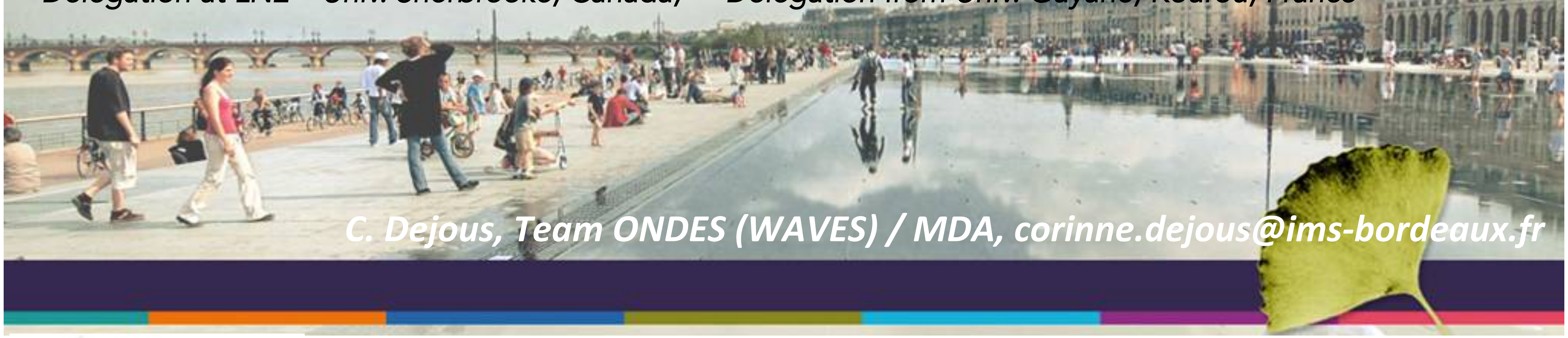
Wave-based resonant microsensors for environmental applications.

Corinne Dejous

Summer School Bordeaux INP - June 25th to July 6th, 2018
Sustainable development in metropolitan Bordeaux

Wave-based resonant MICROSENSORS for environmental applications

C. Dejous,
Presentation based on the team's research work – L. Bechou*, H. Hallil, S. Hemour, S. Joly, J.L. Lachaud, L. Oyhenart, D. Rebière, O. Tamarin**
* Delegation at LN2 – Univ. Sherbrooke, Canada, ** Delegation from Univ. Guyane, Kourou, France



C. Dejous, Team ONDES (WAVES) / MDA, corinne.dejous@ims-bordeaux.fr

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Thematic Organization at IMS Lab.

10 groups *27 teams*

Systems and Interactions	BIOELECTRONICS
	COGNITICS
Systems	SIGNAL
	PRODUCTION ENGIN
	AUTOMATIC CONTR
Hardware Integration	CIRCUITS DESIGN
	DEVICES RELIABILITY
	NANOELECTRONICS
From Materials to Devices	ORGANIC ELECTRON
	ONDES / WAVES *Y. Ousten*

Group ONDES (WAVES), 18 permanent staff

Towards communicating, sustainable, multiphysics and multitechnology systems

MDA *D. Rebière*	EDMINA *L. Bechou*	MIM *F. Demontoux*

Up to 350 staff involved in Information Technologies (IT) & Engineering

www.ims-bordeaux.fr/en

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ONDES (Waves)

Towards Multi-disciplinary Multi-technology Smart and Sustainable systems

Yves Ousten, Professor (C.Dejous, 2011-2017)

MIM : Materials, Interactions, Micro-waves (F.Demontoux, Prof.)

MDA : Acoustic wave-based Detection Microsystems (D.Rebière, Prof.)

EDMiNA : Evaluation of Micro- Nano- Assembled Devices (L.Bechou, Prof.)

A2M, Spin-off on Non-Destructive Material Micro-wave Characterization (G.Ruffié)

OpERaS, Technological platform for Electro-Optical Characterizations (L.Bechou)

Strategic topics

- Microdevices for Optics / Photonics
- Connected Multi-technology Microsensors
- Wireless solutions for micro-energy

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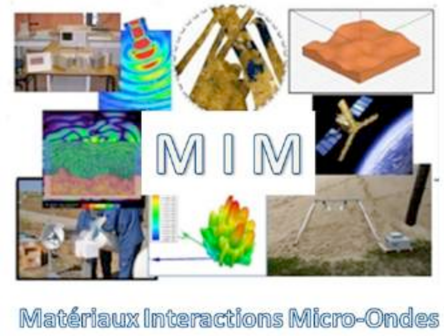
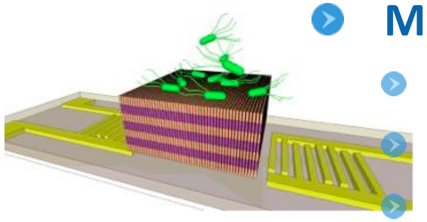
Laboratoire de l'Intégration du Matériau au Système → October, 2016

From Materials to Devices

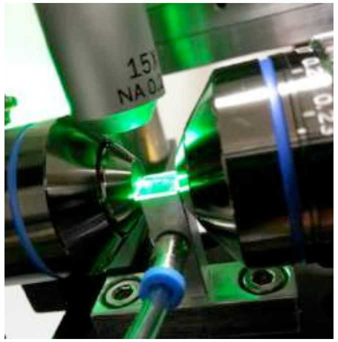
1 → ONDES / WAVES



- **MIM**, Materials, Interactions, Micro-waves
  - Interactions electromagnetic waves (CW to mm-wave) / materials
  - Natural materials : wireless active / passive detection
  - Structured materials with specific properties: photonic crystal, meta-materials, radar stealth (furtivity) …



- **MDA**, Acoustic wave-based Detection Microsystems
  - Resonant devices – microsensors, prototypes
  - Using of innovative sensitive materials
  - Wave propagation in devices, dynamical interaction phenomena with solid, liquid, gaseous medium



- **EDMiNA**, Evaluation of Micro- Nano- Assembled Devices
  - Influence on microelectronic / photonic devices :
    - **Integration of newmaterials**
    - **Assembly, interconnection**
  - Influence of environmental conditions - Reliability

- ***Permanent Staff (18): 6 Prof., 9 Assoc.&Assist. Pr., 3 Ing.***

Towards Multi-disciplinary Multi-technology Smart and Sustainable systems

From Materials to Devices… Pluri- Inter-Disciplinarity

1 → ONDES / WAVES

- Multiphysic Modeling
- Integration
- Characterisation
- Sustainability
- Application Microwaves-Materials (A2M Spin-Off)

- Innovative transduction (acoustical, optical, high frequency)
- Interface Characterisation
- High resolution Electrical / Optical Characterisation
- Reliability

- Heterogeous Integration
- Prototyping
- Evaluation of performances, Tests in controlled environment
- Data treatment / inversion

Materials for High Frequencies and Photonics

Micro- Nano-Assembled Sensors and Devices

Multi-technology Smart and Sustainable Systems

Strategic topics

- **Microdevices for Optics / Photonics**
- **Connected Multi-technology Microsensors**
- **Wireless solutions for micro-energy**

https://www.ims-bordeaux.fr - Research – Research groups - WAVES

Philosophy in agreement with IMS scientific strategy

Four Major Research Projects since 2015

- Two combined paradigms: Interdiscipinarity & Partnership
- The Group is involved into the 4 Major Research Projects at IMS

Environments

Internet of Things

Innovative Systems for Health

Smart Transports

EnvironmentS

1.Societal Environment

2.ICT and Sustainability

3.Operational Environment of Devices

4.Energies

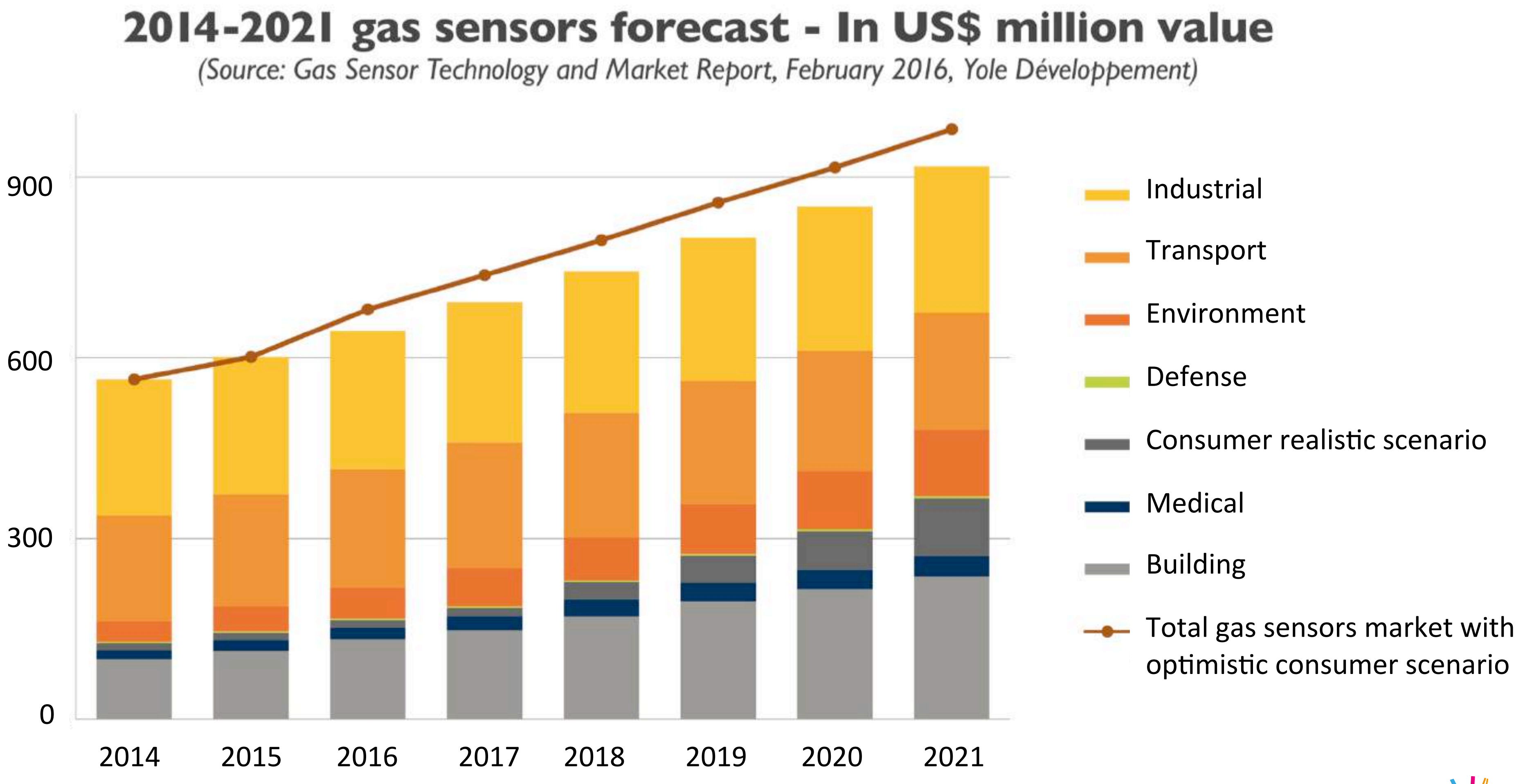
Ingénierie Verte

2018, the year of green engineering, INSIS/CNRS

Environmental and health related applications

- Introduction: Background, **Principle of Wave-based Resonant Microsensors**
- Focus on Acoustic Love Wave Devices
  - Principle
  - Examples of Gas sensing applications
  - Introduction of microfluidics and examples of Liquid-phase sensing applications
- Other Resonant Devices and Applications
  - Flexible imprinted Electromagnetic Devices & Carbon-based Sensitive films
  - Polymer Optical Microring Resonators & Digital Microfluidics
- Conclusion, perspectives

Chemical sensor market

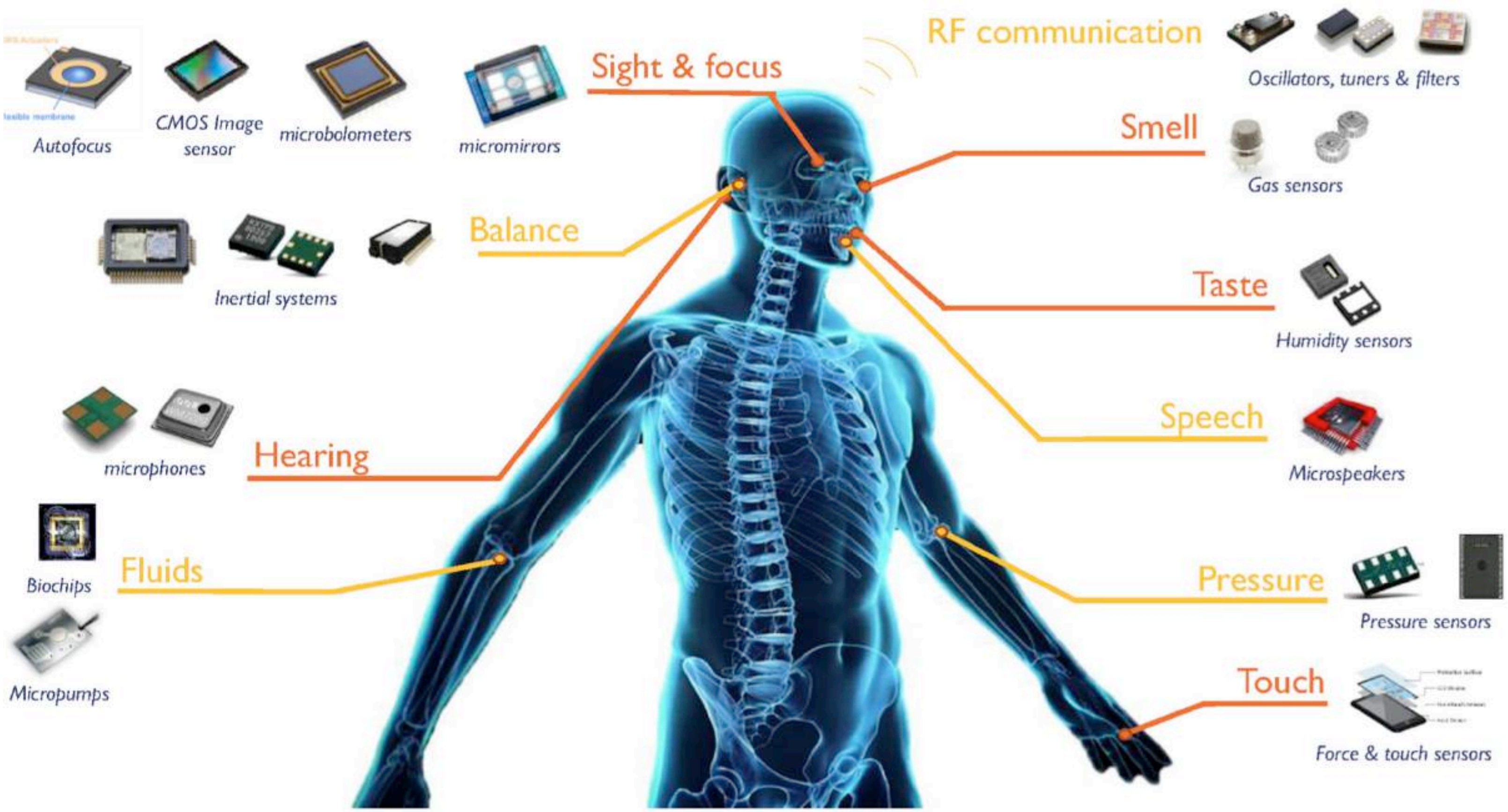


Yole Développement, February 2016,http://www.yole.fr/iso_album/illus_gassensors_forecast_yole_feb2016.jpg

Sensors for healthcare

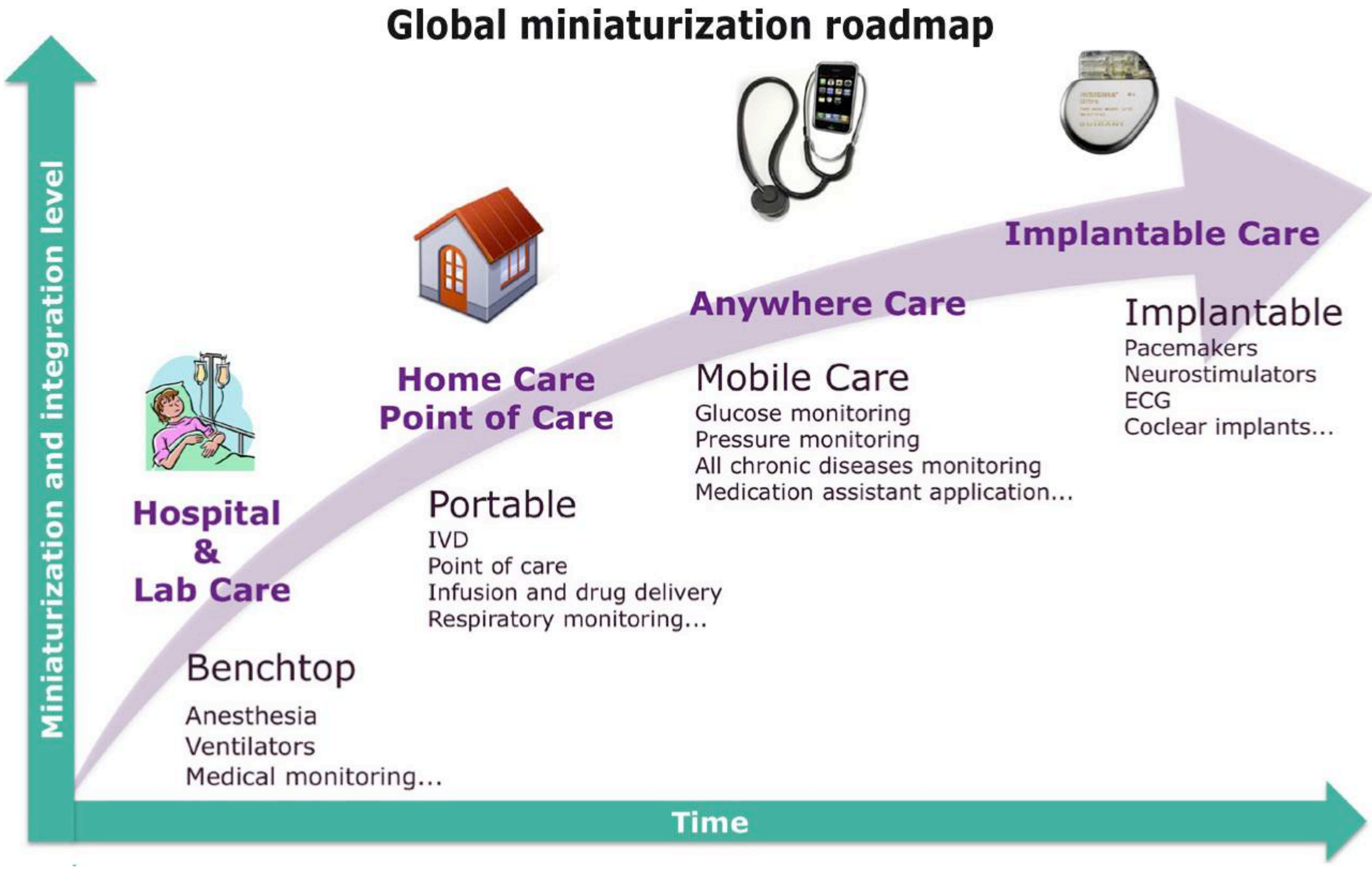
MIMICKING THE 5 SENSES ....AND MORE

(Source: Sensors for Wearable Electronics & Mobile Healthcare, Yole Développement, June 2015)



