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# Recent applications of fibers for optical sensors and particles manipulation in lab-on chip systems

C. Sada

A. Zaltron, G. Bettella, G. Pozza, R. Zamboni

# Lab on a chip

## Outline

2

### • Introduction

- Opto-fluidics: smart solutions
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

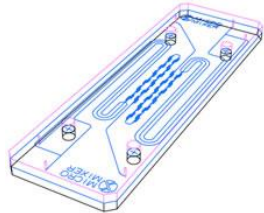
## Optofluidics



Microscope



Fast Camera



Microfluidic chip

Lack of  
integration

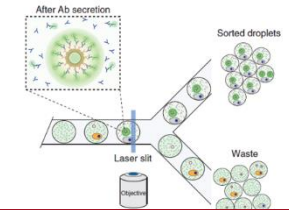
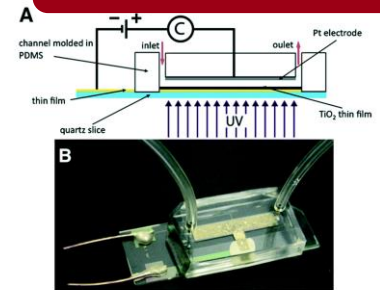


Lab-on-a-Chip

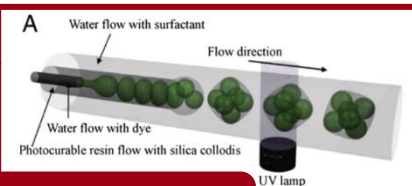


Syringe Pumps

## Chemical synthesis



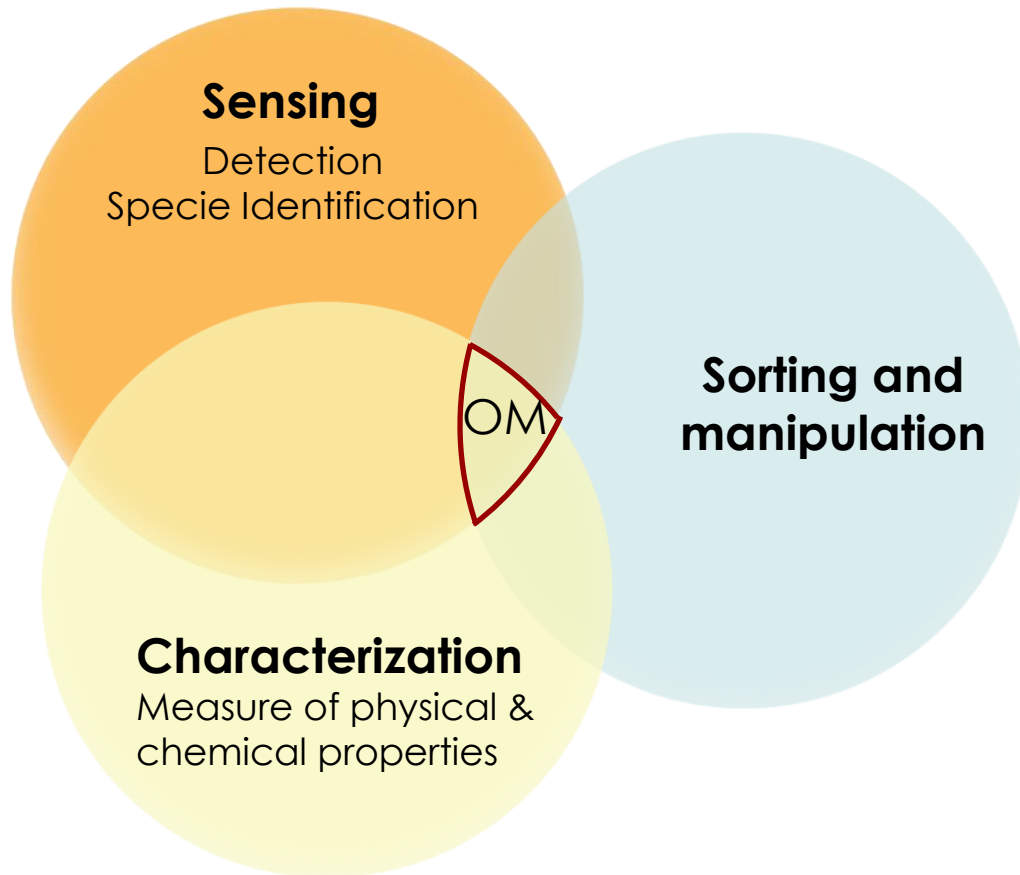
Cells Manipulation



Drug delivery

# Lab on a chip

## ***“Frame”*** of reference



### Outline

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### **OM:Opto-microfluidics**

- Small efficient device
- Multifunctional
- Portable
- Low cost

# Opto-Microfluidics Integration

• Introduction

- Opto-fluidics: smart solutions
- Fibers for coupling
- Fibers beyond coupling
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## Microfluidics circuit

- Laser ablation
- Laser irradiation+ chemical etching
- Casting
- Molding

## Substrates

- Glass/silica
- Silicon + ..
- PDMS, PPMA, ...
- Hybrids

## Optical circuit

- Laser irradiation
- Local doping
- Incorporation/Bonding
- **Fibers coupling**

## External Coupling & Detection (Fibers)

**Issue:** optimisation of the coupling between optical and microfluidics stages on the same substrate

# Outline

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### • Introduction

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## Fibers always needed for :

- Waveguiding;
- Guaranteeing portability and flexibility of the device;
- Laser sources coupling.

## Outline of this talk:

- Examples of **fibers applications** (coupling and beyond coupling);
- Limits of current results;
- What is needed next.

# Absorption Analysis

## Outline

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- Introduction
- Fibers for coupling
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## Fiber coupling

### A hybrid silicon-PDMS optofluidic platform for sensing applications

Genni Testa,<sup>1,\*</sup> Gianluca Persichetti,<sup>1</sup> Pasqualina M. Sarro,<sup>2</sup> and Romeo Bernini<sup>1</sup>

<sup>1</sup>Institute for Electromagnetic Sensing of the Environment (IREA), National Research Council, (CNR), Via Diocleziano 328, 80124 Napoli, Italy

<sup>2</sup>DIMES-ECTM, Delft University of Technology, Feldmannweg 17, 2628 CT Delft, The Netherlands  
[testa.g@irea.cnr.it](mailto:testa.g@irea.cnr.it)

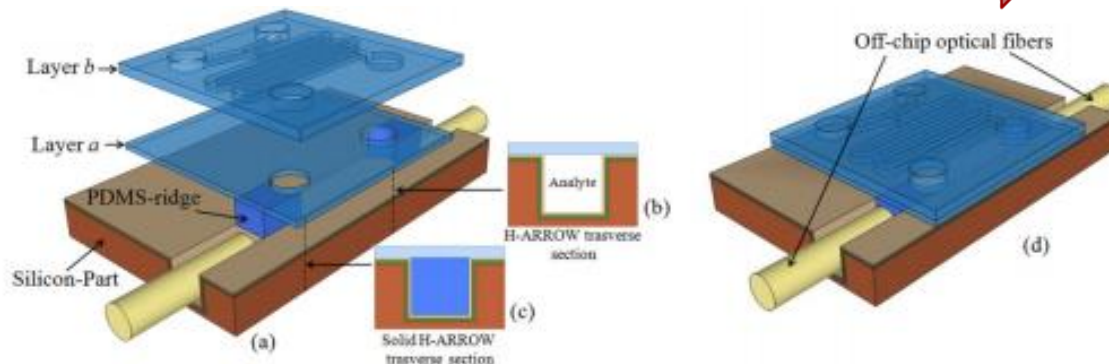
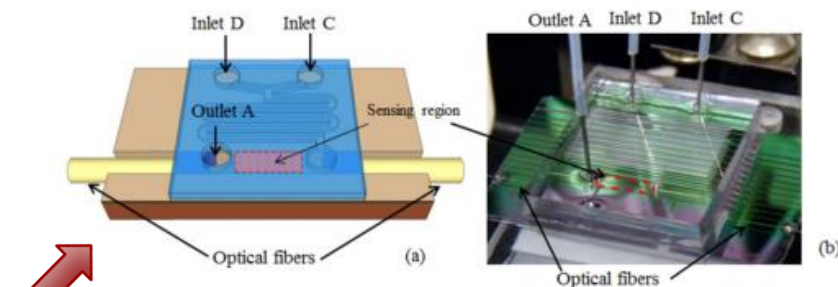
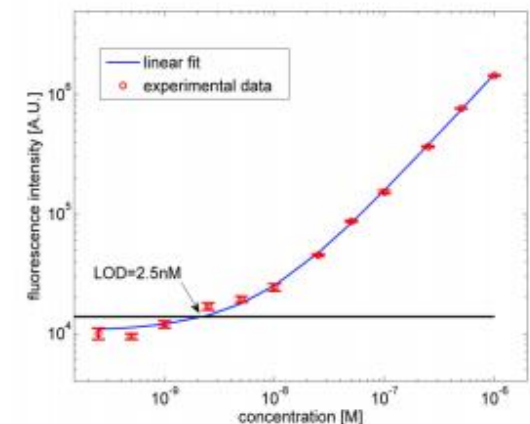


Fig. 1. (a) Exploded view drawing of the proposed hybrid ARROW optofluidic platform with transverse section of (b) liquid h-ARROW and (c) solid h-ARROW. (d) Schematic of the assembled device.



### Fluorescence of Cy5 in H<sub>2</sub>O



# Particle sorting

## Flow cytometer: an integrated device

### Outline

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- Introduction
- Fibers for coupling
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- Conclusions

*L.-M. Fu et al. / Analytica Chimica Acta 507 (2004) 163–169*

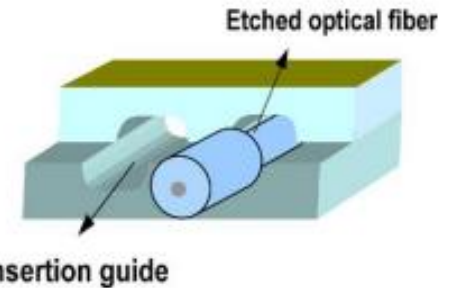
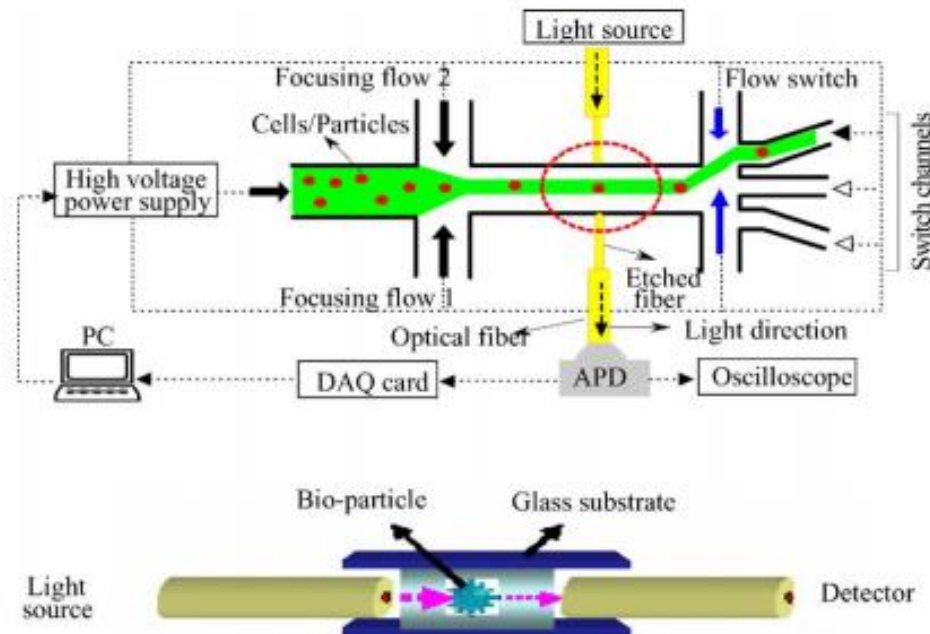
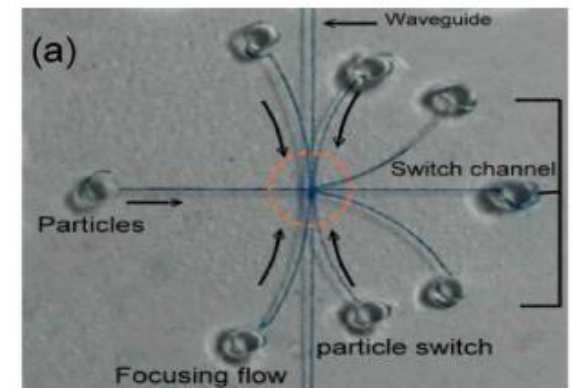


Fig. 3. Schematic of “insertion guide” for etched optical fibers.



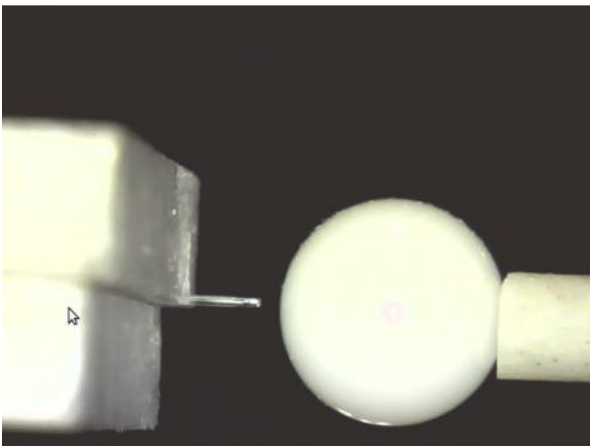
# Fiber shaping: feasibility

- Introduction
- Fibers for coupling
- Fibers beyond coupling
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- Conclusions

## Fiber Cavity

Alternative techniques to shape a fiber tip with concave curvature

Fiber shaped on a sphere at 1200 – 1600°C



Produce really concave spherical tips  
with a larger useful diameter  
(and so more stable cavities)

\*Work in collaboration with Tracy Northup @ UIBK - Austria

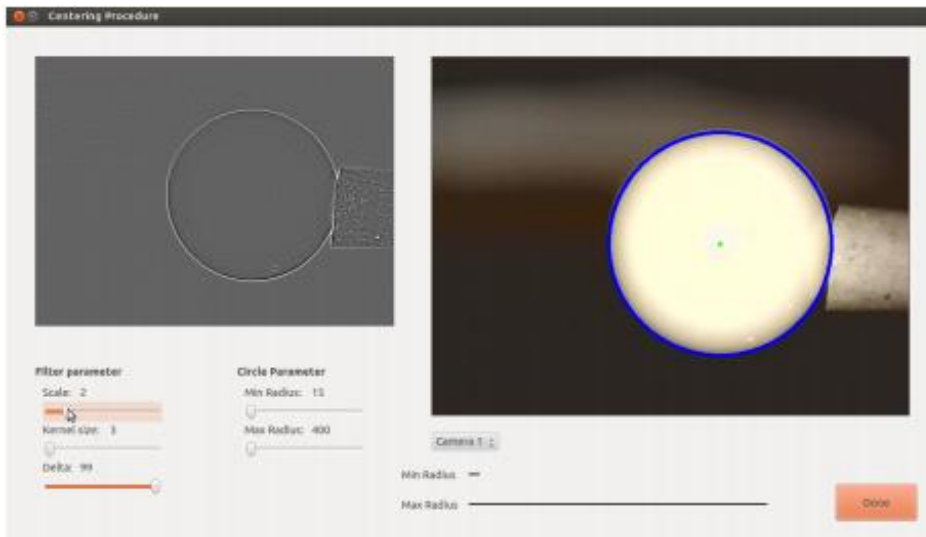
# Fiber shaping: feasibility

## Outline

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## Fiber made Cavity:



