The Abdus Salam
International Centre
for Theoretical Physics

2443-16

Winter College on Optics: Trends in Laser Development and Multidisciplinary Applications to Science and Industry

4 - 15 February 2013

Femtosecond laser micromachining - (part 2)

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Femtosecond laser micromachining (2)

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Outline of the presentation

- Femtosecond laser microstructuring for optofluidics
  - ➢ microfluidic channels by irradiation + etching
  - ➢ new optofluidic functionalities by waveguide-channel integration
- Femtosecond laser microstructuring for solar cells
- Fabrication of microoptical components: Fresnel lenses
- Two-photon polymerisation

Femtosecond laser microfabrication for optofluidics applications

Femtosecond Laser Microchannel fabrication

March 1, 2001 / Vol. 26, No. 5 / OPTICS LETTERS 277

Femtosecond laser-assisted three-dimensional microfabrication in silica

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Mitsuru Watanabe, Masafumi Miwa, Shigeki Matsuo, and Hiroaki Misawa

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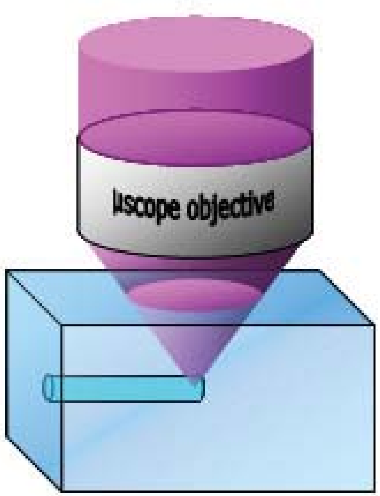
- Novel technique for the fabrication of directly buried microchannels in three dimensions

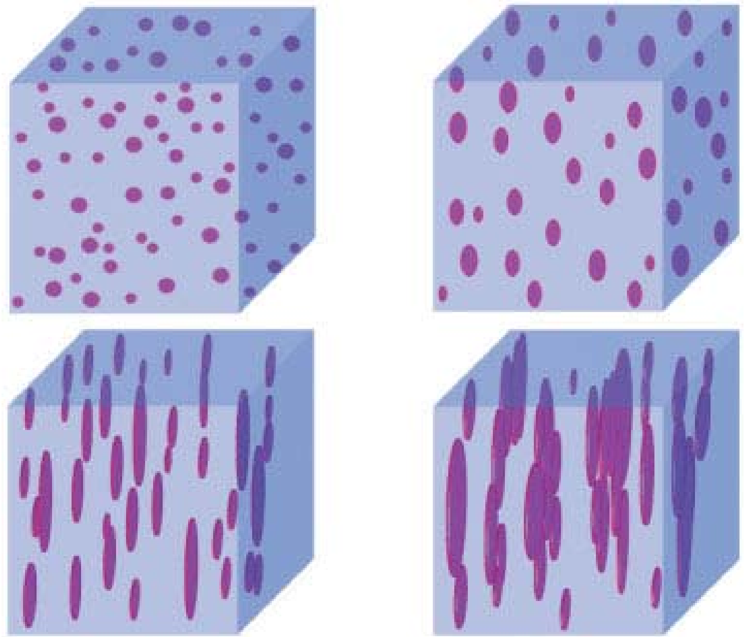
Femtosecond Laser Microchannel fabrication



Femtosecond irradiation

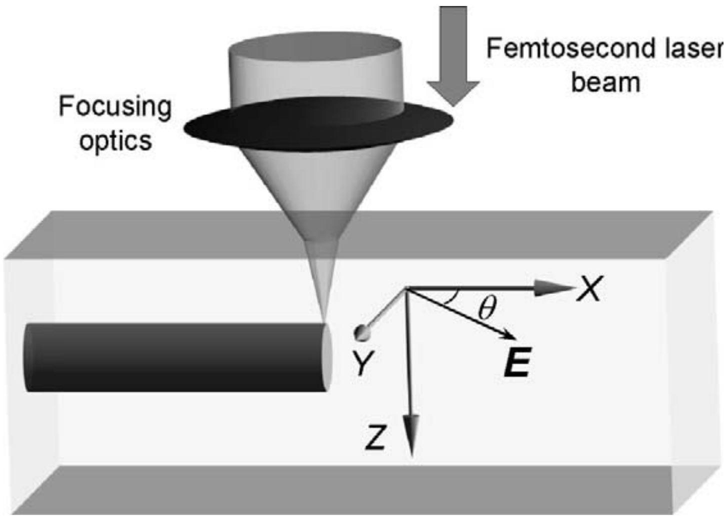
- High intensity femtosecond laser irradiation
- Selective etching in HF solution
- Fabrication of directly buried microchannels in three dimensions

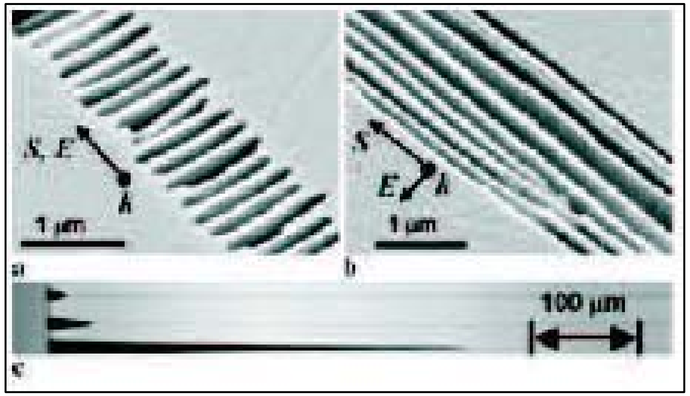
Microchannel fabrication: underlying physical mechanism



- Nonlinear ionization creates randomly localized plasma nanodroplets
- The droplets grow and flatten under the electric field
- They merge to form regular arrays of nanoplanes

R. Taylor et al., Laser Photonics Reviews **2**, 26 (2008)

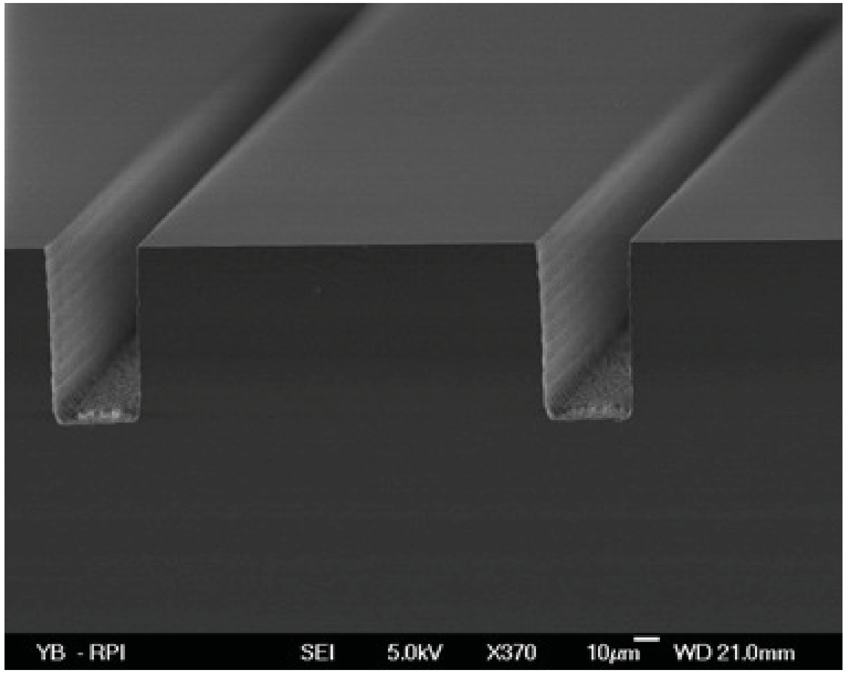


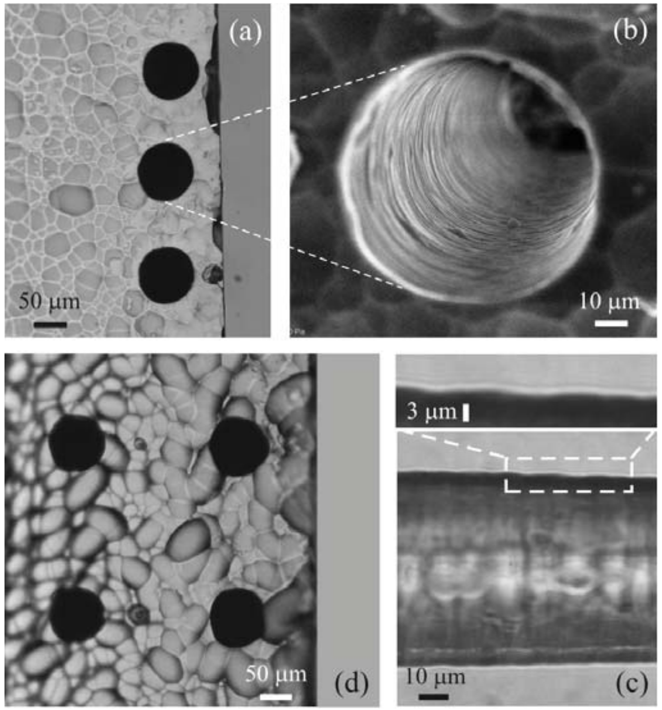


C. Hnatovsky *et al.*, Opt. Lett. **30**, 1867 (2005).

- Femtosecond laser irradiation followed by etching
- Grating planes are perpendicular to the direction of electric field
- Much higher etching rate when gratings are aligned along the writing direction

Microfluidic channels: characteristics





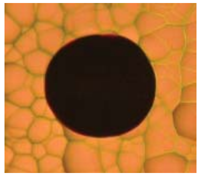
- High aspect ratio surface channels
- 3D directly buried channels

V. Maselli *et al.*, Appl. Phys Lett. **88**, 191107 (2006).

Y. Bellouard *et al.*, Opt. Express **12**, 2120 (2004).

Shape control of the microchannels

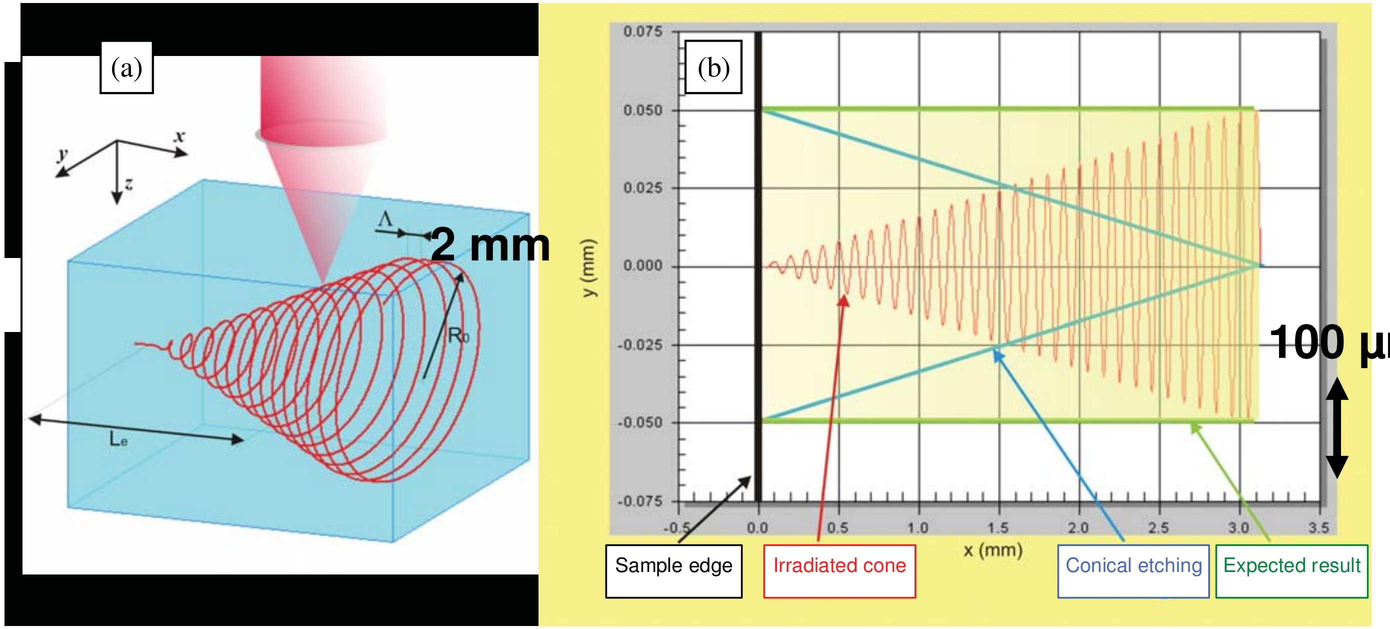
- Conical shape is intrinsic in the etching process of uniform structures



Wobbling the writing laser provides a perfectly uniform cylindrical channel

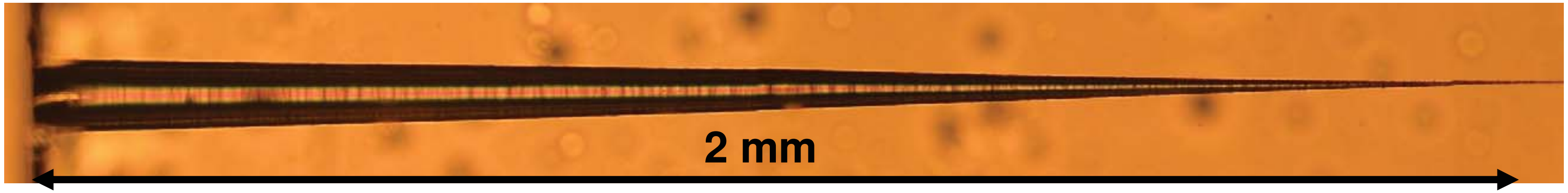
(a)

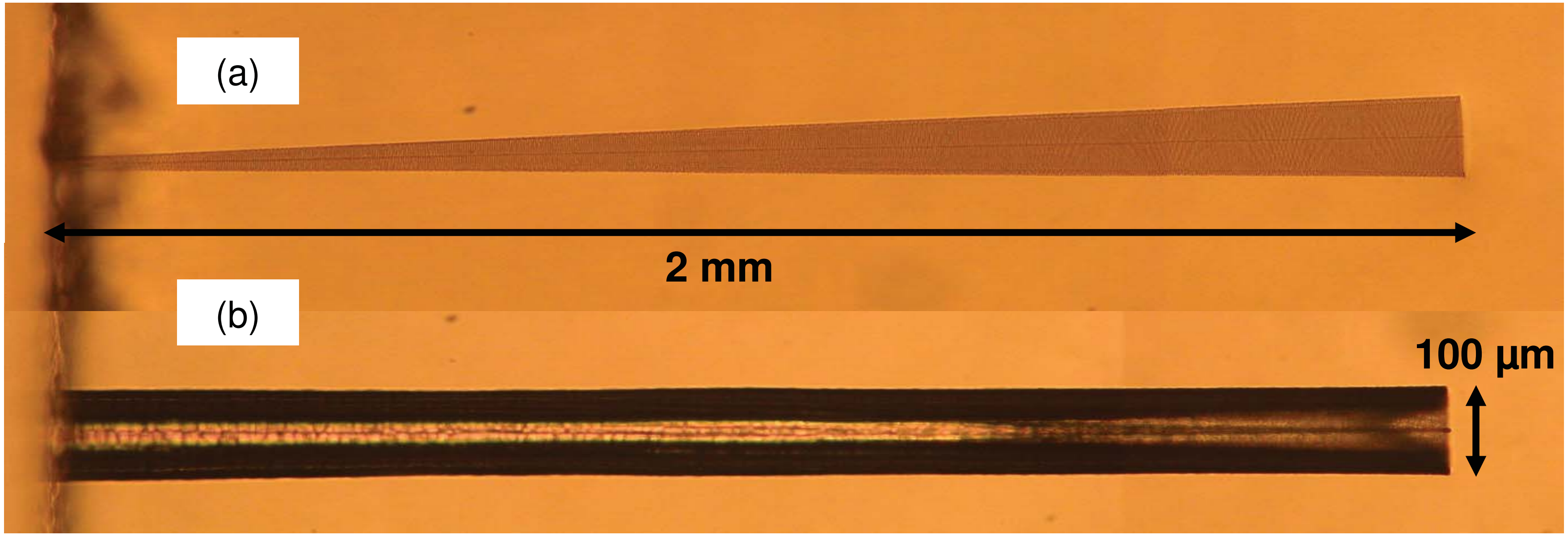
(b)



K. Vishnubhatla *et al.*, Opt. Express. **17**, 8685 (2009).

Shape control of the microchannels





NATURE|Vol 442|27 July 2006|doi:10.1038/nature05060

INSIGHT REVIEW

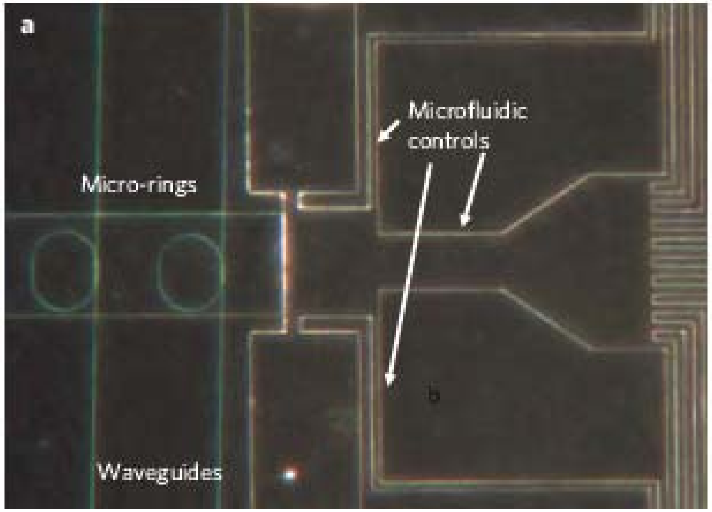
Developing optofluidic technology through the fusion of microfluidics and optics

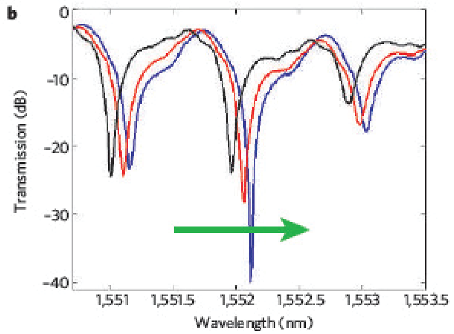
Demetri Psaltis[1], Stephen R. Quake[2] & Changhuei Yang[1]

We describe devices in which optics and fluidics are used synergistically to synthesize novel functionalities. Fluidic replacement or modification leads to reconfigurable optical systems, whereas the implementation of optics through the microfluidic toolkit gives highly compact and integrated devices. We categorize optofluidics according to three broad categories of interactions: fluid–solid interfaces, purely fluidic interfaces and colloidal suspensions. We describe examples of optofluidic devices in each category.