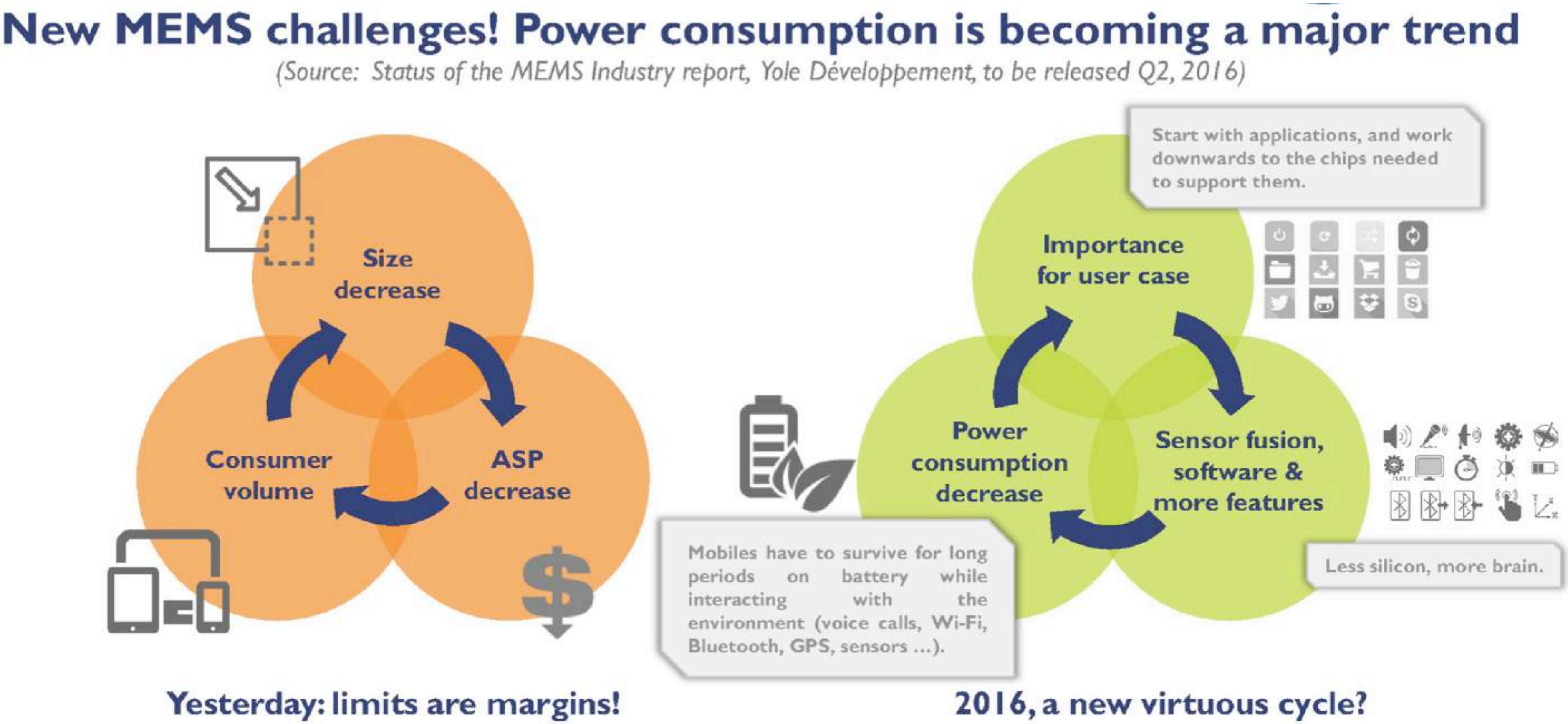
Yole Développement, June 2015, http://www.yole.fr/iso_album/illus_wearableelectronics_5senses_yole_sep2015.jpg

Medical microsystems roadmap



Yole Développement, 2013, www.i-micronews.com/images/Reports/MedTech/Images_reports/Global_miniaturation_roadmap.jpg

Chemical sensor and Energy



Yole Développement, March 2016, http://www.yole.fr/iso_album/illus_memsvirtuouscycle_yole_march2016_2.jpg

Issues of chemical detection

- **System dedicated to an application or a specific compound?**
- **Test cost?**
- **Measuring time?**
- **Duration of phase calibration?**
- **Frequency of maintenance periods?**
- **Sensitive elements form disposable?**
  (typical case for a biomedical application)
- **Major, minor or trace compound?**
- **Staff qualification (to perform the measurement)?**

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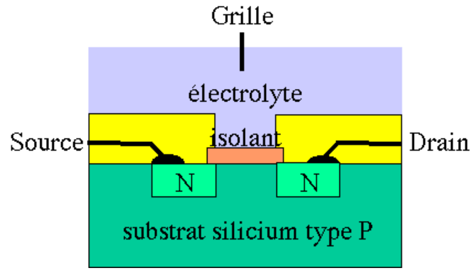
Trend: a wide variety

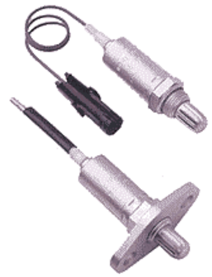
Chemical microsensors:
an economical and efficient alternative

Variation of conductance
Oxide semiconductor



Field effect transistor ChemFET

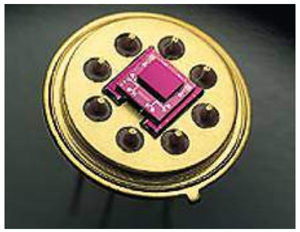
Electrochemical sensor



Micro Détecteur

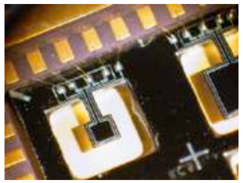
Optical
Fibre
Phototransistor

Thermal conductivity

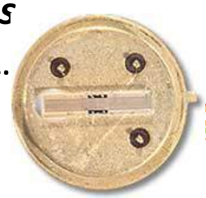


Pellistor

Mobile microstructures



Acoustic waves
Quartz microbalance (BAW)
Surface waves
Plate wave, …



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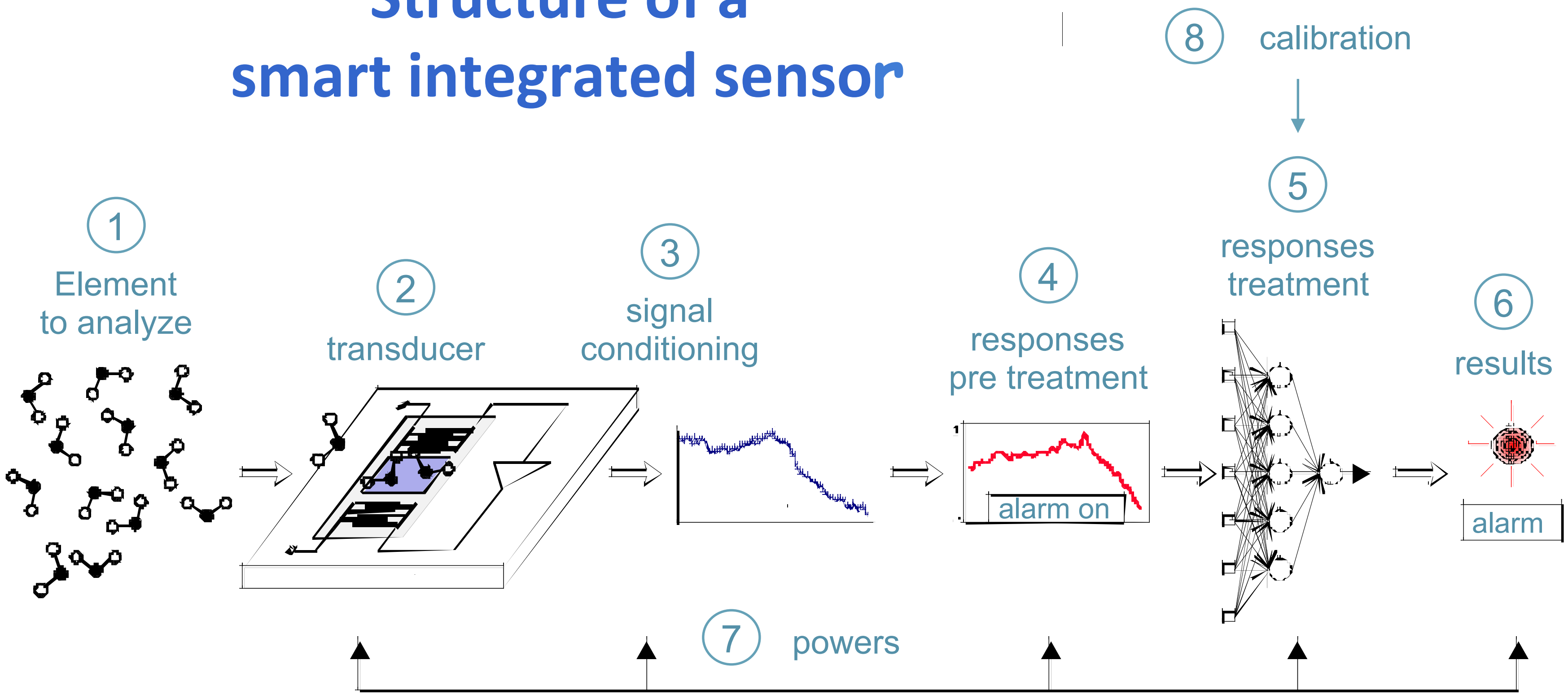
Smart (bio)chemical Sensor

Structure of a smart integrated sensor



Chemical and biological fields

Chemical industry

Military & Security

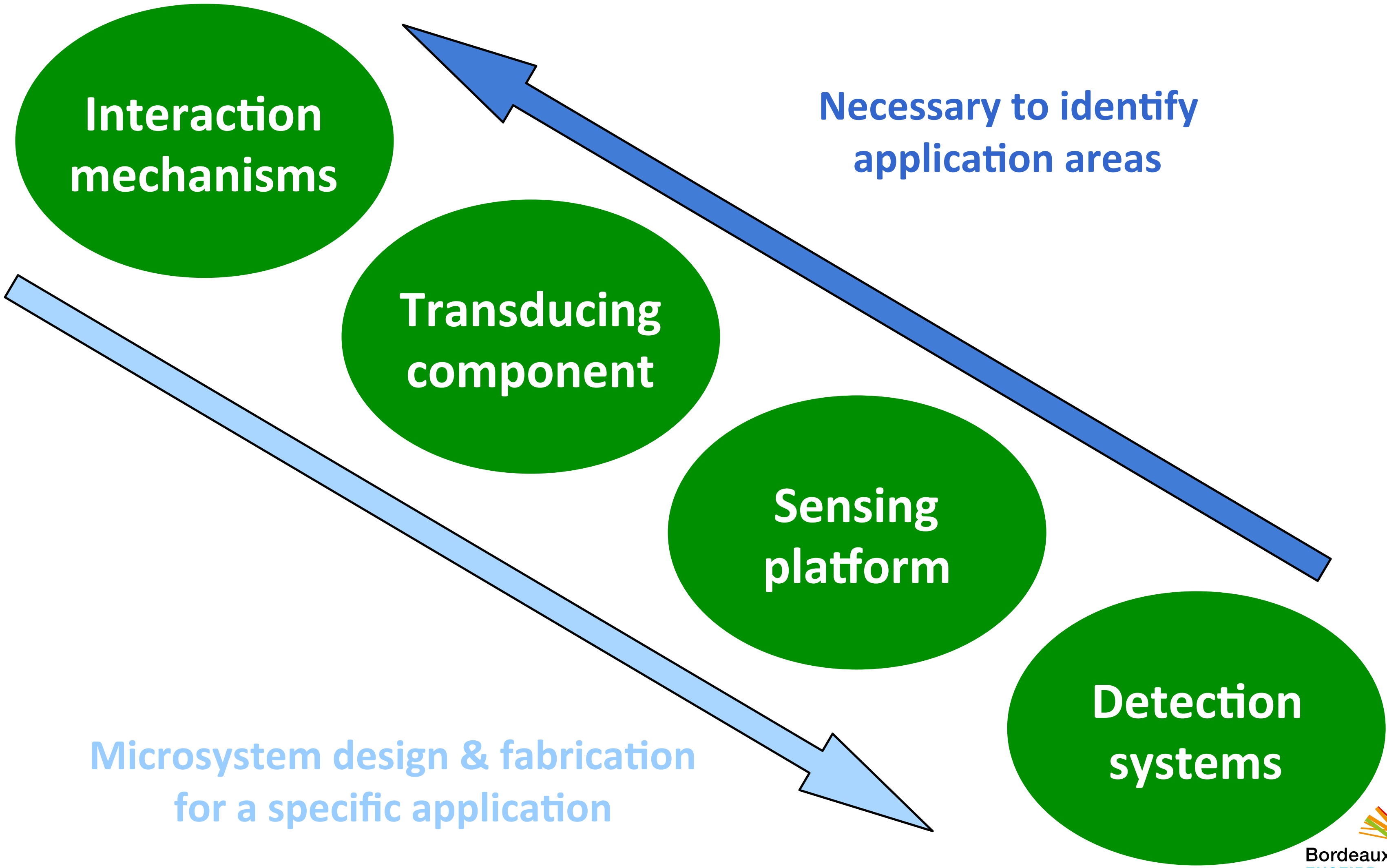
Food industry

Biological and medical field

Fine chemistry cosmetic

Environment

Wave-based Sensor Research activity



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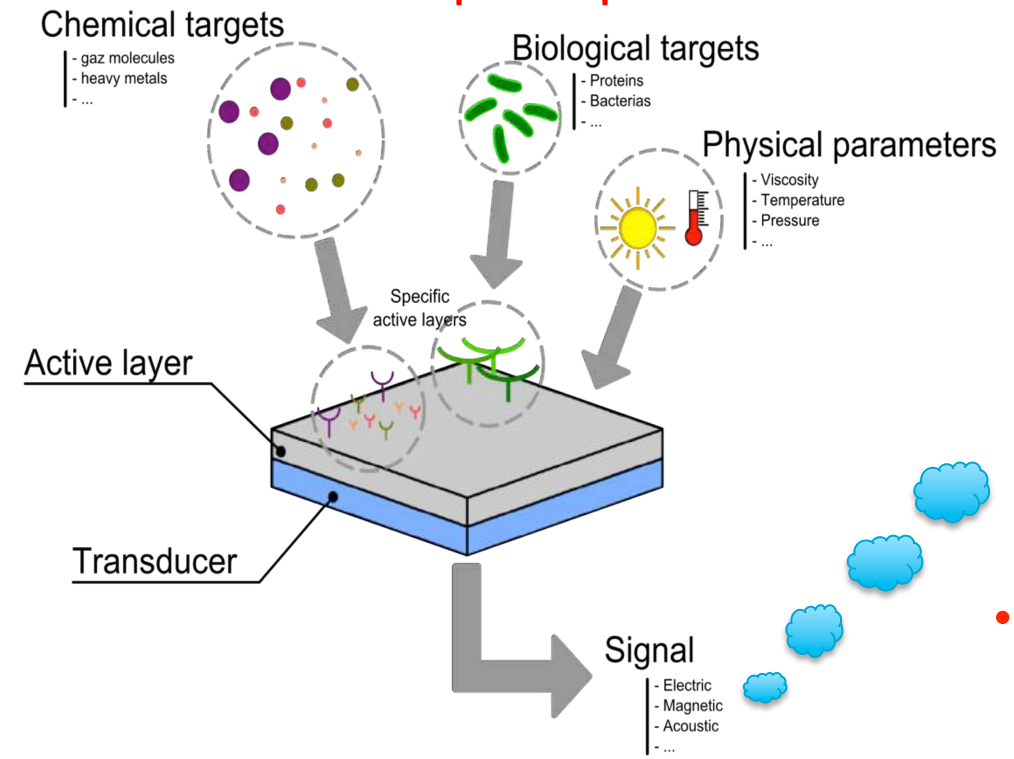
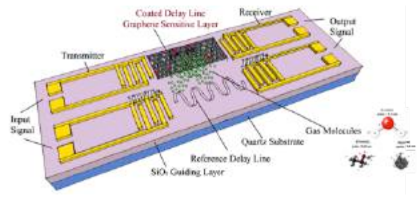
Ultrasensitive Acoustic & Electromagnetic & Optical transducers : Application for gas and bio sensing

Gas or Biosensor principle

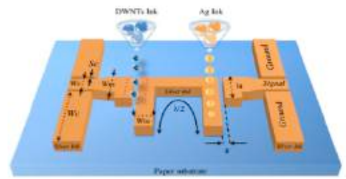


Integrable Technologies
Sensitivity, Selectivity
Sustainability

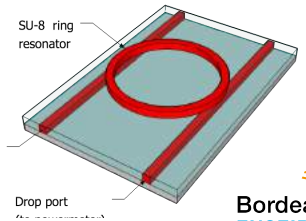
Acoustic platform (piezoelectric substrate)



Electromagnetic platform (flexible substrate)



Optical platform (polymer materials)



- **Materials : polymeric, hybrid, meso- and nano-structured, biomaterials (antibodies, whole micro-organisms), etc.**

- **Overview of devices and applications**

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Environmental and health related applications

- Introduction: Background, **Principle of Wave-based Resonant Microsensors**
- Focus on Acoustic Love Wave Devices
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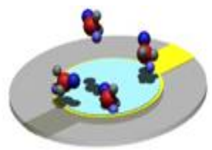
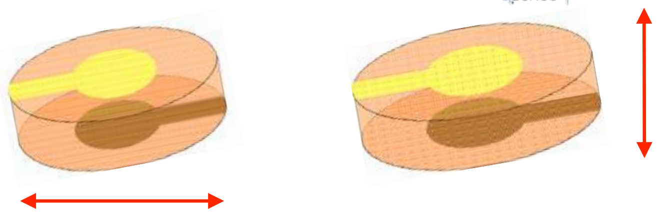
Acoustic wave transducers

Acoustic or elastic wave: mechanical displacement
Generation by piezoelectric effect

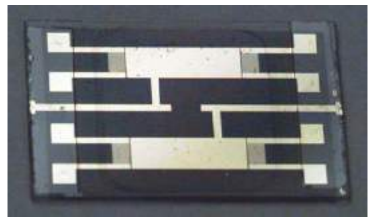
Bulk wave transducer
(QCM : quartz microbalance)



qsense



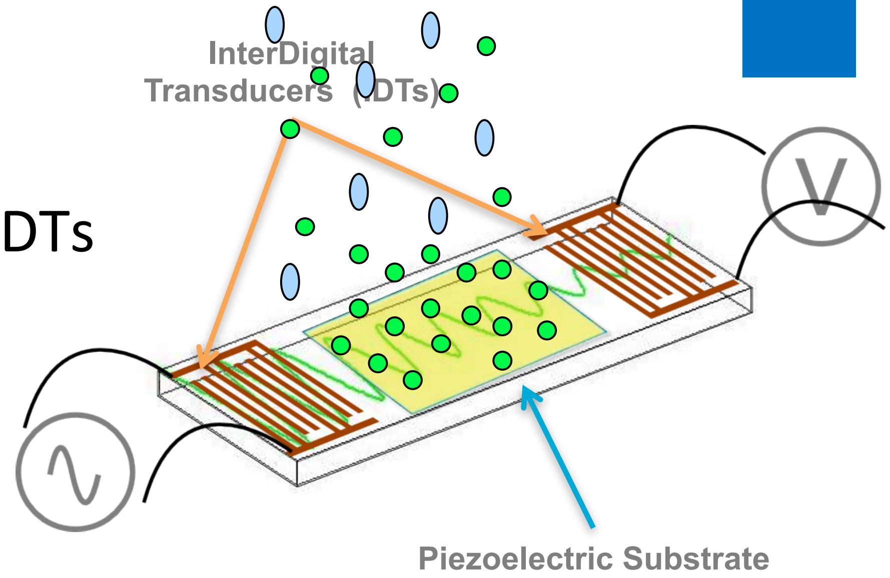
Surface wave transducer

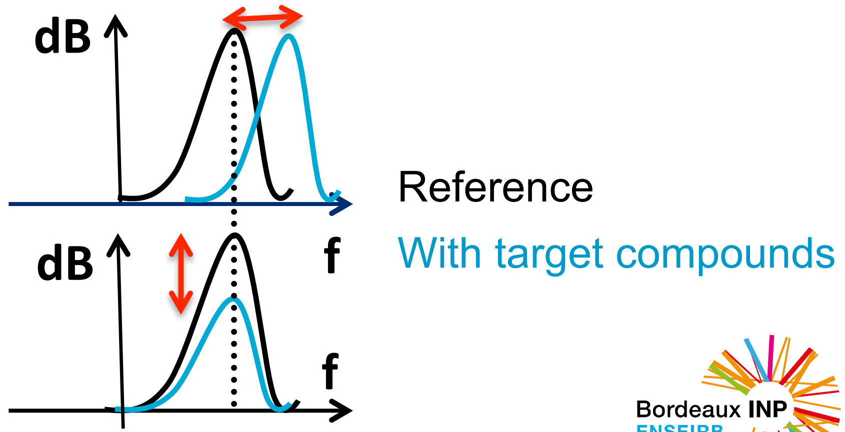


Planar device: collective fabrication
Energy near the surface: sensitivity

SAW Delay Line

- **Piezoelectric effect**
- Acoustic wave propagation using IDTs (Inter Digital Transducers)
- Wave perturbation induces
  - **Phase velocity variation**
  - **Attenuation (Insertion Loss)**
- Design - Modeling
  - **Wave propagation**
  - **Interaction mechanisms**