Process optimization



\*Dario Fioretto @ UIBK - Austria

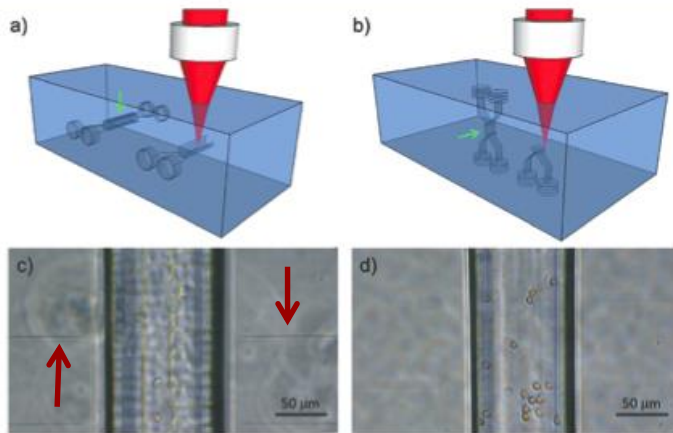
When cavity are used sensitivity increases of at least 2 orders of magnitude

# Particle manipulation

- Introduction
- Fibers for coupling
- Fibers beyond coupling
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## An integrated optofluidic device for single-cell sorting driven by mechanical properties†

T. Yang,<sup>‡a</sup> P. Paiè,<sup>‡bc</sup> G. Nava,<sup>a</sup> F. Bragheri,<sup>c</sup> R. Martinez Vazquez,<sup>c</sup> P. Minzioni,<sup>\*a</sup> M. Vegliione,<sup>d</sup> M. Di Tano,<sup>d</sup> C. Mondello,<sup>d</sup> R. Osellame<sup>bc</sup> and I. Cristiani<sup>a</sup>

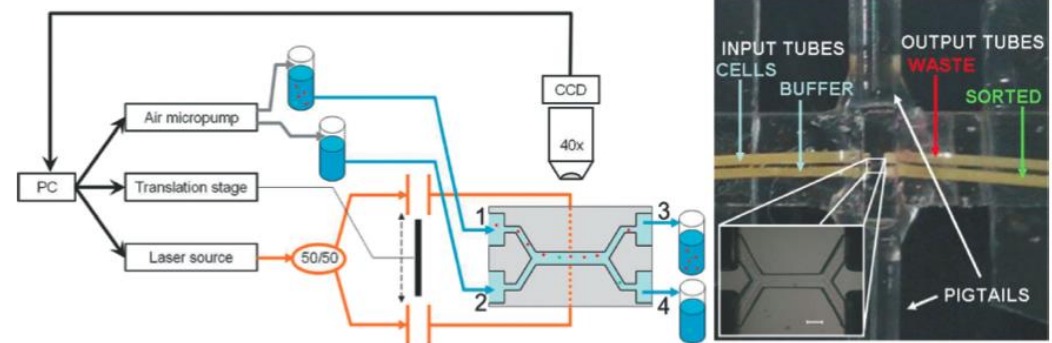


**Fig. 1** Schematic rendering of the double-Y-shaped microfluidic circuit irradiation patterns and phase-contrast microscopy images of swollen red blood cells in the microchannels fabricated with the two geometries: (a, c) transverse and (b, d) longitudinal writing geometries.

\*Lab Chip vol.15 1262 (2015)

Fibers to couple light in/out the device

SUITABLE SHAPE



**Fig. 2** Left: schematic representation of the experimental setup used to validate our **novel** optofluidic microchip. Cell stretching and sorting are based on the optical forces exerted by the same waveguides, which are connected with a fiber-to-fiber U-bench to realize the automatic sorting. Right: picture of the optofluidic chip connected to external capillaries and fiber pigtails. The locally enlarged image shows the inside structure of the microchannel network (scale bar, 100  $\mu\text{m}$ ).

# Beyond light coupling?

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Fibers as a tool for sensing and manipulation of particle

Exploitation of the **evanescent** field at the core-cladding interface

- ➡ Electric field can induce dielectrophoresis / electrophoresis
- ➡ Surface plasmon resonance (SPR)

Modification of the **tip shape** to improve/tailor the fiber output

- Tapering
- Rounding
- faceting

# Evanescent fields (EF)

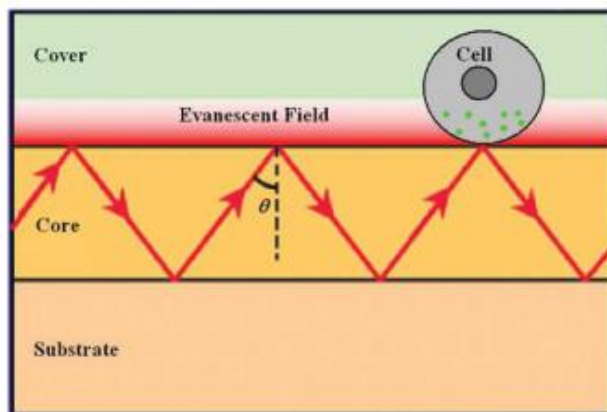
## Outline

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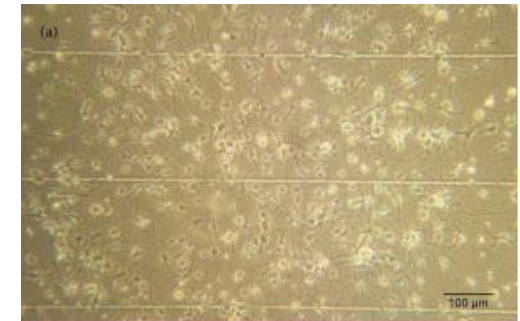
- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Evanescent-Field Excitation of Fluophores in Cultured Neural Networks by Integrated Polymer Waveguides

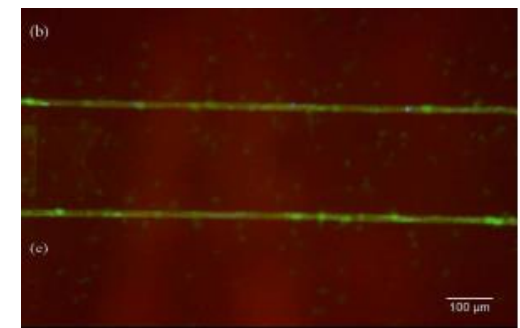
R. Adam Seger, *Student Member, IEEE*, Dominik G. Rabus, *Senior Member, IEEE*, Yasuhisa Ichihashi, Mathias Bruendel, and Michael S. Isaacson, *Member, IEEE*



## Neural cells

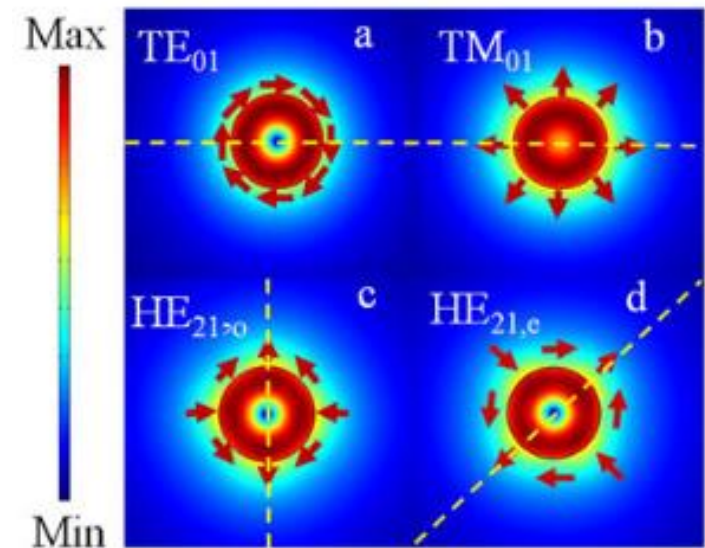
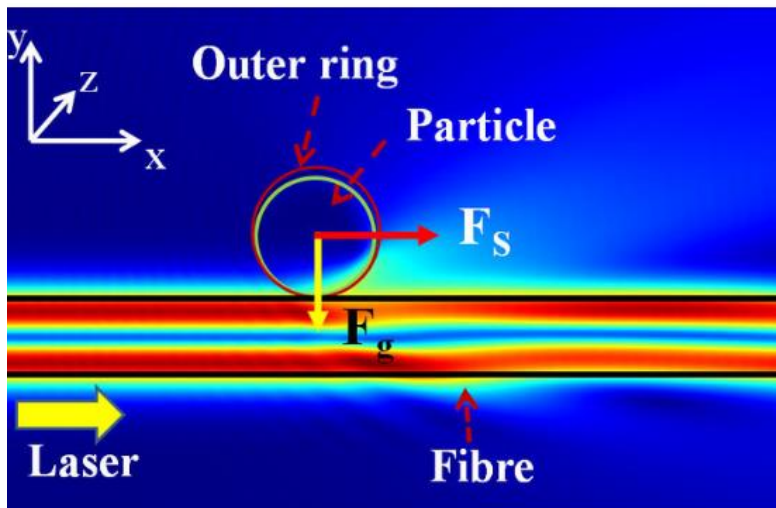


## Evanescent field excitation



- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Higher order microfibre modes for dielectric particle trapping and propulsion



\*Aili Maimaiti et al Scientific Reports 5, n. 9077 (2015)

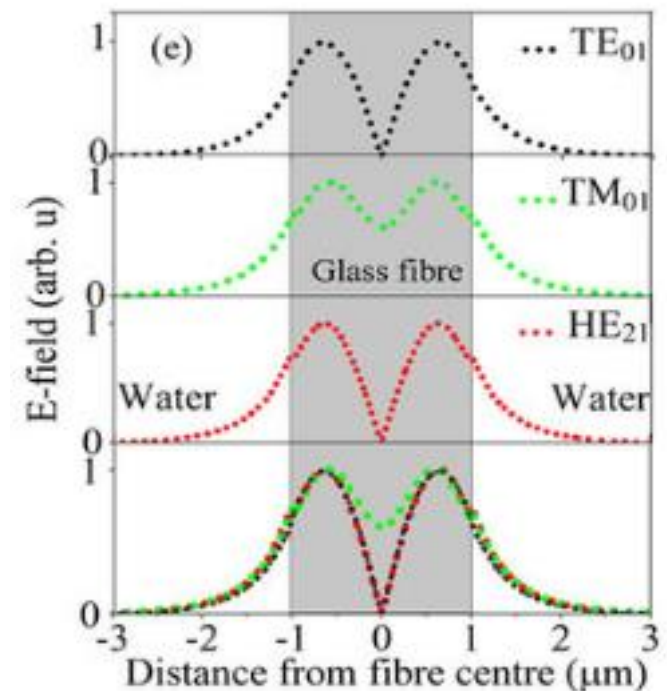
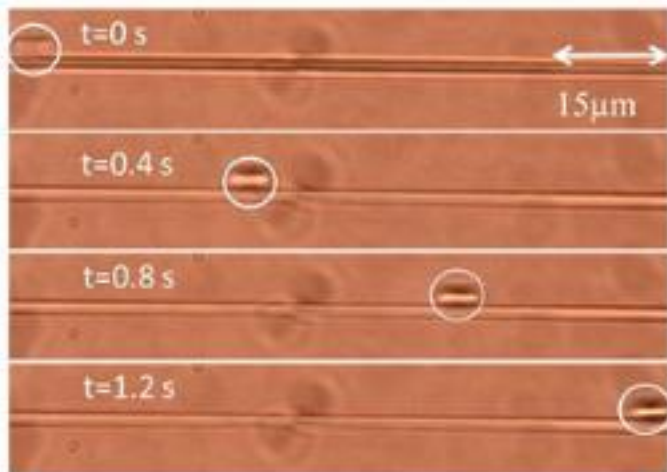
# Fiber for particle propulsion

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Particle Propulsion

Evanescent field drives charged and polarised particles

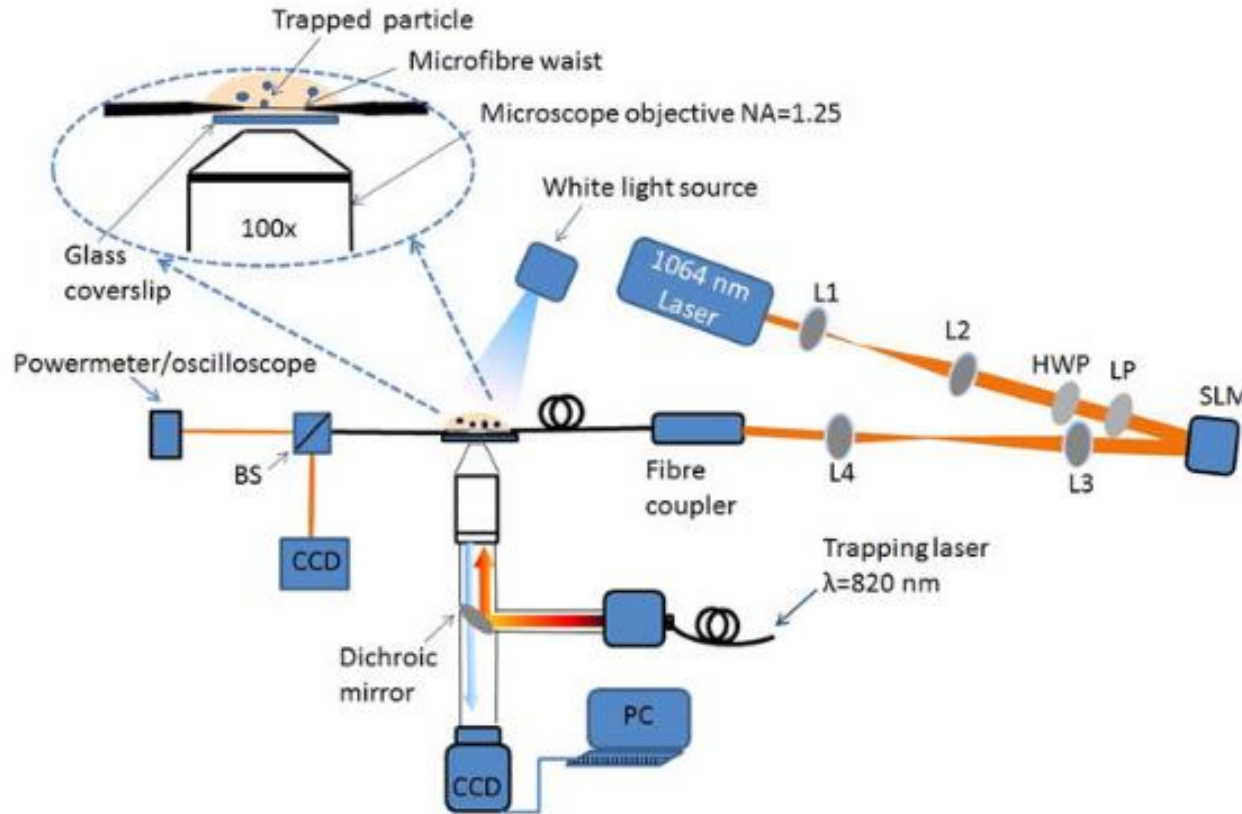
### Example 3 $\mu\text{m}$ polystyrene particles



\*Aili Maimaiti et al Scientific Reports 5, n. 9077 (2015)

# Fiber for particle propulsion

- Introduction
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**Need of integration:**

High power Fiber laser?  
Fiber detection?

