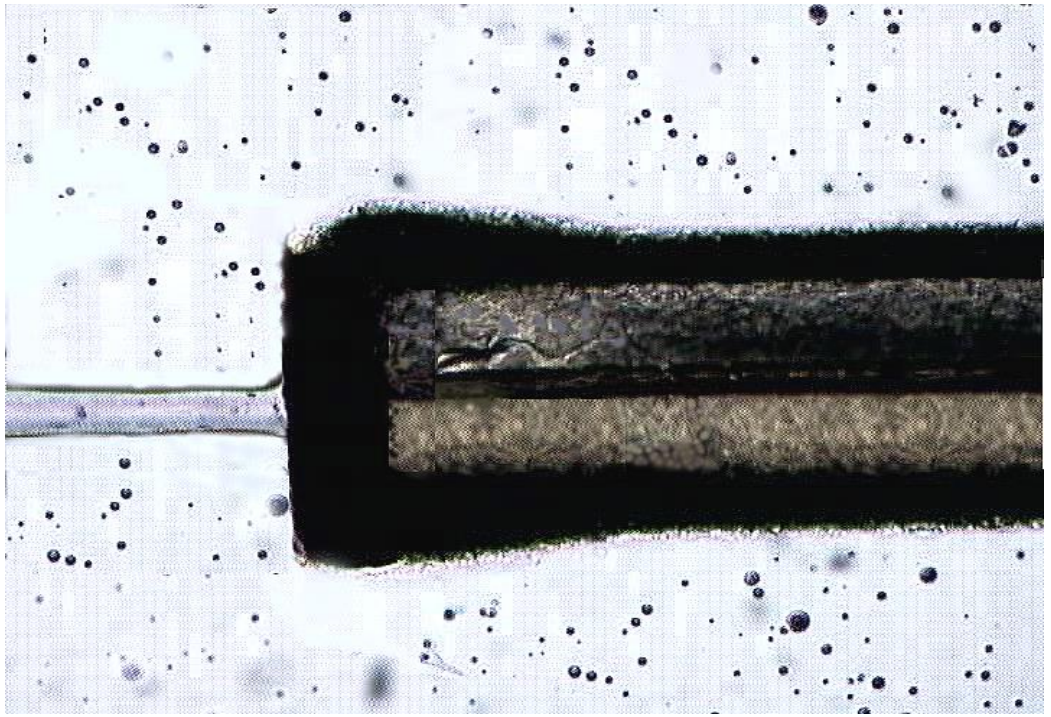
Chip side connectors using powder blasting/gluing

- Low dead-volume connector
- Allows chip integration in commercial capillary detection system



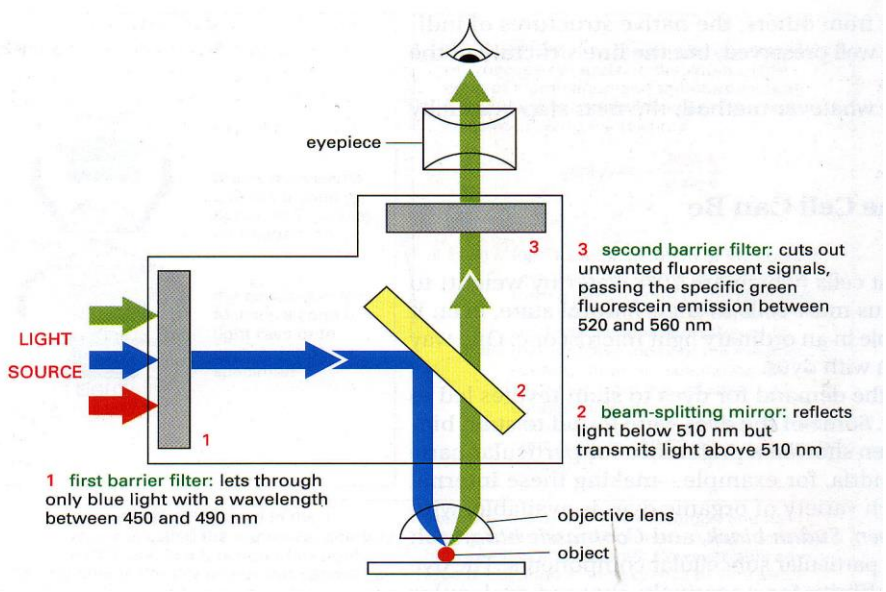
Connectors to the chip (iv)