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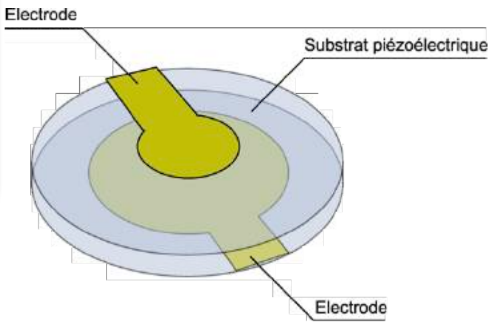
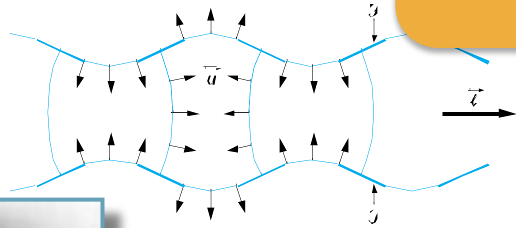
BAW (QCM)

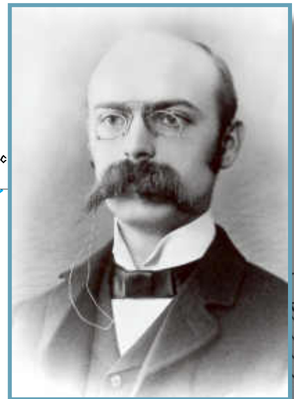
- Shear Honrizontal (SH) polarization
- Low mass-loading sensitivity (working frequency)



FPW (Lamb)

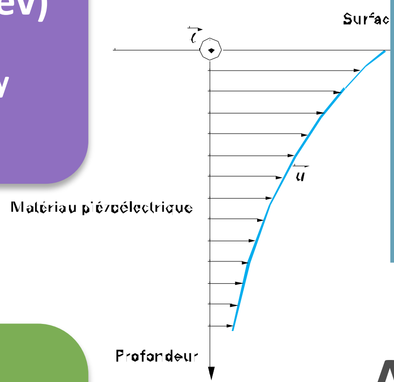
•Longitudinal
+ Shear Vertical
•h < λ : technological difficulties





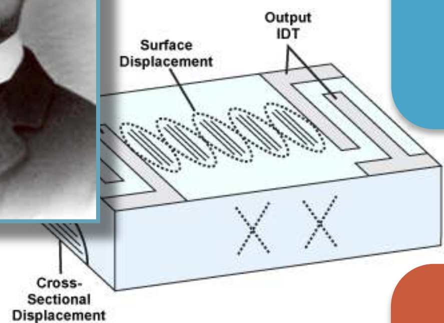
STW (Bleustein Gulyaev)

- SH wave
- Weak trapping energy



SH-APM

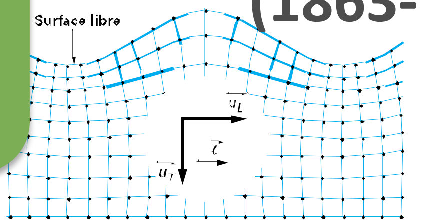
- SH guiding wave
- weak mass-loading sensitivity



A.E.H. Love
(1863-1940, UK)

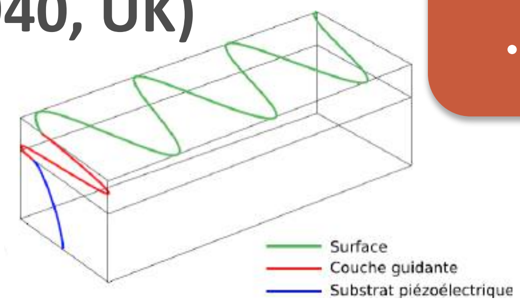
SAW (Rayleigh)

- Elliptic polarization (longitudinal & SH
- Good mass-loading sensitivity



Love

- Guiding SH SAW
- Guiding layer
- High mass-loading sensitivity



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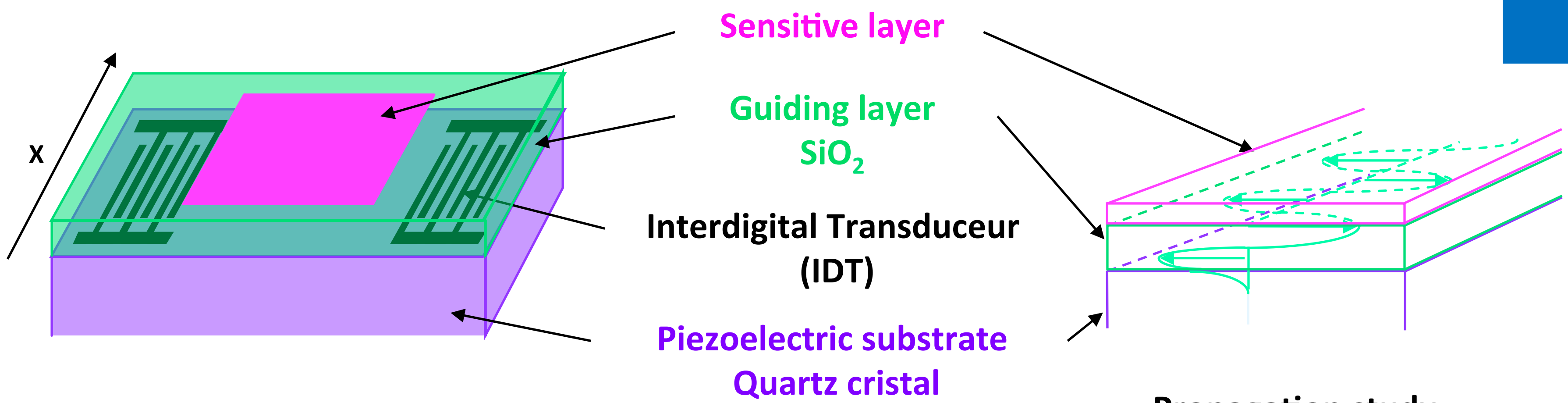
Love waves delay lines



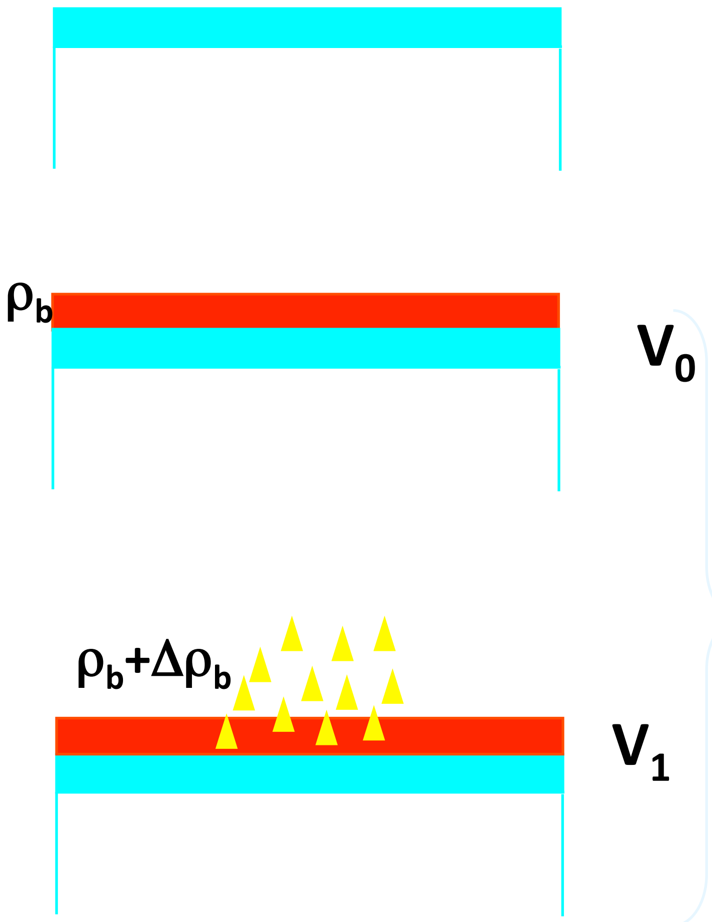
Love waves device

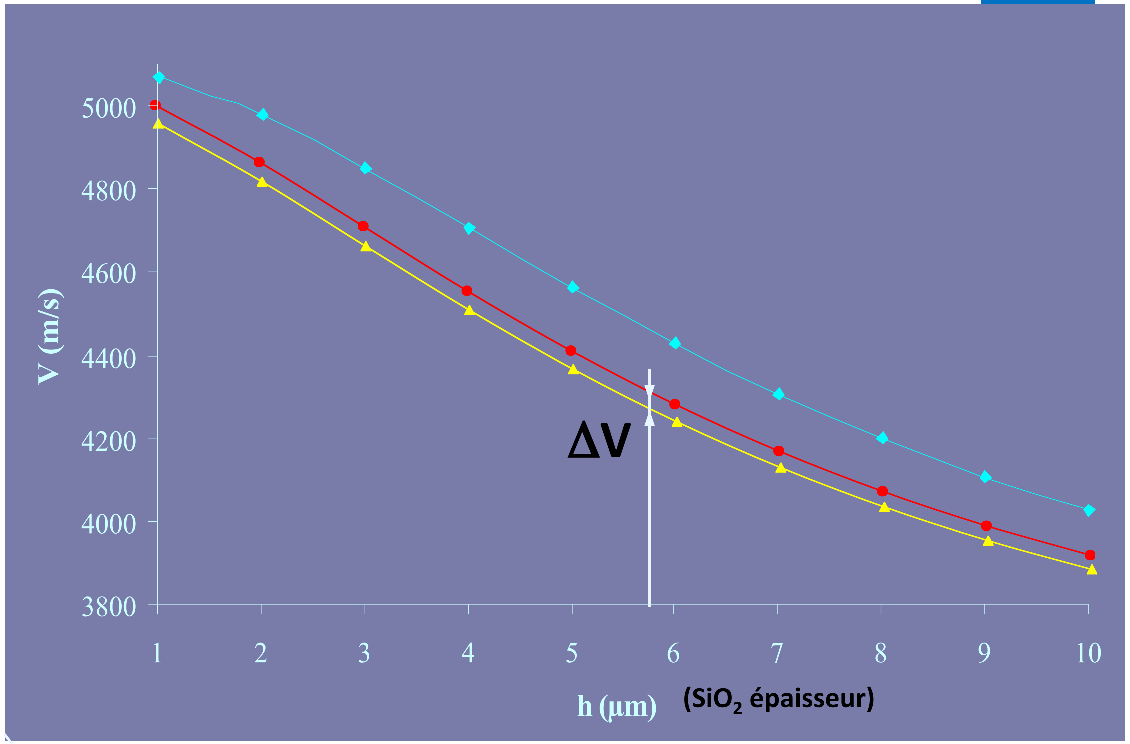
Propagation study model

- Oriented substrate + IDT electrodes: TH waves ➡ liquid
- Guiding layer: energy confinement ➡ sensitivity
- Sensitive layer: amplification, specificity

➡ **Propagation in a multilayer structure**

Sensitivity to mass effect



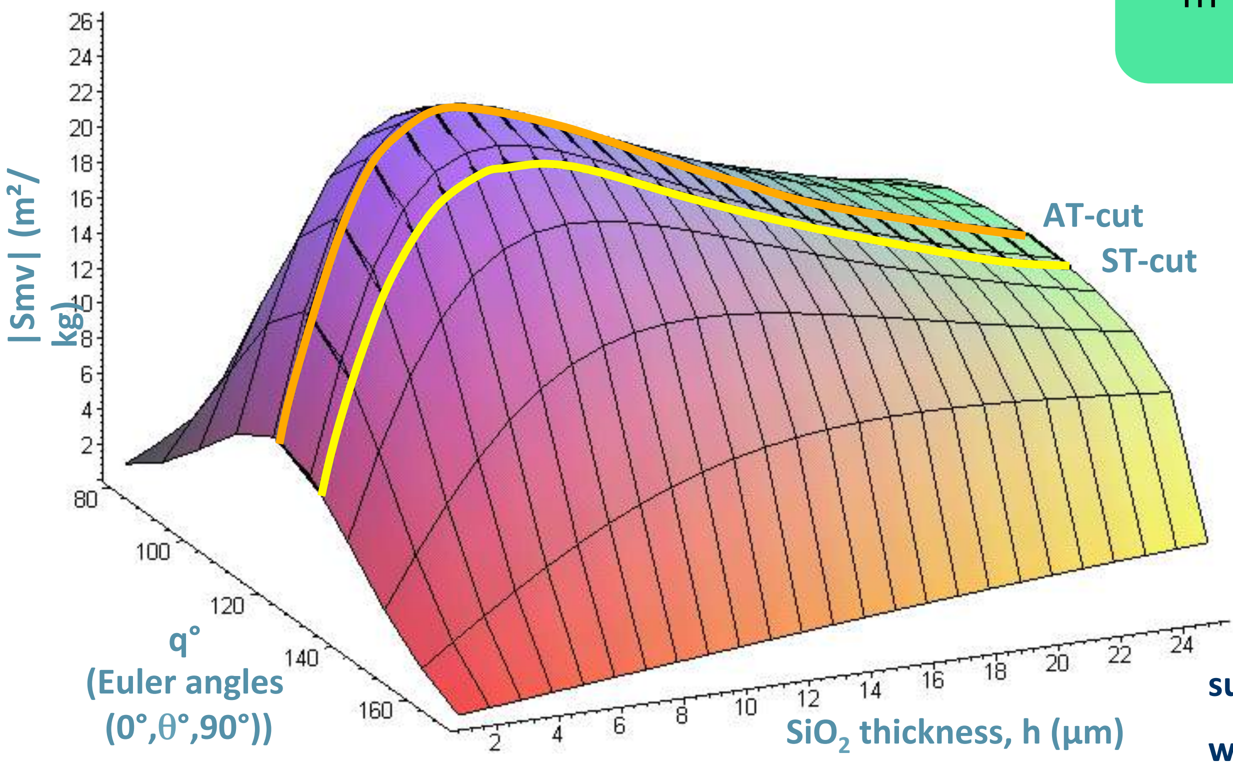


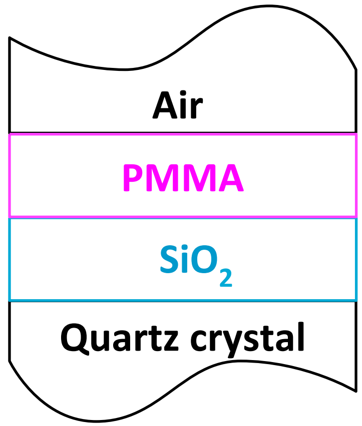
$$Smv = \frac{\Delta V}{V_0}\frac{1}{\Delta\rho_b b}$$

with $\Delta V=V_1-V_0$
due to the detection

Theoretical results
Mass loading sensitivity

$$S_m = \frac{\Delta f_m}{f_m} \times \frac{1}{\Delta m}$$





substrate: quartz

wavelength: λ = 40 µm

guiding layer: SiO_2

sensitive coating: PMMA,
b = 50 nm, $\Delta\rho_b$ = 5%

Maximum Sensitivity to mass effect :
Quartz AT cut, h from 4 to 8 µm

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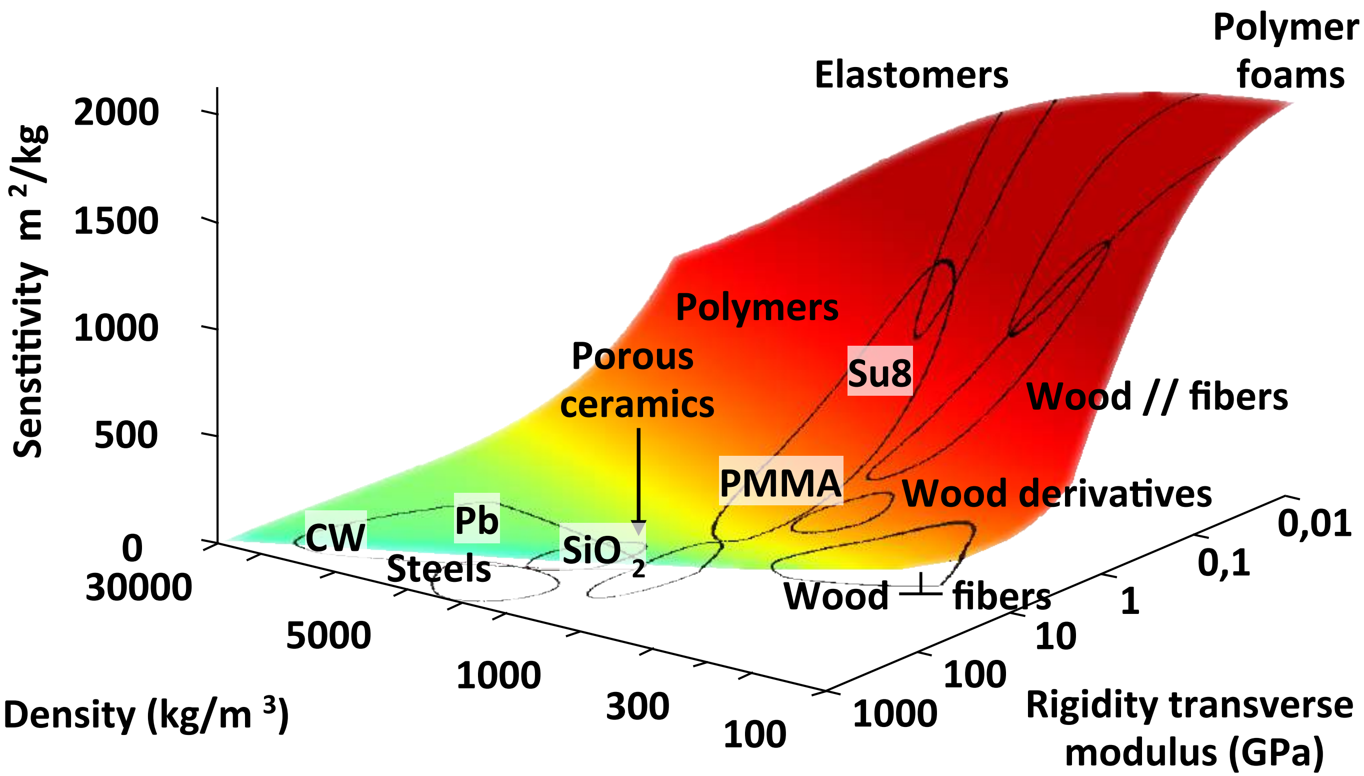
Theoretical results

Choice and characterization of the guiding material



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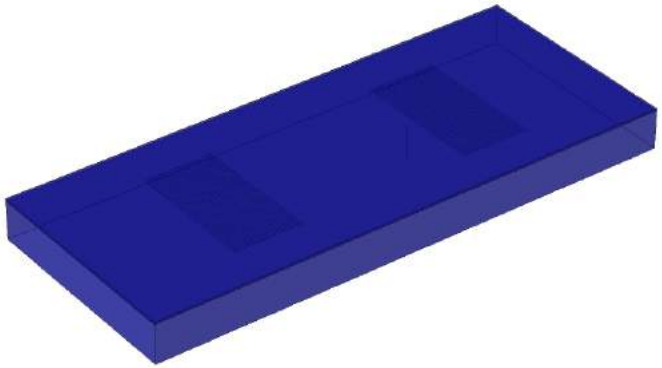
FEM* Simulation

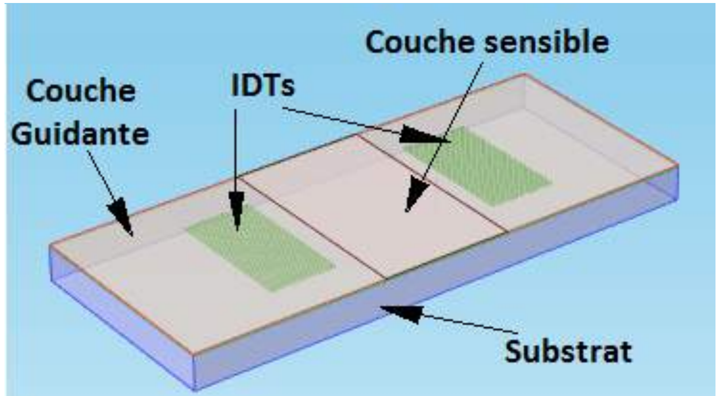
** Finite Element Method*
COMSOL Multiphysics

- Collaboration IRD Guyane - France (O. Tamarin)
- Toolbox 'Piezoelectric devices'