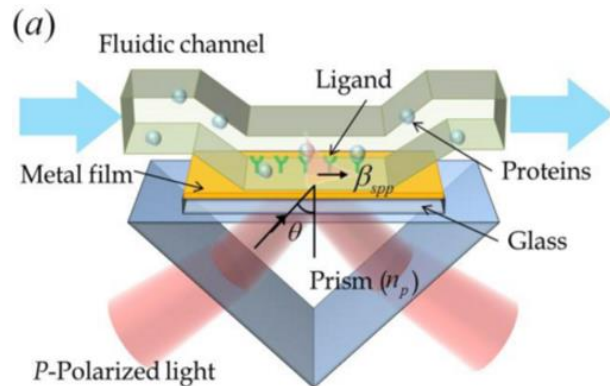
Table 1 | Hydrodynamic velocities of single 3  $\mu\text{m}$  polystyrene beads. Speed of particles is calculated using different correction factors for the LP<sub>01</sub> and LP<sub>11</sub> microfiber modes. The power at the fibre waist regions  $P_{\text{waist}}$  was fixed at 25 mW

Modes	Particle radius $a$ ( $\mu\text{m}$ )	Distance from particle centre to fibre surface, $h$ ( $\mu\text{m}$ )	Scattering force $F_s$ (pN)	Correction	Velocity ( $\mu\text{m/s}$ )
LP <sub>01</sub>	1.5	1.52	0.75	Stokes	29.0
		1.52	0.75	Krishnan	8.9
		1.56	0.67	Faxen	9.6
				Experiment	8.5
LP <sub>11</sub>	1.5	1.52	4.15	Stokes	160.5
		1.52	4.15	Krishnan	49.2
		1.56	3.90	Faxen	56.0
				Experiment	72.5

# EF: tool for sensing

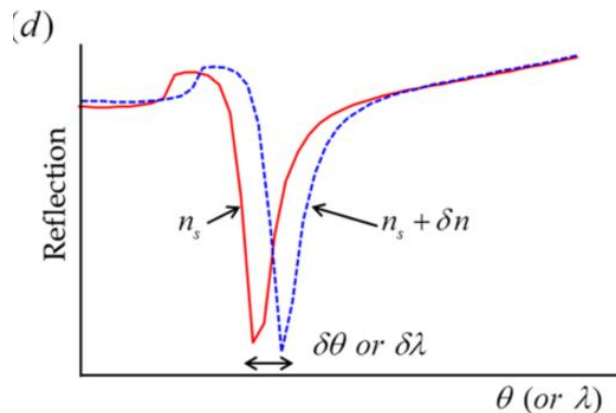
- Introduction
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## Surface Plasmon resonance as a sensing method



Kretschmann configuration based coupling

- **Angle scanning** ( $5 \cdot 10^{-7}$  RIU – 500 deg/RIU<sup>-1</sup>)
- Wavelength scanning (30000 nm/RIU<sup>-1</sup>)
- Often **gratings are implemented**



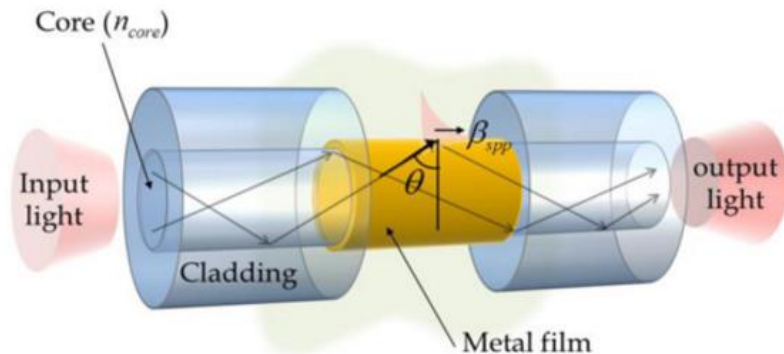
The measure of the **reflectance** carries infos on:

- Refractive index of the fluid
- Concentration of components
- In some cases particle identification

**LOD close to pM**

# EF: fibers for sensing

- Introduction
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**Fiber core deposited with metal**  
mimes Kretschmann configuration  
based coupling

$$S = 4000 \text{ nm/RIU}^{-1}$$

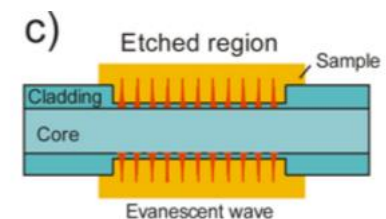
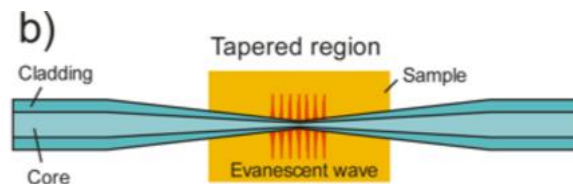
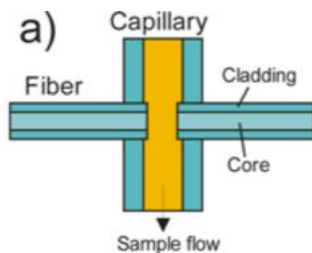
## Problems in angle scanning

Wide range of configurations

In grating coupling  $S$  close to 200-600 deg/RIU<sup>-1</sup>  
at 950nm ( $\lambda=350\text{nm}$ )

$$\frac{2\pi}{\lambda} n_p \sin \theta = \beta_{ev} = \text{Re}(\beta_{spp}),$$

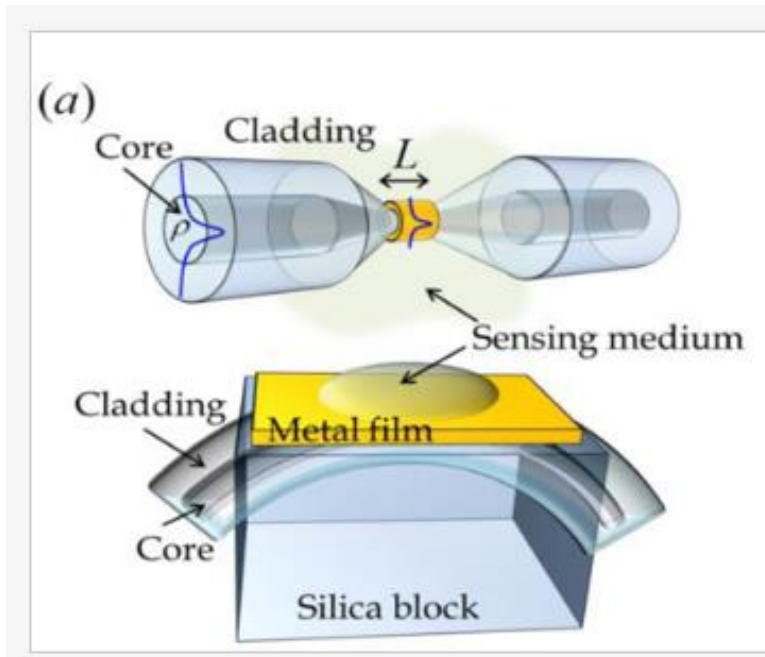
$$\beta_{spp0} + \Delta\beta, \beta_{spp0} = \frac{\omega}{c} \sqrt{\frac{\epsilon_m n_s^2}{\epsilon_m + n_s^2}}$$



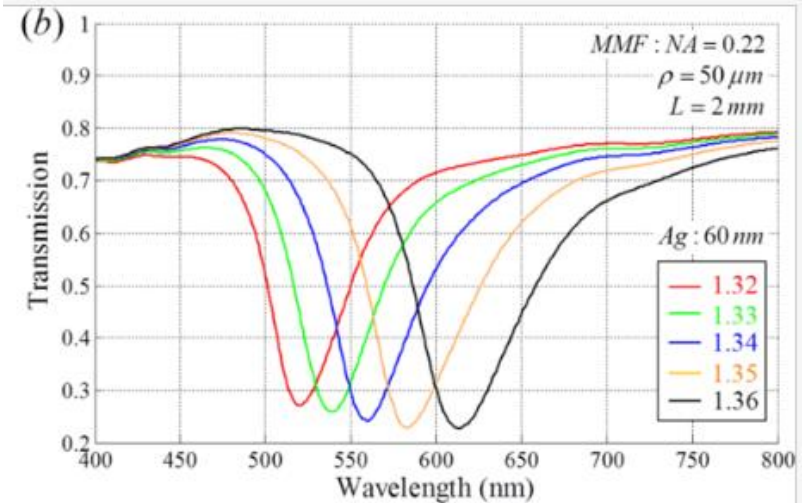
# EF: fibers for sensing

- Introduction
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## Micro- and Nano-Structured Surface Plasmon Resonance Sensors



## Wavelength scanning



Sookyoung Roh et al.  
Sensors 2011, 11(2), 1565-1588

## Refractive index resolution

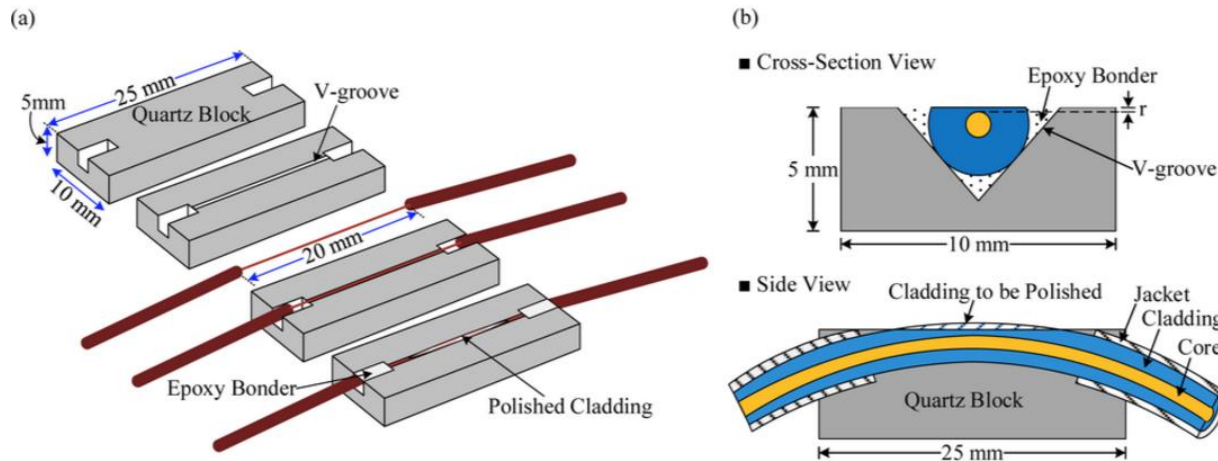
$9.9 \times 10^{-4}$  at  $n=1.410$

$4.2 \times 10^{-4}$  at  $n=1.440$

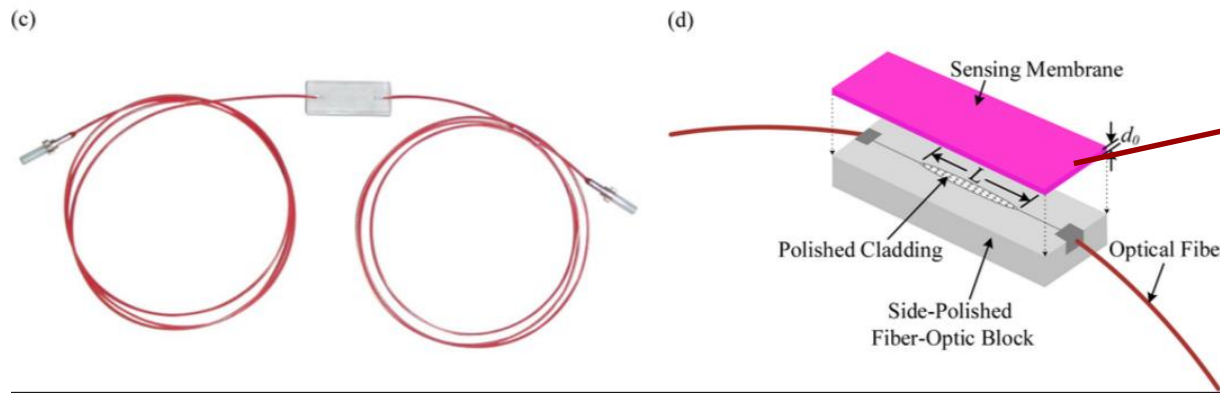
# Fibers for sensing VOC gas

- Introduction
- Fibers for coupling
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## A High Sensitivity and Wide Dynamic Range Fiber-Optic Sensor for Low-Concentration VOC Gas Detection



Sensors 2014  
14(12) p.23321-23336



$$d_p = \frac{\lambda}{2\pi n_1} \left\{ \sin^2 \theta - \left( \frac{n_2}{n_1} \right)^2 \right\}^{-0.5}$$

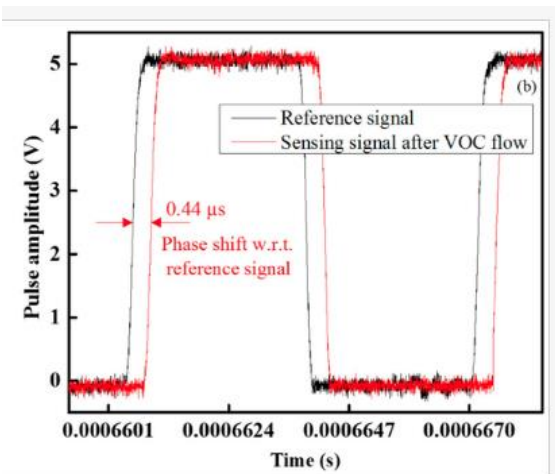
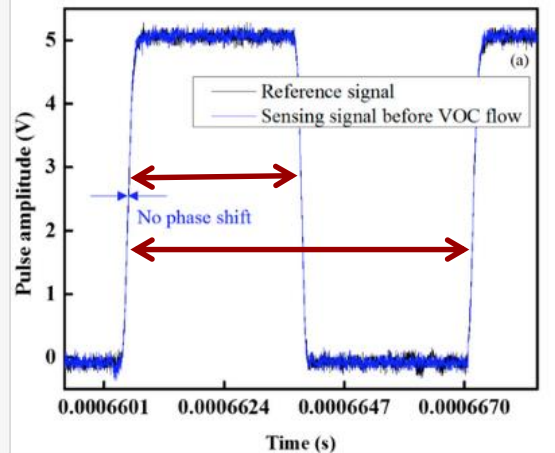
$$P_L = P_0 \exp(-\gamma CL)$$