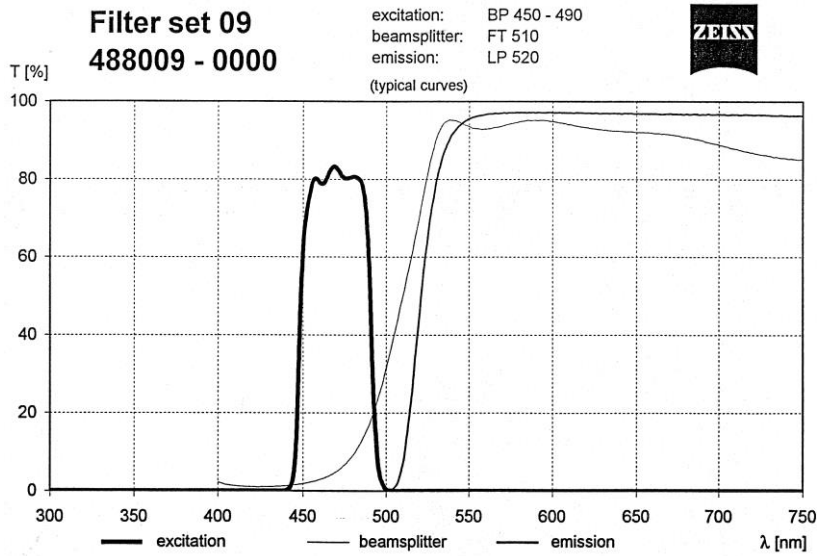
Chip/capillary side connectors using powder blasting/gluing



Chip detection platform (i)

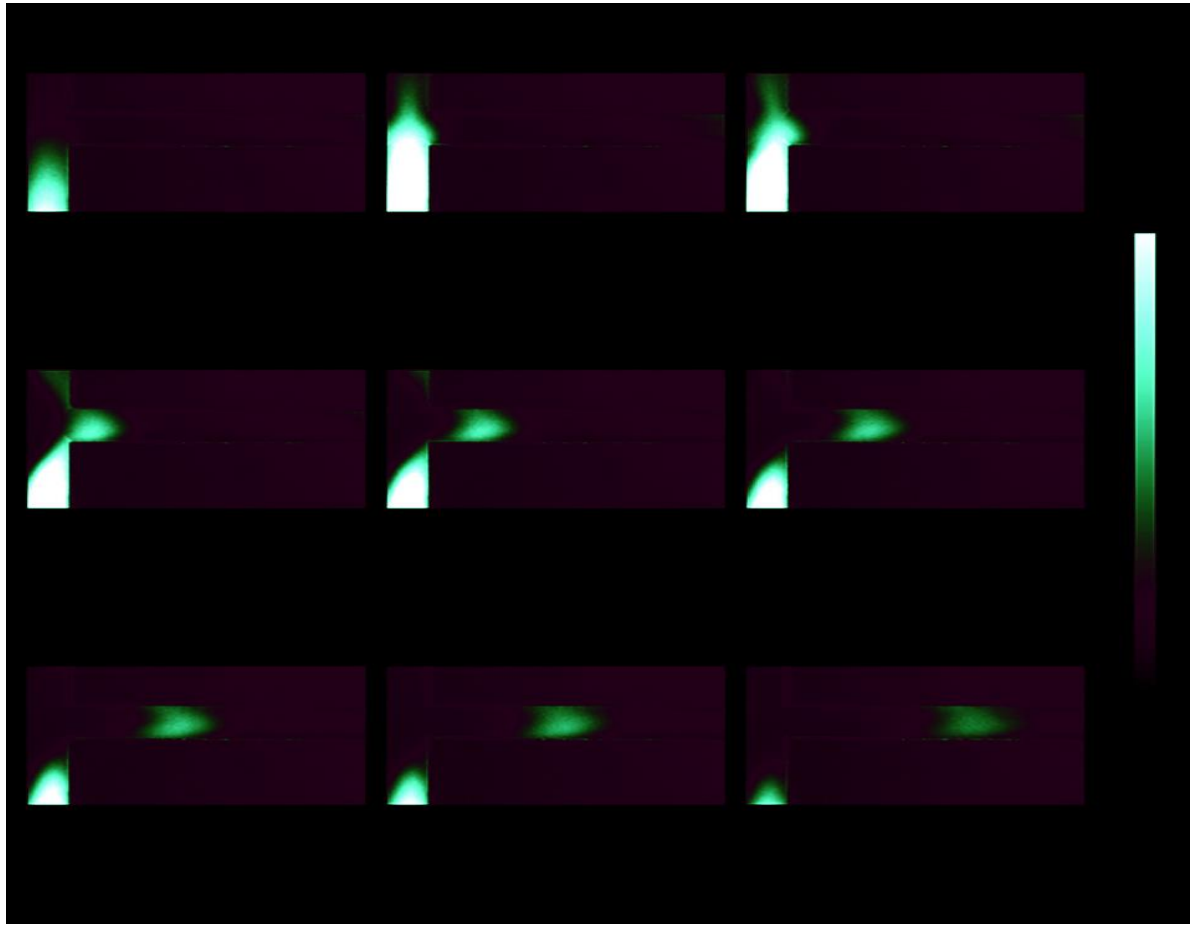
Fluorescent detection using an inverted microscope