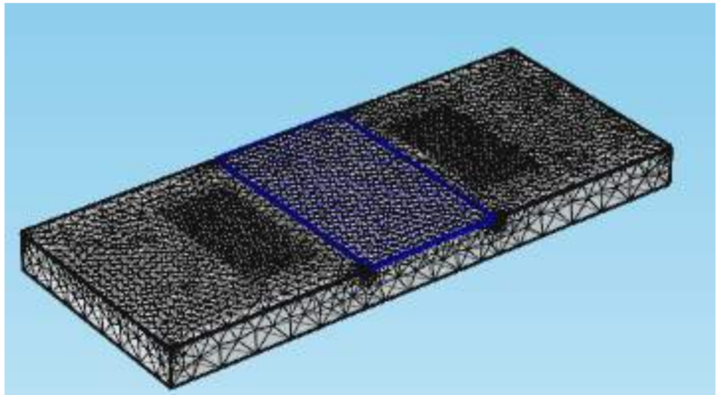
➢ *Inhomogeneous materials such as nanostructured thin films*



R. Djoumer – Master Thesis - 2012



Meshing '*Free tetrahedral*', Size '*Normal*'



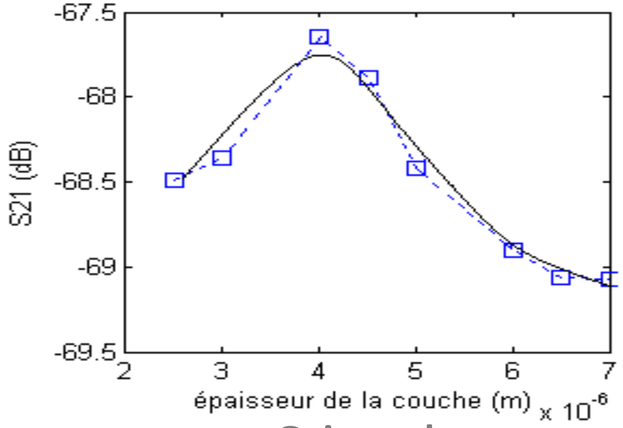
Meshing '*Free triangular*', Size '*Extrafine*'

FEM Simulation

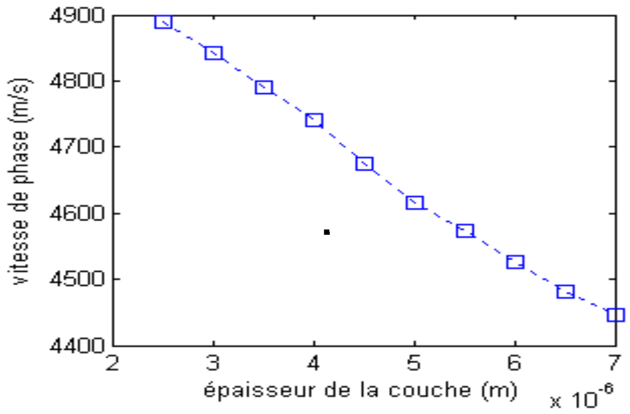
- Parametric study, effect of SiO_2 thickness (h) of a structure without any sensitive film
- Gain max: h = 4µm

- Phase velocity decreases from 4900m/s downto 4450m/s with h in the range [2.5µm – 7 µm]

R. Djoumer – Master Thesis - 2012



Gain vs. h



Vp vs. h

PhD, C. Zimmermann - 2002

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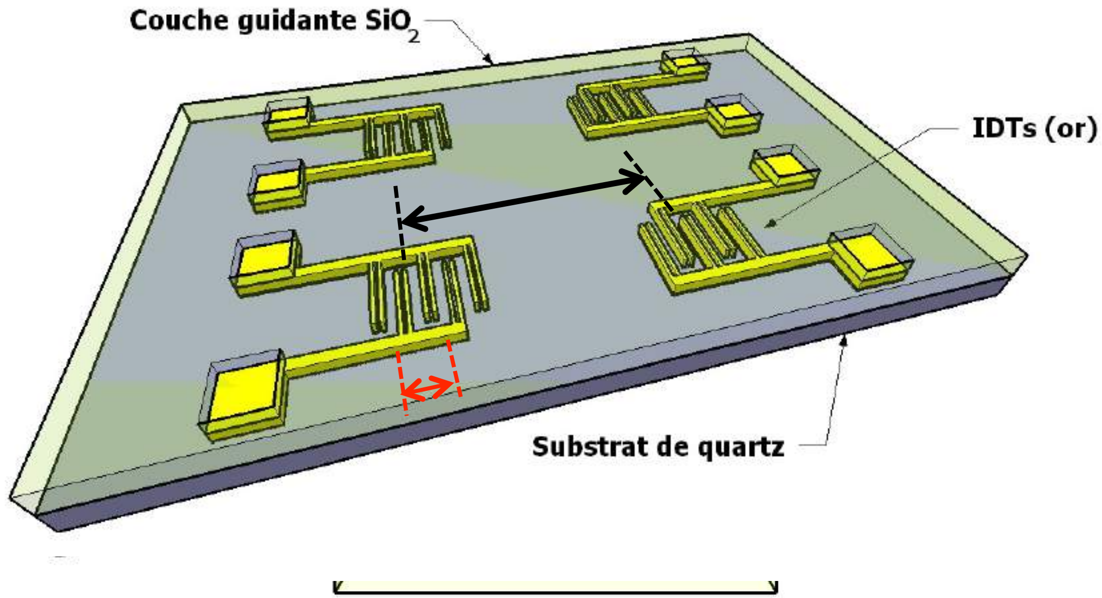
Towards Love-waves devices



1. Piezoelectric substrate: AT cut (0°,121.5°,90°)
   - ➢ Shear Horizontal polarization
   - ➢ Working in liquid medium
2. Interdigital transducers (IDTs) (λ=40 µm, Lcc=8.4 mm, e=200nm)
3. SiO_2 guiding layer (4µm, PECVD)
   - ➢ Trapping Energy → near free surface: attractive way for sensing applications

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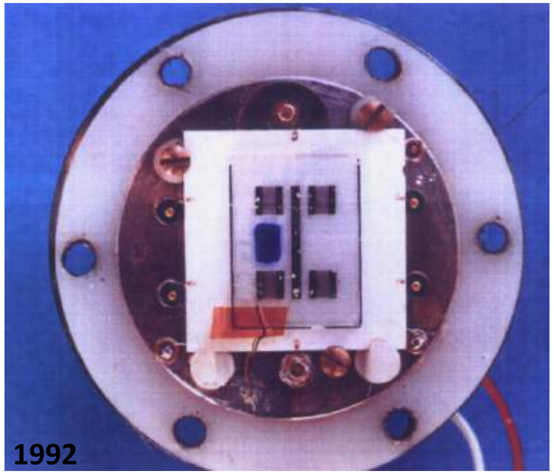
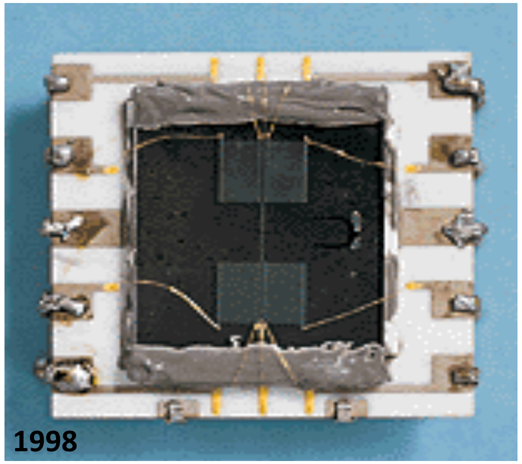
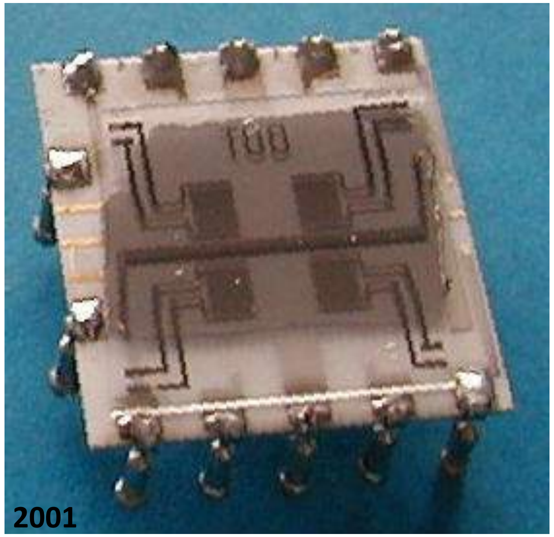
Technology achievements

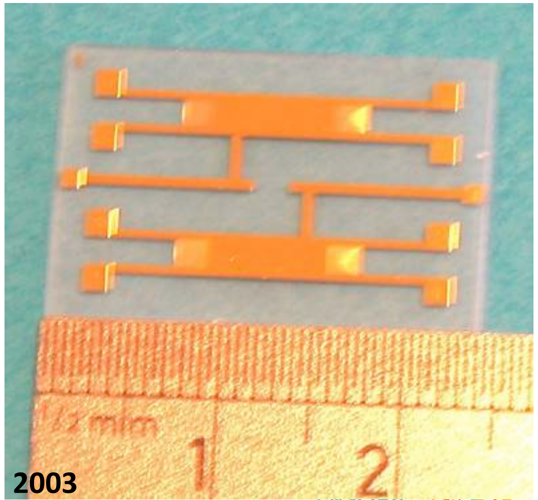
Delay lines :
- **Rayleigh waves (SAW)**
- **SH-APM**
- **Love waves**

Love wave devices :
Quartz cut AT, λ=40µm,
Électrodes : Al, Ti/Au
Guiding layer SiO_2, 4,6 µm









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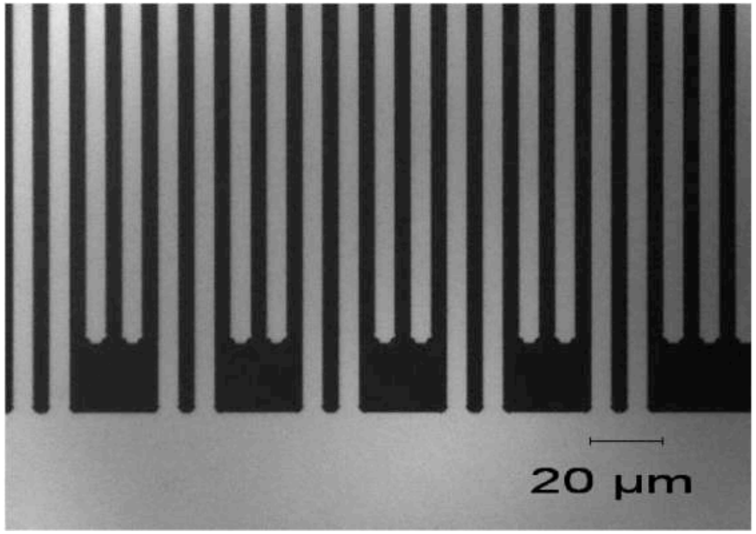
SAW delay-line devices

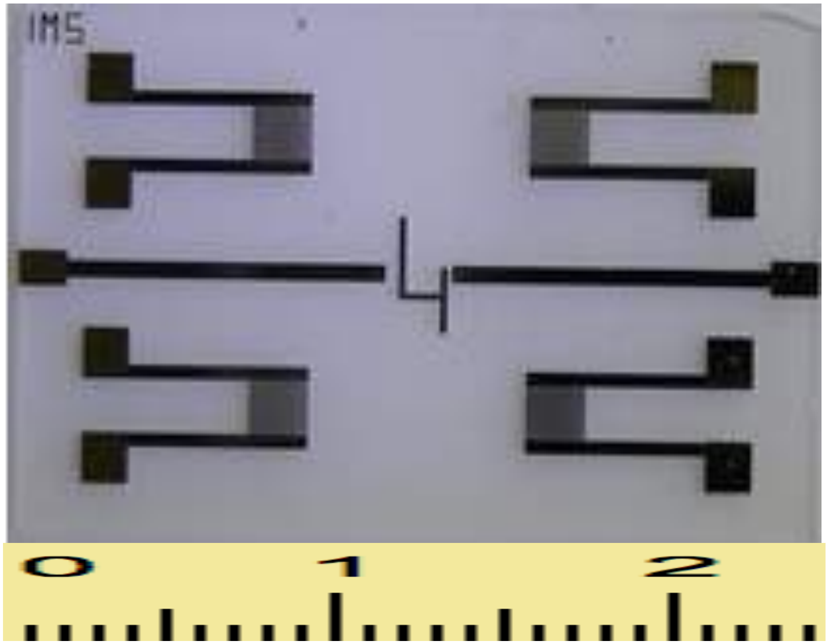


Quartz substrate: AT-cut

Wavelength λ = 40 µm

Synchronous frequency

f ≈ 117 MHz



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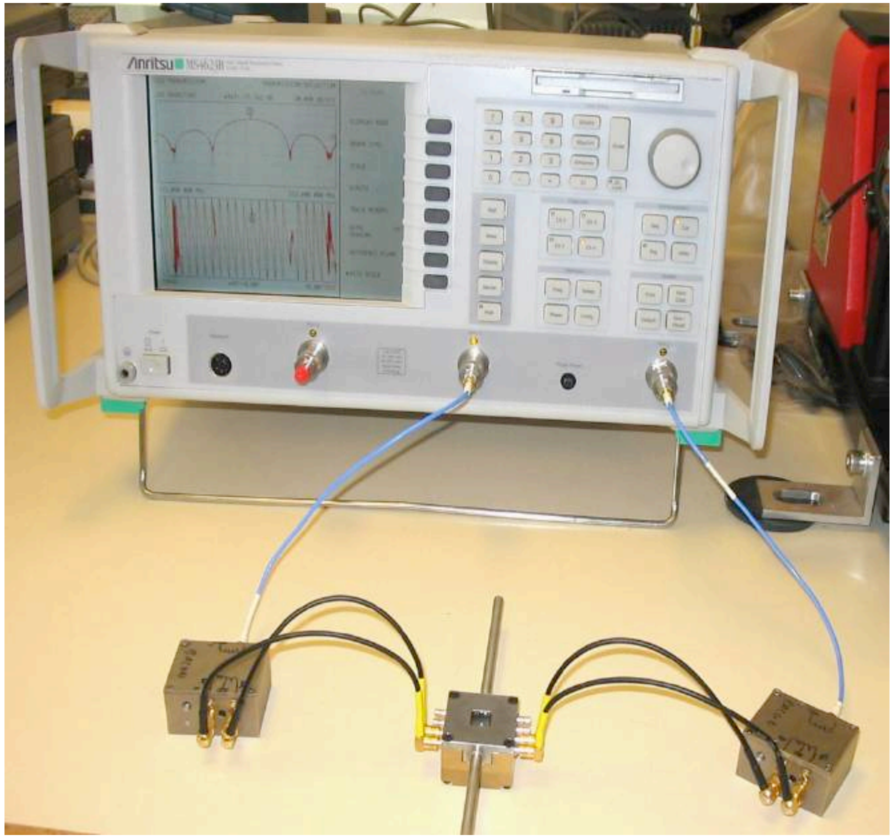
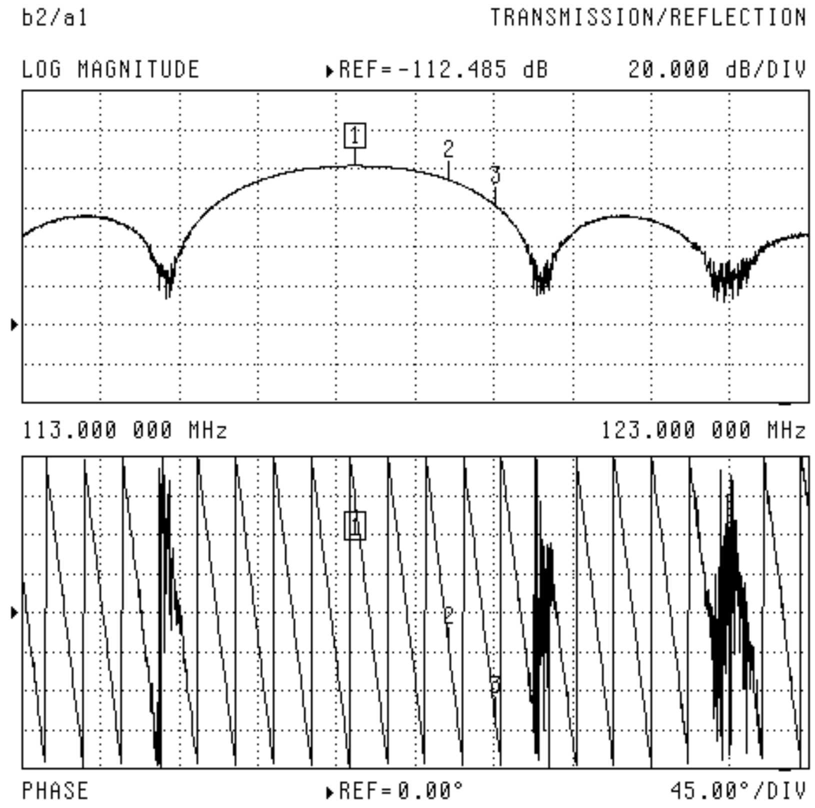
From the delay line to the oscillator

Transmission response characterization
of a delay line using a network analyzer

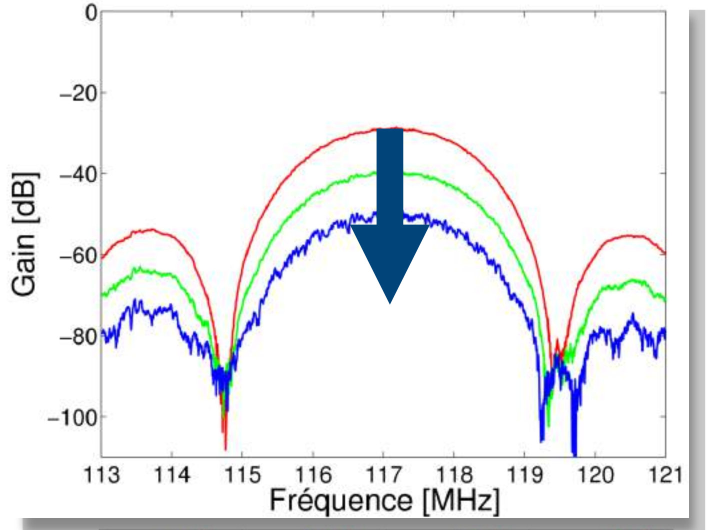


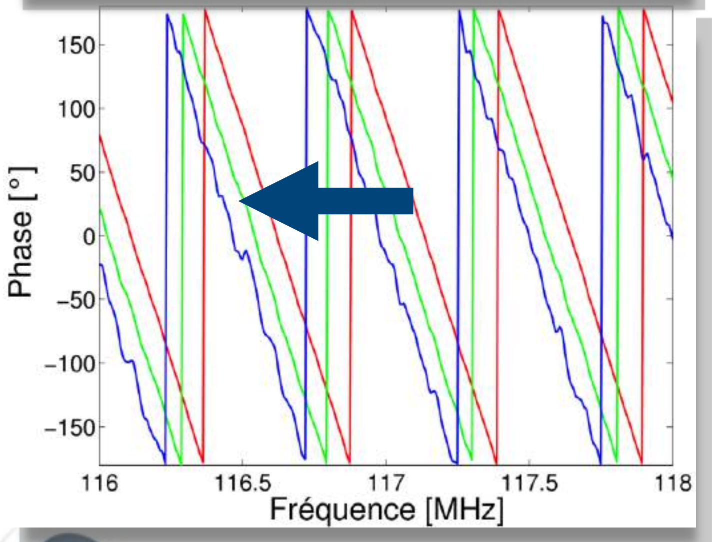


Operating frequency : 117 MHz

Test cell designed and realized according to the one
proposed by F.Josse et R.Cernosek, Sandia Labs

Electrical characterizations using Network Analyser



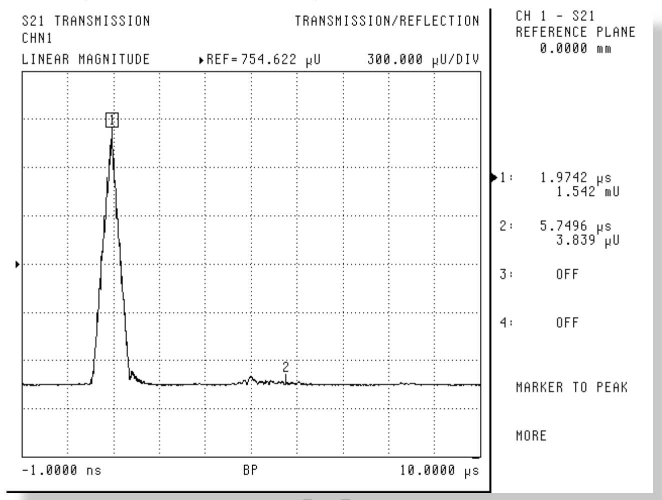


S_{21} (magnitude & phase)