# Fibers for sensing VOC gas

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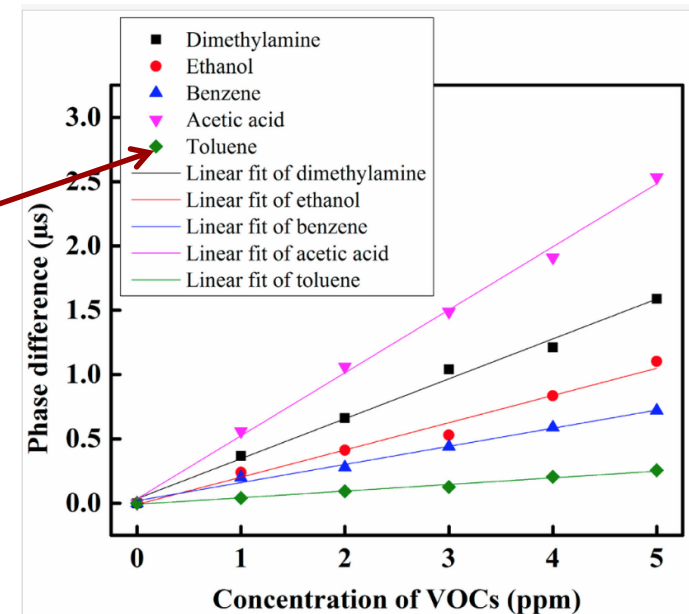
## Pulsed Width modulation technique

Volatile organic compounds (VOCs)  
are common air pollutants: ppm  
detection sensitivity



$$d_p = \frac{\lambda}{2\pi n_1} \left\{ \sin^2 \theta - \left( \frac{n_2}{n_1} \right)^2 \right\}^{-0.5}$$

$$\phi = \frac{2\pi n}{\lambda \gamma C} \left( \frac{T}{T_H} \right)^2$$



# Focus: opto-fluidics for sensing

## Outline 21

- Introduction
- Fibers for coupling
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- Optical sensing
- Conclusions

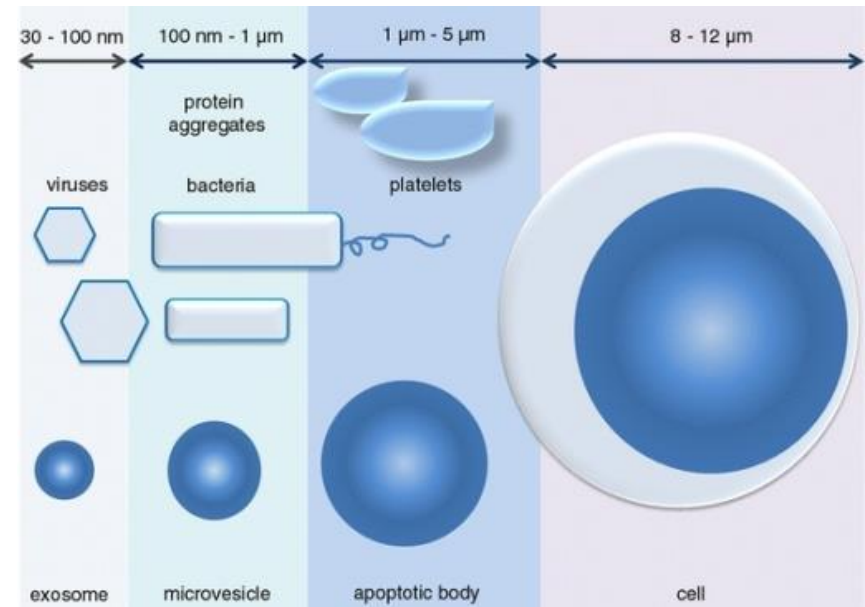
## ISSUE: Target counting and identification

### Working principle

- Target Quantification
- Size measurement
- Sorting

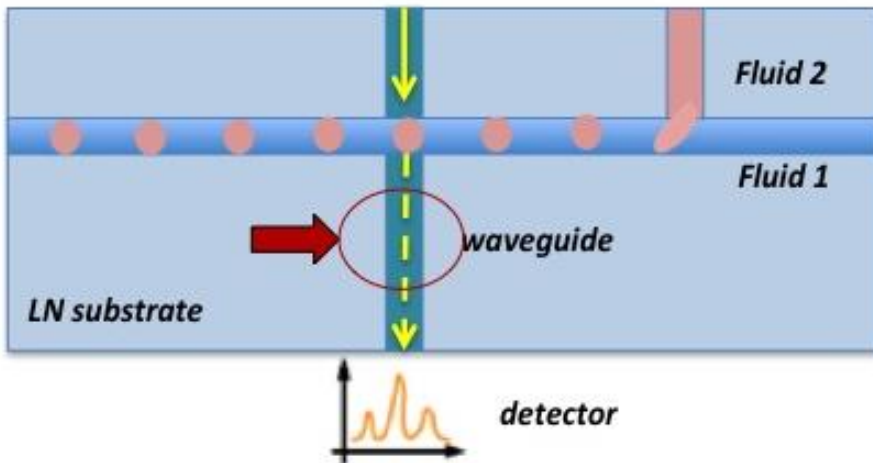
### Key parameters

- High sensitivity
- Size domain  $< 12 \mu\text{m}$
- Complementary to other techniques



# Integrated Opto-microfluidic prototype

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions



- Droplets generator: controlled dilution
- Droplets counter and analysis

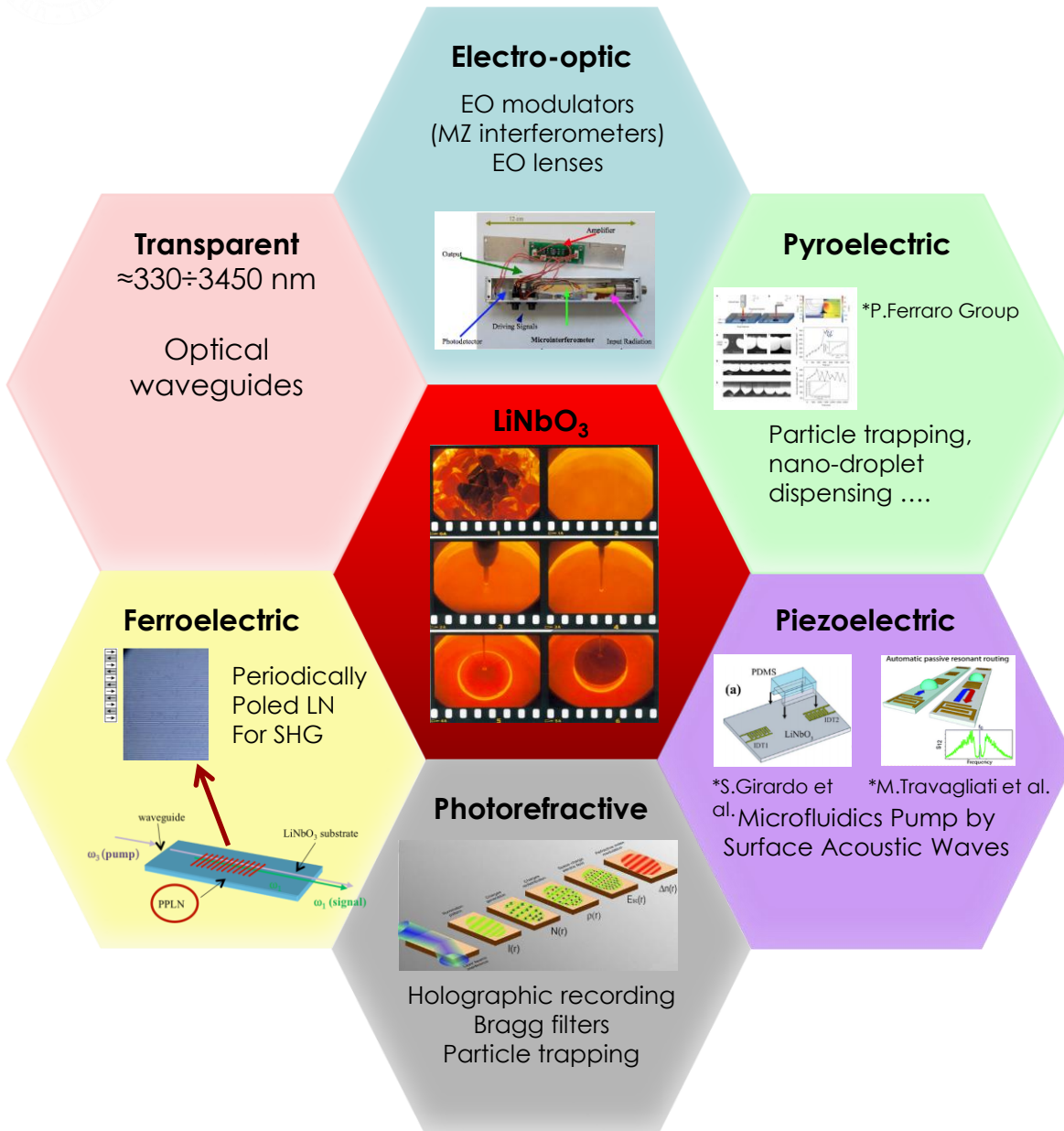
## Applications to:

- Targets counting
- Target dimension
- Target identification

***“Integrated opto-microfluidics platforms in lithium niobate crystals for sensing applications”***

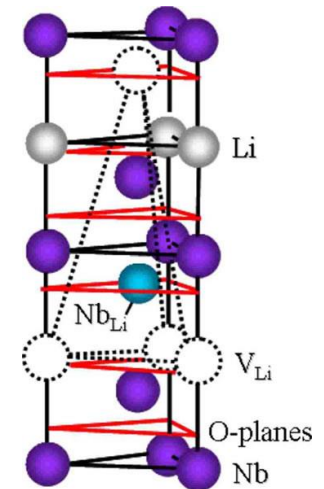
G. Bettella ; G. Pozza ; A. Zaltron ; C. Sada ; M. Chauvet et al. Submitted to LabOnChip 2015

# Why not using $\text{LiNbO}_3$ ?



## Outline 23

- Introduction
- Fibers for coupling
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- Conclusions



Local and bulk doping



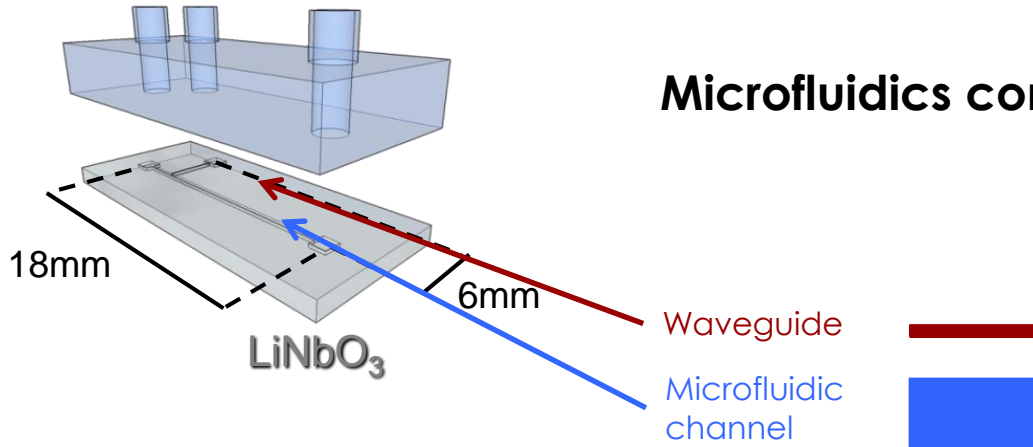
# Opto-microfluidic prototype

Outline

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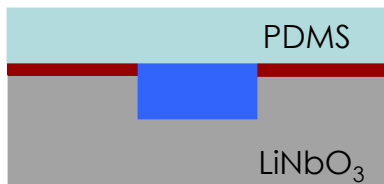
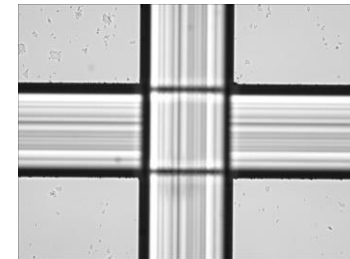
- Introduction
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## Sealing

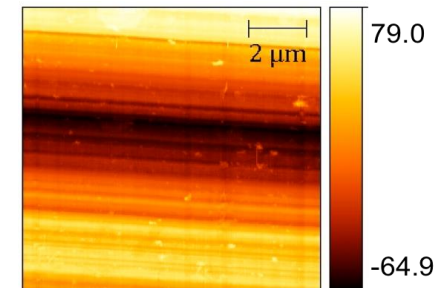
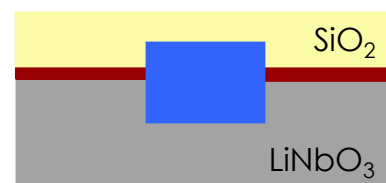
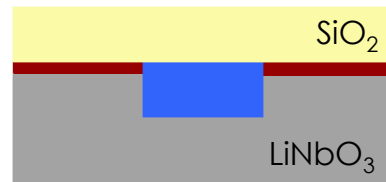
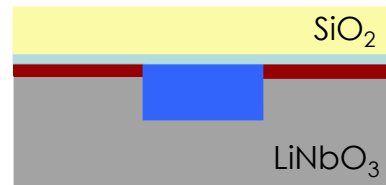
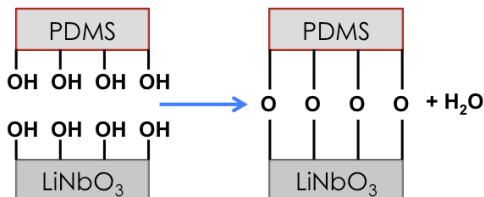


## Microfluidics configurations

DICING @ FEMTO-ST prof. M. Chauvet



Sealing with a PDMS layer: O<sub>2</sub>-plasma



## Average roughness

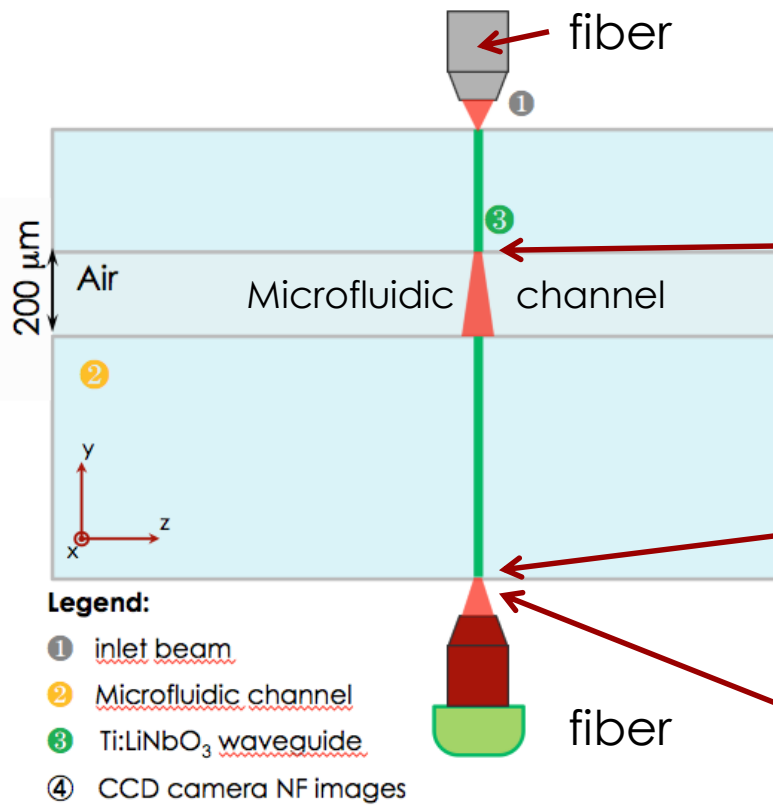
Bottom  $R_a = (19 \pm 3) \text{ nm}$

Lateral walls  $R_a = (7 \pm 1) \text{ nm}$

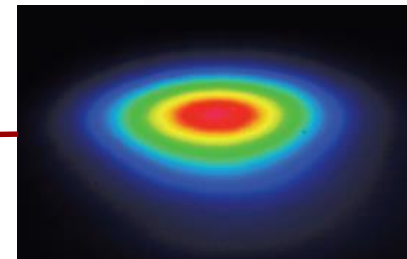
# Optical Coupling

- Introduction
- Fibers for coupling
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## Integrated waveguide: Ti-indiffusion in LN