Optofluidics

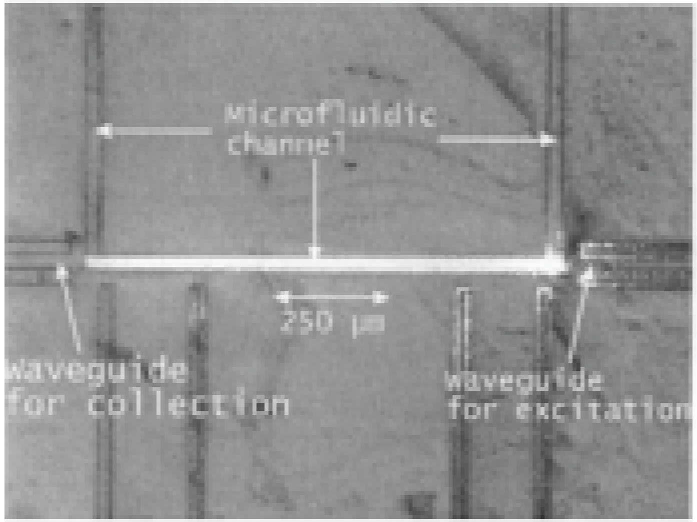
- Liquids may improve or extend the functionalities of integrated optical devices

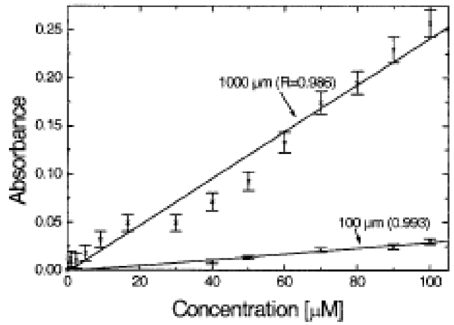




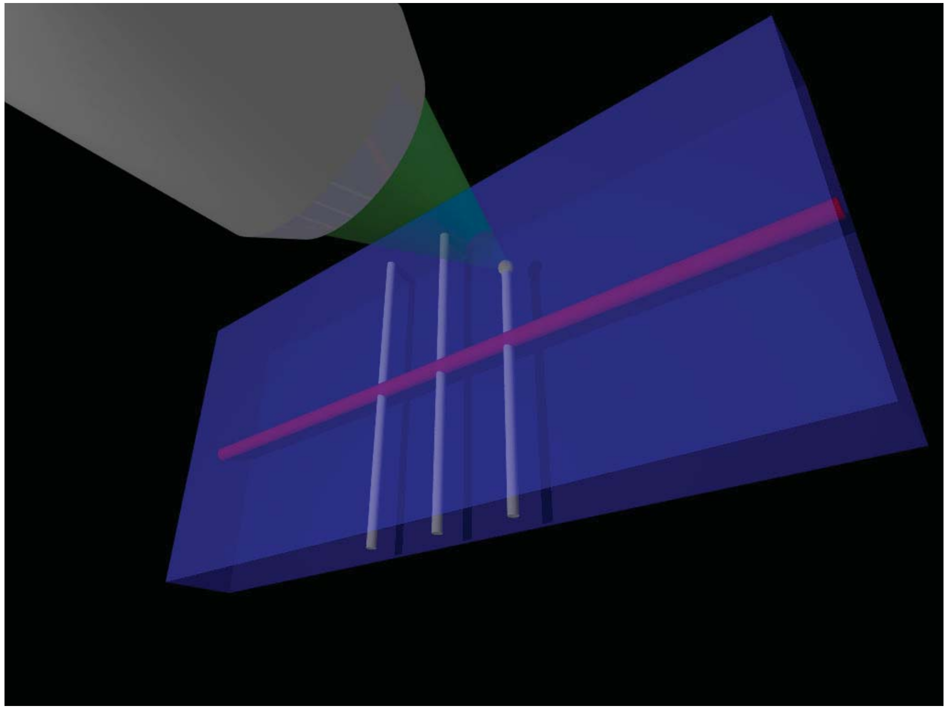
U. Levy et al, APL 88,111107 (2006)

- Integrated optics may enhance the sensing capabilities in fluidic devices



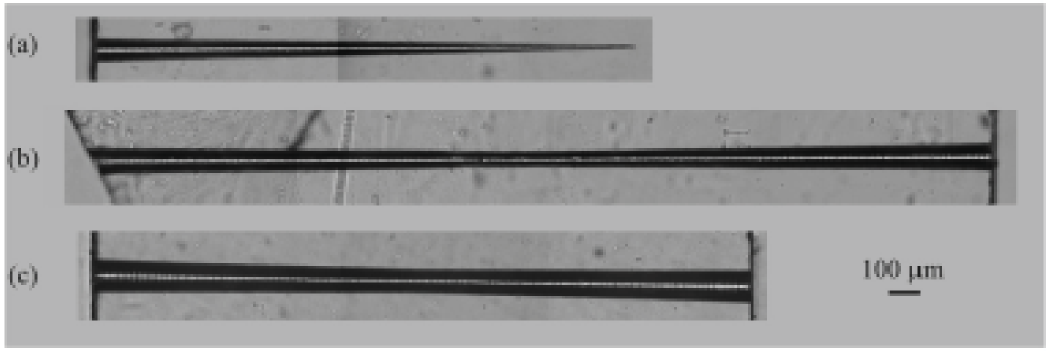


K.B. Mogensen et al, AO 42, 4072 (2003)



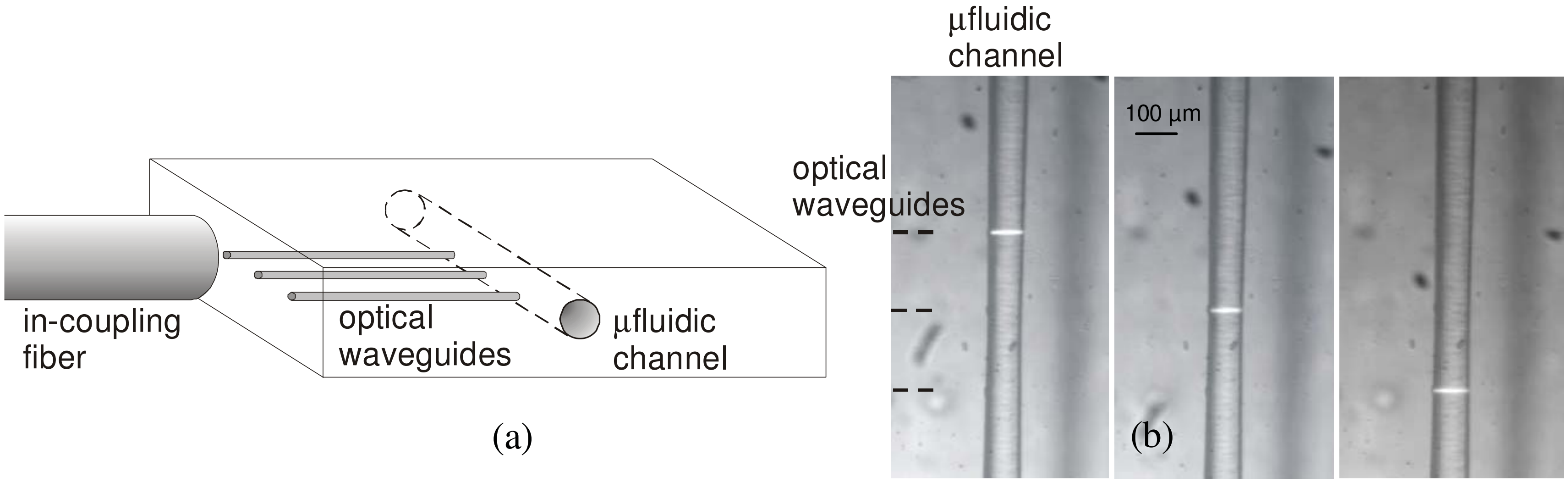
- Direct femtosecond laser fabrication of integrated waveguides and microchannels
- Use laser as post-processing tool adding new functionalities to microfluidic devices

Integration of femtosecond written waveguides and microchannels



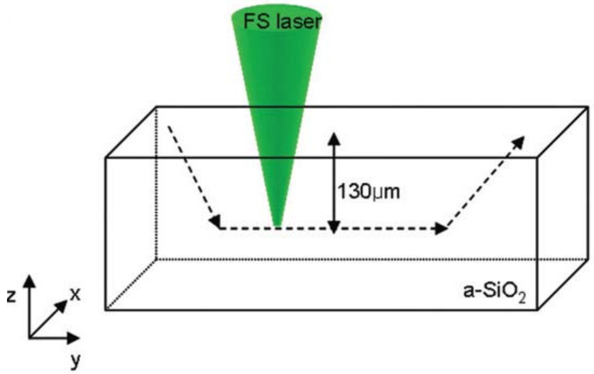
- Single side: 1.8mm, ∅90µm
- Double side: 3mm, ∅100-50µm or 2.2mm, ∅110-90µm

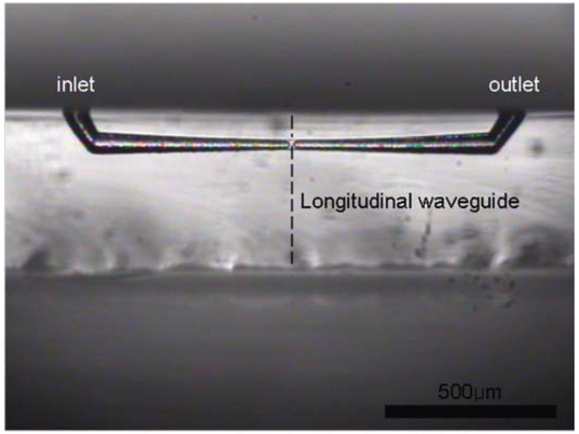
- Waveguides provide selective excitation of a Rhodamine solution in the channel

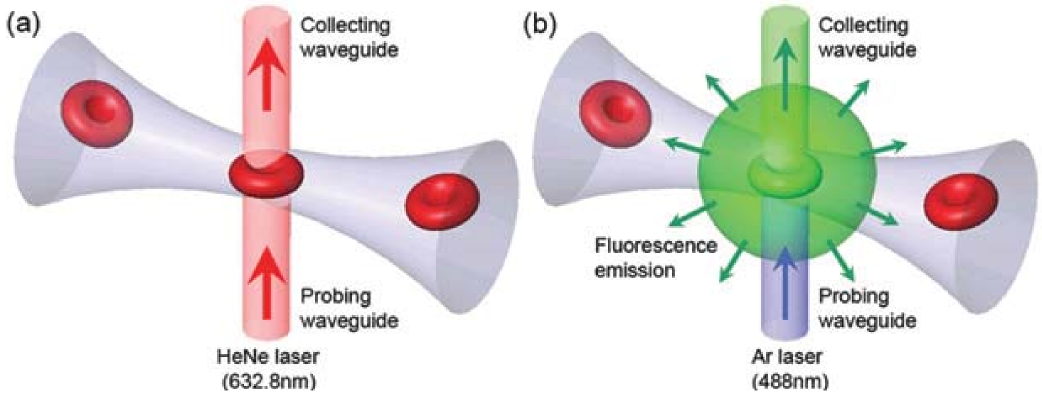


R. Osellame *et al.*, Appl. Phys. Lett. **90**, 231118 (2007).

Femtosecond laser written optofluidic cell detector





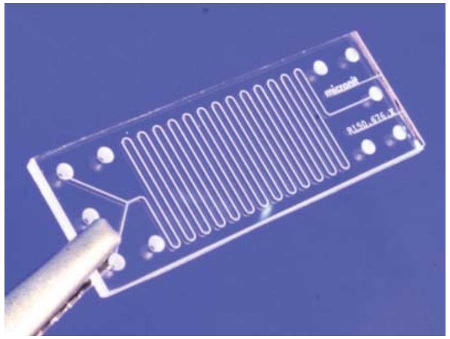


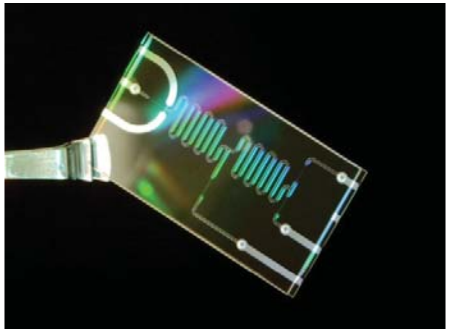
M. Kim *et al.*, Lab on Chip **9**, 311 (2009)

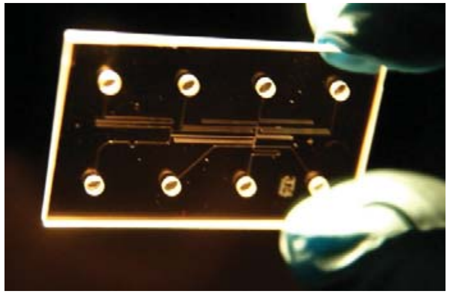
Lab-on-a-Chip

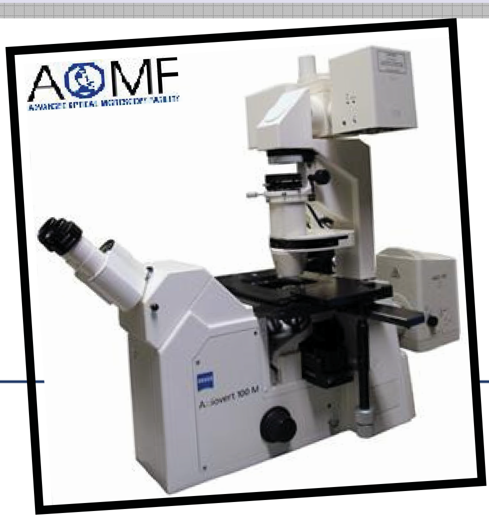
A **network of microfluidic channels** allows to perform **chemical processes** or **bio-analysis** with very small amounts of fluids

- Extreme miniaturization:
  - Rapid and automated processes
  - Limited reagents consumption
  - Capillarity and surface interactions
- Multifunction integration
  - microfluidics + analytical techniques
  - replicate a real chemi/bio-lab on chip

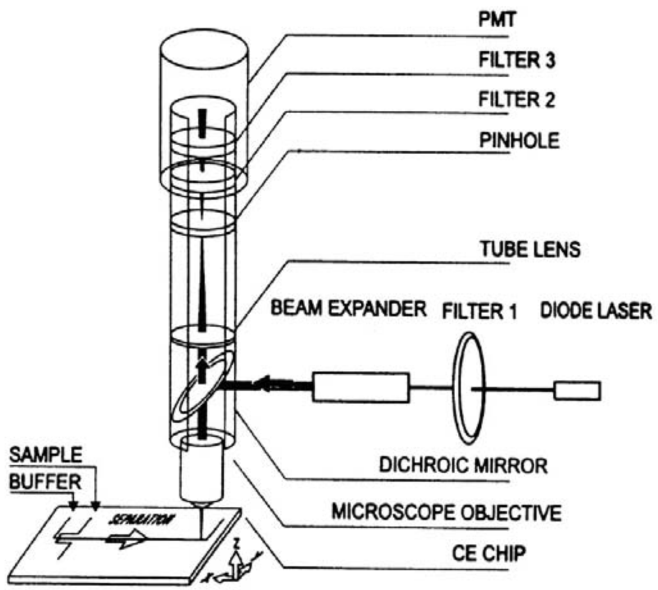








Detection techniques



Confocal microscope for *off-chip* detection

Optical detection

- Fluorescence
- Absorbance
- Refractive index

Off-chip approach

External bulky equipment that needs precise alignment

⇓

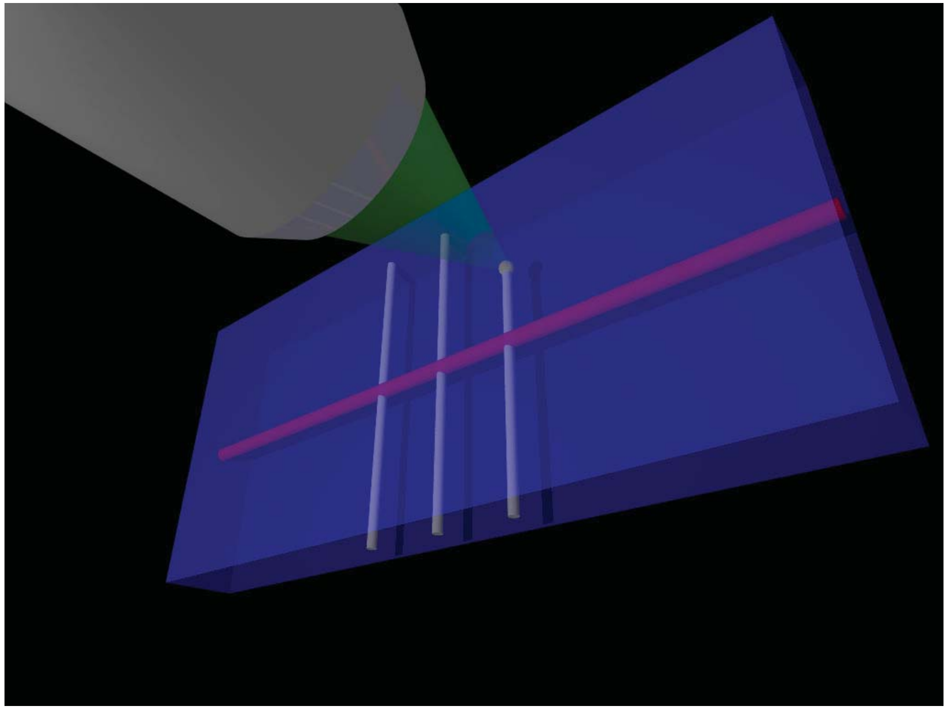
Lab-on-chip miniaturization is frustrated