LOVE Wave delay line

- f_0 = 117 MHz
- Insertion Loss: 25 dB
- Delay: 1.542µs

$V_{phase\ velocity} \approx 4300 m.s^{-1}$



Tme Domain, [1] acoustic signal

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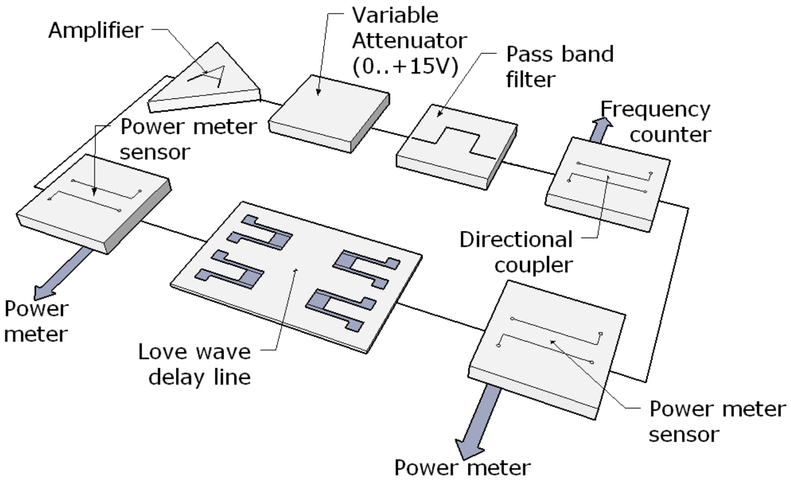
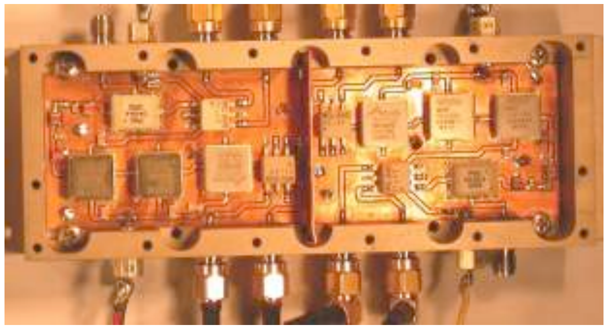
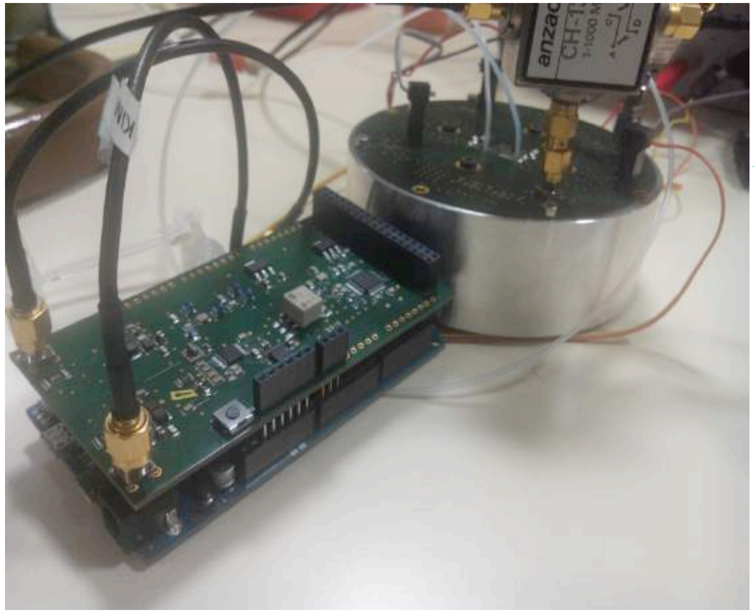
Read-out electronics

Oscillator configuration





Direct Digital Synthesizer (*DDS*)



- ✓ **Arduino-based read-out**
- ✓ **Communicating object (Ethernet)**
- ✓ **Sensor network ability**

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Acoustic sensors: various applications

- Gas sensors
  - ➲ CO, CO_2, NO, NO_2, …
  - ➲ Volatile organic compounds, organophosphorus & organosulfur, …
- Electronic noses
  - ➲ Gas mixture, odors, …
- Liquid sensors
  - ➲ Viscosity meter, conductivity meter, organic compounds, …
- Biosensors
  - ➲ Bacteria, viruses, toxins, heavy metals, hydrocarbons, …
- Thin film & complex fluid characterization

- Sensitive coating
  - ➲ Material
  - ➲ Deposition method
  - ➲ Structuration
- Result
  - ➲ Sensitivity
  - ➲ Selectivity
  - ➲ Reversibility
  - ➲ Response time
  - ➲ Recovery time
  - ➲ Reproducibility, Reliability

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Technological facilities (TAMIS)

- **Clean room class 10 000 (hood 100)**
- **Thin films microprocessing:** spin-coater, spray-coater, Graphtec, mask aligner, hotplates, wet etching of photoresist
- **Thick films processing**
  screen printers, ovens, inks formulation
- **Wire-bonding assembly**
  ball-bonder, wedge-bonder, flip-chip
- **Films and devices characterization:** profilers, AFM, SEM, vibrometer, gain/phase analyzer, network analyzer…
- **Soft lithography**
- **Mechanical micro-fabrication**
- **Partnerships**

➔ Device customization

Environmental and health related applications

- Introduction: Background, **Principle of Wave-based Resonant Microsensors**
- Focus on Acoustic Love Wave Devices
  - Principle
  - Examples of Gas sensing applications
  - Introduction of microfluidics and examples of Liquid-phase sensing applications
- Other Resonant Devices and Applications
  - Flexible imprinted Electromagnetic Devices & Carbon-based Sensitive films
  - Polymer Optical Microring Resonators & Digital Microfluidics
- Conclusion, perspectives

Sensitive materials : polymers

Molecular interactions

- van der Waals’s forces - *Non specifics*
  Low energy
- Hydrogen bondings - *specifics*
  Intermediate energy level (40 kJ)
  A preference for good
  reversibility & selectivity

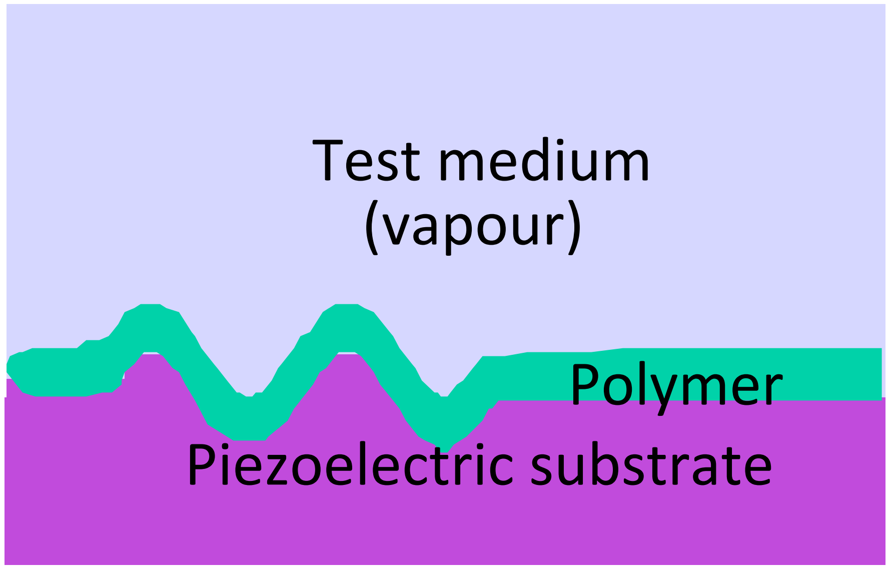
Low glass transition temperature (T_G)

For a fast diffusion of species in the polymer

Selection tool : solubility energy linear relation

Determination of partition coefficients polymer/gas (K)
Abraham’s method - LSER

Interaction sensitive layer /gas



Thermodynamic equilibrium
partition coefficient

$K = C_s/C_v$

Molecules sorbed in the polymer

mass effect,
rigidity,
viscosity, ...

K function of:
- polymer
- nature of gaseous compound
- temperature

Sensitivity to the vapor A : high value of K_A

Selectivity with respect to the compound B : $K_A > K_B$

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Polymer Sensitivity and Selectivity

Abraham's method: Linear Solvation Energy Relationship (LSER)*

Polarisability — **Ability to provide hydrogen bonds Acidity** — **Dispersive interactions**

$$\log K = c + r.R_2 + s.\pi_2^* + a.\alpha_2^H + b.\beta_2^H + l.\log L^{16}$$

Constant — **Polarity** — **Ability to accept hydrogen bonds Basicity**

r, s, a, b, l : solvant parameters (polymer)

R_2, p_2*, a_2^H, b_2^H, log L^{16} : solute parameters (vapor)

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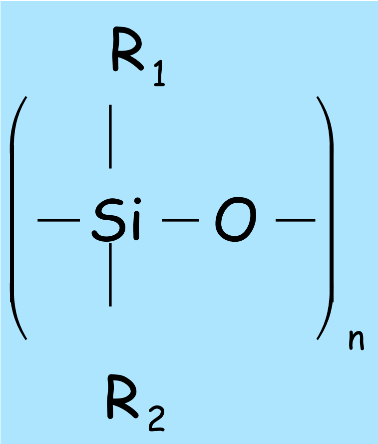
Interest of Polysiloxanes



Ability to interact with compounds acceptors of hydrogen bonds (bases)

	polarisability	polarity	basicity	acidity	dispersion
	c	r	s	a	b	ℓ
PHG	-0,331	-0,979	0,774	1,324	4,269	0,810
PMHS	-0,077	0,139	0,203	1,025	-0,469	0,846
PLG	-0,296	-1,161	1,325	0,971	4,785	0,674
PMTFPS	-0,328	-0,757	1,443	0,112	1,221	0,721
PCPMS	-0,258	0,167	1,480	1,997	0,694	0,674

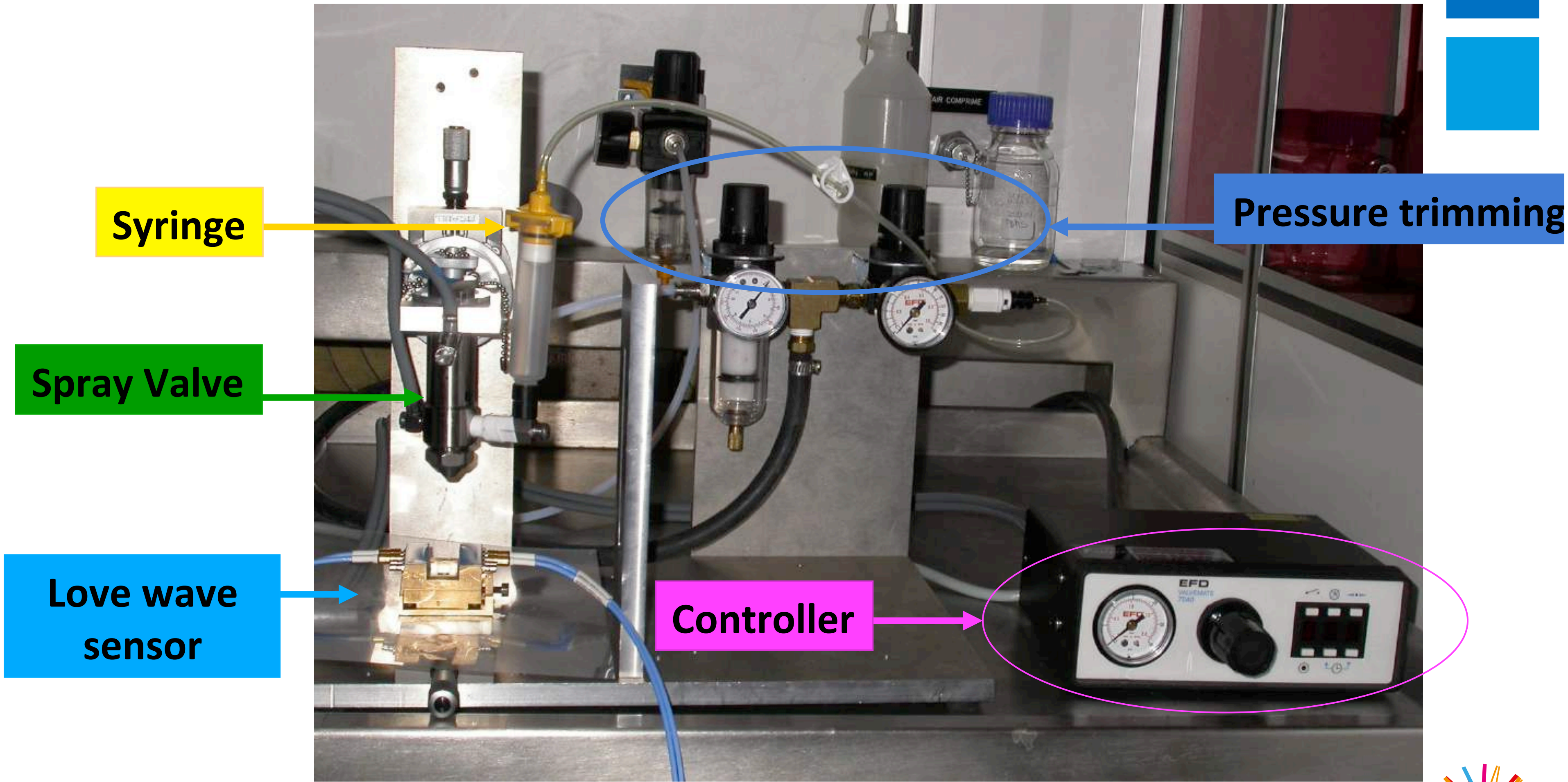
PLG : Polymethylhydrosiloxane **grafting of hexafluorodimethylcarbinol ($C(CF_3)_2OH$) : fluorinated linear polymer**

A good candidate for the detection of organophosphates

Log K	PMHS	PLG	PHG	PMTFPS	PCPMS
Toluène	2,882	2,659	2,813	2,592	3,675
H_2O	0,910	2,650	2,808	1,0280	2,631
DMMP	3,107	7,105	6,775	4,559	4,600

Works achieved by Thales TRT (formerly Thomson, LCR)

Polymer deposition by spray



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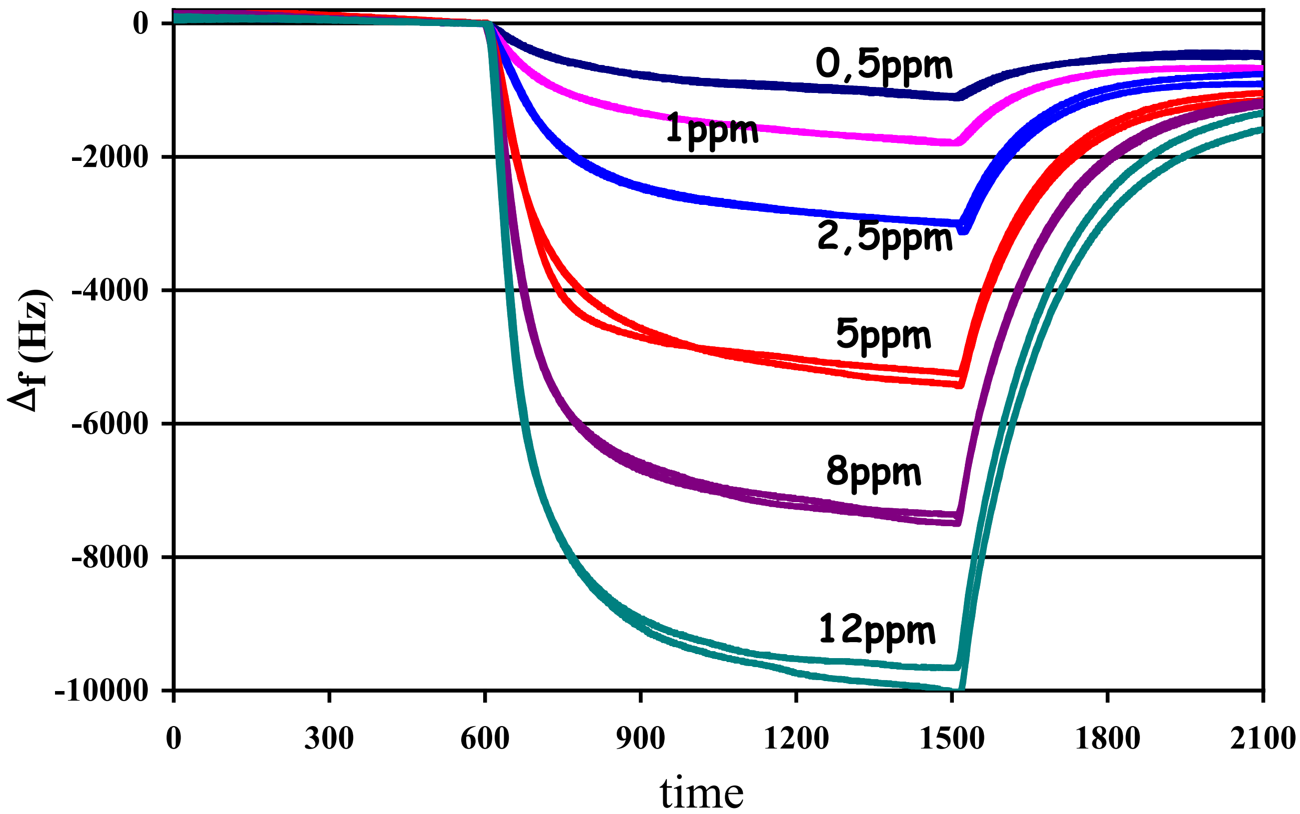
Vapor Detection (GB)



MINISTÈRE DE LA DÉFENSE
DGA

LCR Thalès

f_0 = 109 MHz, SAW Rayleigh wave, Sensitive coating:

- Grafted Polysiloxane PLG, Δf_0 = 138 kHz
  ➔ emphasize hydrogen interactions (sensitivity / reversibility)
  ➔ low glass-transition temperature (diffusion @ ambient T°C)
- Spray-coating ➔ High effective surface

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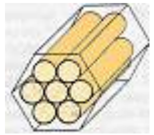
Sensitive material : mesoporous

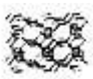
- ✓ Large specific surface
- ✓ Thin film technology
- ✓ Microstructure functionalization

UNIVERSITE PIERRE & MARIE CURIE



Different structures

Porosity Domain



Zeolite

Mesoporous silica

Protonic Crystal

Mesoporous

Microporous ◄······► **Macroporous**

0.1 nm **1nm** **10nm** **100nm** **1µm**

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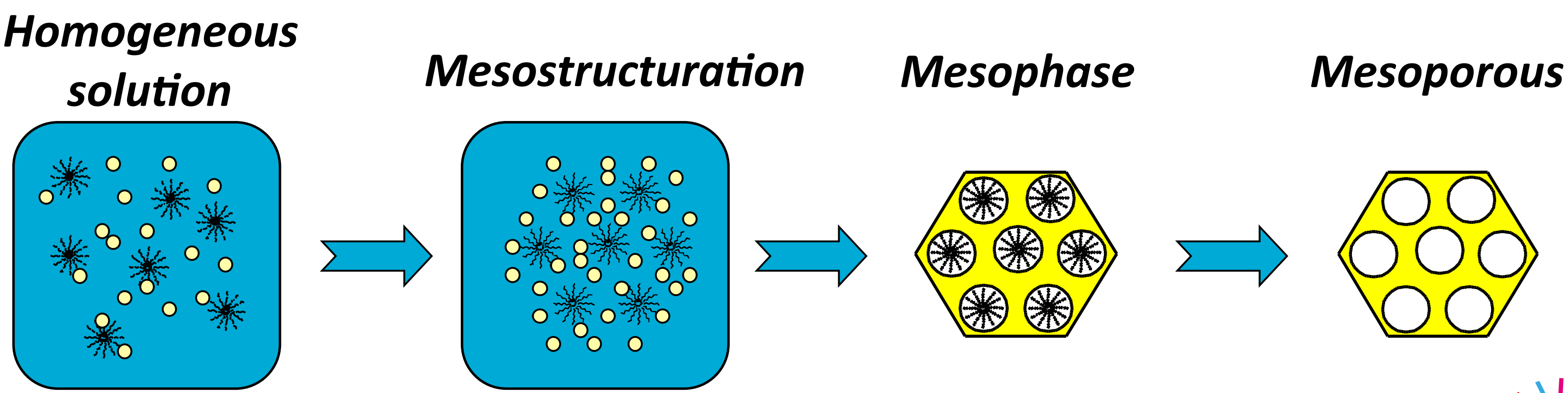
Mesoporous sensitive layer