### Near field



Input WG's exit

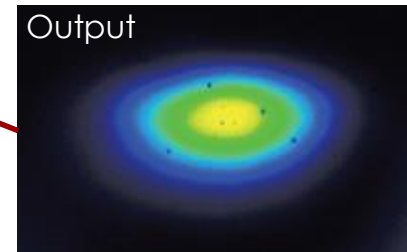
$$I/I_0 = \text{REF } 100\%$$



Output

Air in the channel

$$I/I_0 = 38\%$$



Output

Hexadecane in the channel

$$I/I_0 = 76\%$$

$$I_0 = 2.2 \times 10^5 \text{ W/m}^2$$

$$DT = \frac{T_L}{T_0} = \frac{2n_{LN}n_L}{n_{LN}^2 + n_L^2} \frac{2W_{GB}W_{gap}}{W_{GB}^2 + W_{gap}^2} / T_0$$

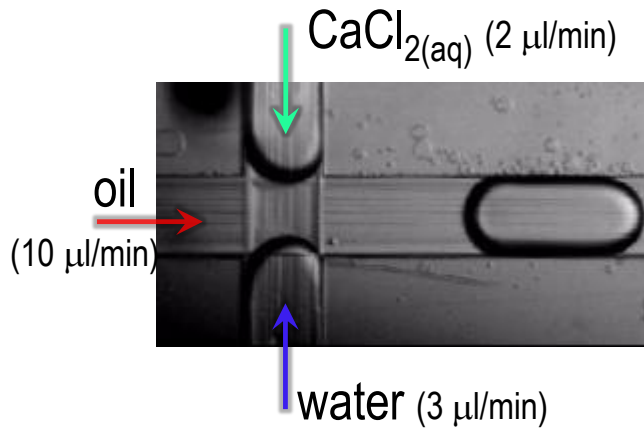
# Counting

## Outline

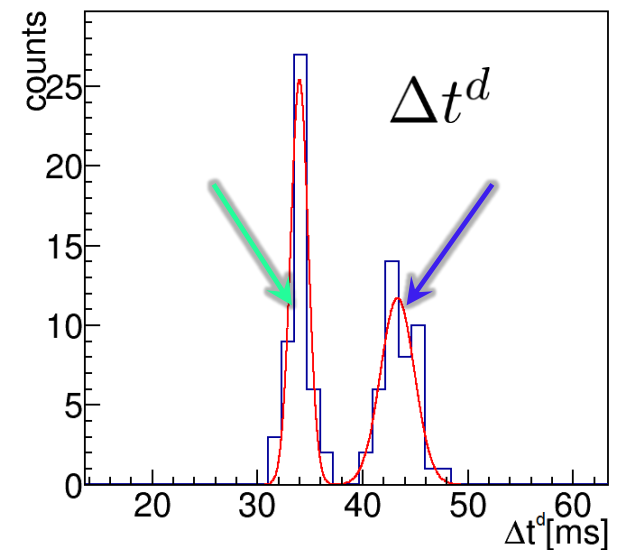
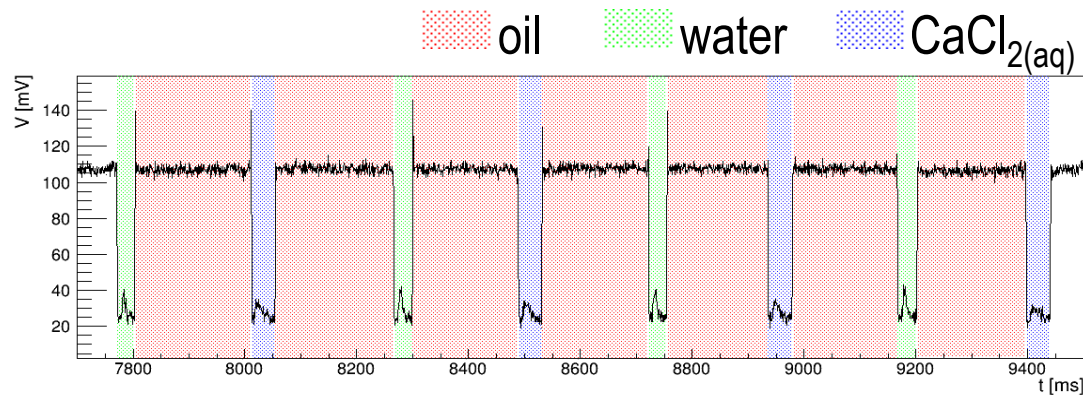
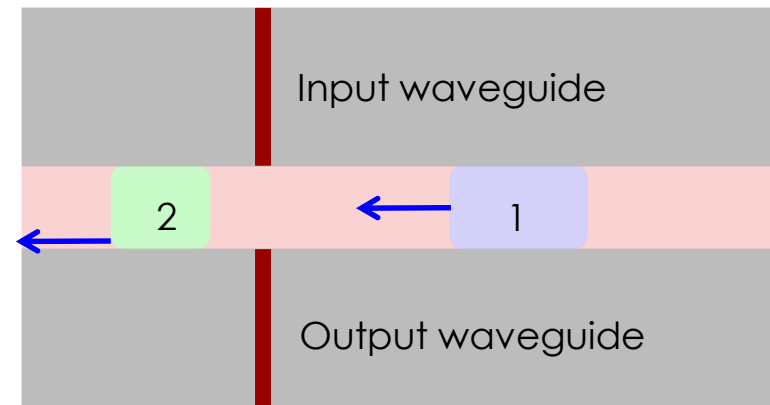
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- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Target identification



1= reference  
2=target to detect

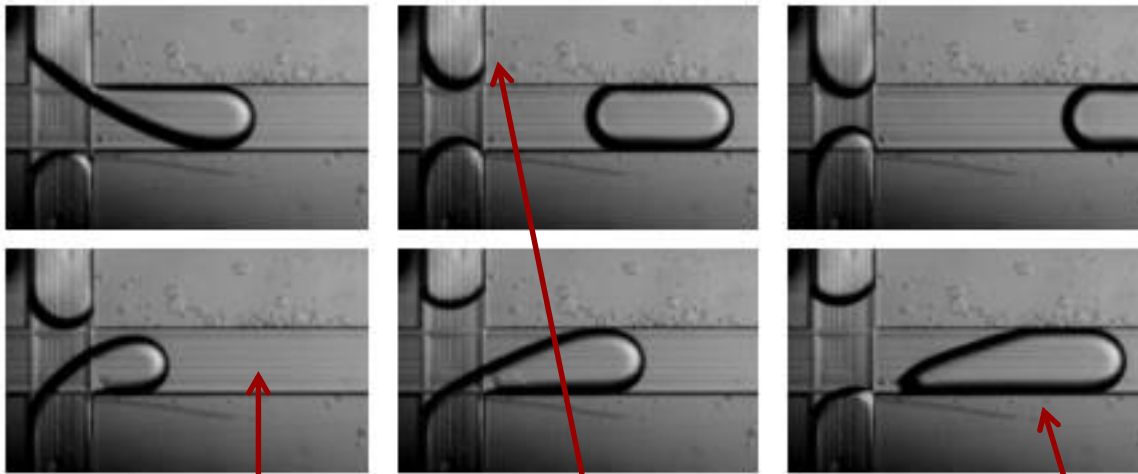


# Microfluidics in $\text{LiNbO}_3$

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Cross-junction

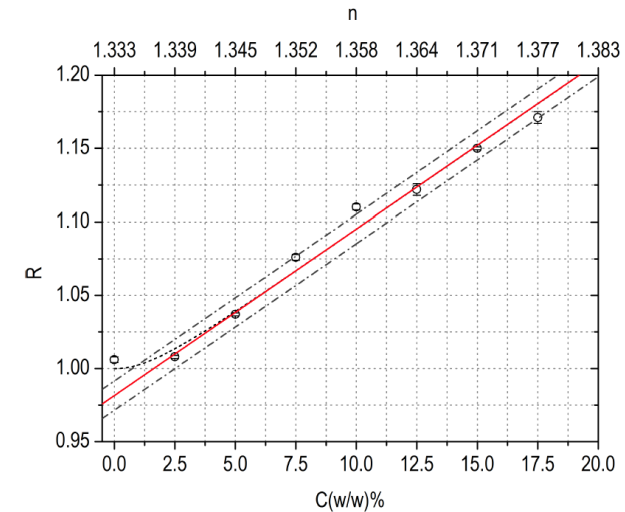
### Alternating Droplets of different composition



$Q_C = 10 \mu\text{L}/\text{min}$

$Q_D^{(w)} = 3 \mu\text{L}/\text{min}$

$Q_D^{(w+\text{CaCl}_2 \text{ 5\%})} = 2 \mu\text{L}/\text{min}$

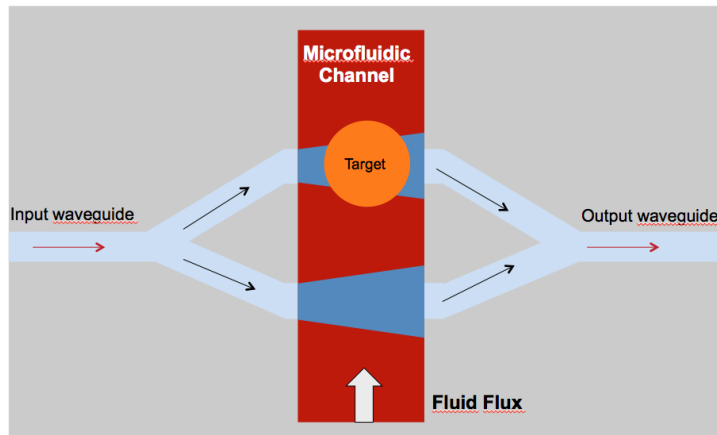
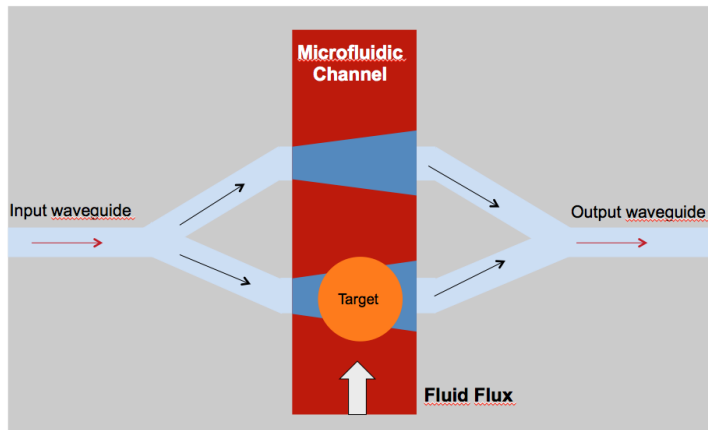


# Sensing application

## Outline 28

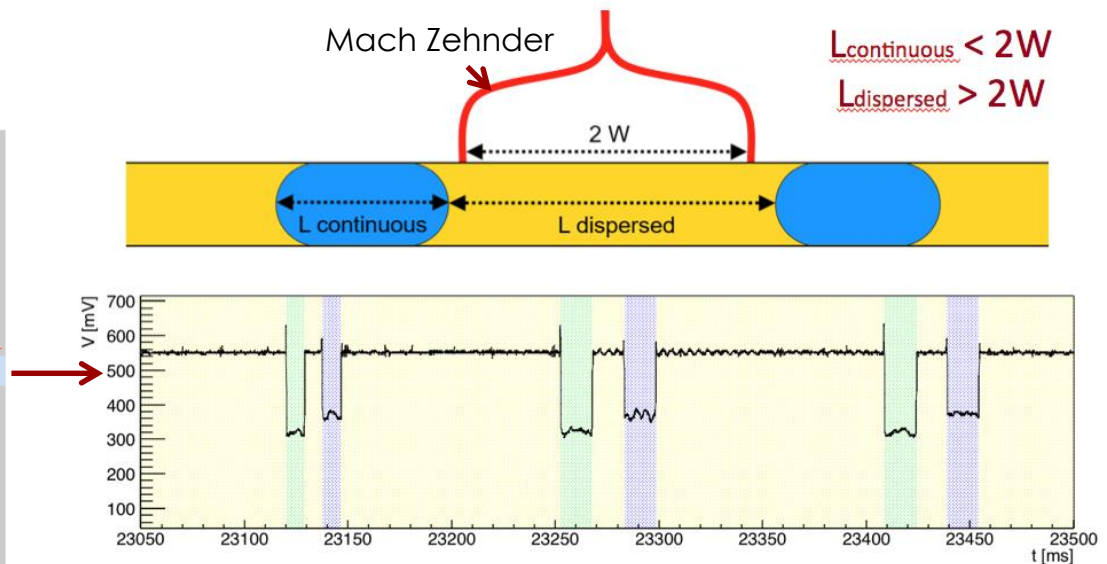
- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## Optofluidic platform: target detection