On-chip approach

Monolithic integration of photonic devices with one-time alignment

Increased compactess and portability but increased fabrication complexity

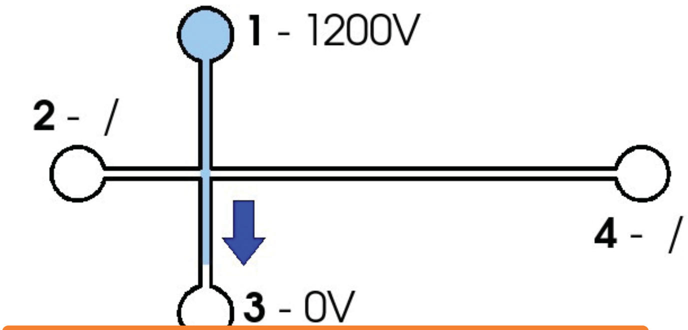
Femtosecond laser post-processing of lab-on-chips



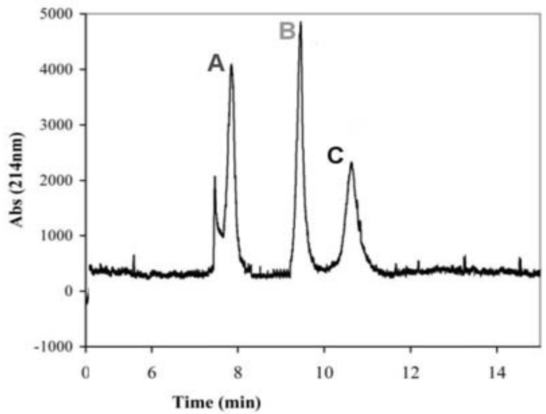
- Post-processing on an already made Lab-on-a-chip.
- Fabrication of three-dimensional devices.
- High versatility and limited equipment costs.

Capillary electrophoresis in *Lab on a Chip*

1) Injection

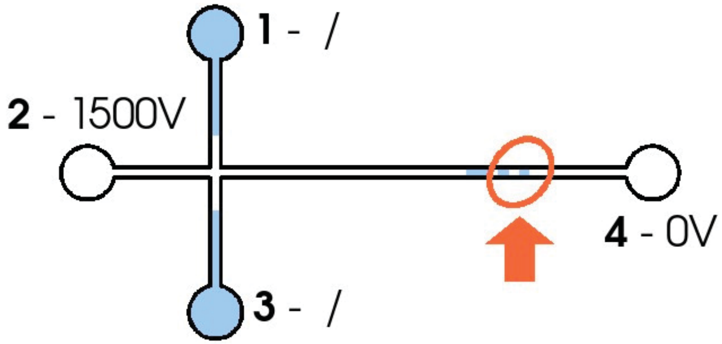


3) Detection



The **flow of molecules** is driven by **voltages** applied to the reservoirs

2) Separation



Molecules will separate in the channel according to the **different mobility**

They are **identified** on the basis of the **arrival time** at the detection point

Microchip capillary electrophoresis

Application: separation of DNA fragments to perform bioassays for the detection of a variety of diseases.

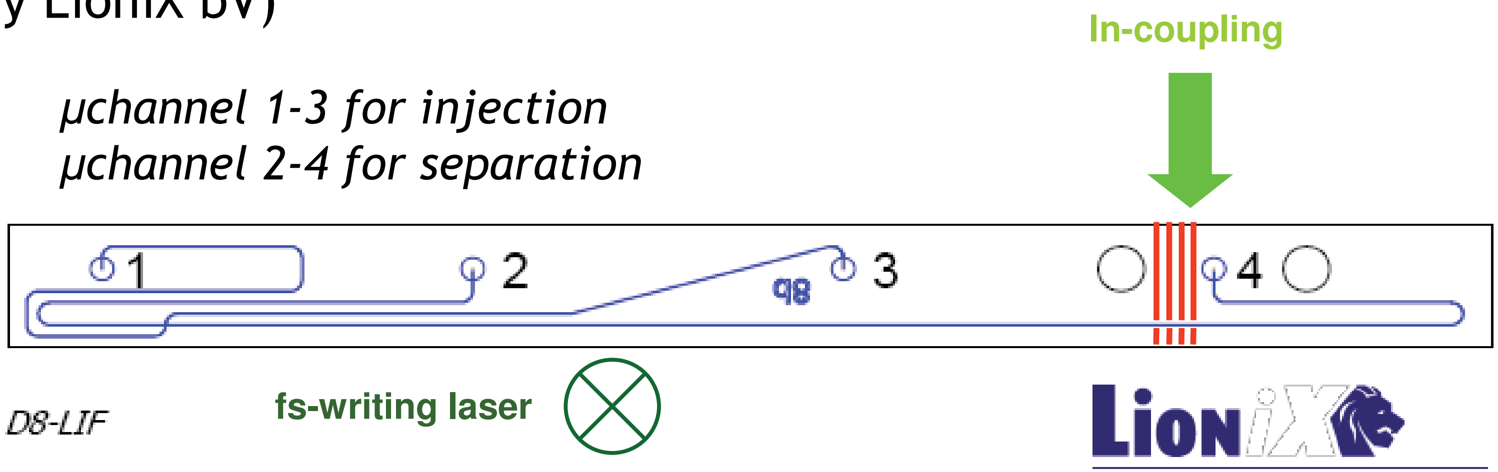
Exogenous DNA detection: viruses and bacteria.

Endogenous DNA mutation detection: cancer, hereditary genetic diseases

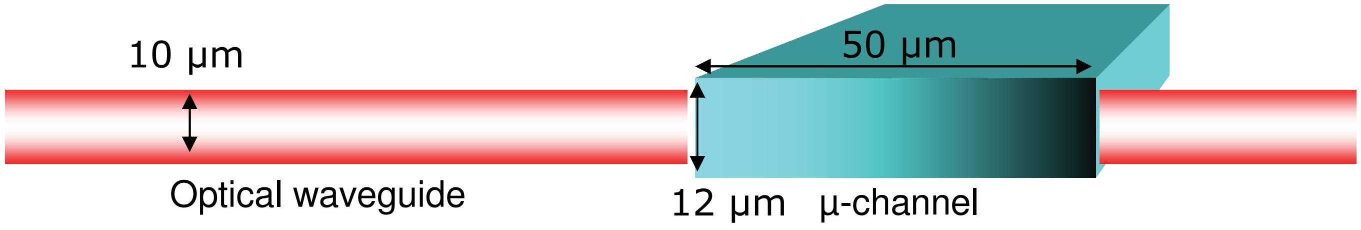
On-chip waveguide integration

- **Commercial** microfluidic chip for capillary electrophoresis (by LioniX bV)

μchannel 1-3 for injection
μchannel 2-4 for separation

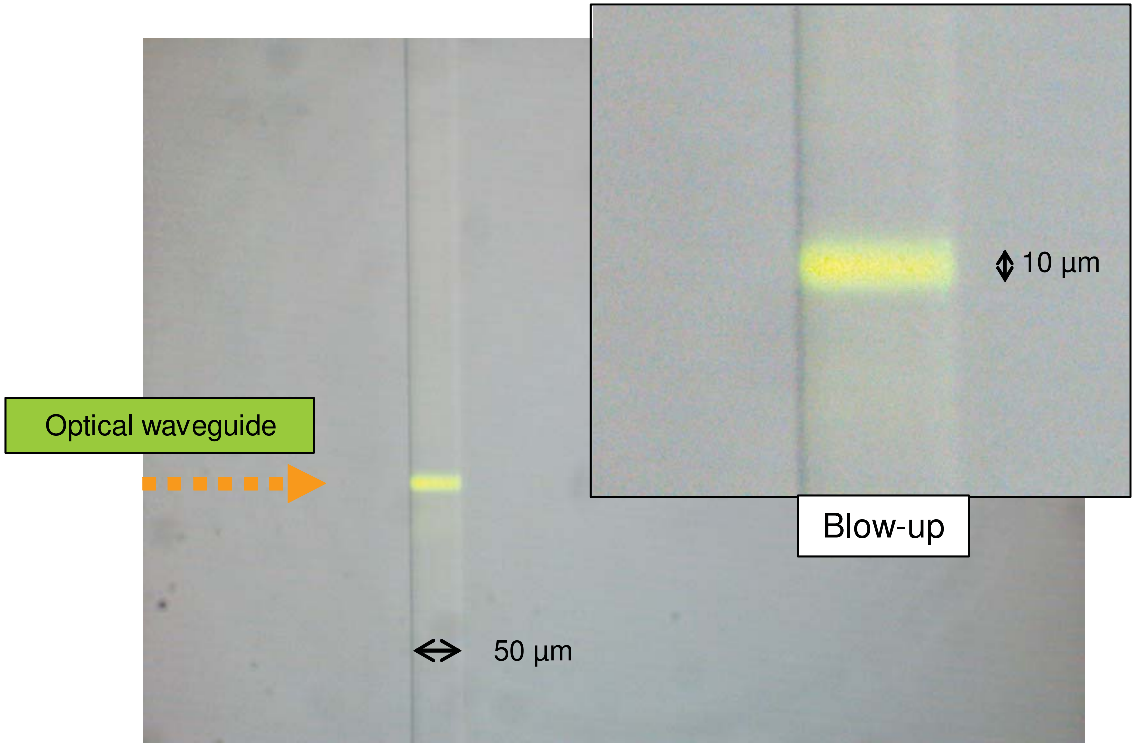


- Microchannel cross-section



On-chip waveguide integration

- Inscribed optical waveguide allows **selective excitation** in the channel

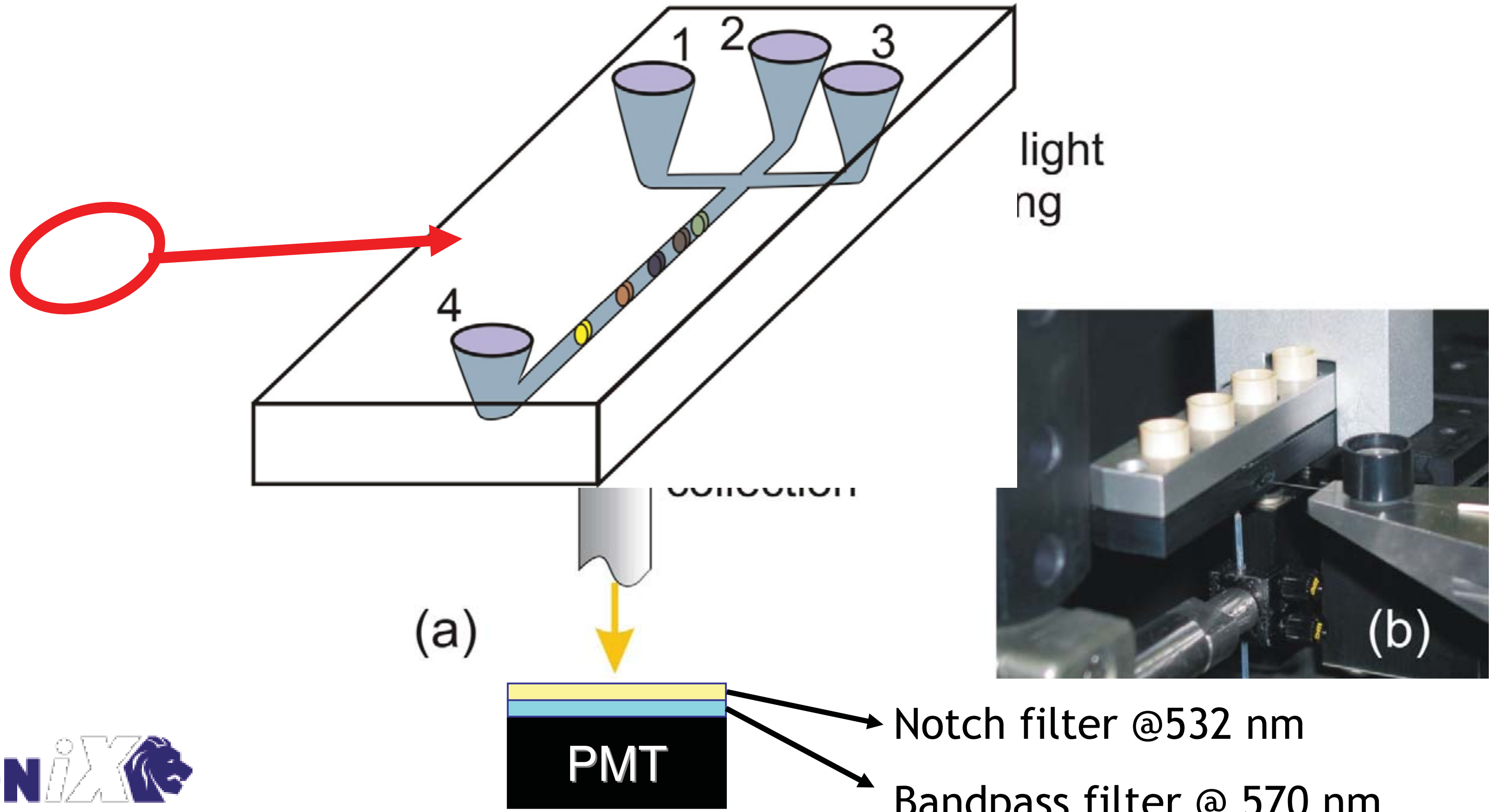


Microchannel filled with rhodamine 6G
Microscope image through a cut-off filter at 570 nm

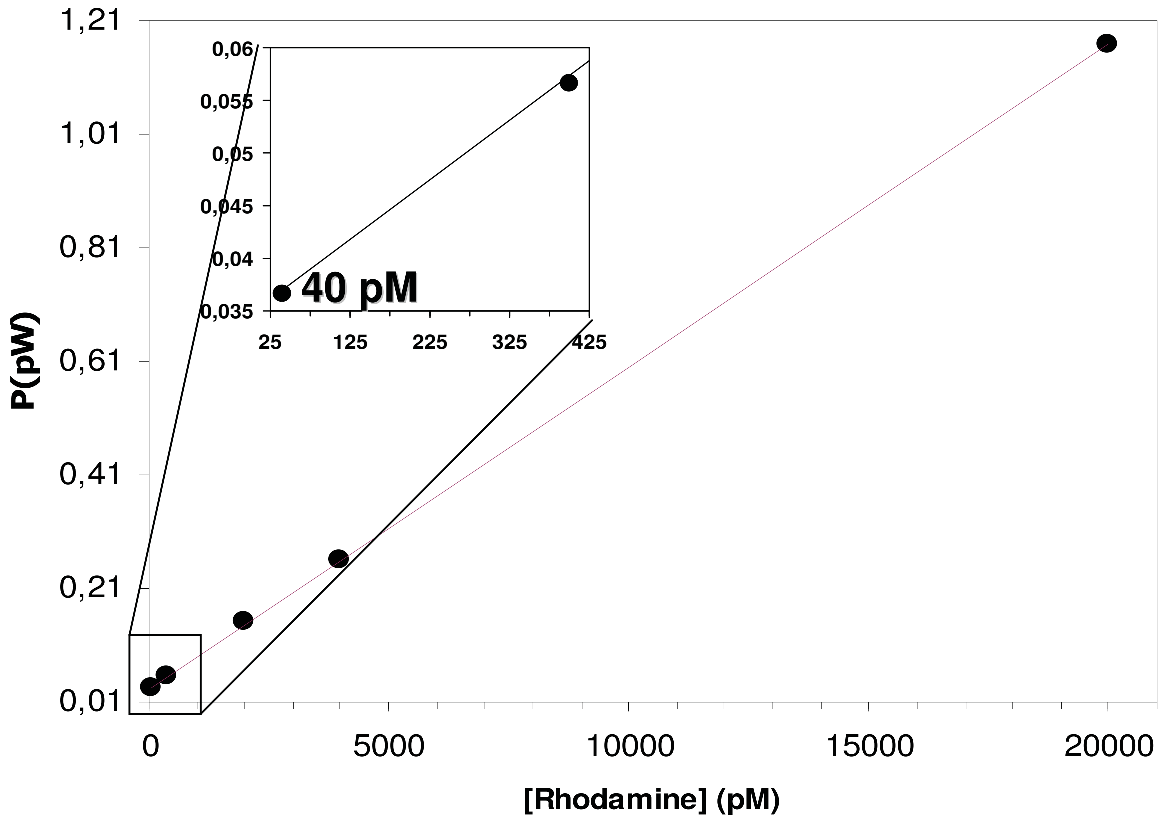
Laser at 532 nm coupled into the waveguide

On-chip waveguide integration

- Pigtailed excitation and collection fibers provide a very **compact** and **portable** unit