Chemical sensor = **Delay line** + **Sensitive layer**

MINERAL layer : MESOPOROUS Material

Silica and CethylTrimethylAmmonium Bromide

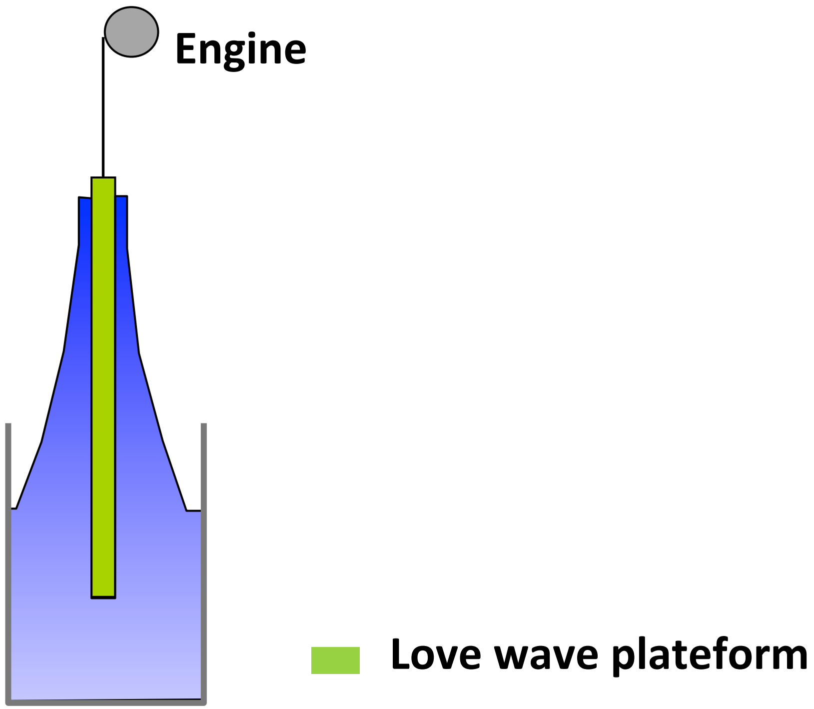
$(C_{16}H_{33}N(CH_3)_3Br$ or CTAB



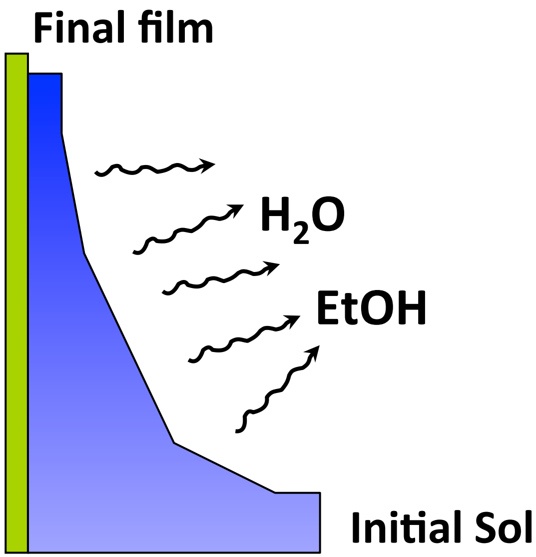
T-T. TRUONG, G. LEDOUX, T-H. TRAN-THI, D. GROSSO, C. SANCHEZ, Benzene chemical sensors based on porous sol-gel materials
The 16th European Conference on solid state transducers, Prague, Czech Republic, pp 1237-1240 (2002)

Mesoporous sensitive layer

1. « Dip coating »



2. Self-assembly by evaporation



- Initial solution : Surfactant (CTBA) + soluble silica + H_2O + EtOH
- Mesostructure: depends on the sol-gel chemistry

(nature of the compound, experimental conditions: Humidity, Temperature)

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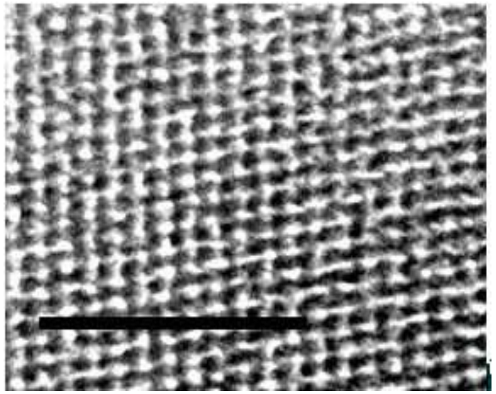
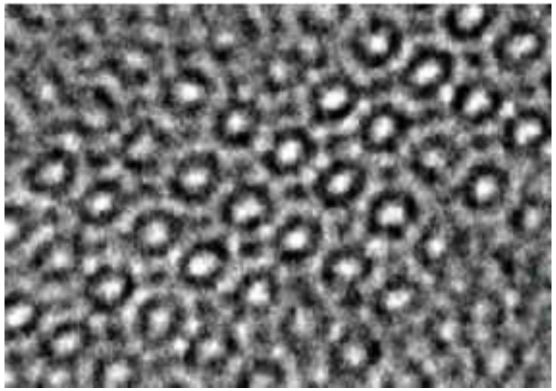
Mesoporous sensitive layer

- **CTAB/silica = 0.14**
- **2 nm mesopores periodic organization**
- **Thickness : 50 – 500 nm**





TEM pictures

- **High apparent surface (>700 m^2/cm^3)**
- **High volume porosity (0.5 cm^3/cm^3)**

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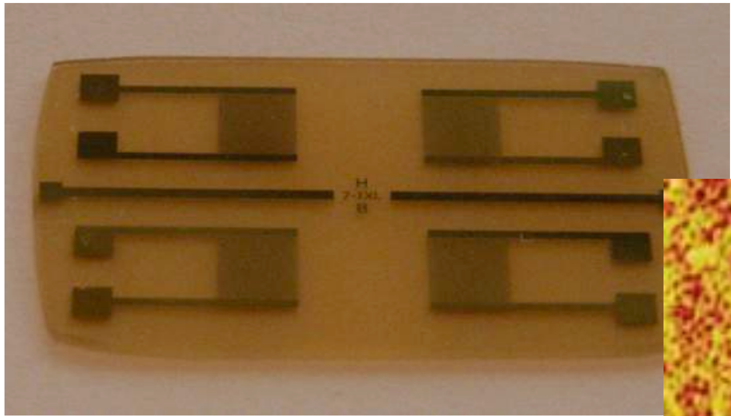
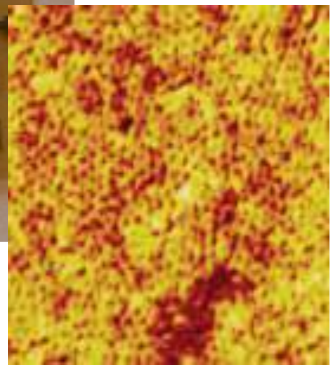
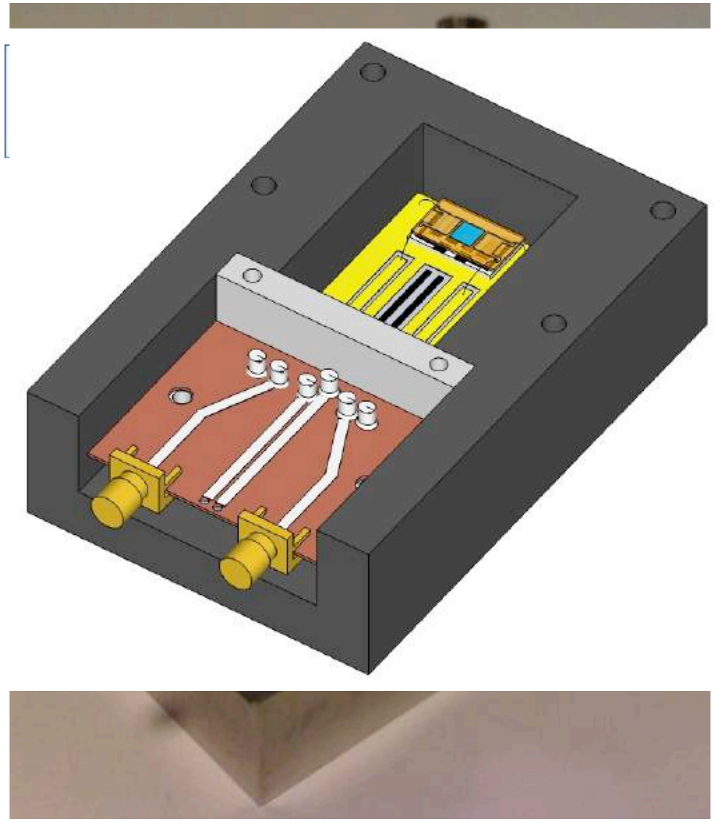
High temperature SAW Platform using LGS
Harsh environmental applications





- SAW delay-line on LGS
- Mesoporous sensitive film
  - ✓ Mesoporous : 2 - 50 nm
  - ✓ Sol-gel technique, spin or dip-coating
  - ✓ Stability: thermal, mechanical, chemical …
  - ✓ High specific surface : 10 - 1000 m^2/g



PhD G.Tortissier 2009

Complete assembly :

• Stainless steel shell

• Inlet/Outlet integrated cover for safe gas detection

• PCB-SMA RF connector

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COLLÈGE DE FRANCE 1530

RENAULT

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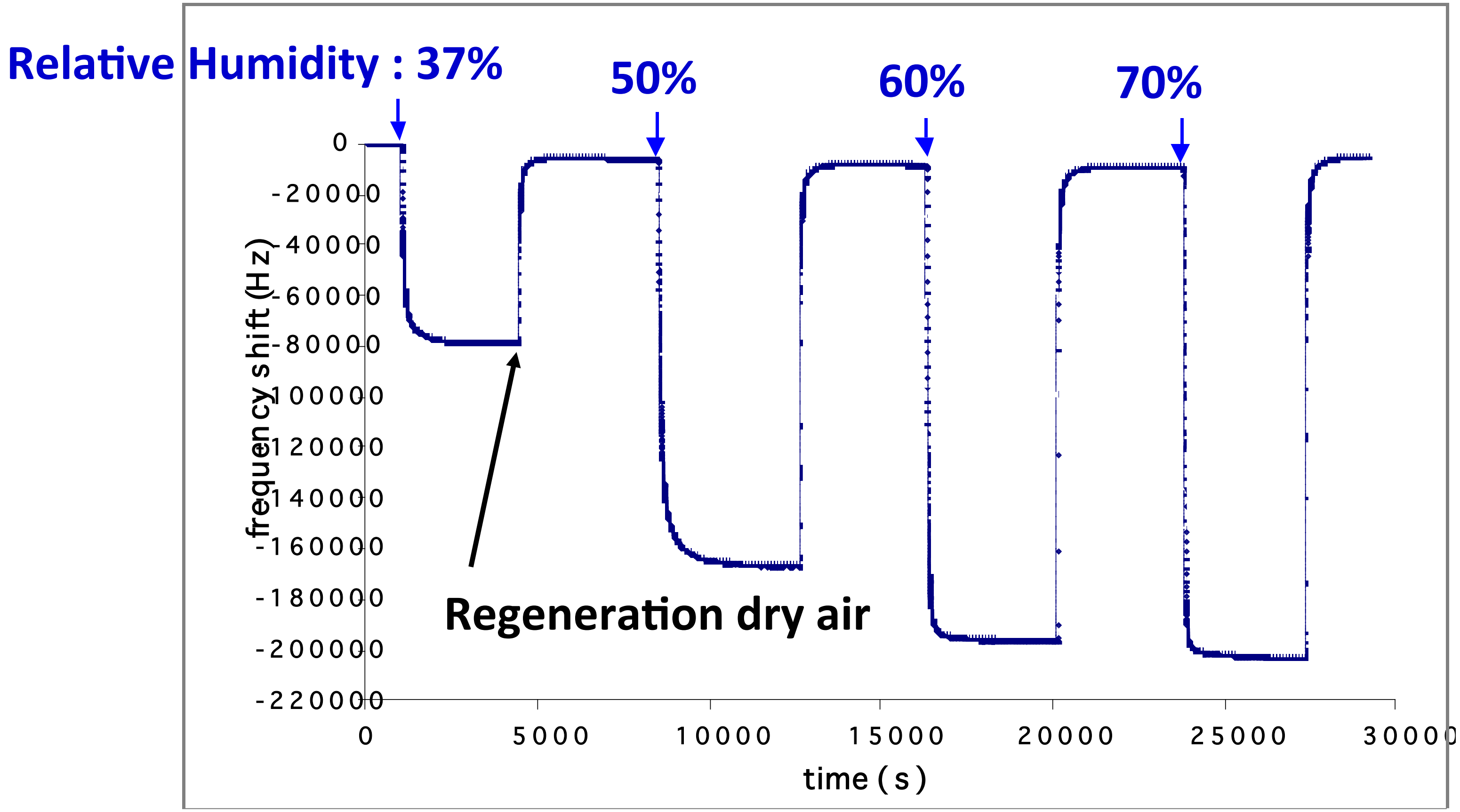
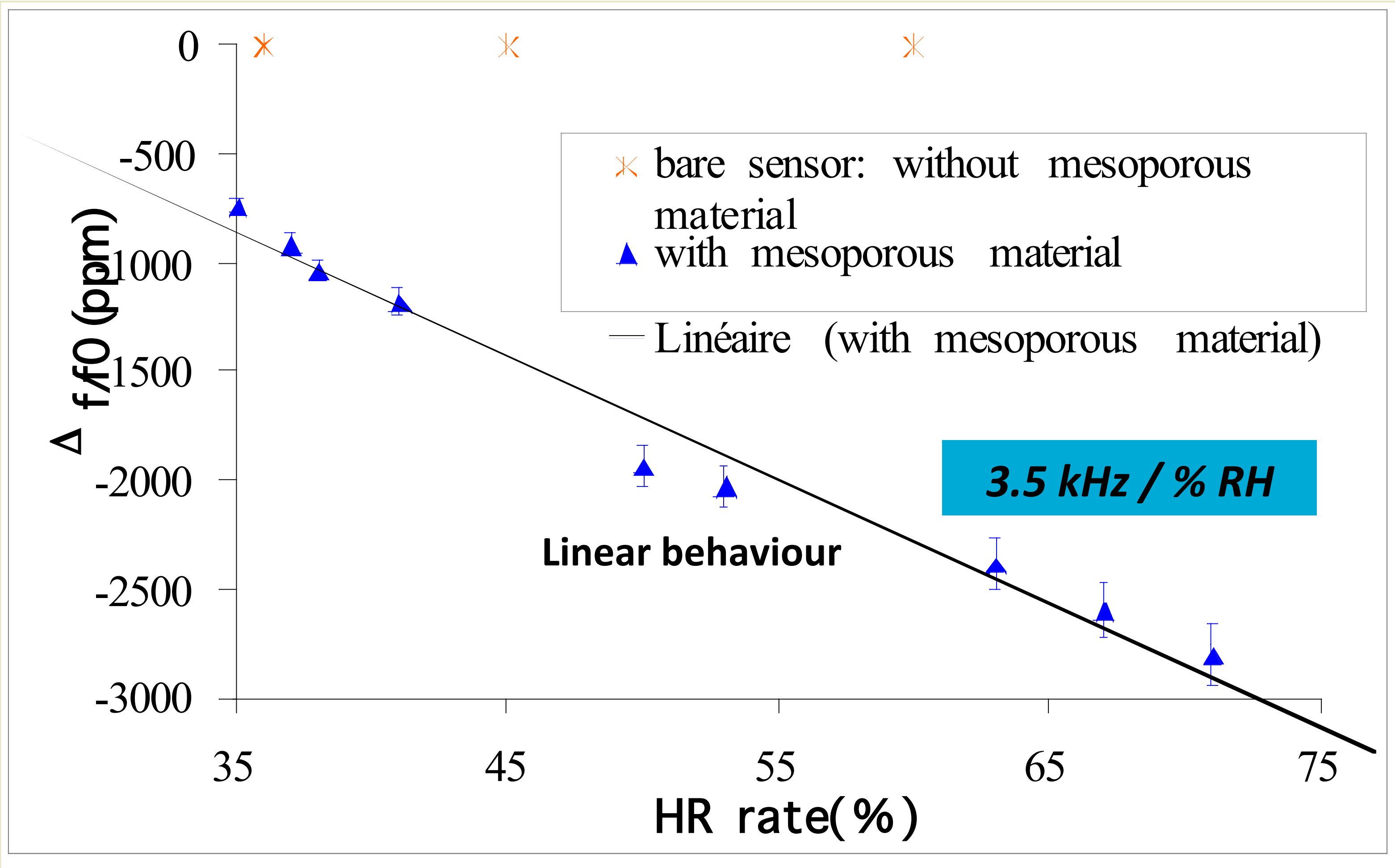
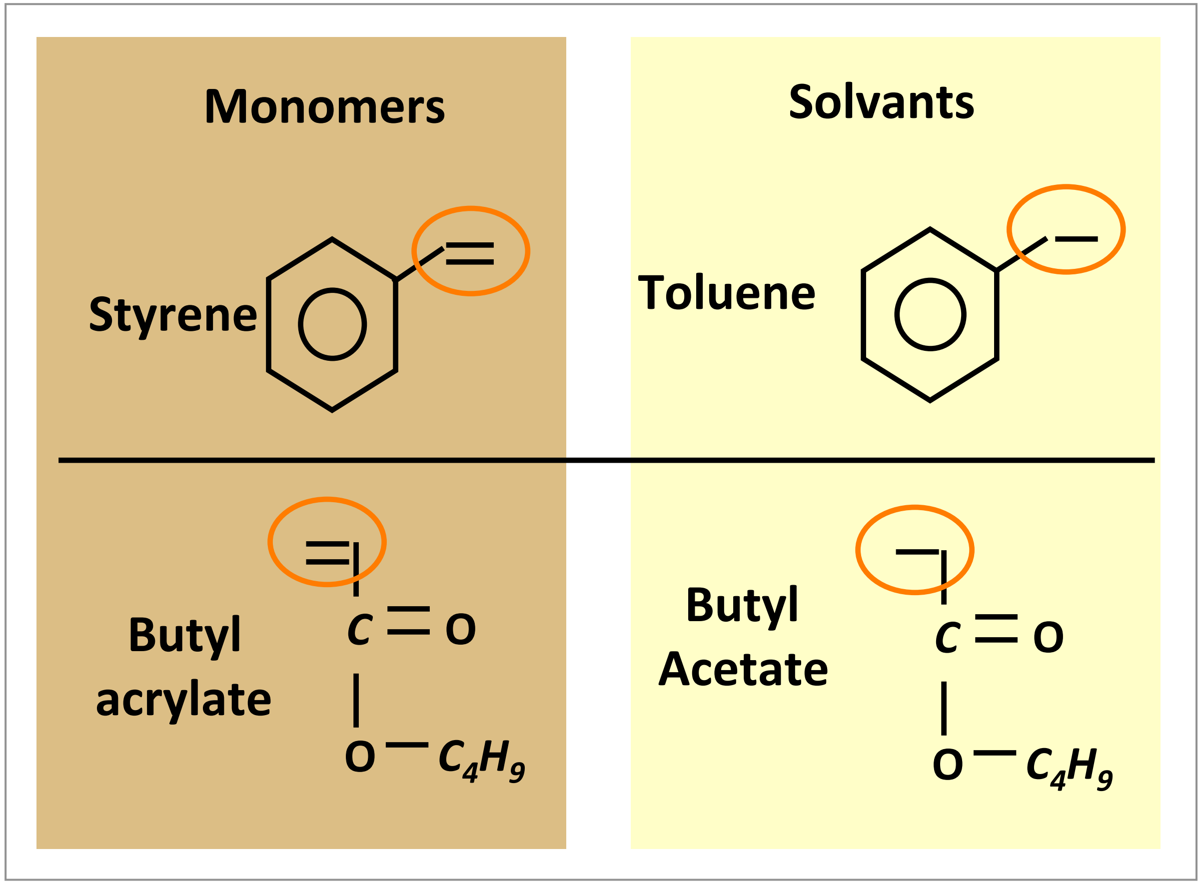
Mesoporous sensitive layer