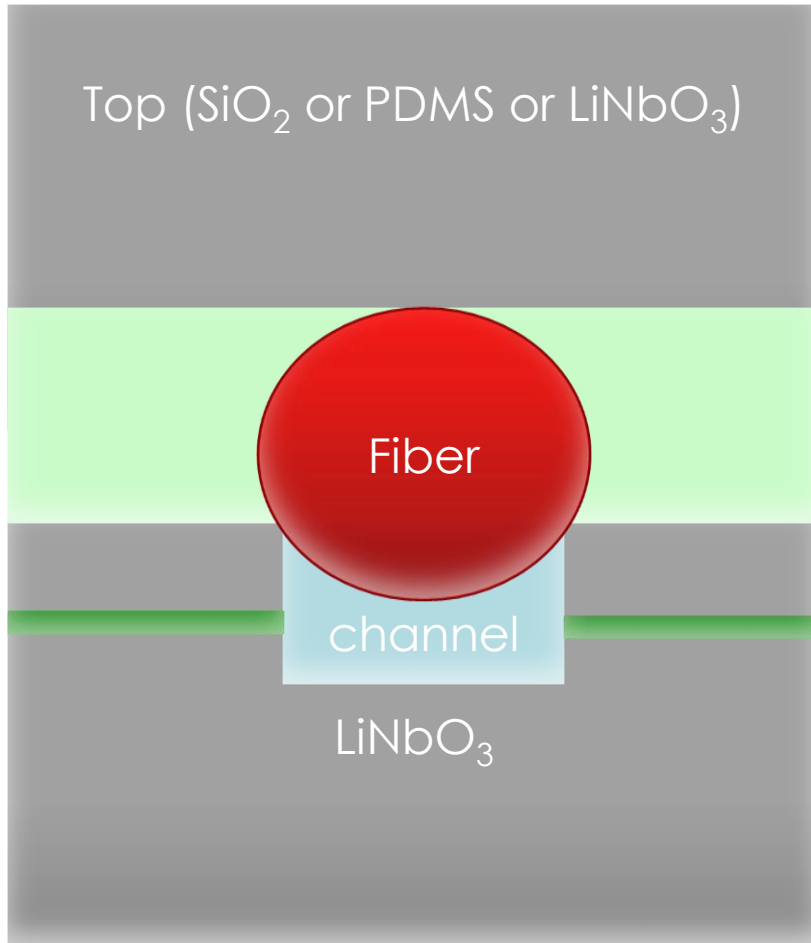
Transmitted intensity modulation

Infos on  $n(t)$



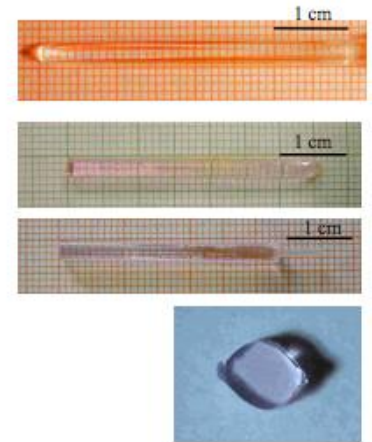
# Sensing application

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions



## Evanescent field to particle driving

$\text{LiNbO}_3$  fibers growth

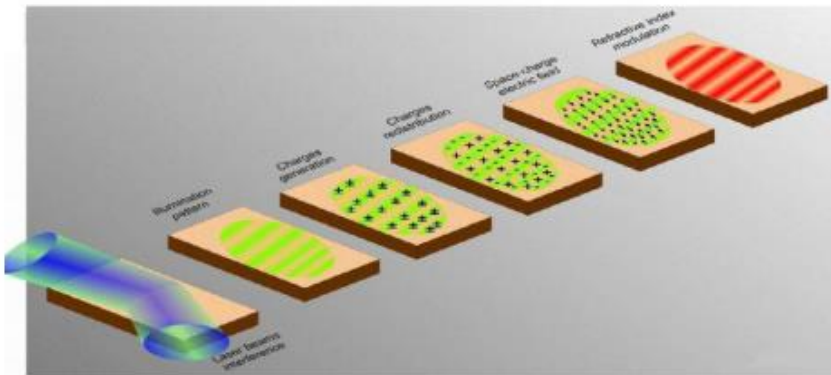


Journal of Applied Physics 104, 103114 (2008);  
A. Arcangeli, M. Tonelli, A. Toncelli, C. Sada et al.

# Trapping of particles

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

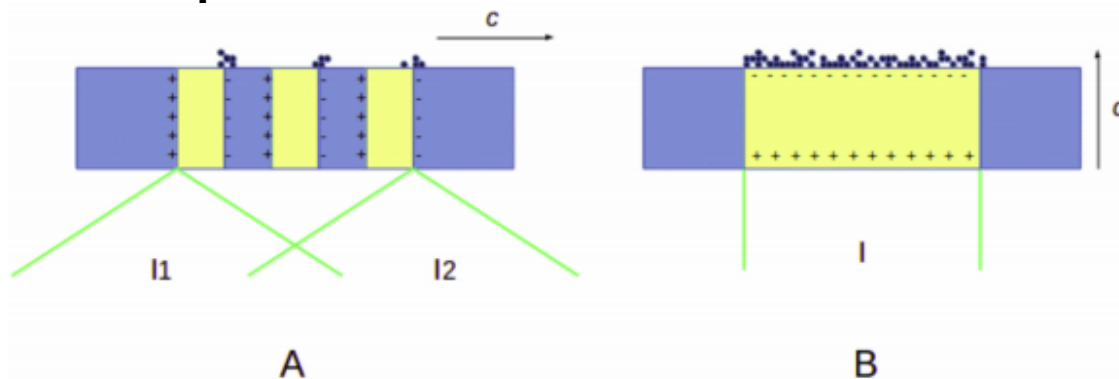
$$I(\mathbf{r}) \rightarrow N_{e,h}(\mathbf{r}) \rightarrow \mathbf{j}(\mathbf{r}) \rightarrow \rho(\mathbf{r}) \rightarrow \mathbf{E}_{sc}(\mathbf{r}) \rightarrow \Delta n(\mathbf{r})$$



## LiNbO<sub>3</sub> photorefractivity

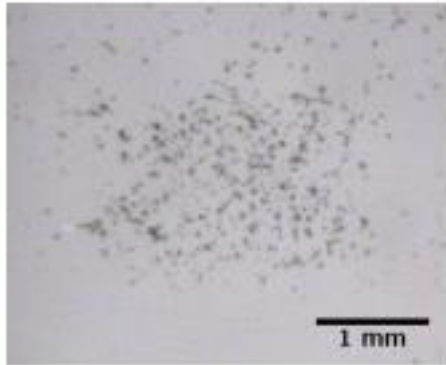
Pattern of light modulation induces a pattern of evanescent field

## Electrophoresis

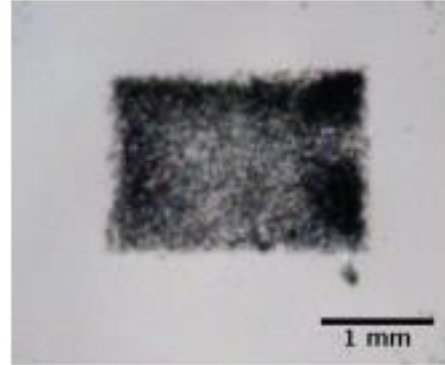


# Trapping of particles in fluids

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions



(a) Camp. 117.1,  $t_{ill}=40\text{min}$



(b) Camp. 210.6,  $t_{ill}=6\text{min}$

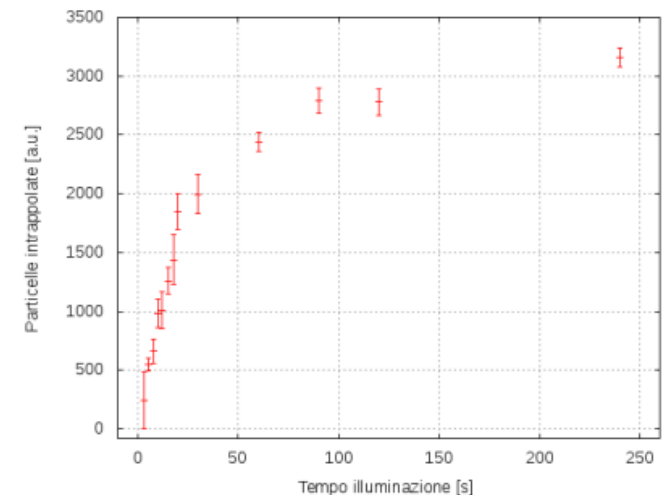
Particle trapping

Electrophoresis

Integrated Round Shape Fibers To illuminate



Local definition of the Trapping region



# What to do next?

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

**Fibers to be improved** focusing on the following aspects:

- Wide range of refractive index (work on new materials);
- Tailored cladding characteristics along the fiber length;
- Tailored fiber tip (new procedures for tapering, rounding,...)

## **AIMS**

- Reduce optical losses (propagation and coupling losses)
- Improve material compatibility to simplify the integration process
- Simplify fiber processing

# Conclusions

- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions

## In summary:

- Thank you for your attention!
- Thank you for this opportunity to meet this community!
- I forgot.. As Editor, Welcoming you to submit papers for publication

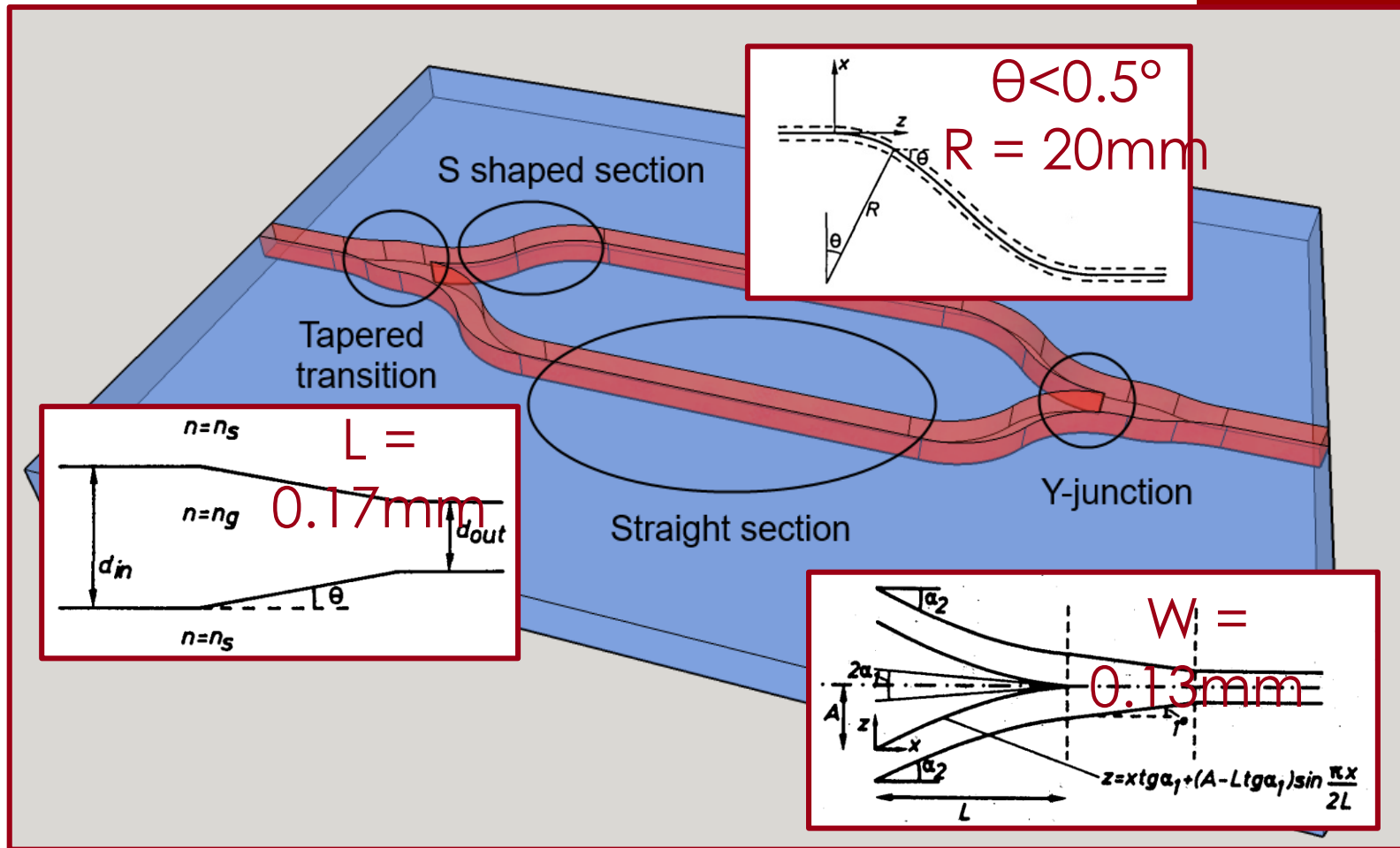
**Optofluidics, microfluidics and nanofluidics Journal**

De Gruyter Open Ed Open Access

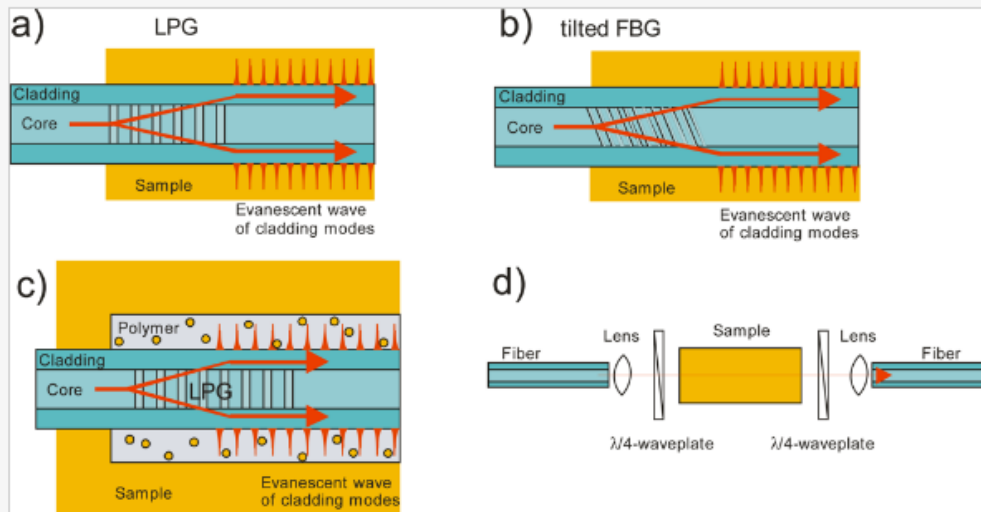


# Optical sensing

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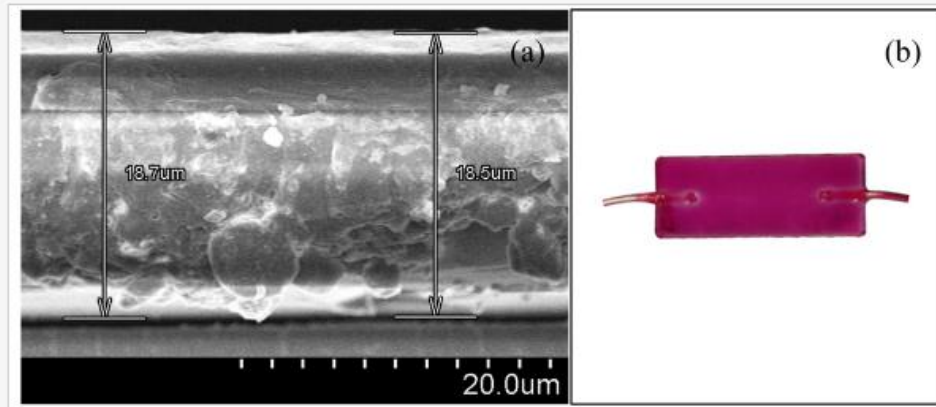


**Figure 6.** Different sensor elements for refractive index change measurements. Refractive index sensitive elements like (a) long-period gratings (LPG) or (b) tilted fiber Bragg gratings (FBGs) are often used. (c) By coating an LPG with a polymer the sensor element can be made more sensitive and more selective. (d) Circular birefringent samples have different refractive index for left or right circularly polarized light. In this sensor element the linear polarized light from the fiber is converted to circular polarized light by a quarter waveplate before being transmitted through the sample. A second quarter waveplate restores the linear polarization before coupling the light back into the cavity fiber.