On-chip Laser Induced Fluorescence detection



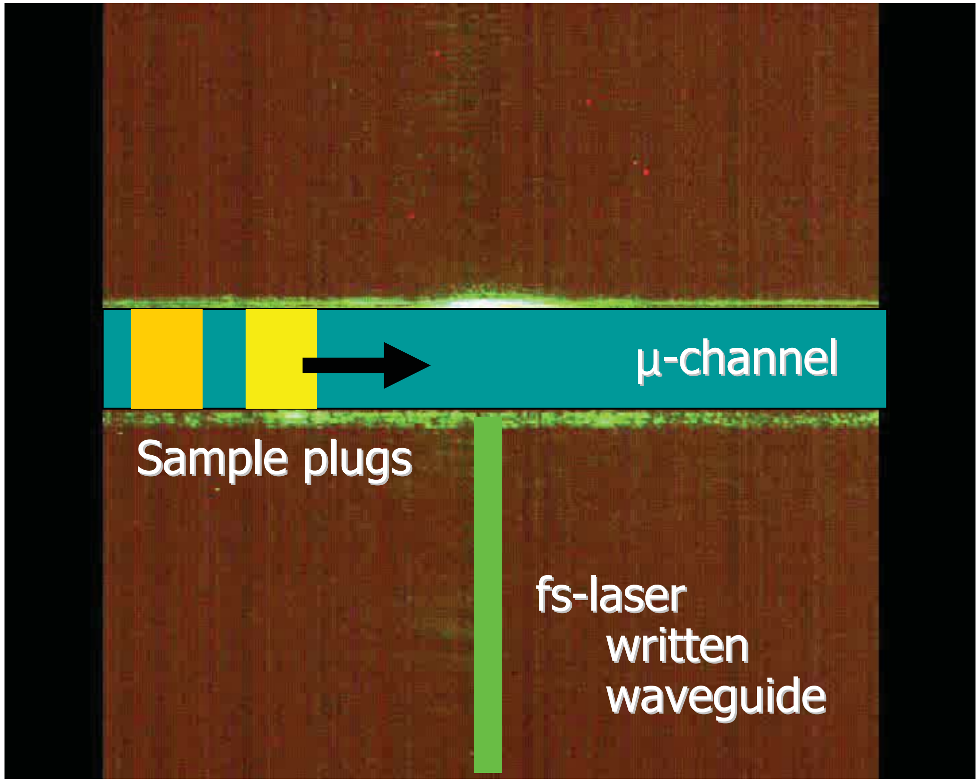
- Rhodamine 6G
- 100 μW of 532 nm excitation
- Detection filter 570±5 nm

➢ **Limit of detection ~10 pM**, among the best results for integrated detection

R. Martinez Vazquez *et al.*, Lab Chip **9**, 91 (2009)

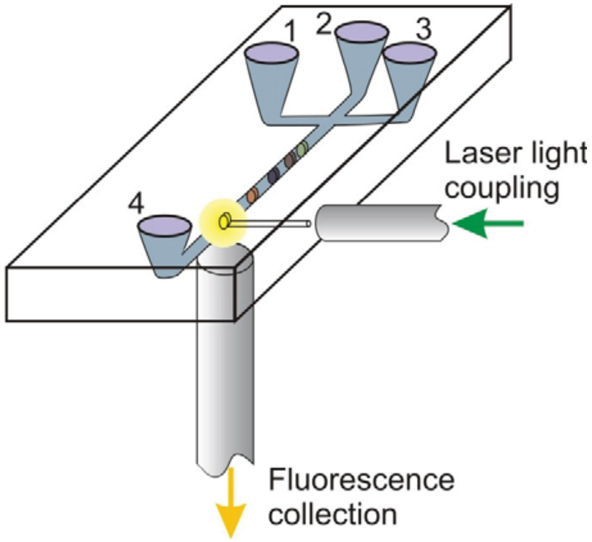
Dynamic detection of separated molecules

- CCD imaging of laser induced fluorescence by a fs-laser written waveguide
- Sample is a highly concentrated solution of two different dyes (Rhodamine 6G and Rhodamine B)

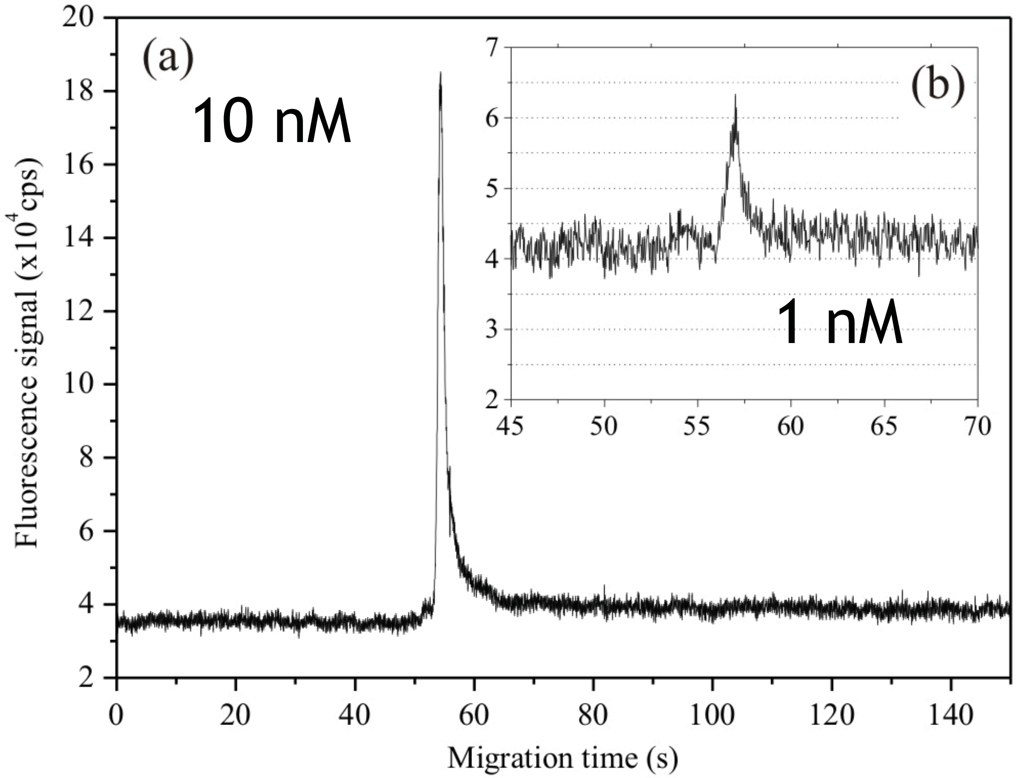


C. Dongre *et al.*, Opt. Lett. **33**, 2503-2505 (2008).

Dynamic Detection of labelled DNA fragments



- **Oligonucleotides** (23 mer) labelled with Cy3
- Polymer coating of the inner walls of the channels to inhibit electroosmotic flow
- Electrophoretic flow under electric fields

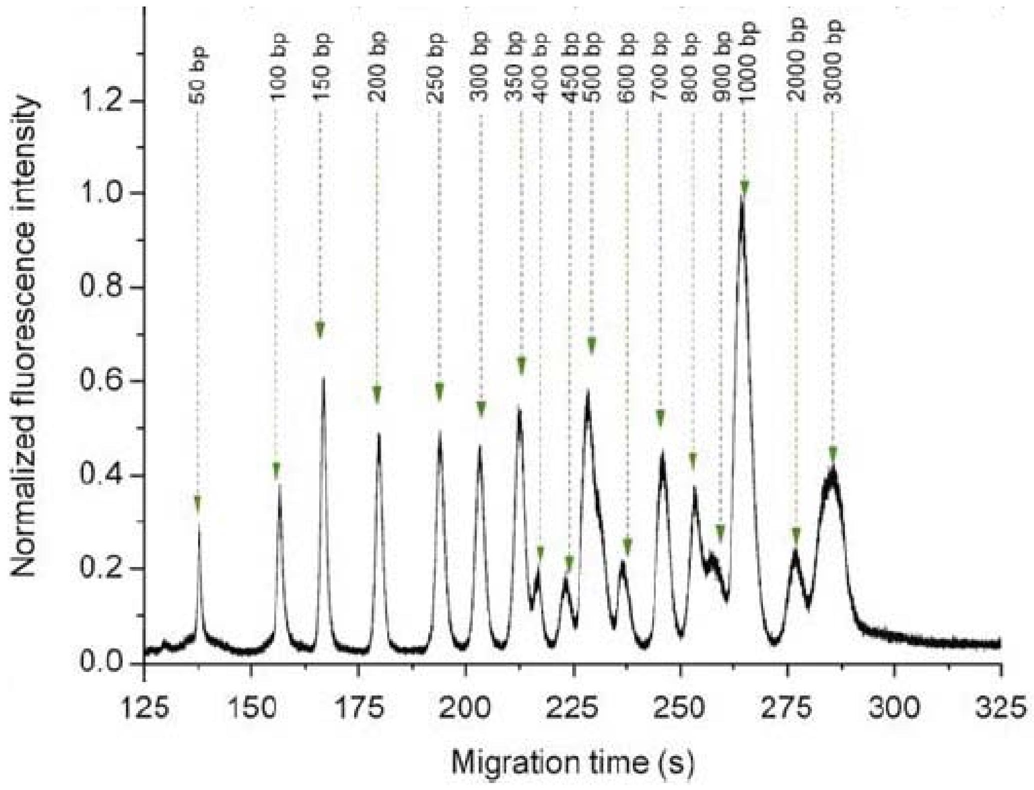


1 nM dynamic LOD

SNR ~ 10

DNA fragments separation

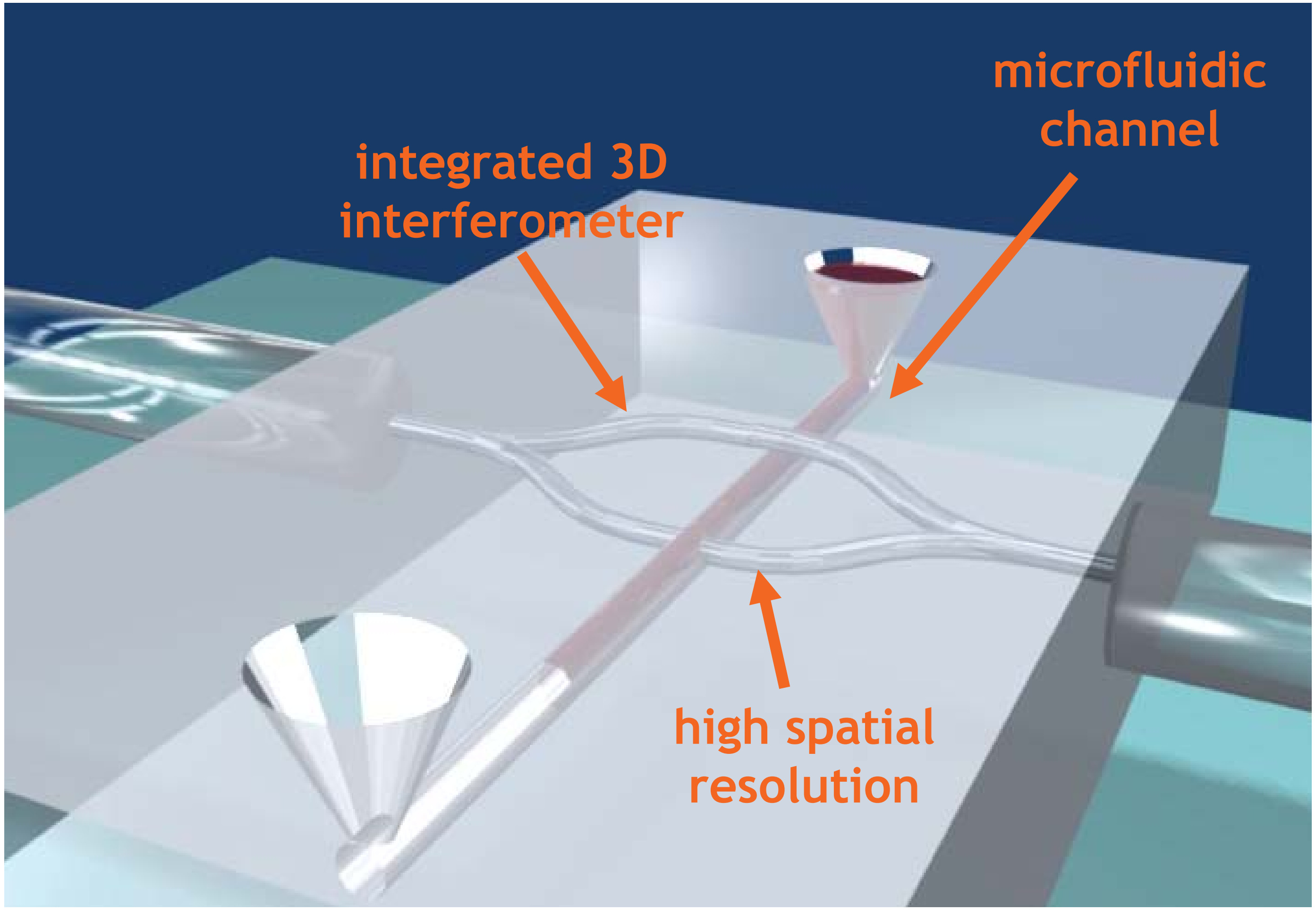
- Sample is a commercial DNA ladder from 50 bp to 3000 bp



C. Dongre et al., Electrophoresis 31, 2584 (2010)

C. Dongre et al., Lab Chip 11, 679 (2011)

Label-free detection



A. Crespi et al., Lab Chip 10, 1167-1173 (2010)

Refractive index sensing

Monitoring chemical reactions in microreactors through interferometry in the **Mach-Zehnder** configuration

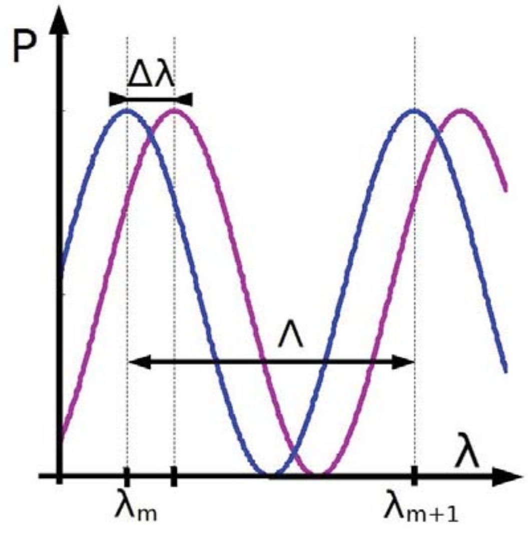
- Need for **spatial resolution** ⇒

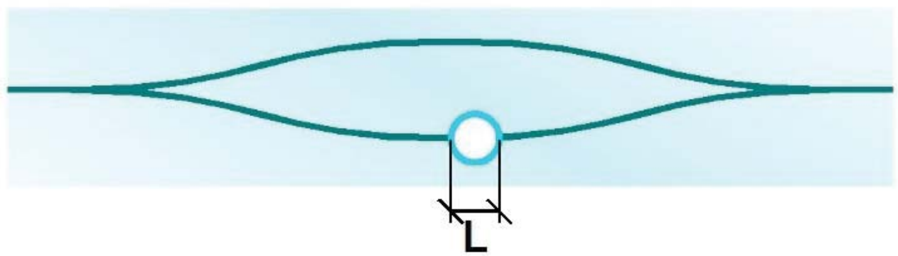
  interferometer **orthogonal** to the separation channel