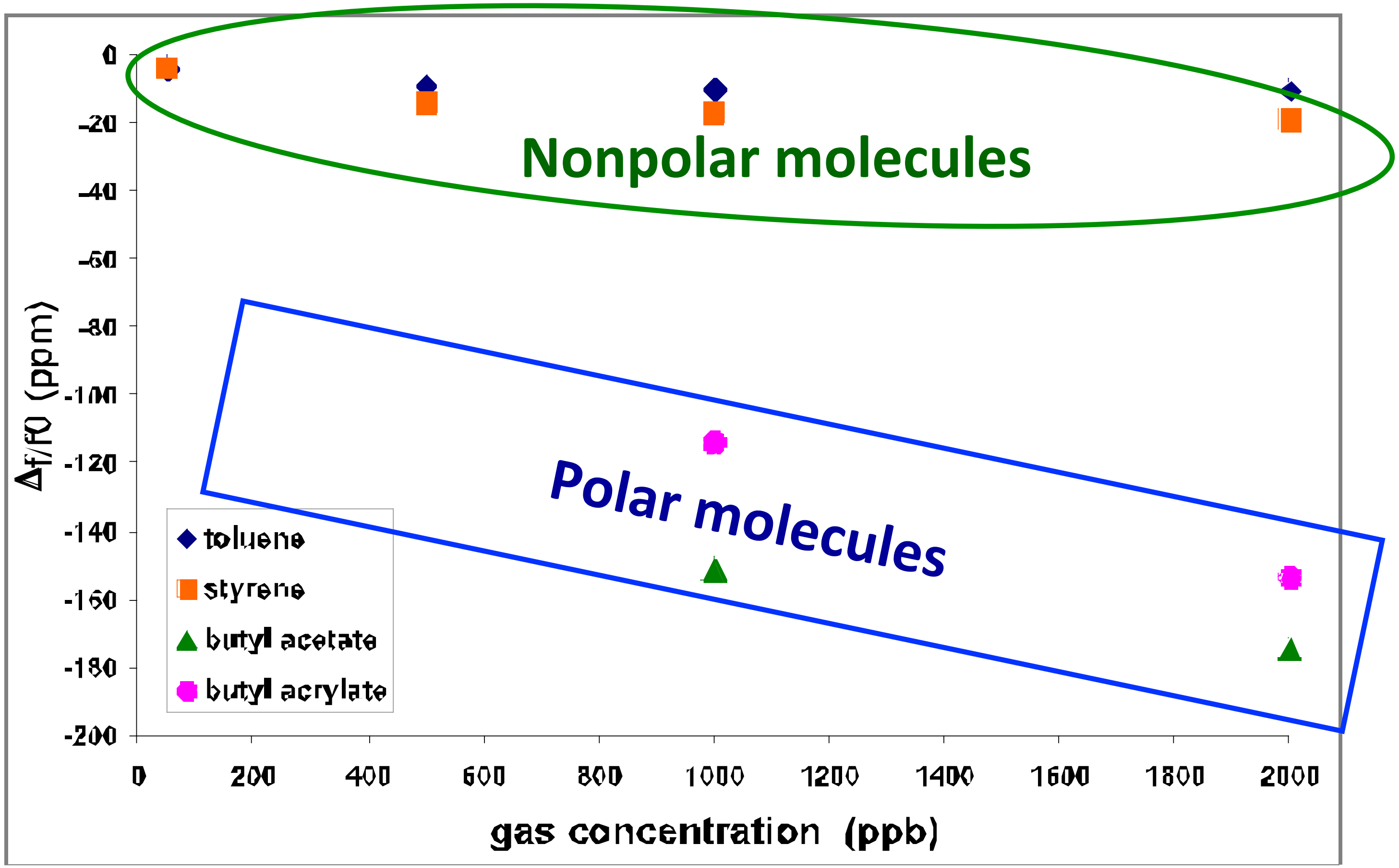
Sensitivity to humidity



Solvant and monomers discrimination



Solvant and monomers discrimination



Polar & Non polar molecules separation in real time

Future : specificity by functionalization of the pores

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SAW transducers: Gas sensing with carbon-based materials

LAAS-CNRS Toulouse (France)

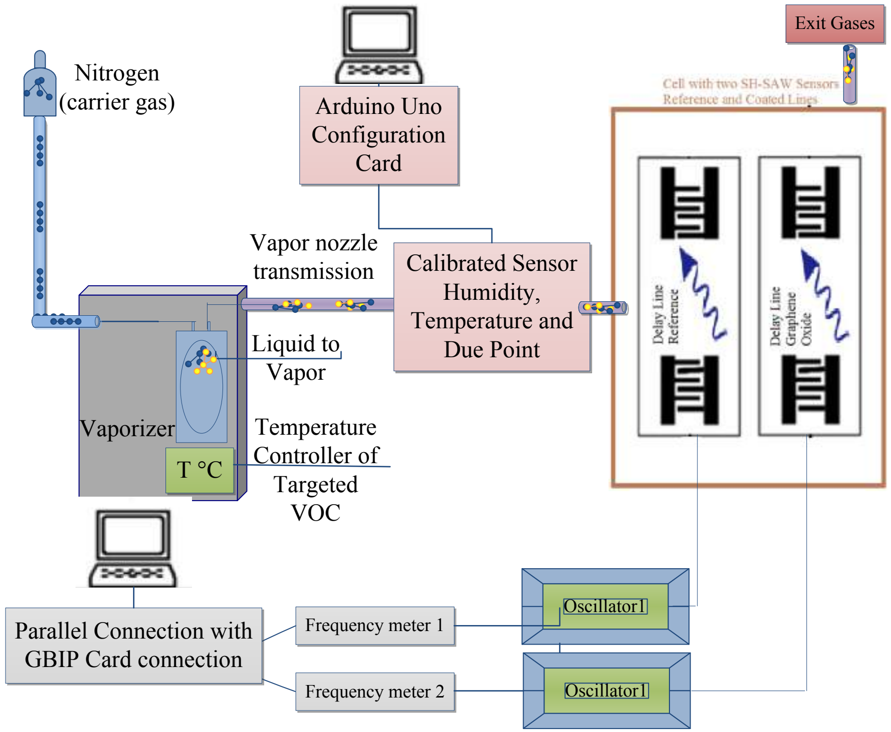
Instituto de Tecnología Química Valencia (Spain)

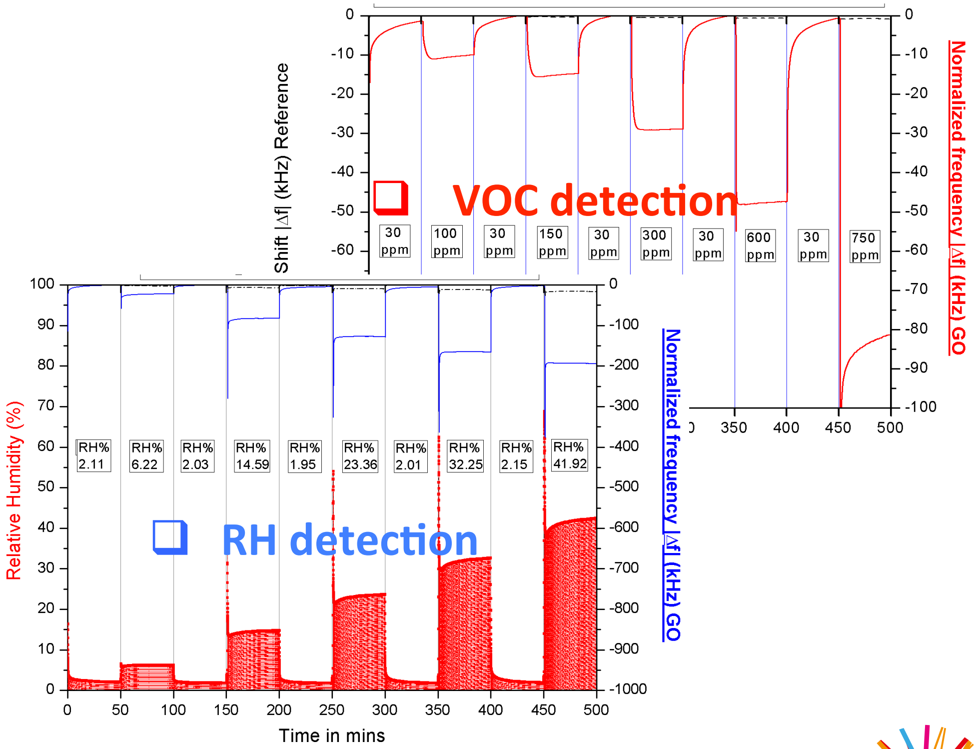
FORTH Institute of Electronic Structure and Laser Crete (Greece)

Graphene oxide on Love wave sensor

Real time detection of Ethanol (VOC) and humidity (RH)

PhD I.Nikolaou, on-going





I. Nikolaou, H. Hallil, G. Deligeorgis, V. Conedera, H. Garcia, C. Dejous, D. Rebière , Novel SH-SAW gas sensor based on Graphene, SPIE Microtechnologies, Paper 9517-38, 2015.

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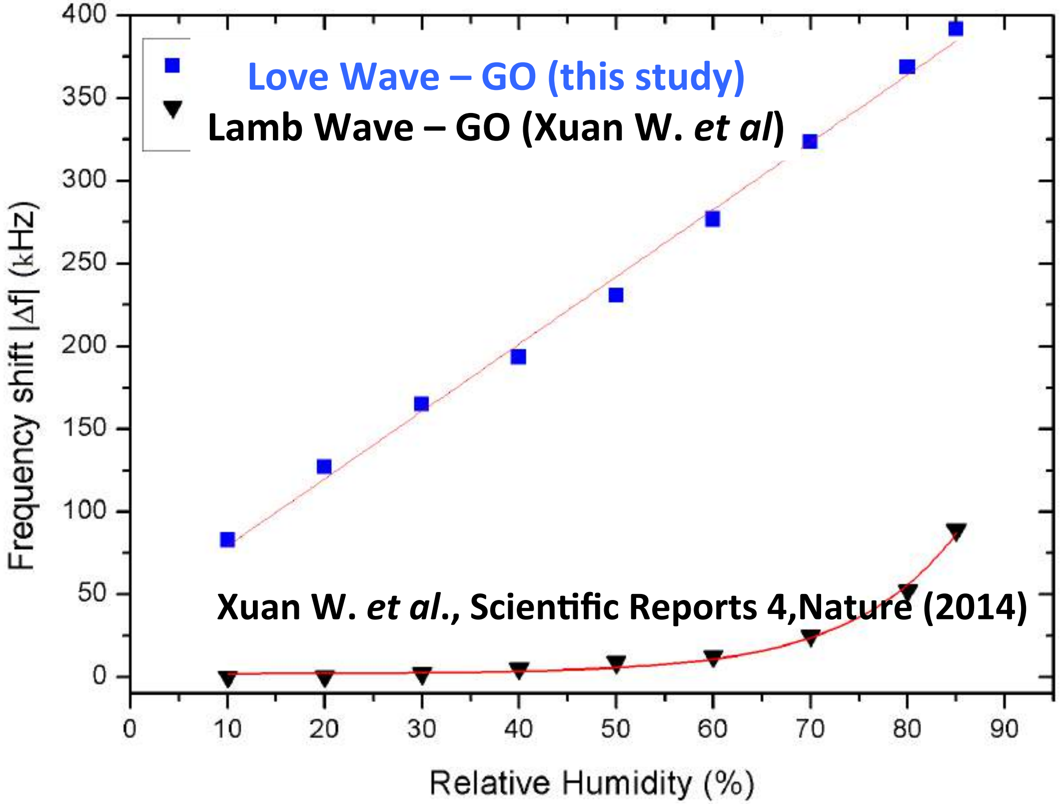
SAW transducers: Gas sensing with carbon-based materials

Evaluation of the Love wave sensor: Humidity detection

Comparison of Graphene Oxide based-devices (Lamb sensor in Xuan W. *et al* study) with similar conditions under Relative



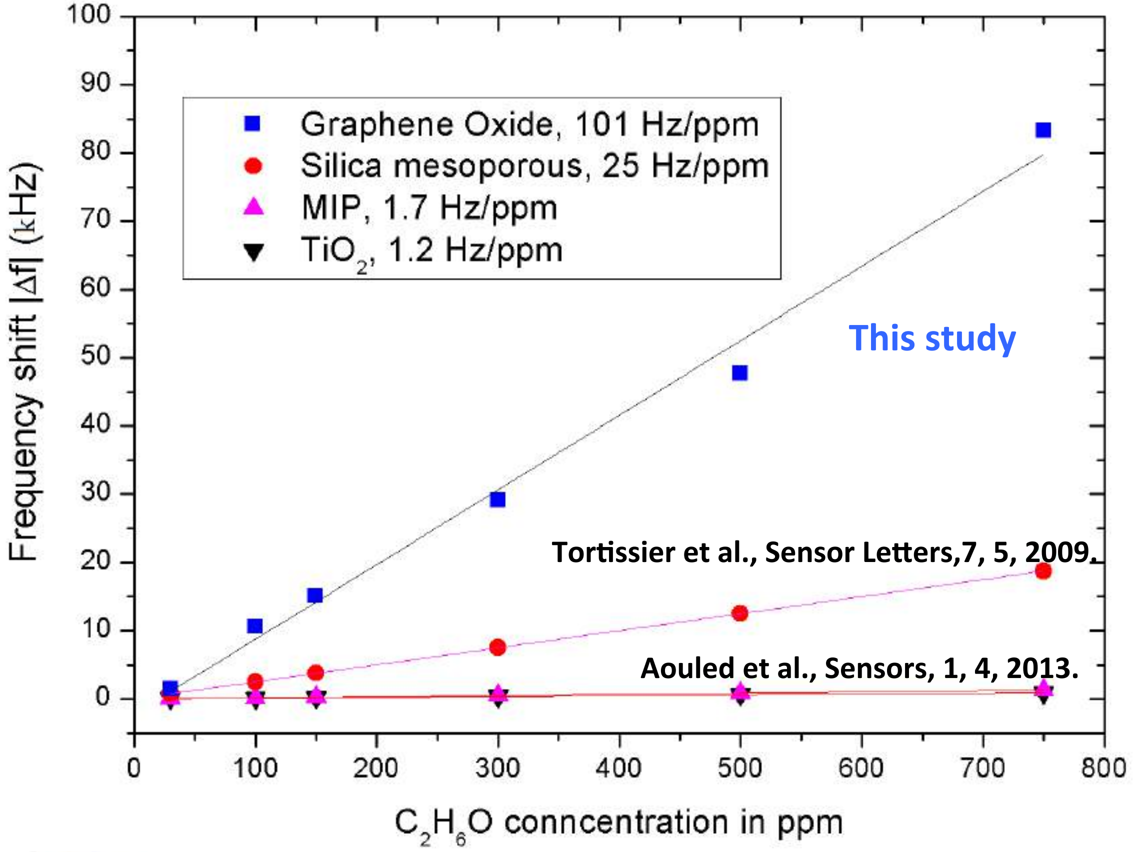
GO-LW Device:

- \+ Sensitivity
- ++ Low RH% !

SAW transducers: Gas sensing with carbon-based materials

Evaluation of the GO-LW: Ethanol as OCV

Comparison of Graphene Oxide with alternative functional materials under C_2H_6O vapors



Sensitivity with GO compared to:

- Silica mesoporous x4
- MIP x50
- TiO_2 x100

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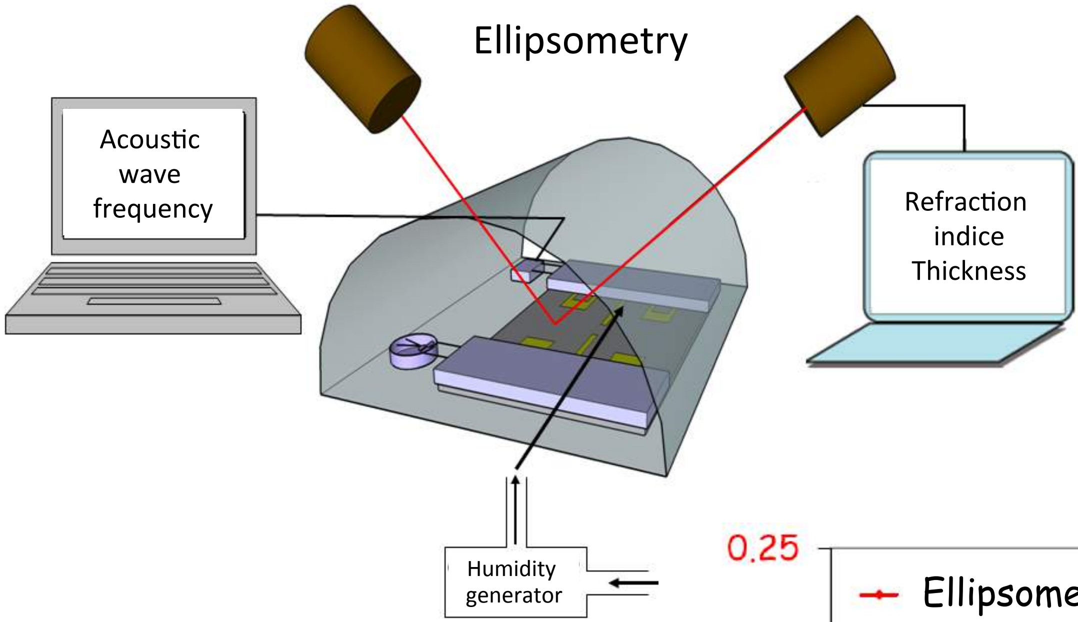
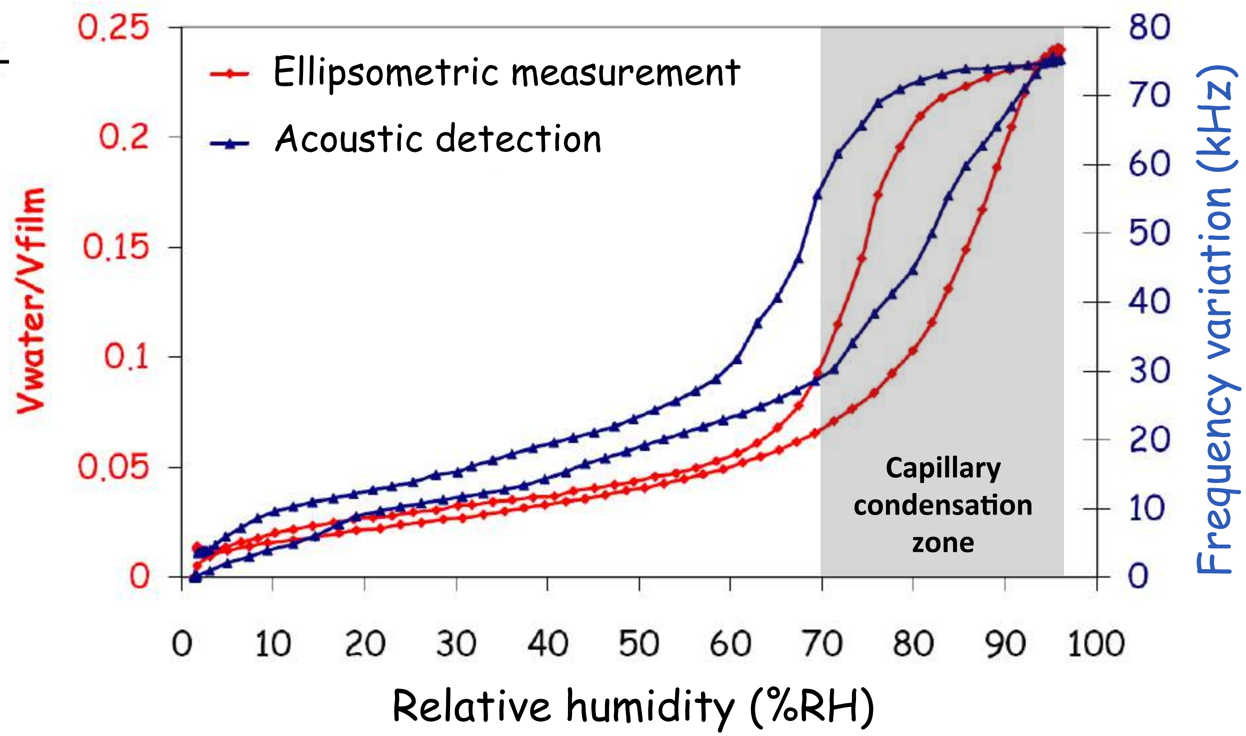
Mesoporous thin film characterization