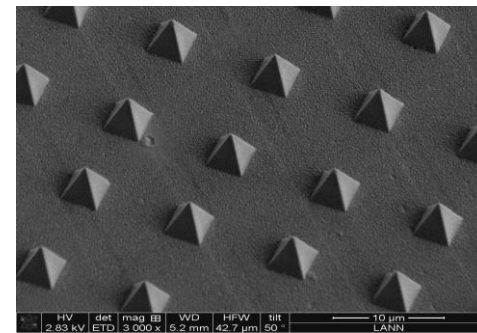
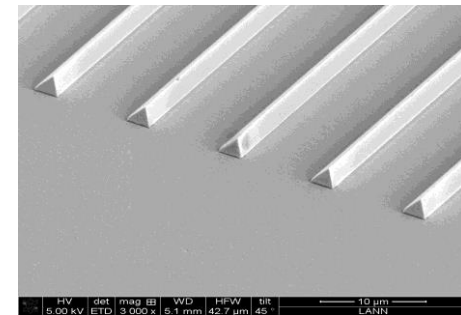
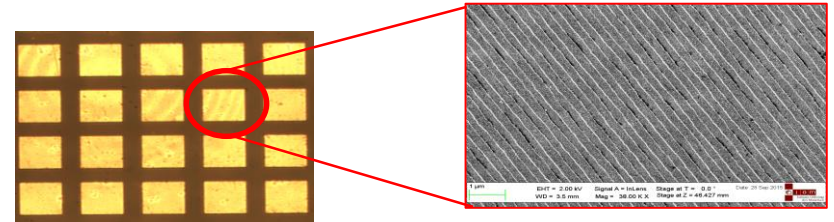
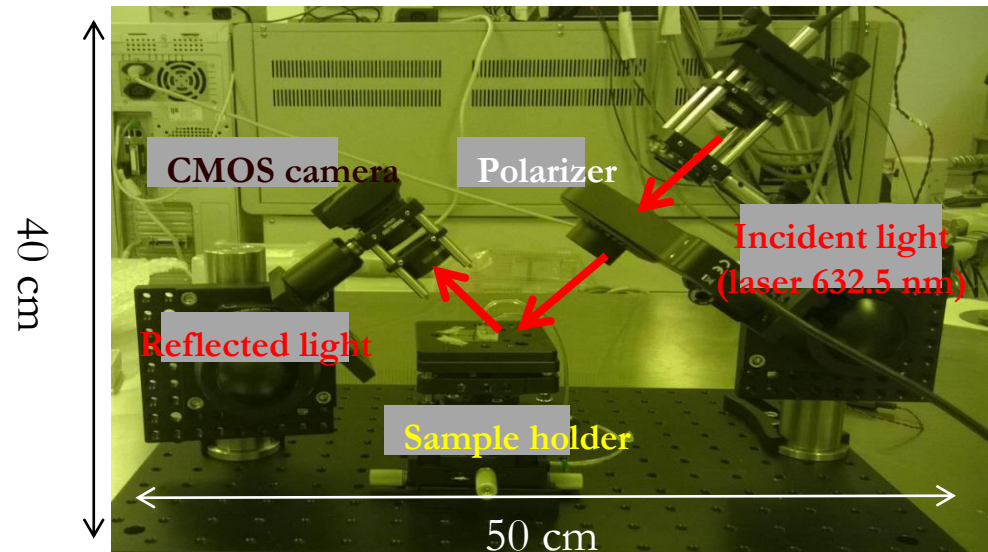
## solvatochromism

**Figure 2.** VOC sensitive sensing membrane: (a) SEM image of the thickness of the VOC sensing membrane and (b) photograph of the fabricated side-polished fiber-optic device after incorporation of the Nile Red-containing sensing membrane.



Nile Red dye, N,N-dimethylacetamide (DMAC, 99%), and PVP were used to prepare the sensing solution

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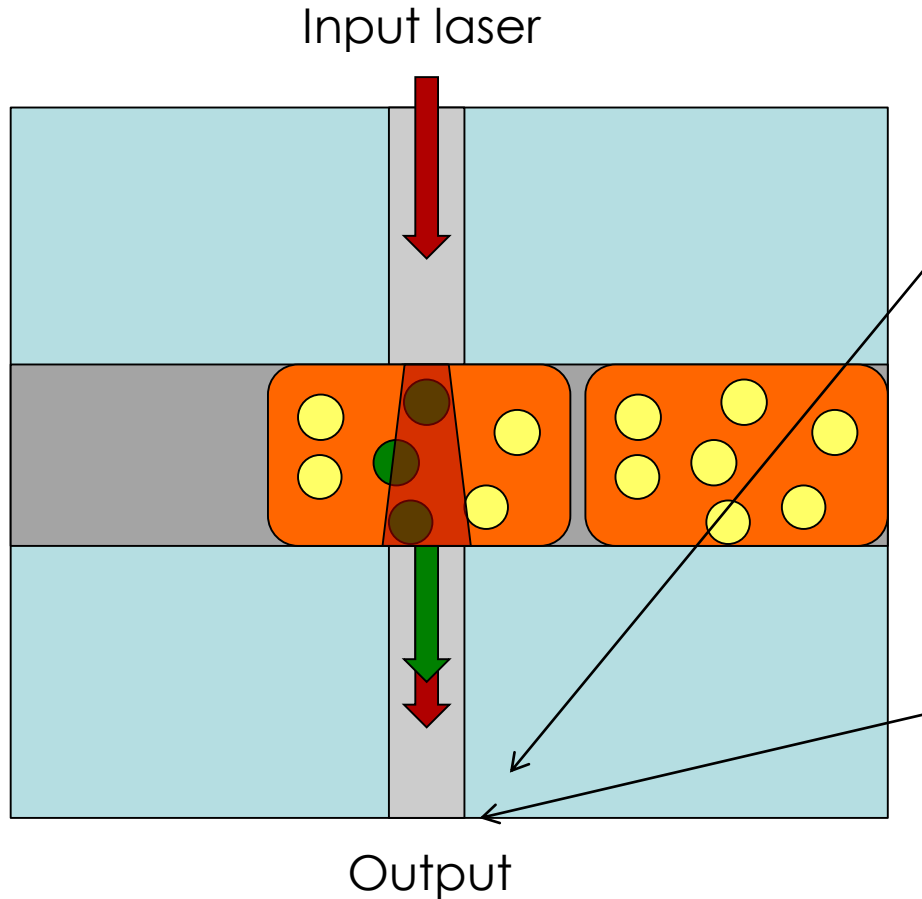
# Spectral analysis

## First Tests on fluorescence detection

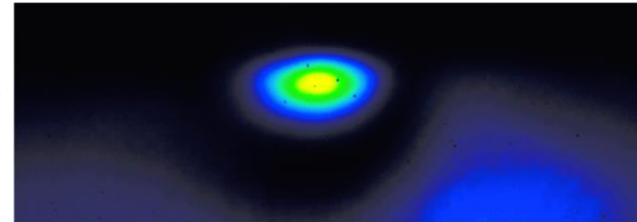
### Outline

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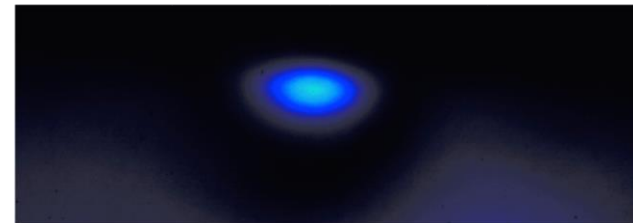
- Introduction
- Fibers for coupling
- Fibers beyond coupling
- Optical sensing
- Conclusions



Output: Transmitted light



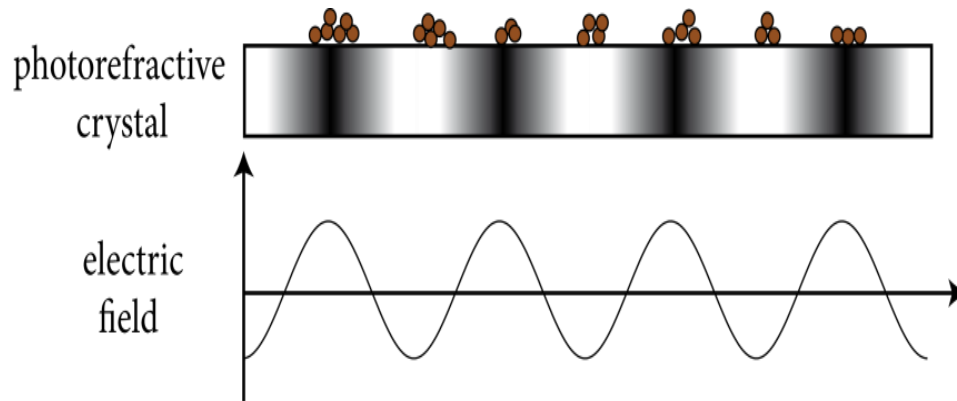
Output: Fluorescence light



Input He –Ne laser intensity coupled into the waveguide:  $2.2 \times 10^5 \text{ W/m}^2$   
Test made with: Rhodamine 6G and Cy5 in water

**Collaboration with Prof. Denz's research group @ Münster (Michael Esseling)**

Non homogeneous illumination  $\rightarrow$  Space charge field modulation  $\rightarrow$  Dielectrophoresis force



LiNbO<sub>3</sub> crystals possess unique properties  
for particles trapping

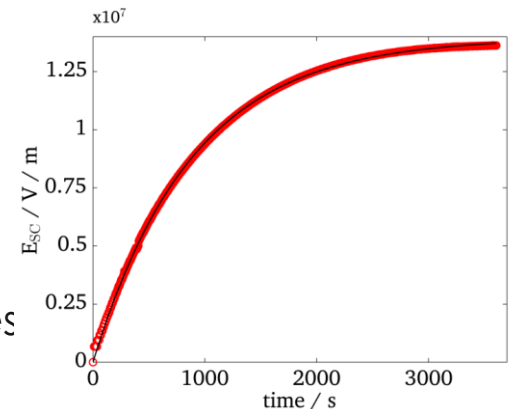
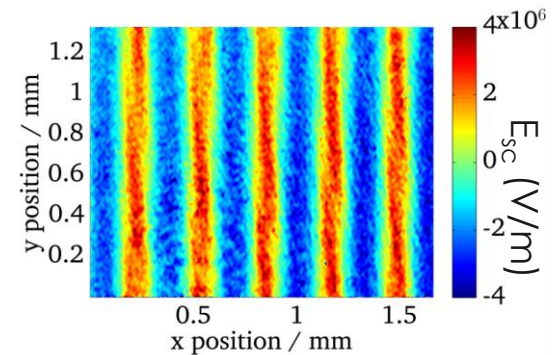
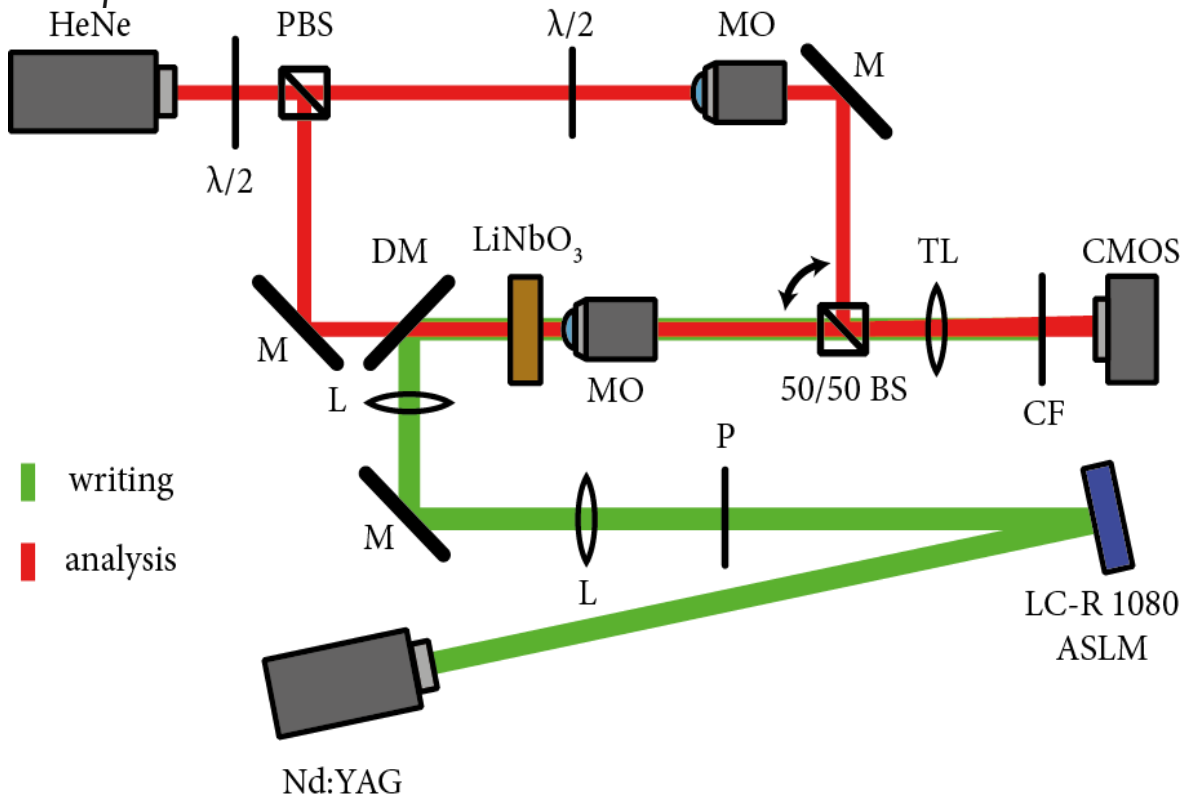
$$F_{DEP} = 2pr^3 e_m \operatorname{Re} \left[ \frac{e_p(\nu) - e_m(\nu)}{e_p(\nu) + 2e_m(\nu)} \right] \nabla E^2$$

- high internal fields up to kV/mm
- low light intensity  
(good for living cell)
- easy reconfiguration  
(uniform white light)

## Digital holographic microscopy (DHM)

provides 2D information about :

- the magnitude of  $E_{sc}$
- temporal evolution of  $E_{sc}$
- internal structure of  $E_{sc}$



- Fe-doped Lithium Niobate (enhancement of PR properties)
- Reduced Fe:LiNbO<sub>3</sub> samples (to speed up PR response)