- Channel width of only L = 50 µm ⇒

  evanescent field sensing too weak ⇒

  one arm of the interferometer is **crossing the channel**

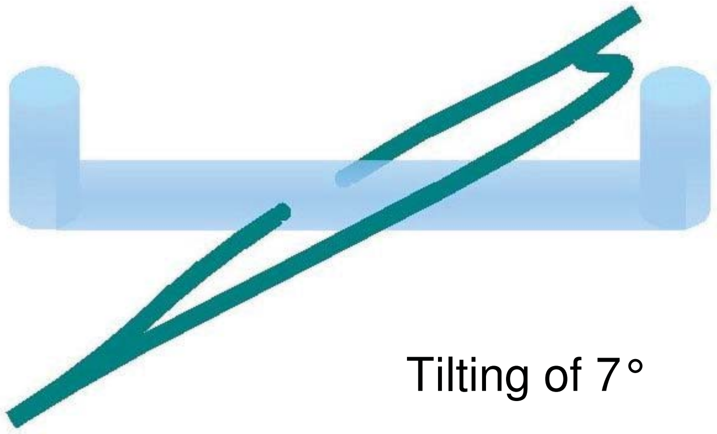


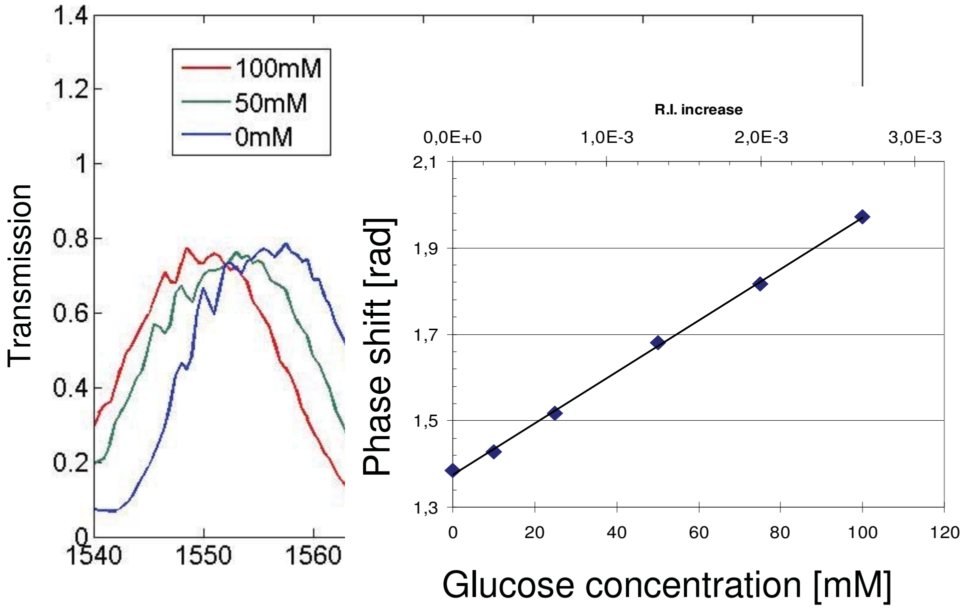


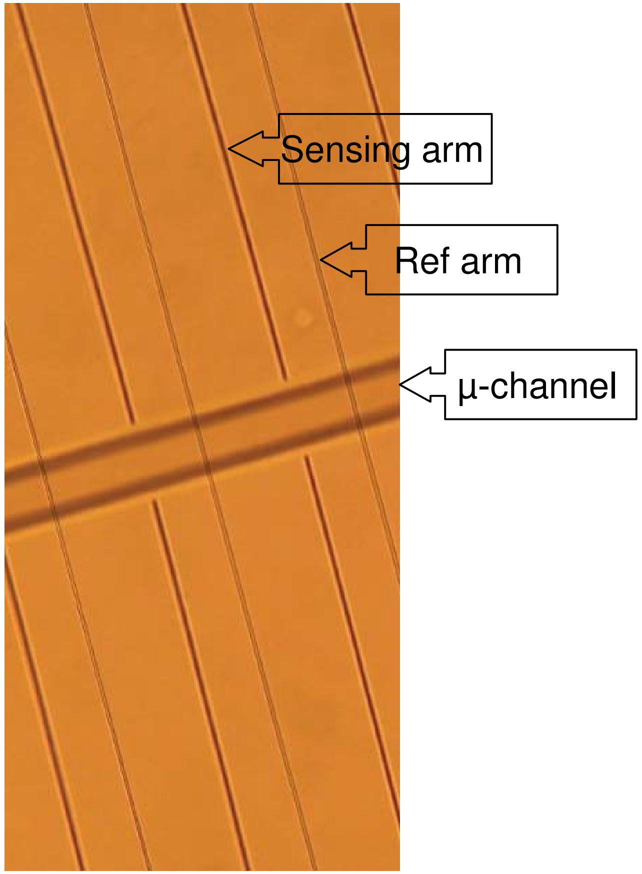
Fringe shift

The phase shift acquired in the channel induces a fringe shift

Mach-Zender integration

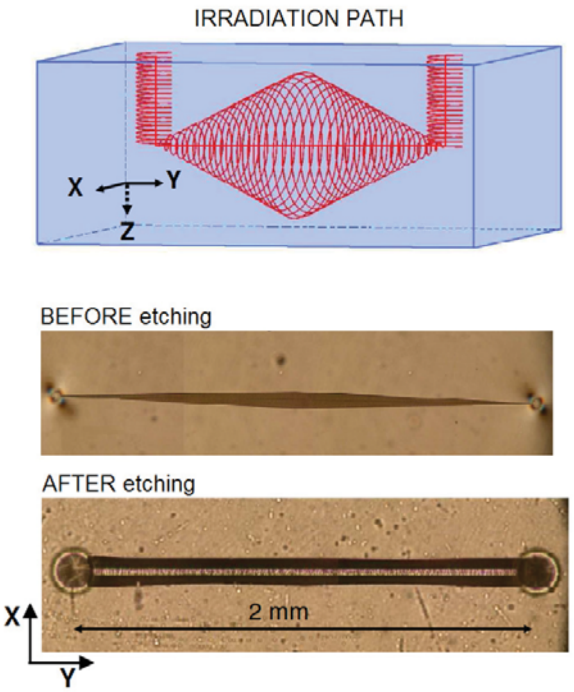




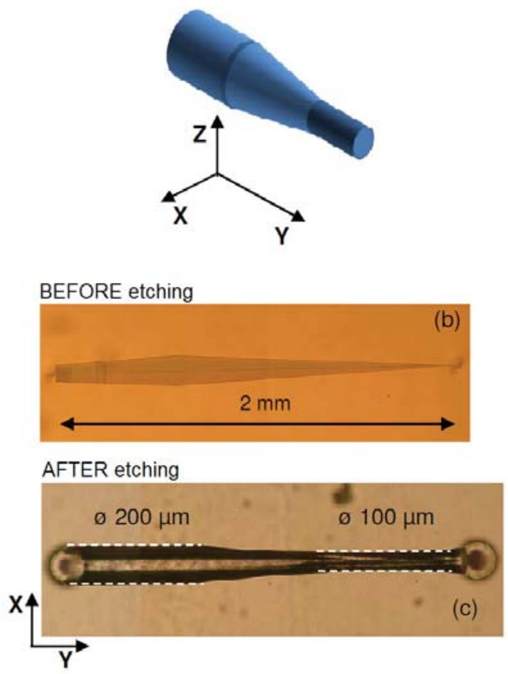


10 mM of glucose provides a very clear fringe shift
$\Rightarrow$ Sensitivity of at least $\Delta n=1\times10^{-4}$

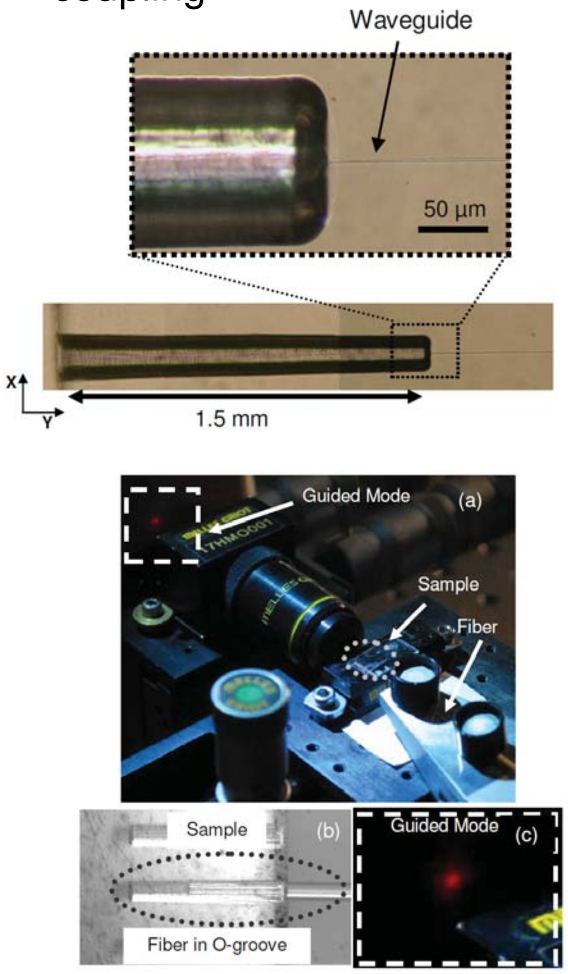
- Channel with top access holes (U-shape)



- Fannel-shape channel (shape control along the channel axis)

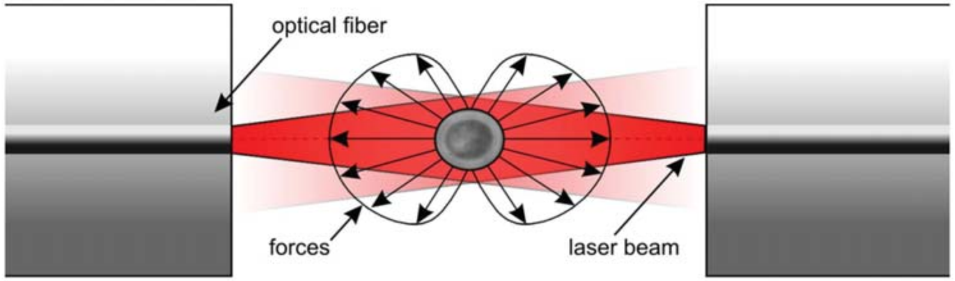


- O-groove for fiber coupling



Optical stretcher

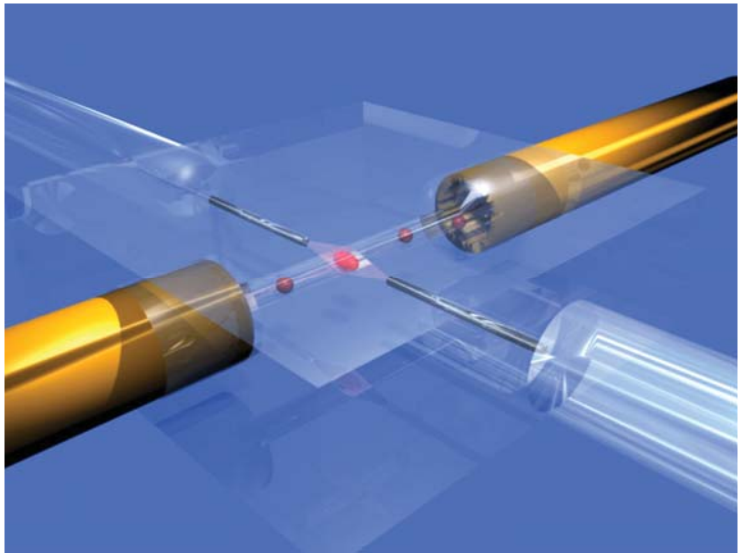
Optical dual-beam trap for single cell (*1μm wavelength*):
- non-focusing counter-propagating beams can trap single cells
- increasing the optical power the trapped cell is stretched along the beam axis



standard configuration: cell suspension in between 2 counter-propagating *fibers*

• misalignement between discrete optical and fluidic components

• system suffers for vibrations and fluctuations



monolithic configuration: optical waveguides crossing the microfluidic channel with the flowing cell suspension in a *glass chip*

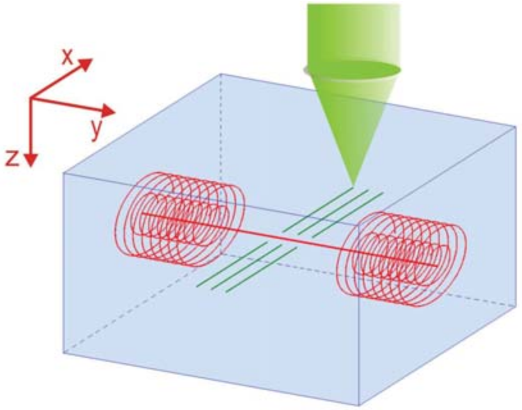
• no alignement + vibration problems

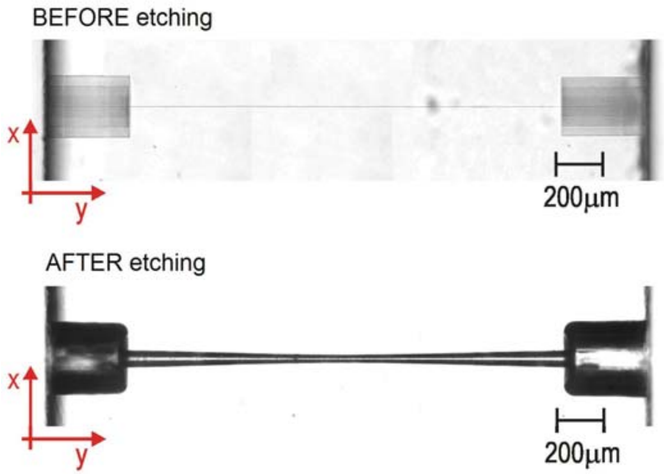
• higher system portability

• possibility of adding other waveguides for further optical funcionalities

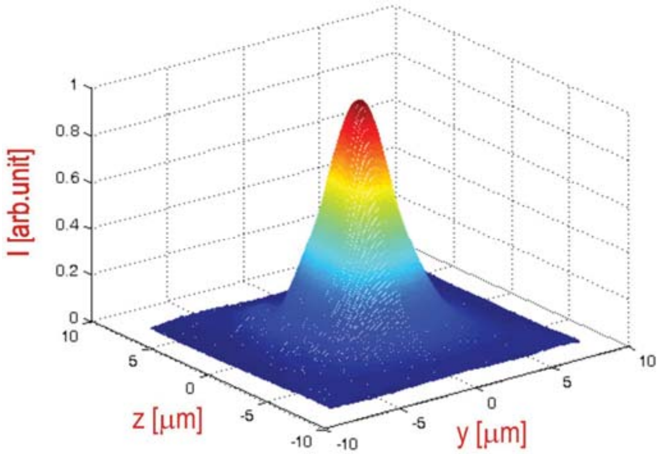
Optical stretcher: fabrication

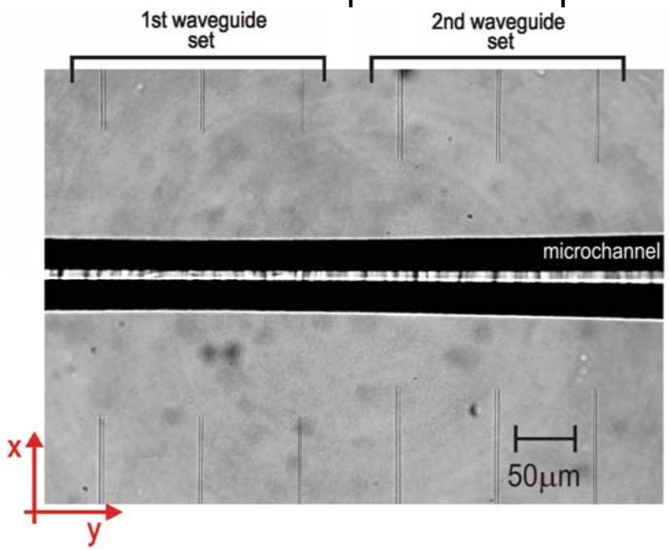
- Microchannel fabrication with larger access holes for capillary connection





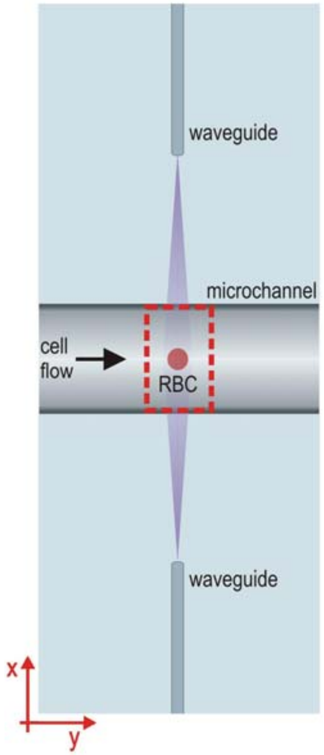
- “self-aligned” optical waveguides at different distance and depth with respect to the channel

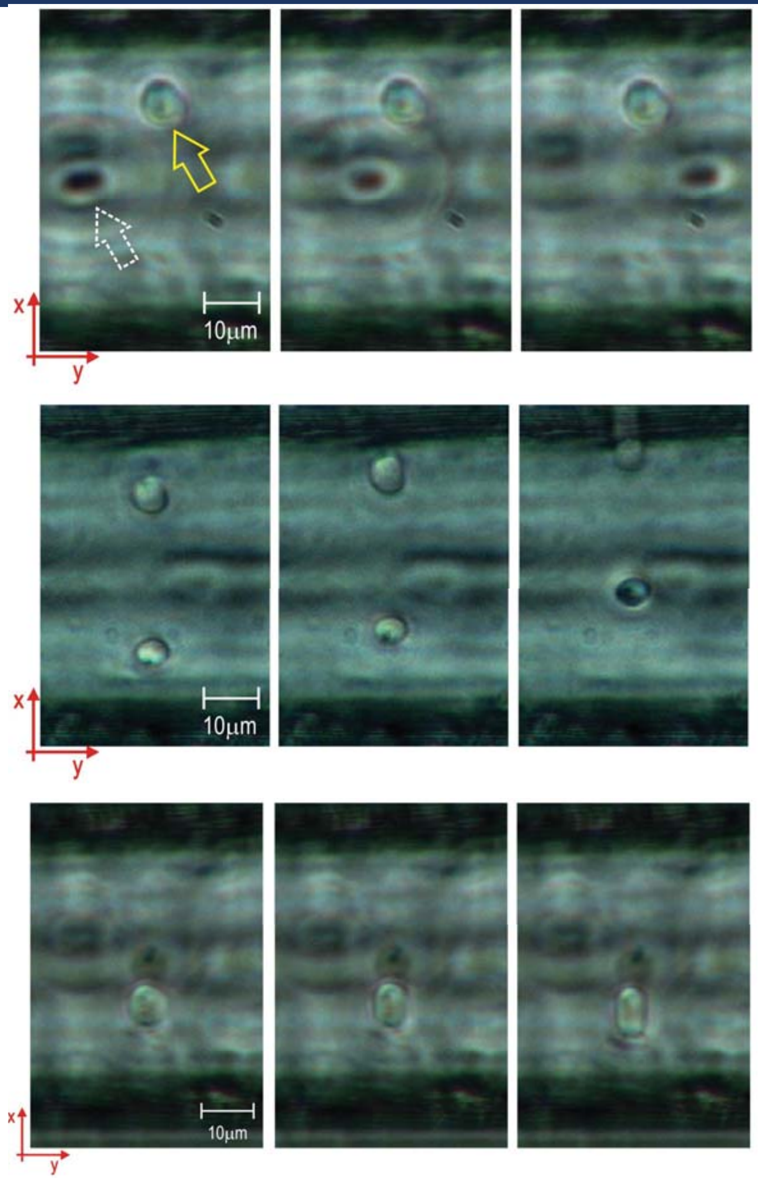




Optical stretcher: experimental results

Test the system on
red blood cells (RBCs)





TRAPPING

balancing optical power at the two waveguides (P_{opt}≈20mW)