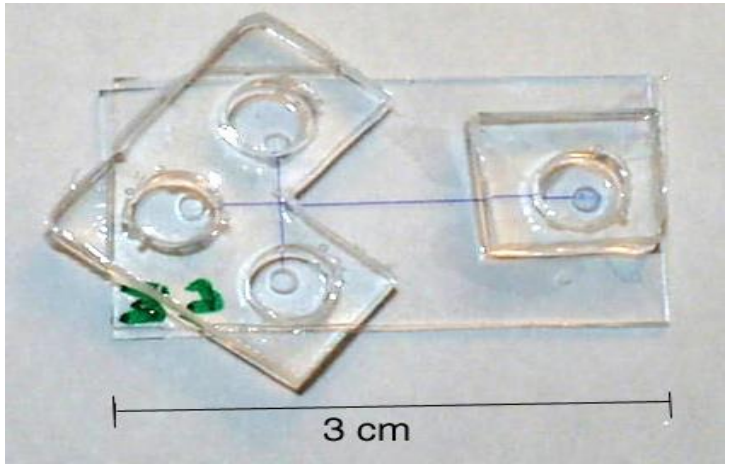


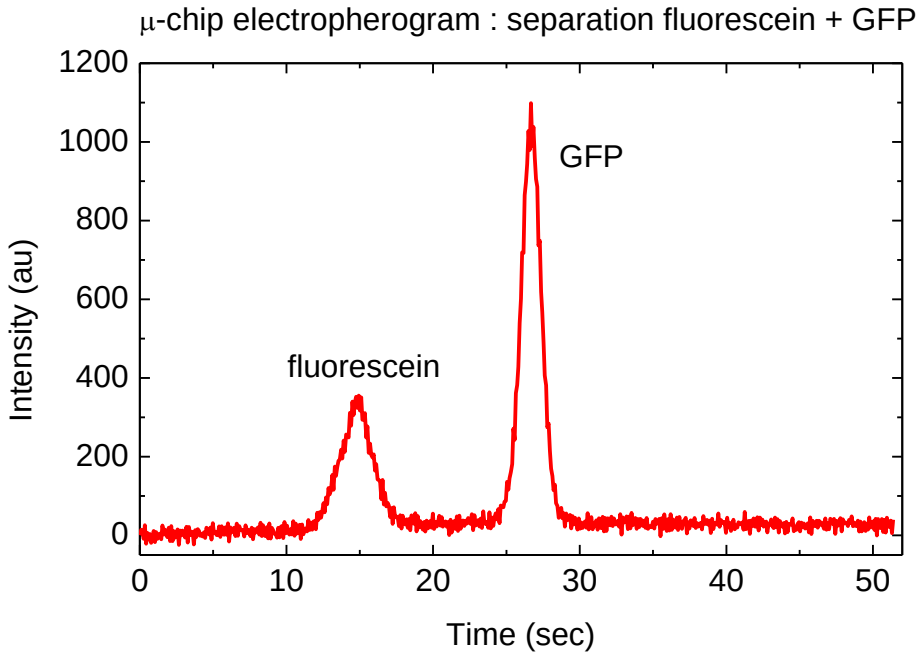
Chip detection platform (ii)

Injection of fluorescent plug



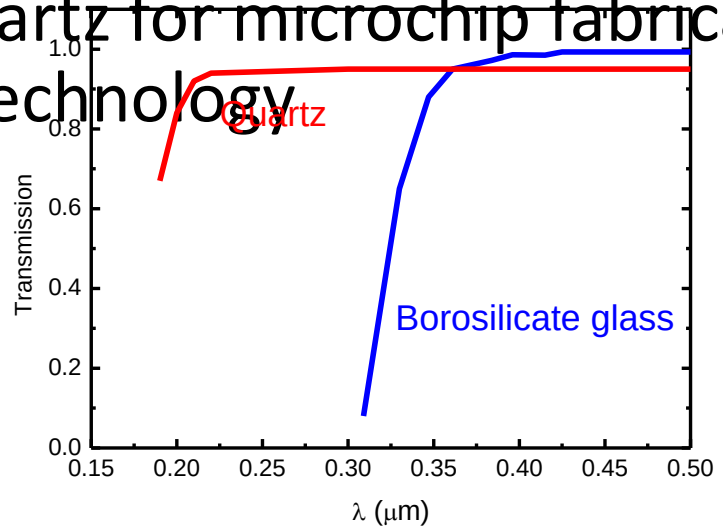
Fluorescein – GFP separation electropherogram





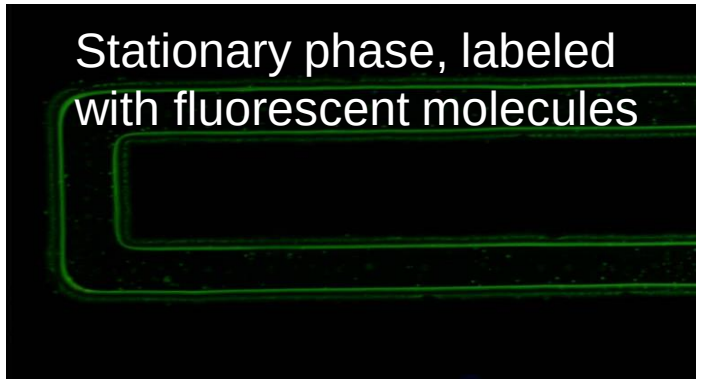
Towards a universal detection scheme

- Only fluorescently labeled molecules can be visualised by fluorescence
- All proteins absorb in UV → universal means for detection
- Also glass absorbs in UV
- Solution : use quartz for microchip fabrication → develop quartz technology



μ-chip chromatography using sol-gel stationary phases

- Inject a liquid solution in the channel
- Let it polymerise to a **stationary phase**, which covers the channel walls
- Inject a liquid **mobile phase** containing the molecules to be separated
- Bio-molecules will selectively adsorb/desorb on the formed stationary phase → chromatography application
- Driving force for the flow is electro-osmosis → μ-chip Capillary Electro Chromatography



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