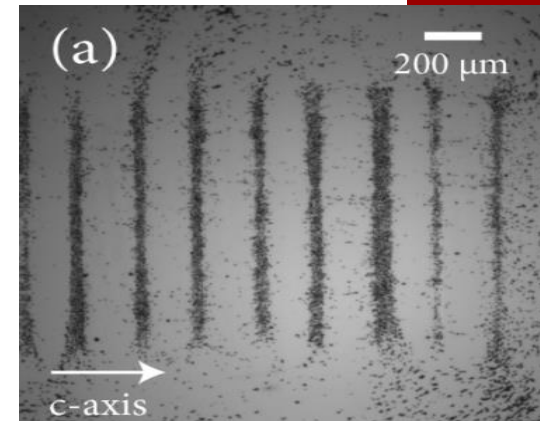
*y-cut Fe:LN samples* → light patterns with k-vector along c-axis of  $\text{LiNbO}_3$



anisotropy of the bulk photovoltaic effect



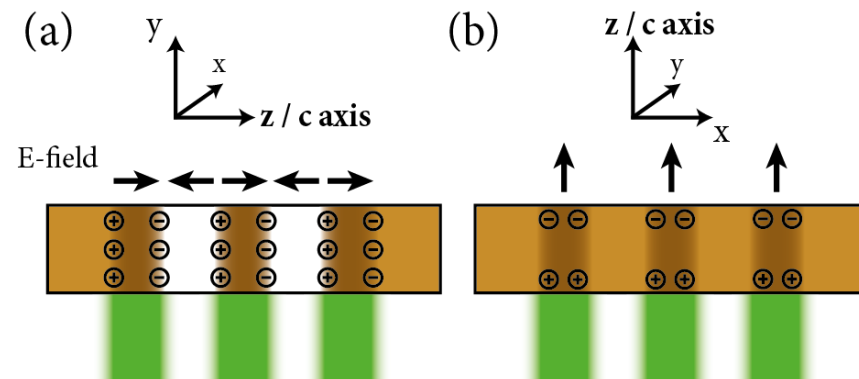
no arbitrary 2D patterns



*z-cut Fe:LN samples* → charges also accumulate on  $\pm z$  faces



- electrophoretic attraction to accumulated charges on  $\pm z$  face
- negligible dielectrophoresis perpendicular to c-axis



## z-cut Fe:LN samples

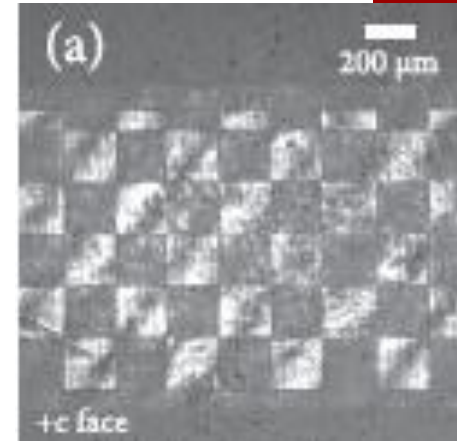
- ✓ Arbitrary 2D patterns (particles trapping)



→ next step: integration of particle sorting device

- ✓ charge sensor

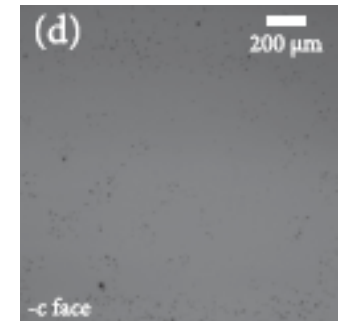
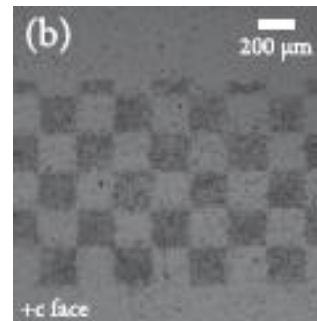
Light pattern on  $\pm c$  faces



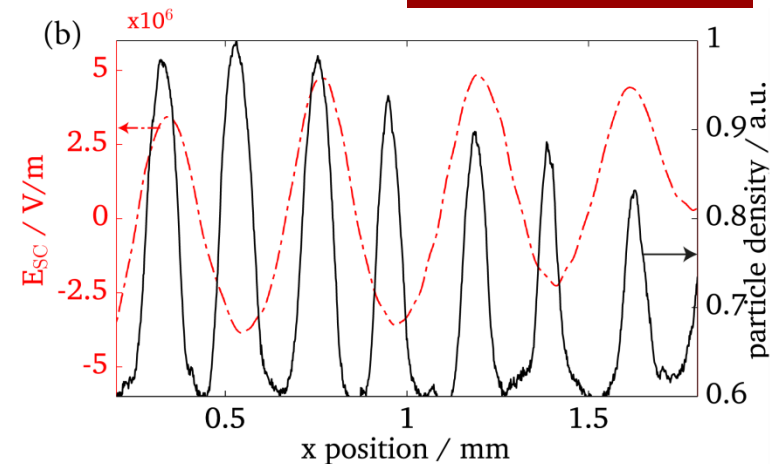
Positive particles are:

*trapped*

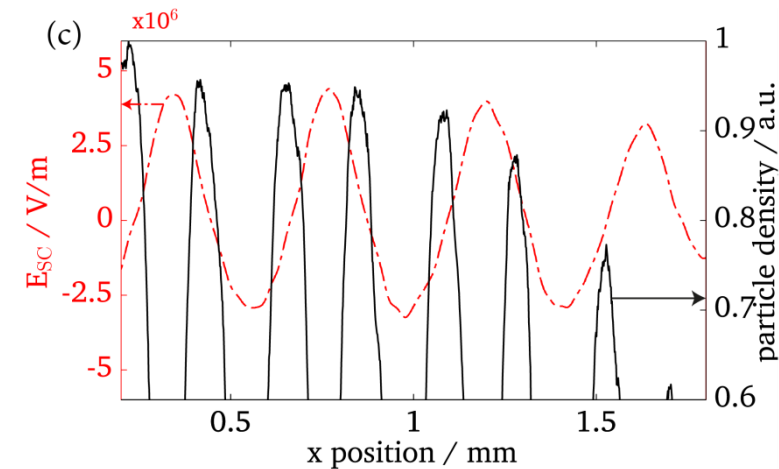
*repulsed*



- glassy carbon (GC) spheres (2 -12  $\mu\text{m}$ ) in tetradecane: **uncharged, manipulated by DEP**
- GC in Novec 7500/7300: **charged, influenced by electrophoresis (EP)**
- charge state determined by phase shift between electric field and trapped particles (y-cut geometry)
- DEP attracts GC towards high field intensity
- EP attracts to regions of high charge density



tetradecane



NOVEC 7500

-> in NOVEC 7500, both charge states exist (!)

# Optical sensing

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# Optical sensing

# Optical sensing

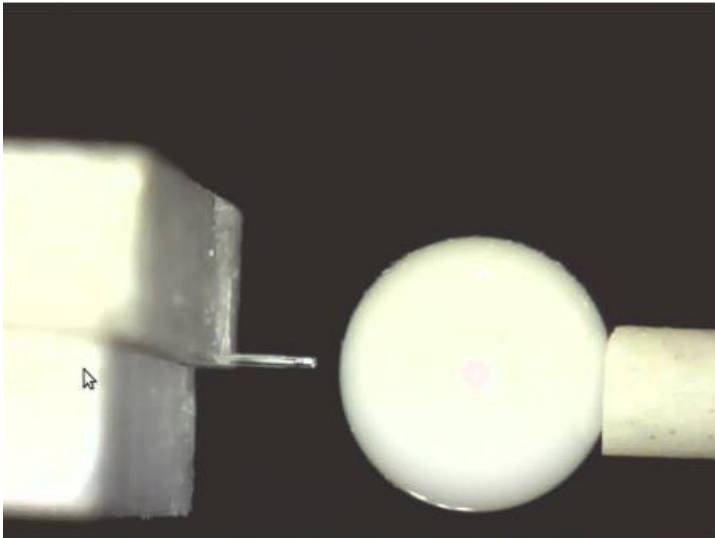
# Optical sensing

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Growth and characterization of Er-doped single crystal lithium niobate fibers

# Optical sensing

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fiber to a sphere at high temperature (1200 – 1600°C)

Produce really concave spherical tips with a larger useful diameter (and so more stable cavities)

# Optical sensing

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Life time 2.3 ms

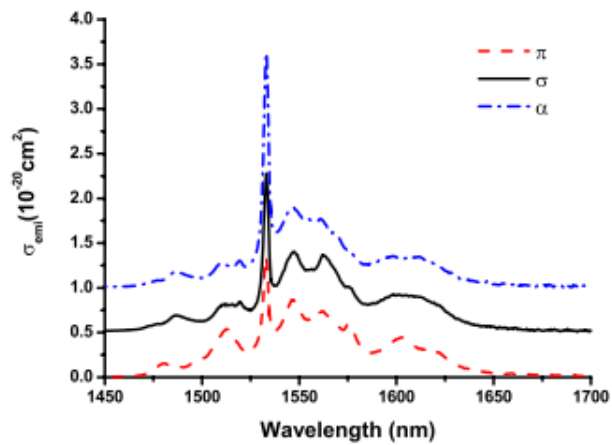
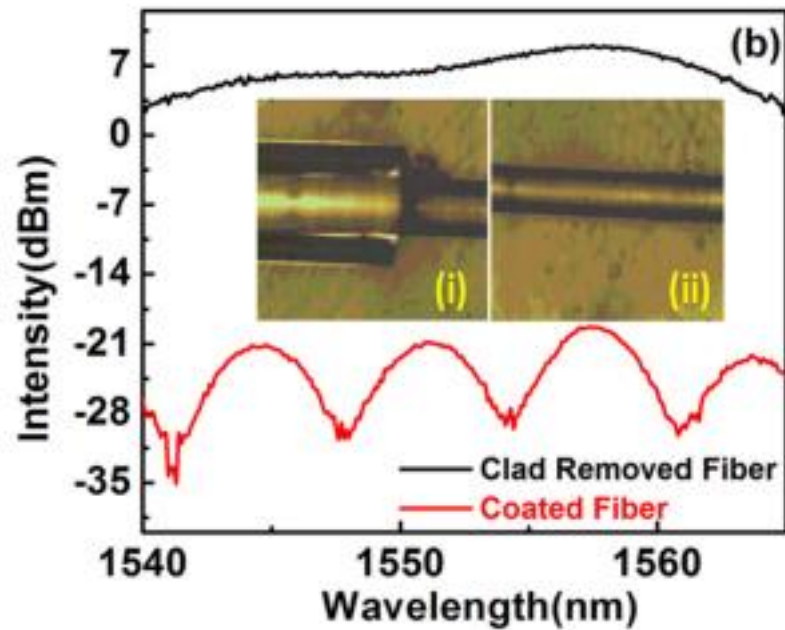
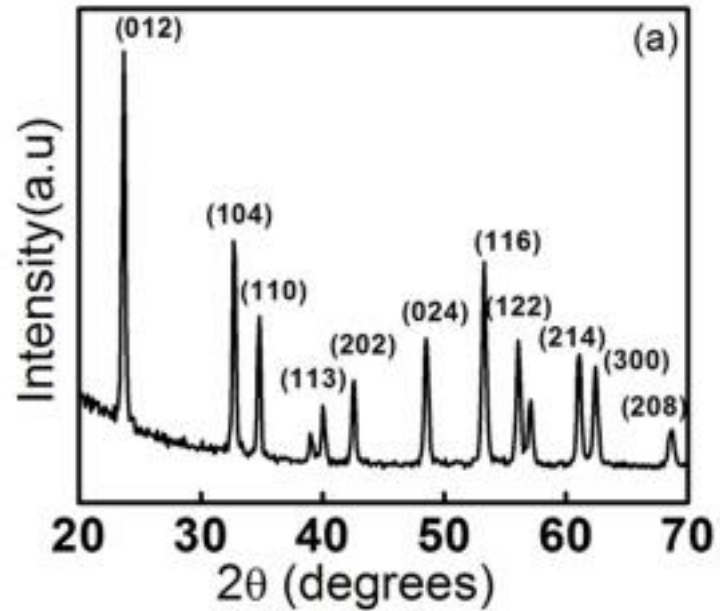


FIG. 8. (Color online) Polarized emission cross section of the  $^4I_{13/2}$  level.



Volatile organic compounds (VOCs) are common air pollutants

VOCs can contain:  
C, O, F, Cl, Br, S N

VOCs can:  
evaporate easily at room temperature;  
Give strong health risks

Several types of sensors were developed

surface acoustic wave sensors

polymer composite sensors

carbon nanotubes

CMOS

capacitive sensors

**Fiber optics sensors**





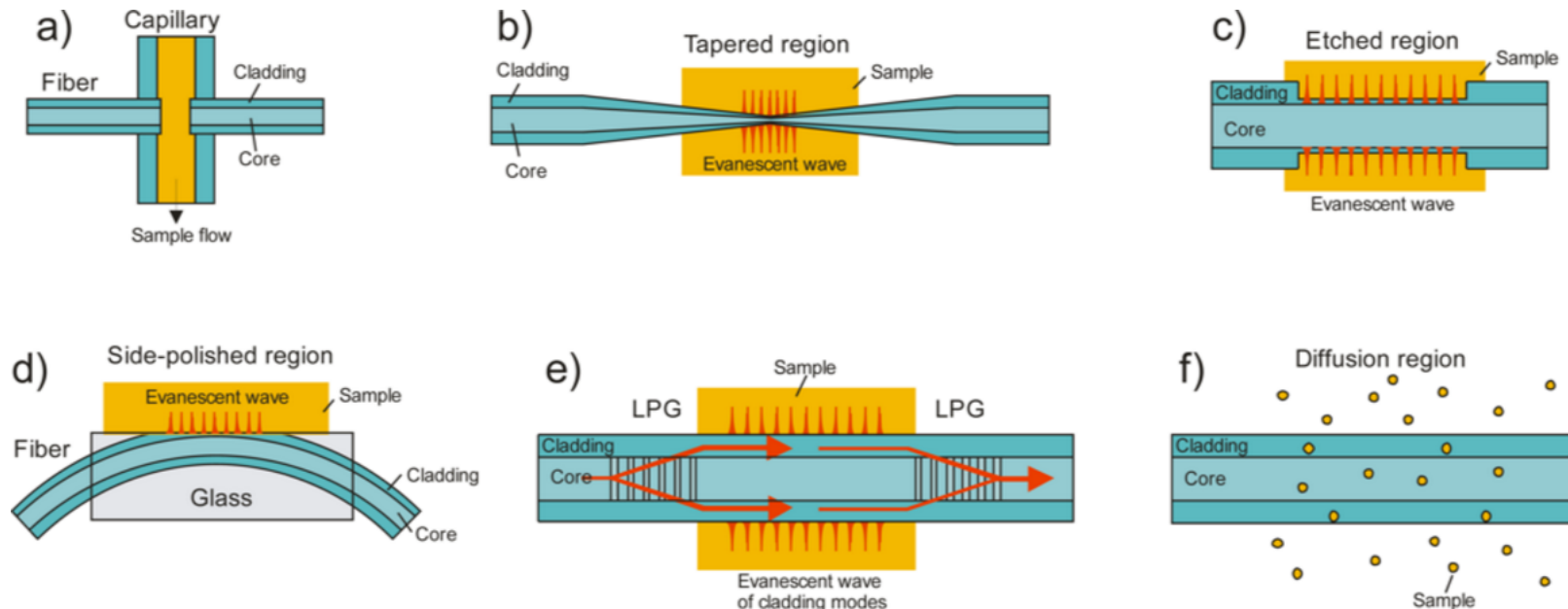






# EF: fibers for sensing

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Sensors 2010, 10(3), 1716-1742

Helen Waechter, Jessica Litman,  
Adrienne H. Cheung, Jack A. Barnes  
and Hans-Peter Loock