MOVING

unbalancing the power at the two waveguides

STRETCHING

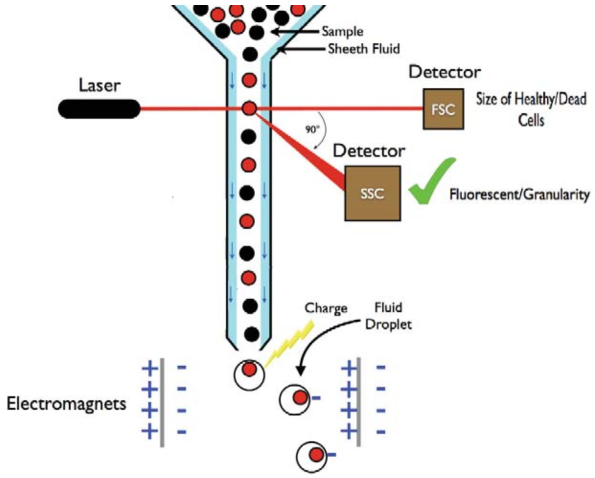
simoultaneously increasing the power at the two waveguides (P_{opt}≈300mW)

Fully-integrated fluorescence-activated cell-sorter

Cytofluorimeters are widely used in many applications (e.g. studies on cancer cells, immunology...) to select and isolate specific cells from an heterogeneous population

Microfluidic lab-on-chip cytofluorimeter would benefit from these advantages:

- Small sample volumes
- High selectivity
- High automation → low sample contamination
- Device compactness and portability



Advantages of the **optical sorting** technique:

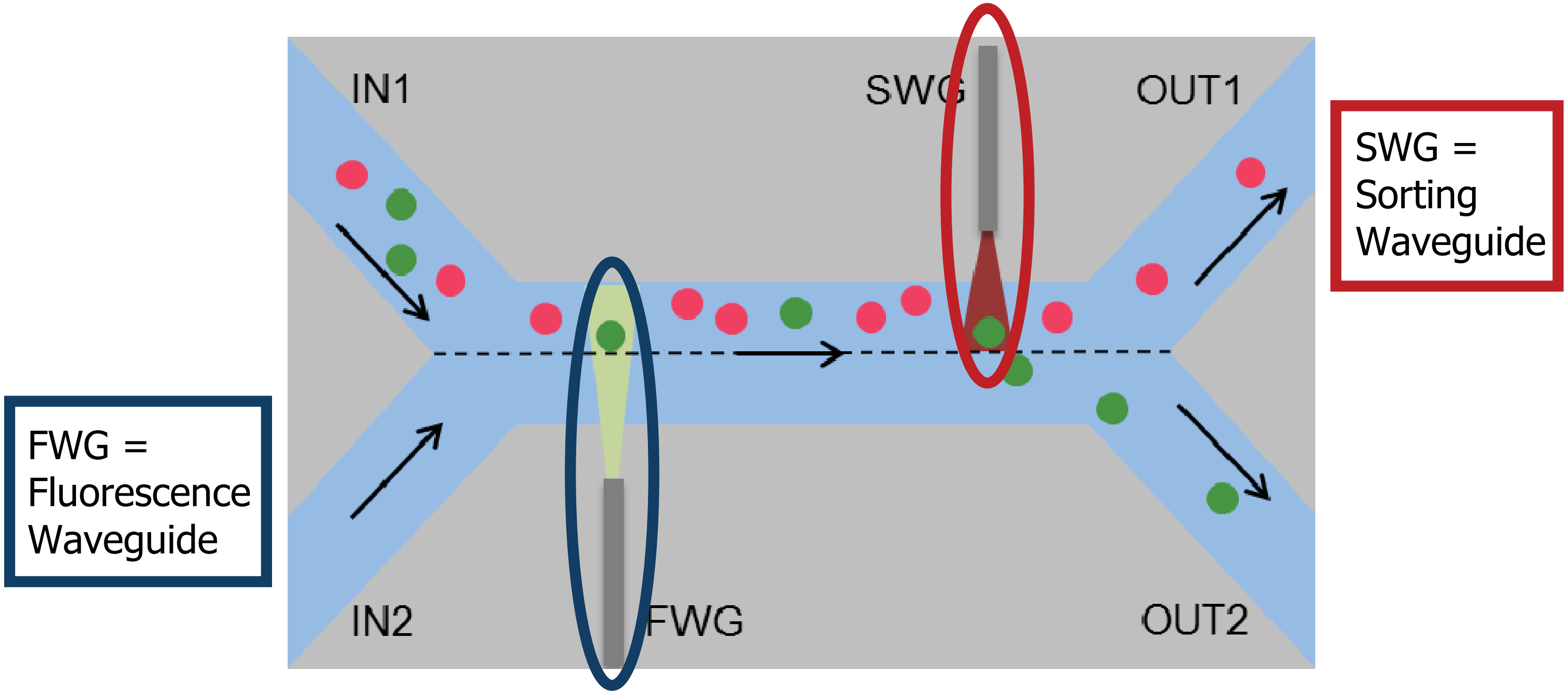
- Non-invasive technique → negligible damage on the sample
- Cell manipulation is allowed without the need for sample pre-treatment
- Analysis at **single cell** level

Optofluidic cell sorter working principle

•Fully–integrated Fluorescence Activated Cell Sorter (μ**FACS**)

↓

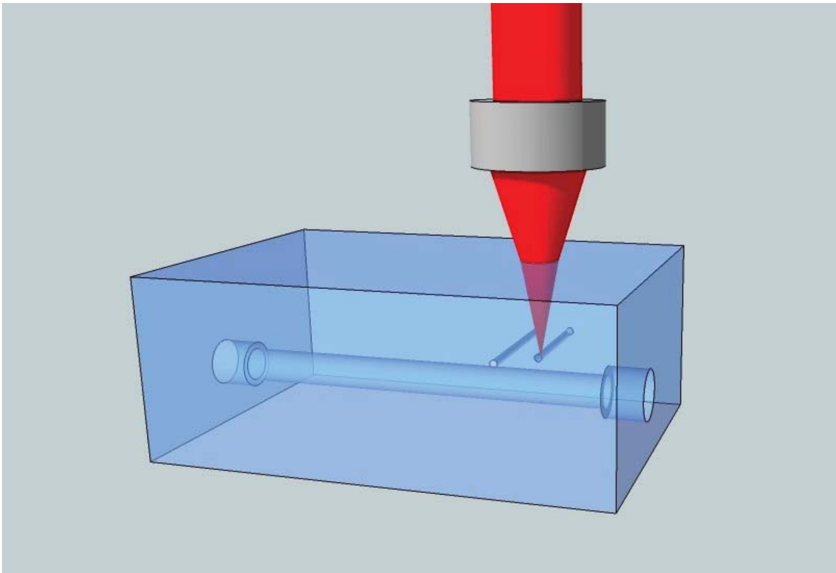
•The device is able to separate through optical forces specific cells from an heterogeneous population, on the basis of their fluorescence properties



Femtosecond laser micromachining

Femtosecond Laser Micromachining (FLM)

Optofluidic integrated device fabrication



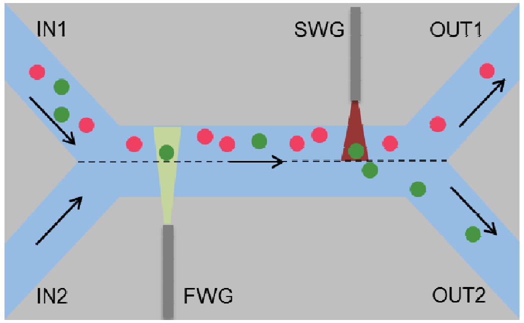
very accurate alignment between fluidic and optical components!!!

Optical waveguide fabrication

Buried microchannel fabrication

Optofluidic cell sorter fabrication

- Microchannel
- Optical waveguides (ortogonally intersecting the channel)

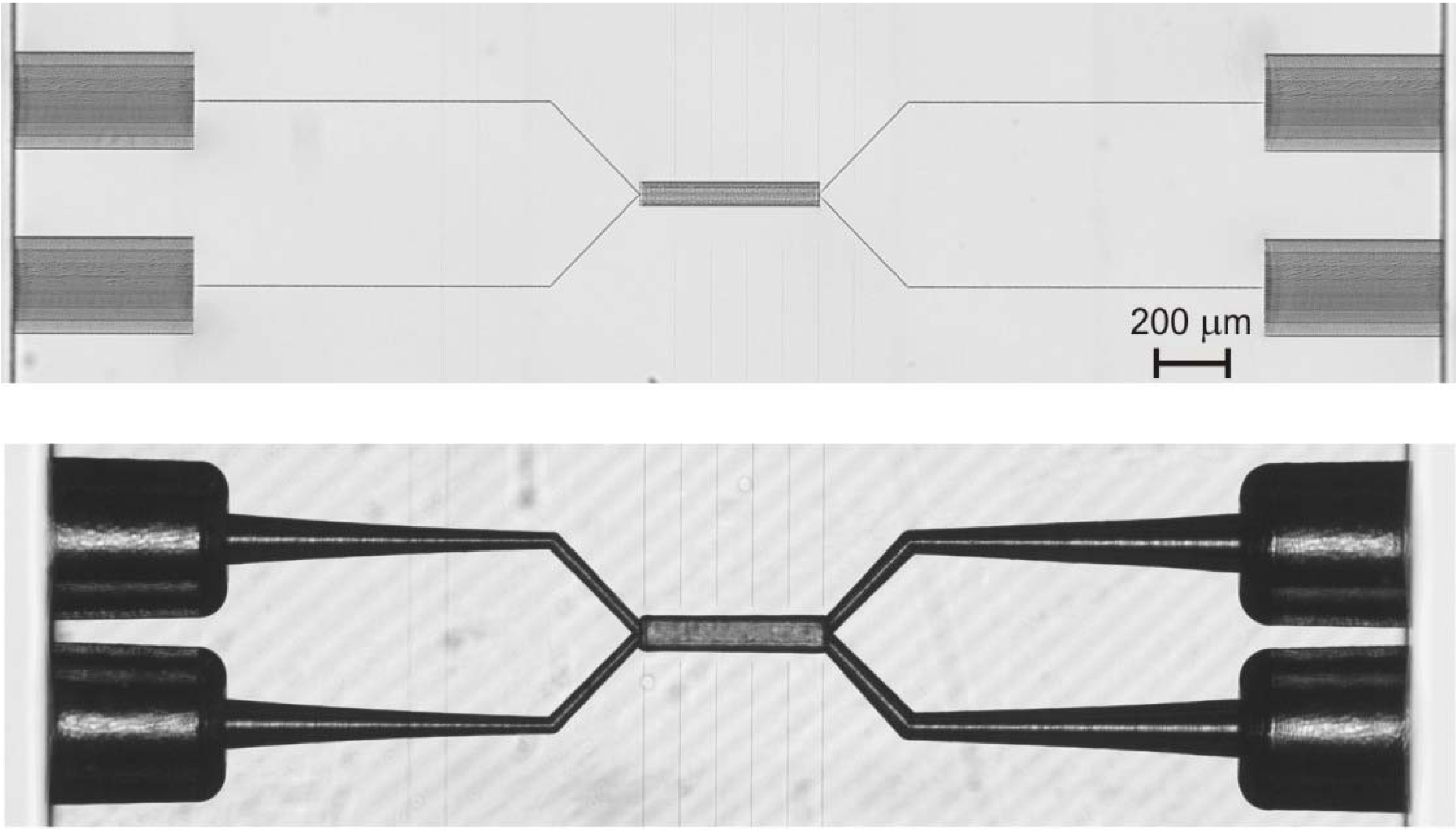


1. **FLM**: HighQ laser, II-harmonic (520 nm), RR=500KHz, Obj=50x, 0.6 N.A.

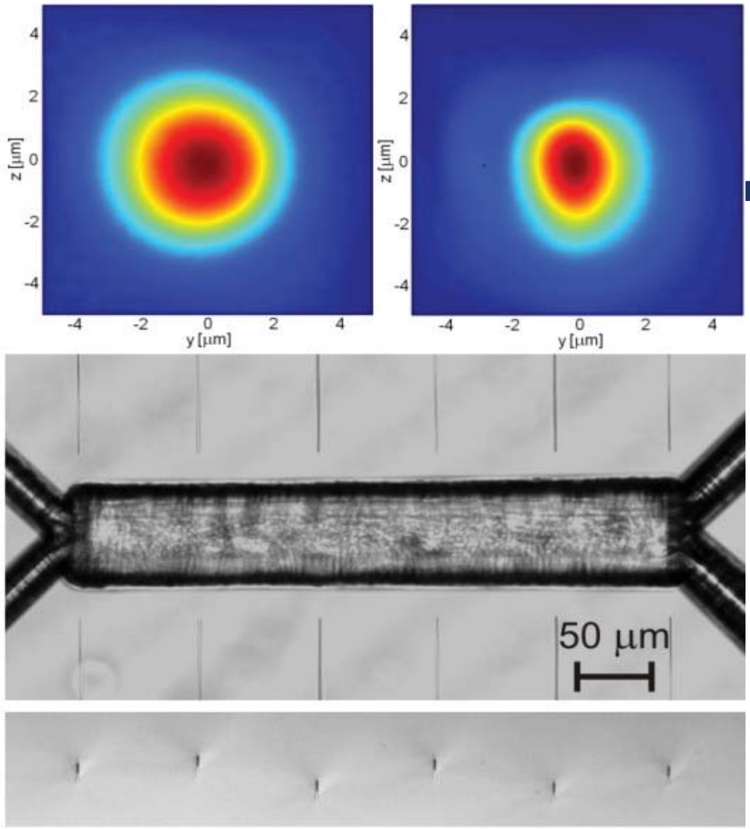
Parameters	Energy	Scan velocity
Microchannel	700 nJ	1 mm/s
Optical waveguide	100 nJ	0.1 mm/s

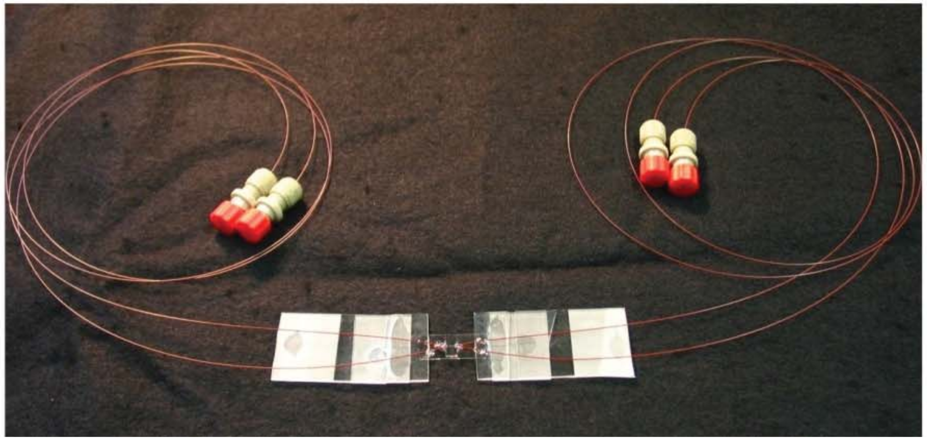
2. **Chemical etching** (HF 20%, T=35°C, ultrasonic bath)

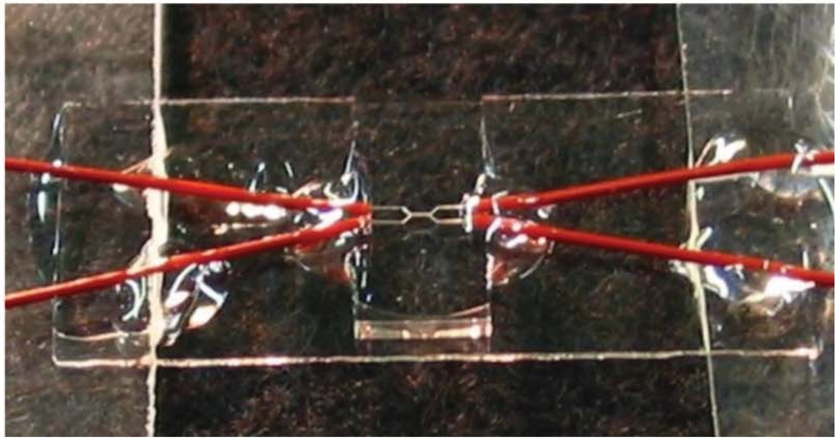
Fabrication



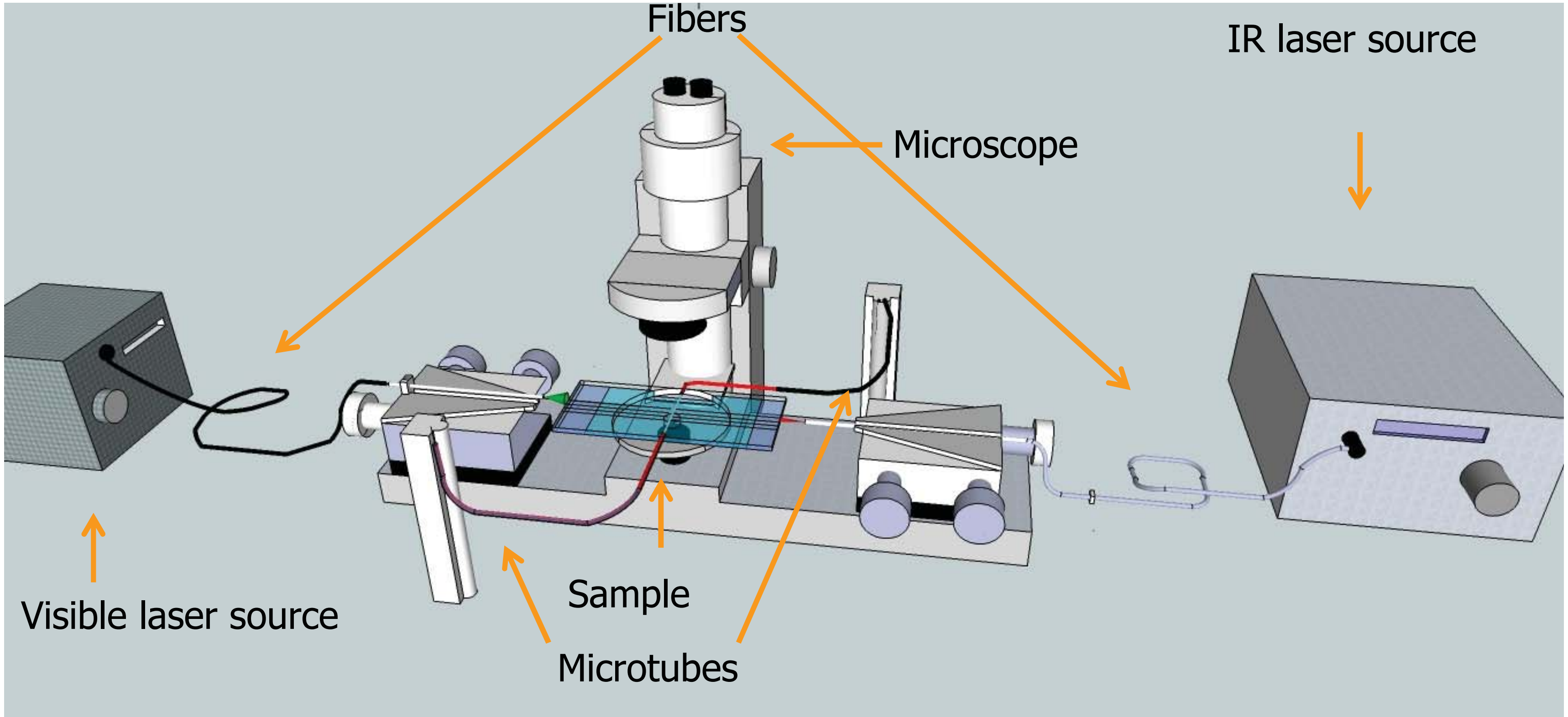
Fabrication







Experimental Set-up

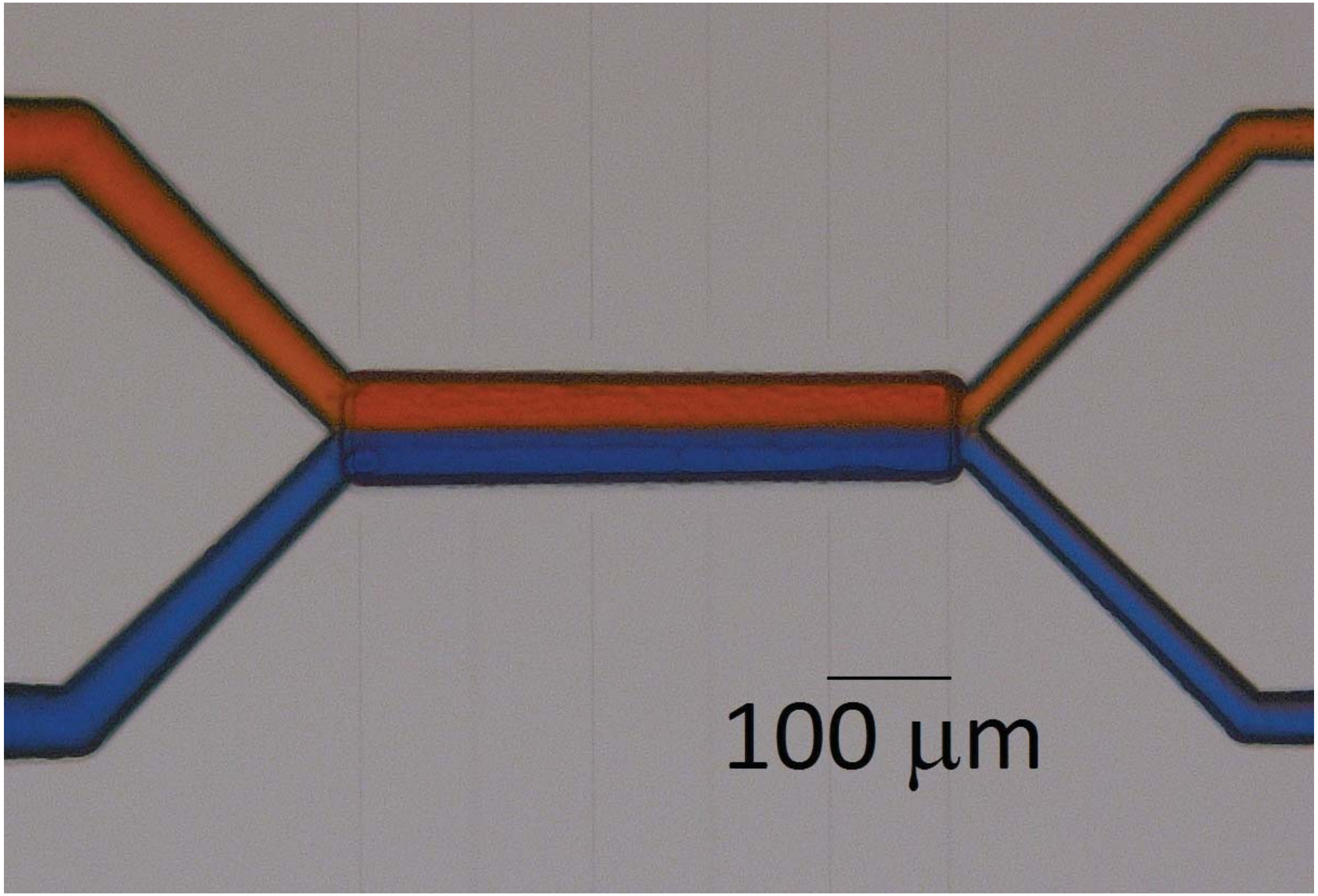


Device validation

1. Flow laminarity
2. Fluorecence excitation capability
3. Optical sorting capability
4. μFACS operation →
   - Polystyrene beads
   - Cells

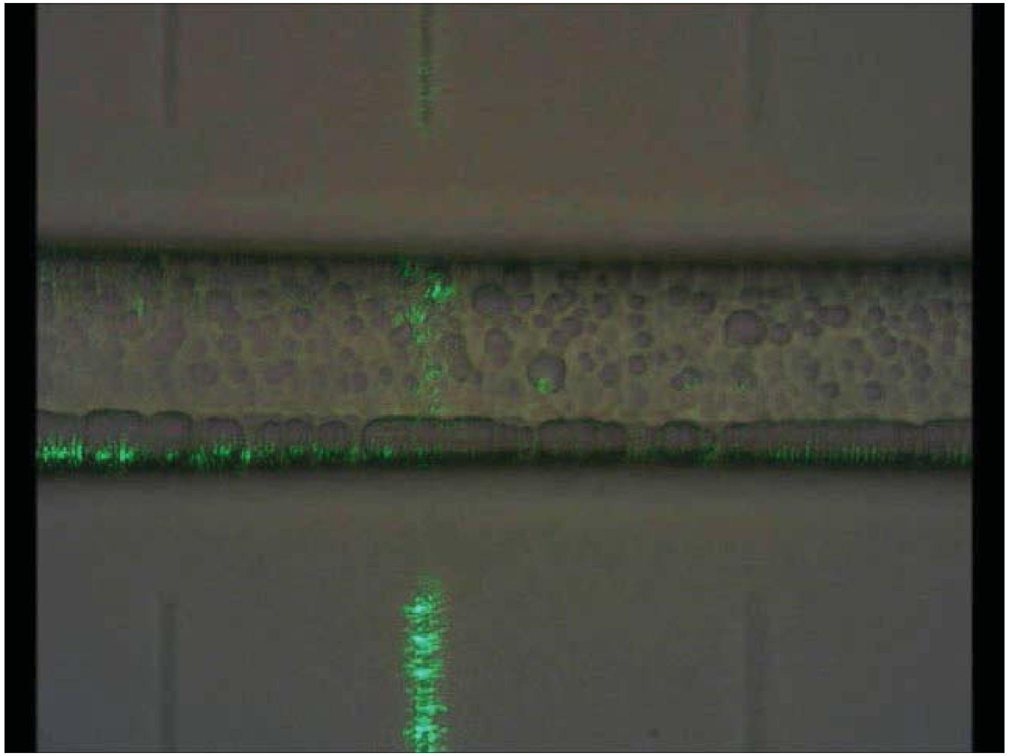
Flow laminarity

1.



Fluorescence excitation capability

2. Efficient fluorescent excitation of every bead flowing in the microchannel



Fluorescent polystyrene beads
(diameter ≈ 7μm, λ_{ex} = 543 nm, λ_{em}= 640 nm)

Optical sorting capability

3.

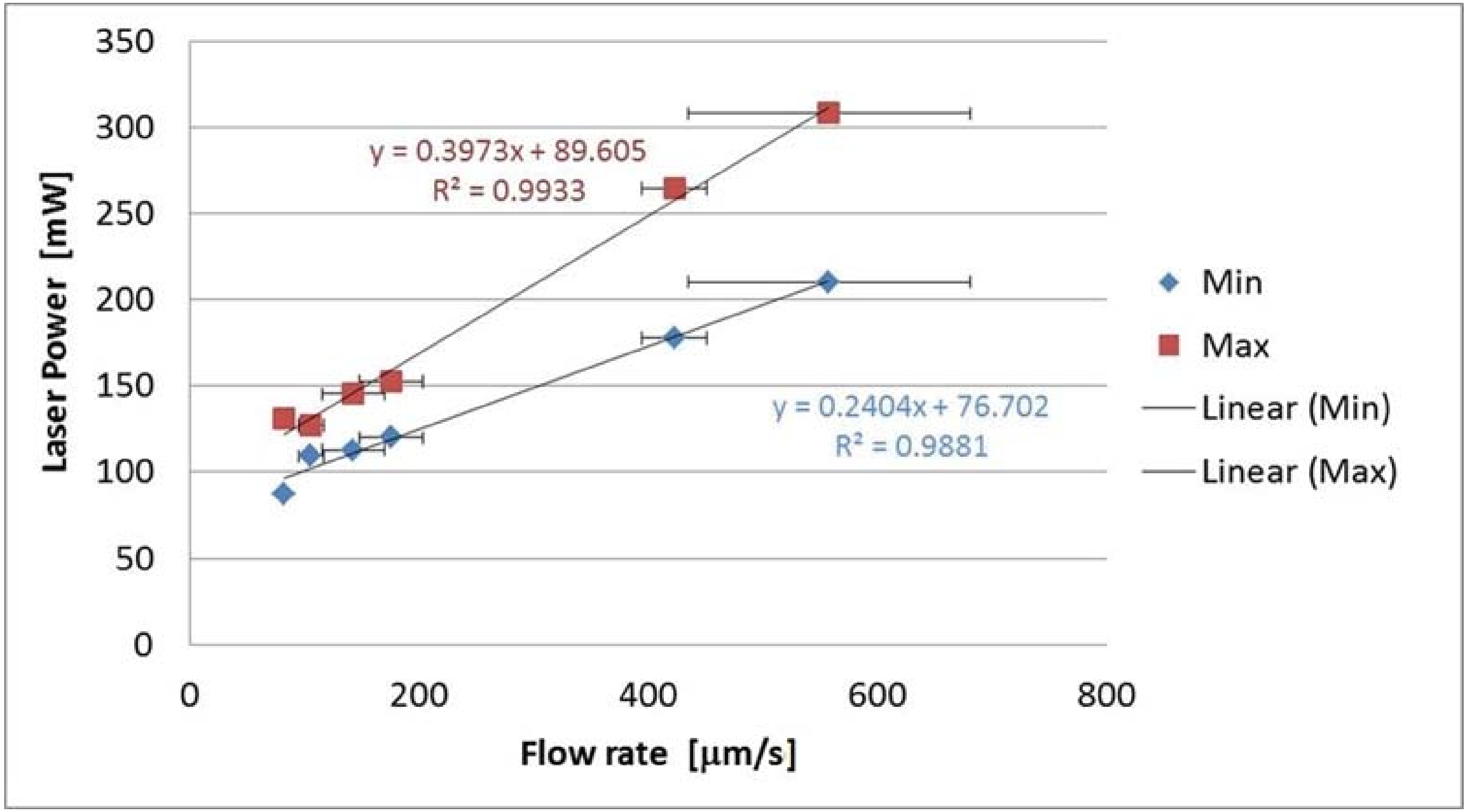
Efficient single cell sorting through optical forces
(λ_{sorter} = 1070 nm, P = 120 mW)

Polystyrene beads (diameter ≈ 7μm), v = 180 μm/s

Optical sorting capability

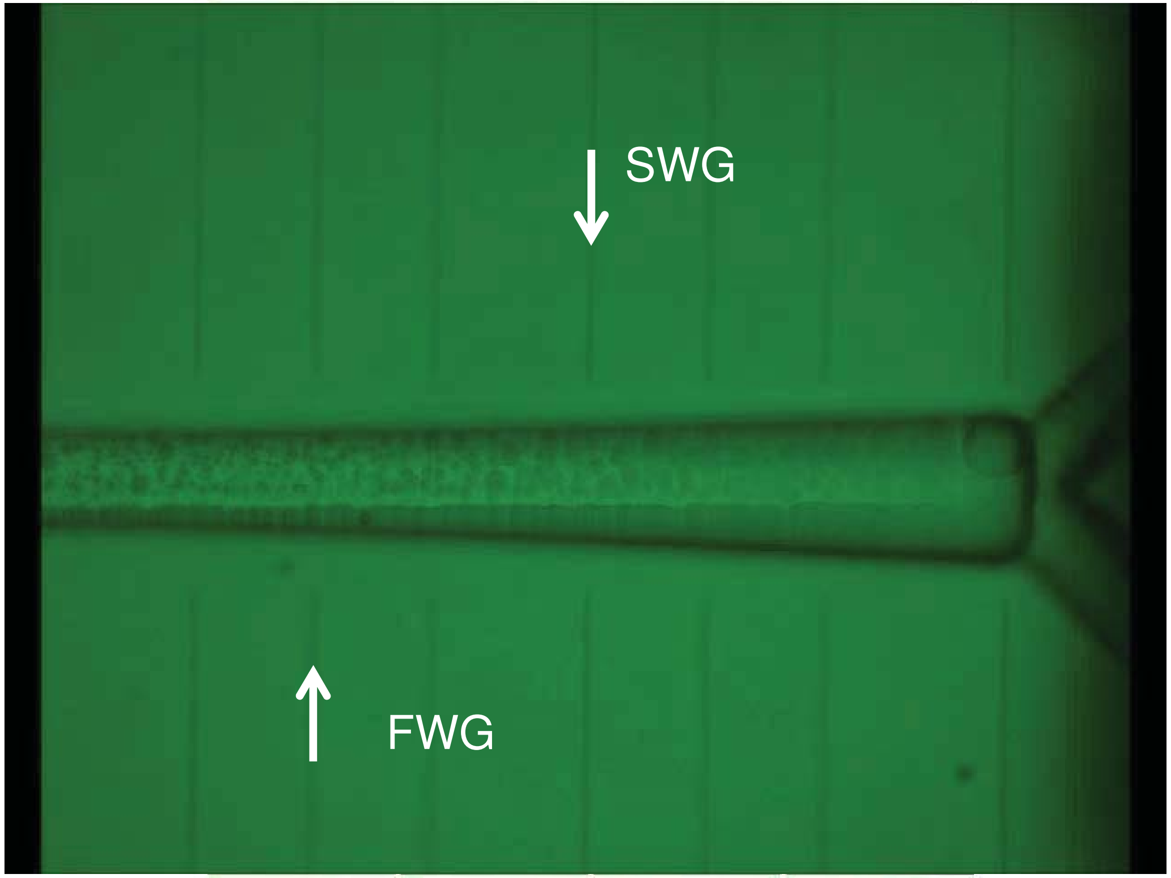
3.

- Minimum and maximum laser power to sort each bead



4.

Device operation as a micro fluorescence activated cell sorter



Human fibroblast marked with fluorescent protein
(EGFP- λ_{ex}= 488 nm , λ_{em}= 505 nm).

Polymeric Solid state devices are well established

Conjugate Polymers in ***solutions*** have additional ***advantages,*** e.g. good isolation of PFO polymer chains in solutions enables to have Ultrafast optical gain switching

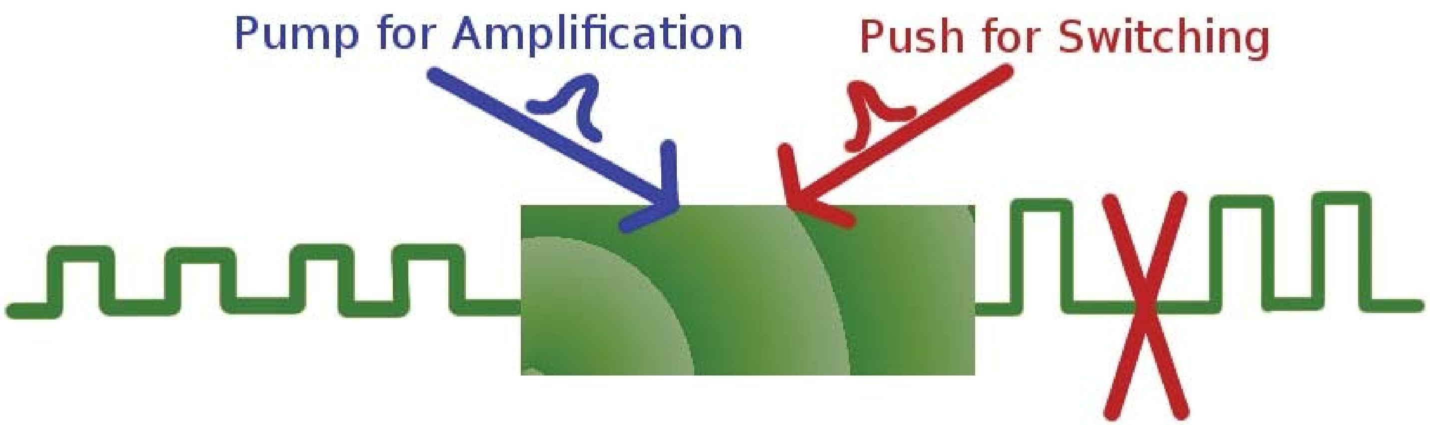
Advantages of Optofluidic devices

- Integration of microfluidic and optical functions
- Ease of reconfiguration, tailoring the device characteristics by simple replacement of solutions

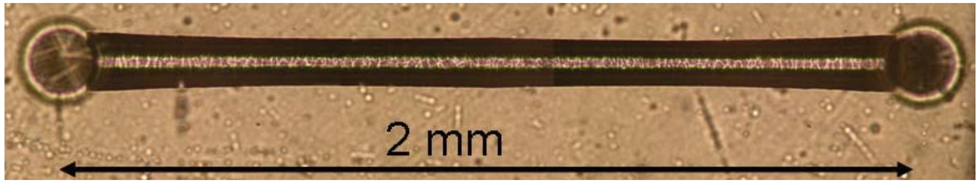
Image: http://www.epmm.group.shef.ac.uk

Gain Switching Concept

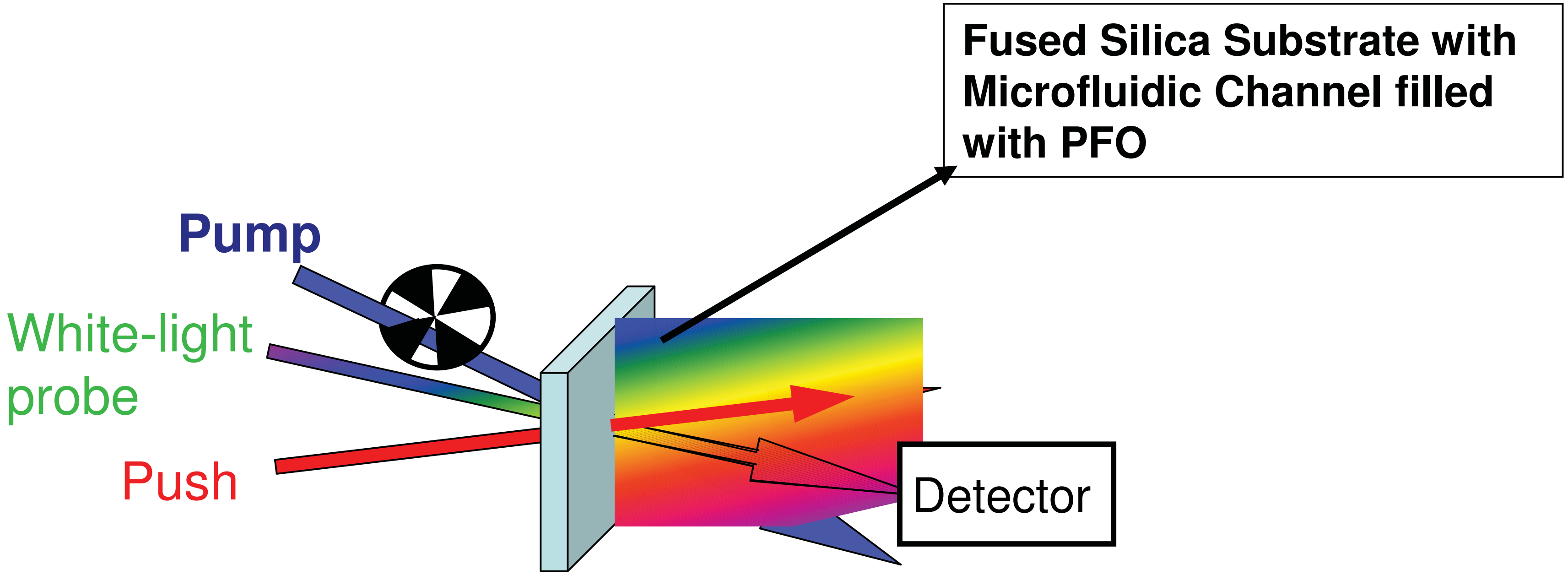
- When isolated, conjugated polymers such as poly(9,9-dioctylfluorene) (PFO) can support short-lived charges. Their photoinduced absorption spectrally overlaps with the stimulated emission region. Therefore, optical generation of these charges by a gating pulse (push) causes a switching off of the stimulated emission



- Good isolation achieved in cuvettes, but not practical for integration
- Solid state shows clustering and very poor quality

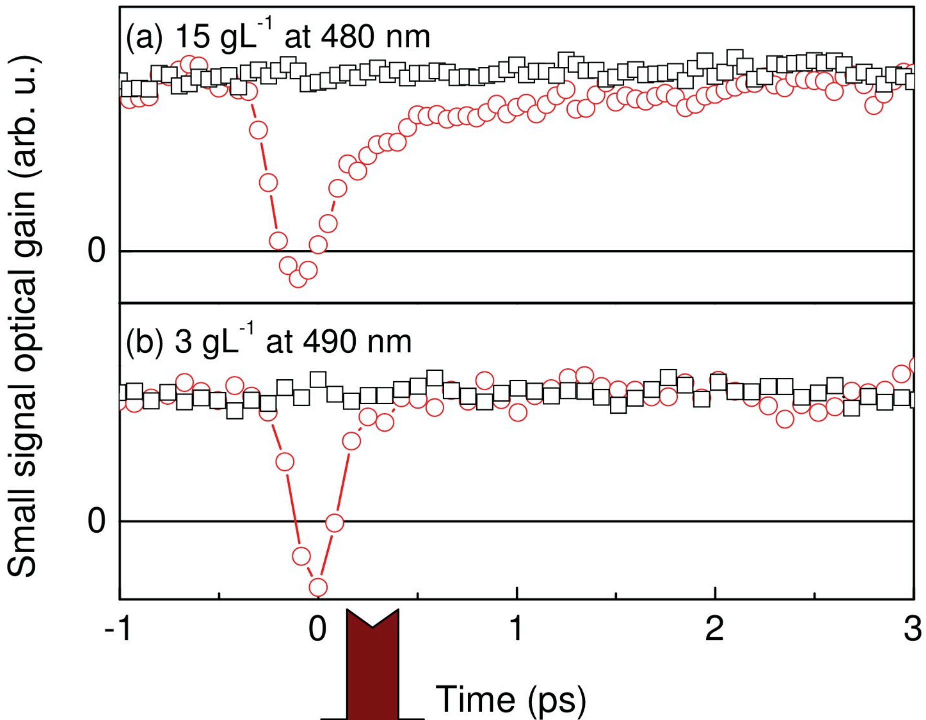


Solution of PFO [poly(9,9-dioctylfluorene)] in Decaline is filled into the U-shaped channel

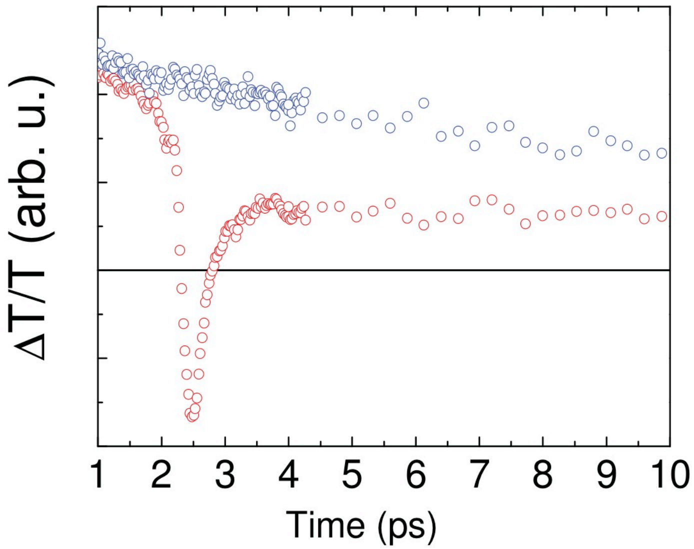


Ultrafast Optofluidic Gain Switch

Optofluidic device



Solid state device



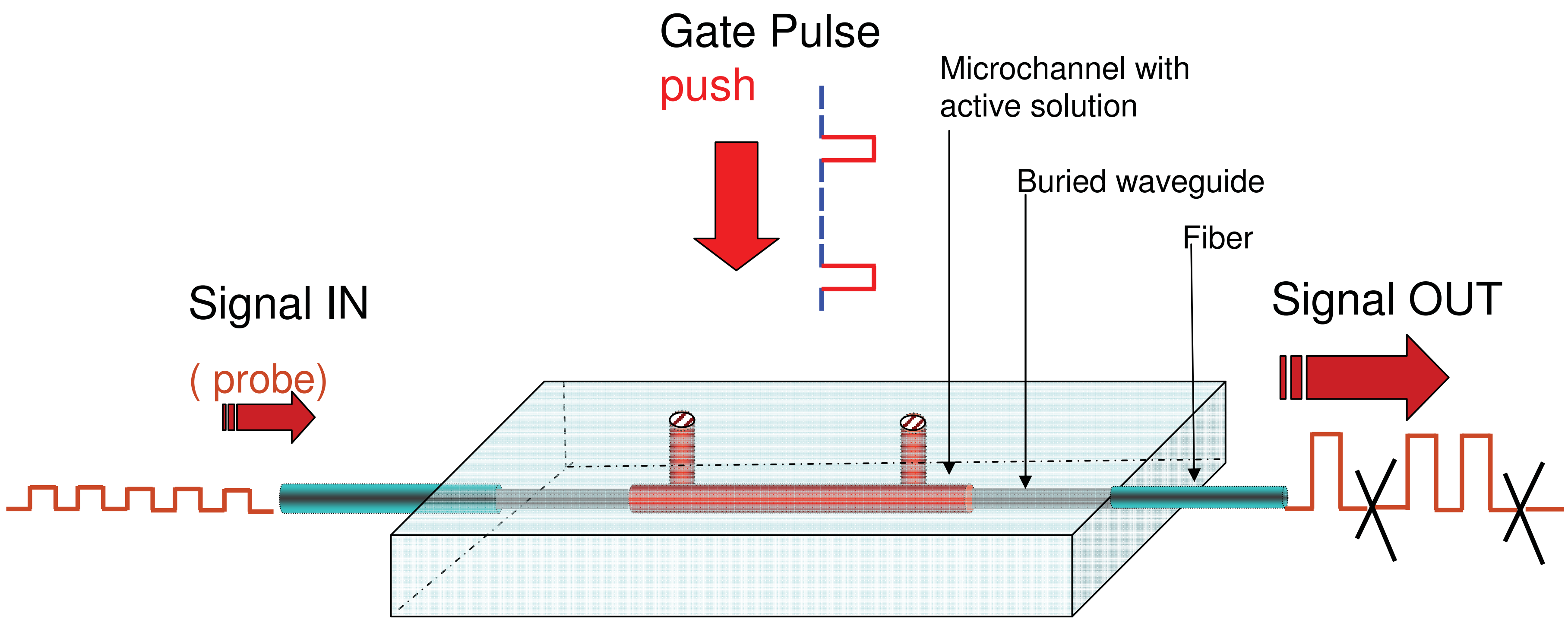
Recovery constants

15 gL^{-1} : 350±20 fs

3 gL^{-1} : **< 150 fs**

Optical Switching speeds well into **THz regimes**

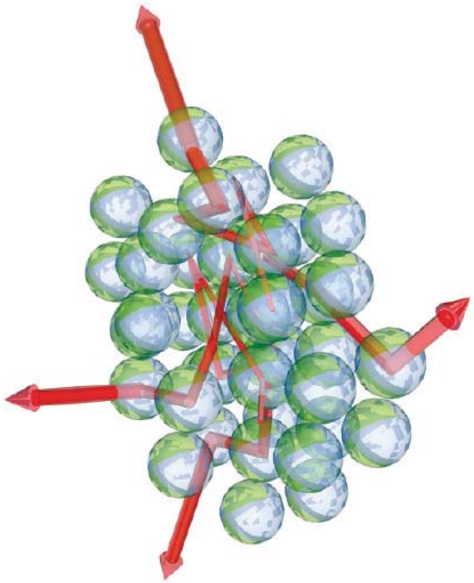
Schematic Diagram of Integration



Applications

- Amplifier, Optofluidic gain switch
- Signal encryption

Random Lasing



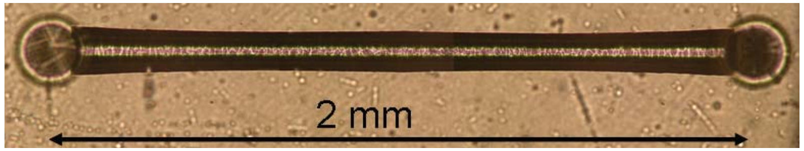
Definition of random lasing:

(1) light is multiply scattered owing to randomness and amplified by stimulated emission, and

(2) there exists a threshold, due to the multiple scattering, above which total gain is larger than total loss.

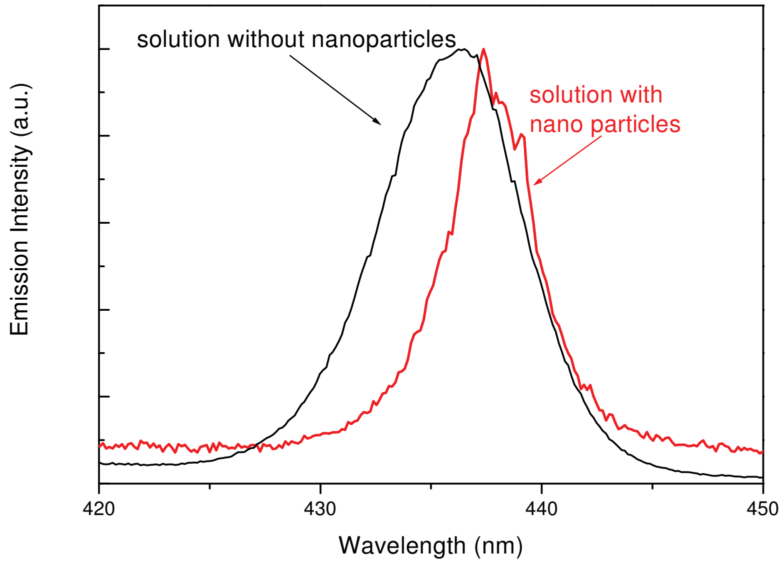
DIEDERIK S. WIERSMA, Nature Physics, vol. 4, May2008

Random Lasing from Microchannels filled with PFO and TiO_2 nanoparticles

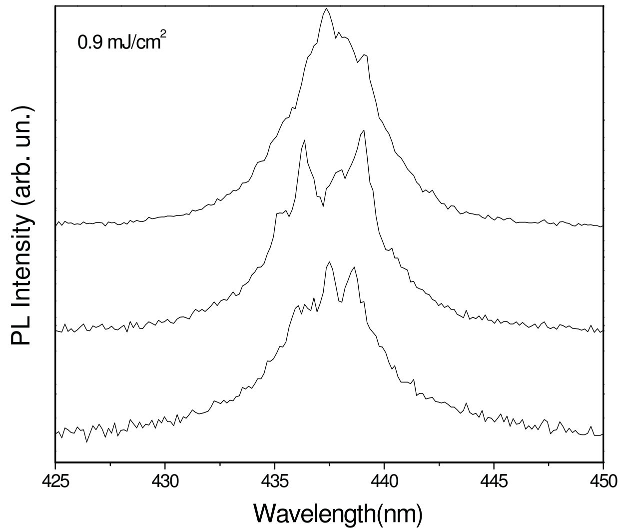


Microchannels were filled with PFO solution and

TiO_2 nanoparticles (~ 100nm)



Random Lasing from Microchannels filled with PFO and TiO_2 nanoparticles (2)

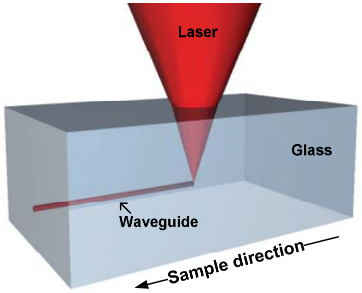


Random lasing modes overlapping the PL emission spectra.
(taken at intervals of 0.2 seconds from the same nanoparticle-dispersed PFO solution)

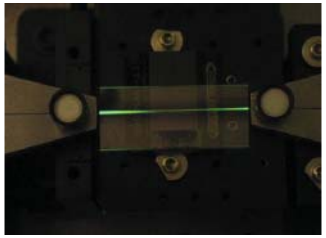
The competition between the different modes is a signature of random lasing.

Conclusions

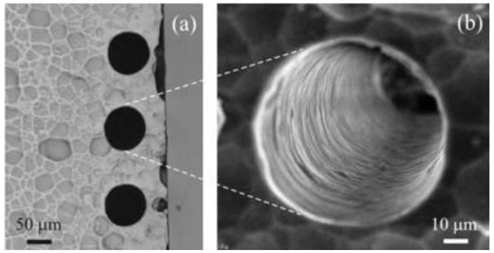
- Femtosecond writing is a simple and powerful technique for the direct fabrication of high quality optical waveguides



- A variety of passive and active devices, both 2D and 3D, can be manufactured in various glass substrates



- Femtosecond laser irradiation + etching provides directly buried 3D microchannels



- Waveguides and channels can be integrated in different geometries to implement optofluidic functionalities and to fabricate optofluidic devices for a wide range of applications