Ellipsometry and acoustics
- **adequacy**
- **complementarity**

Mesoporous film characterization
- **Isotherms of type IV (BET)**
- **Porosity : 25%**
- **Capillary condensation at 70% relative humidity**



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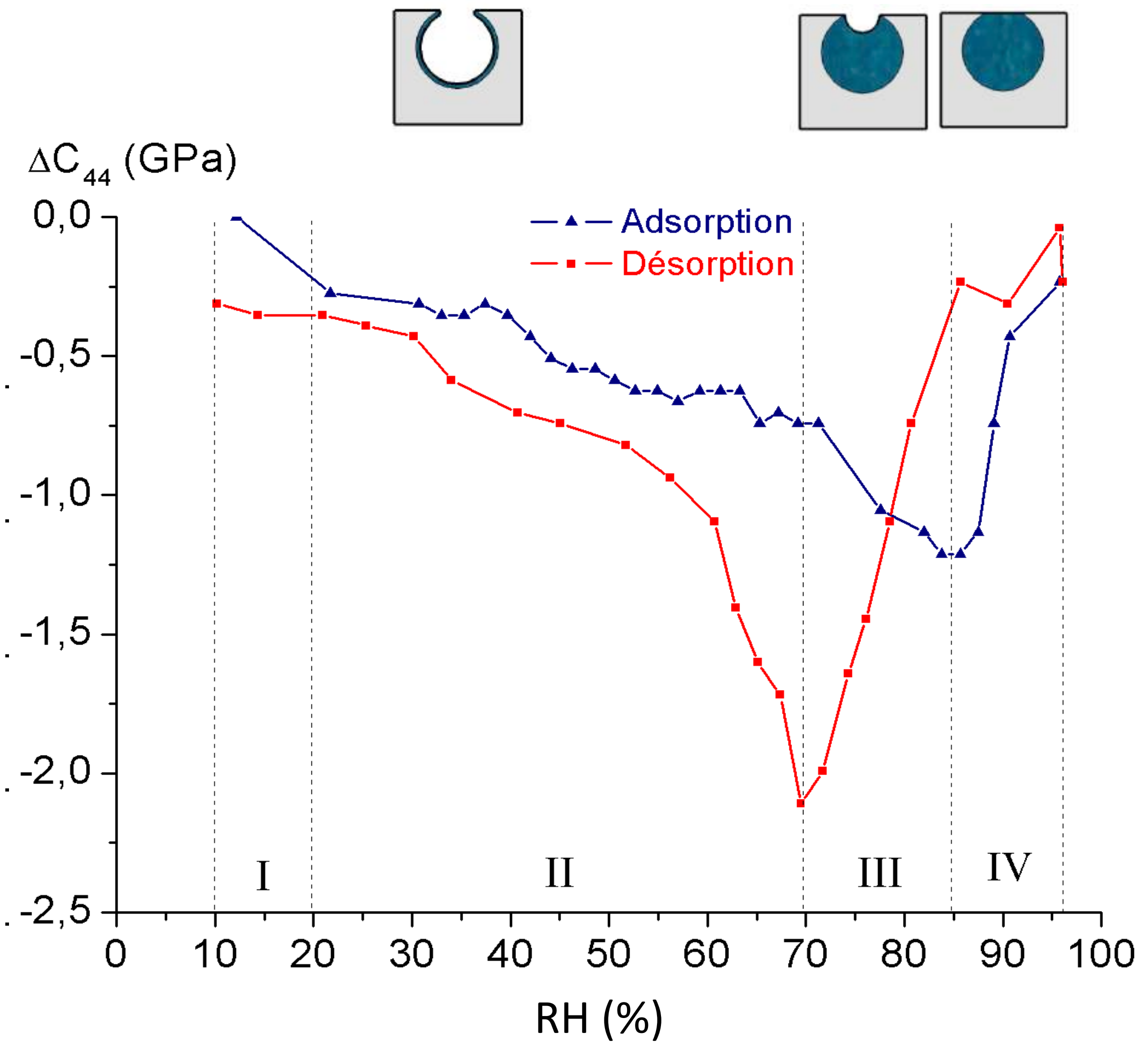
Film characterization: mesoporous TiO_2

Young modulus vs. relative humidity

L. Blanc – PhD, 2011



I : Micropores (< 2 nm)

II : Film of water

⇨ Adsorption of water in grains boundaries

⇨Stress relaxation

III : Sharp pore filling

⇨ Capillary condensation

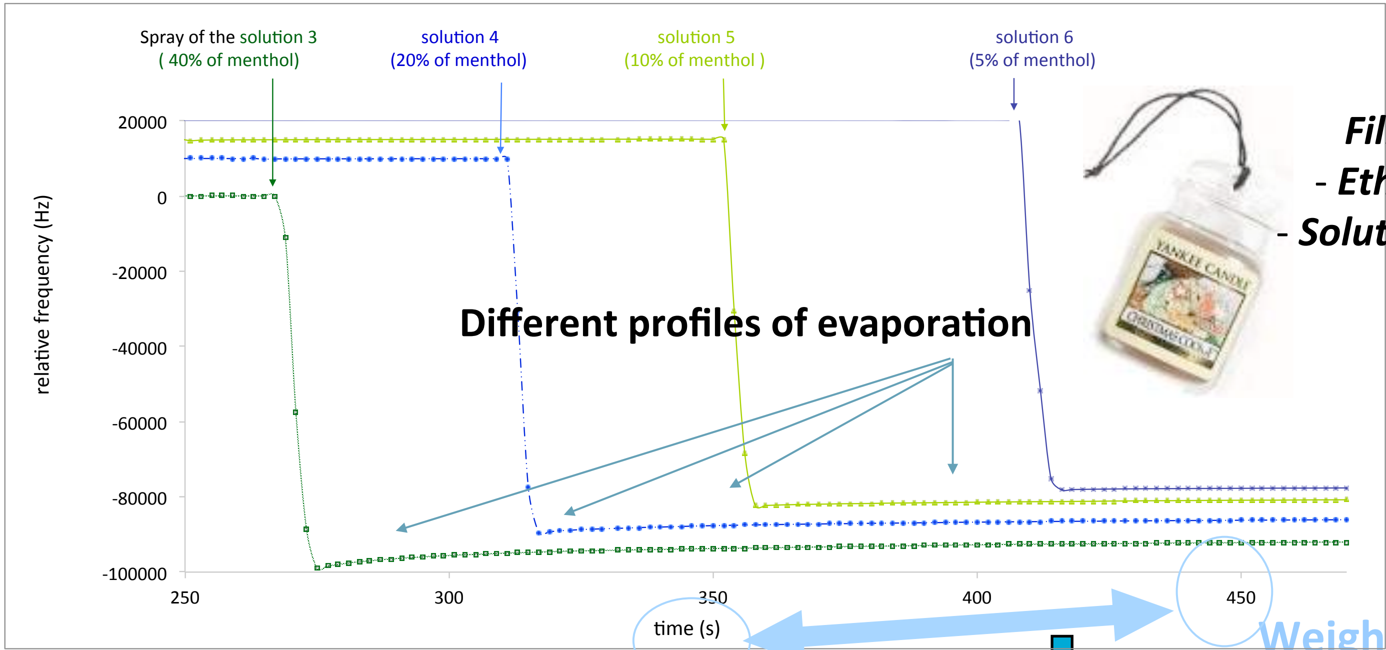
IV : Filled pores

⇨ Disappearance of the curvature of liquid meniscus

⇨ Decrease of ΔP

⇨ Starting value

Kinetics – Monitoring of menthol evaporation

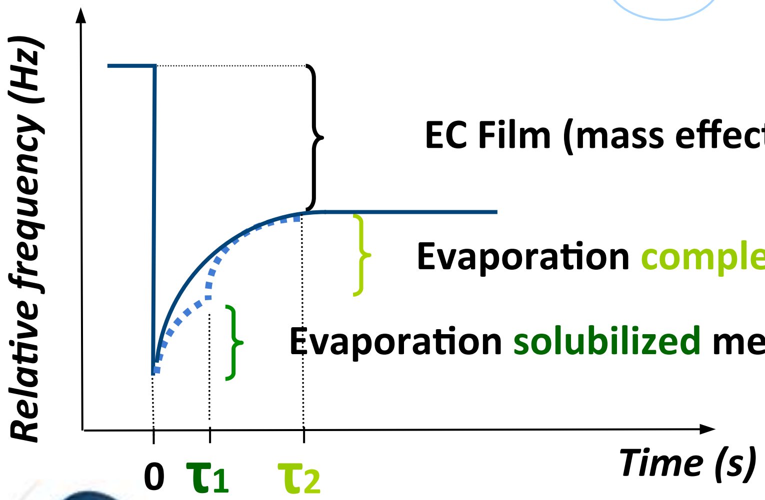


Film on SAW device:
- Ethyl Cellulose Matrix
- Solution menthol + solvent

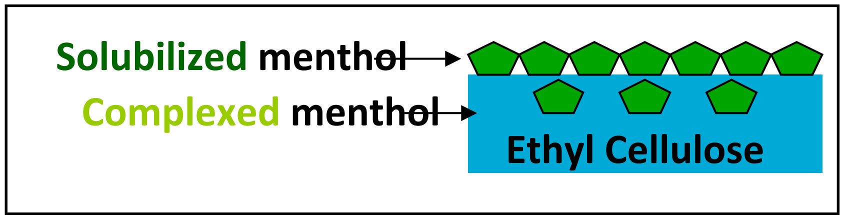
Rhodia

Few minutes

…Classic method :
Weighing film 100µm, …weeks



$$f = F\left(1-(1-\alpha)e^{-t/\tau_1} - \alpha e^{-t/\tau_2}\right)$$



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D. Monin, M. Joanicot, C. Dejous, D. Rebière, F. Razan, Procédé de détermination de la variation de masse d'un système chimique, procédé de criblage comprenant un tel procédé de détermination et installation correspondante, FR2887631, 2006-12-29

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Environmental and health related applications

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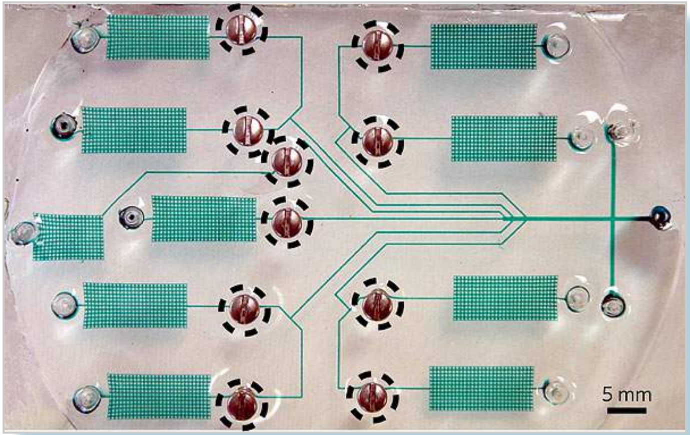
  - Polymer Optical Microring Resonators & Digital Microfluidics
- Conclusion, perspectives

Acoustic Waves & Microfluidic

Interest of both…



Weibel, D. B. et al. - Anal. Chem. 77, 4726–4733 (2005).

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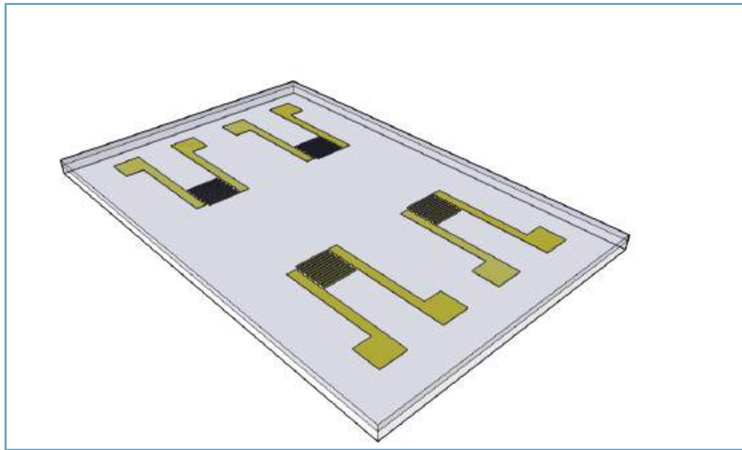

- **Microfluidic**
  - ➲ **Small volumes**
  - ➲ **Parallelization**
- **Acoustic platform**
  - ➲ **High sensitivity**
  - ➲ **Microelectronic fabrication process**

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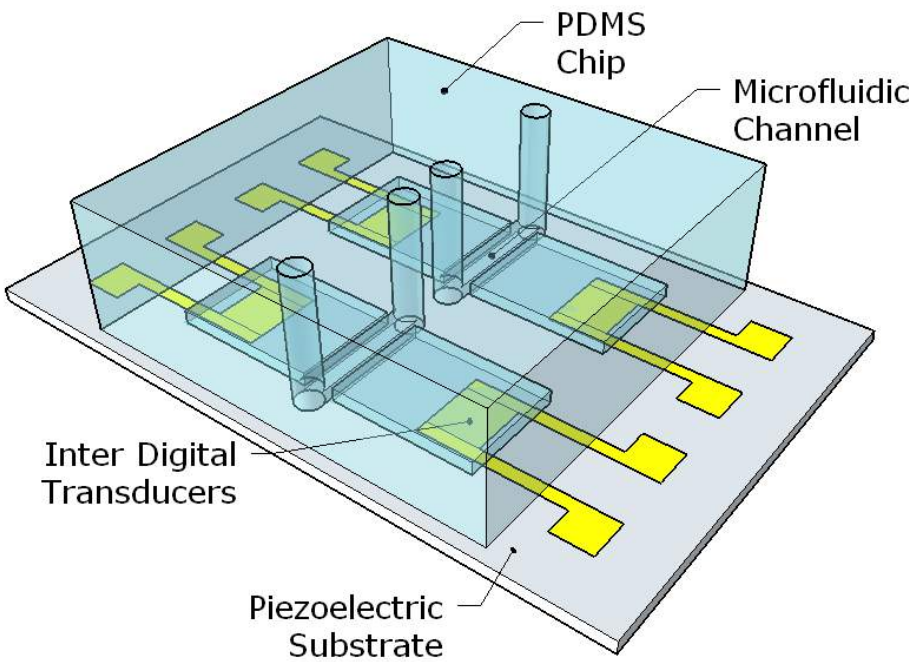
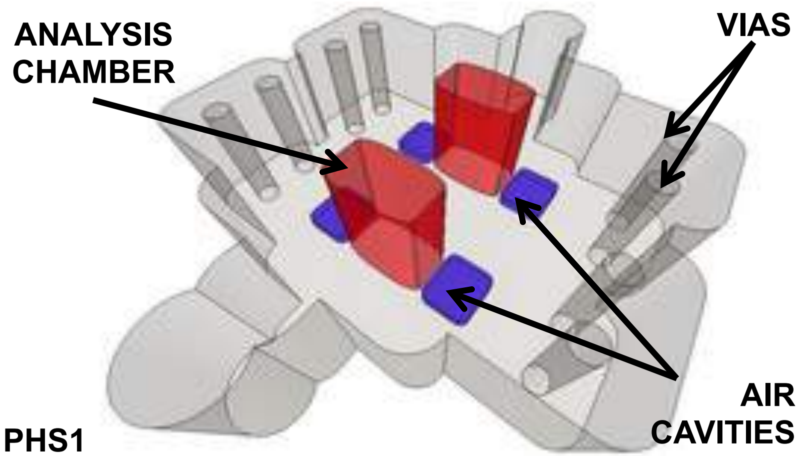
PDMS Microfluidic chip

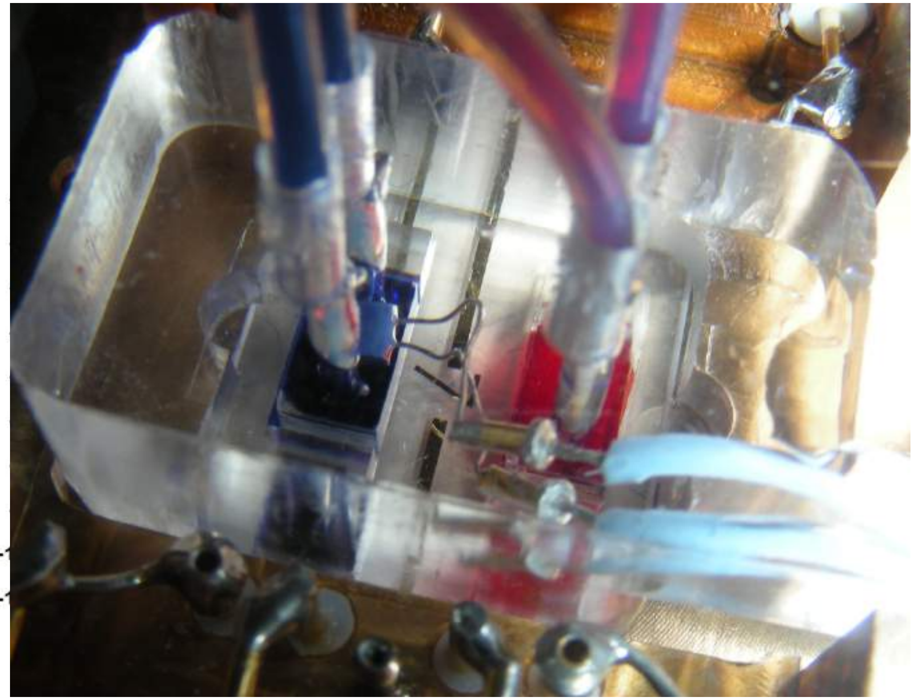


➡ Microfluidic chip

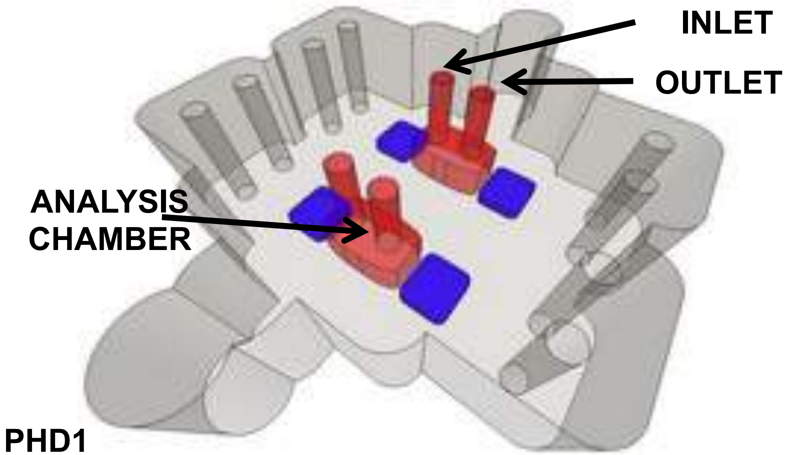
- ❑ Isolates transducers
- ❑ Decreases liquid volume (< µL)
- ❑ Limits acoustic losses
- ❑ Minimizes acoustic reflections
- ❑ Low cost and green technology

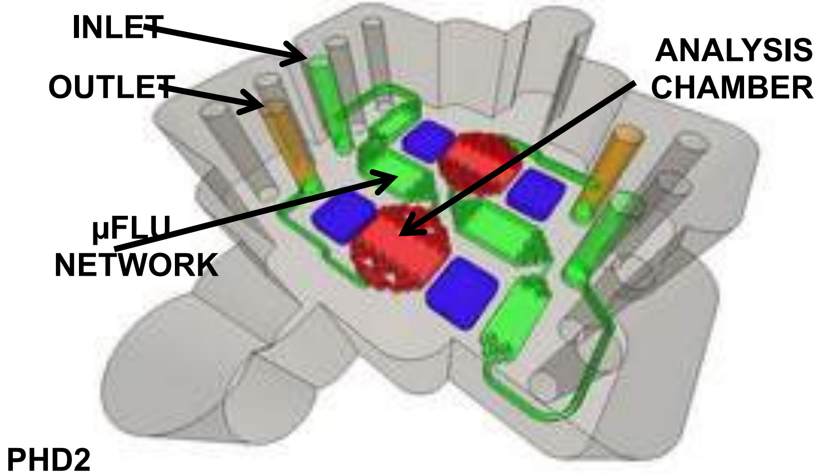
Hydrostatic configuration

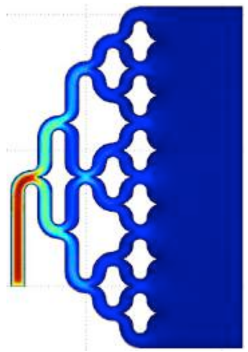




Hydrodynamic configuration







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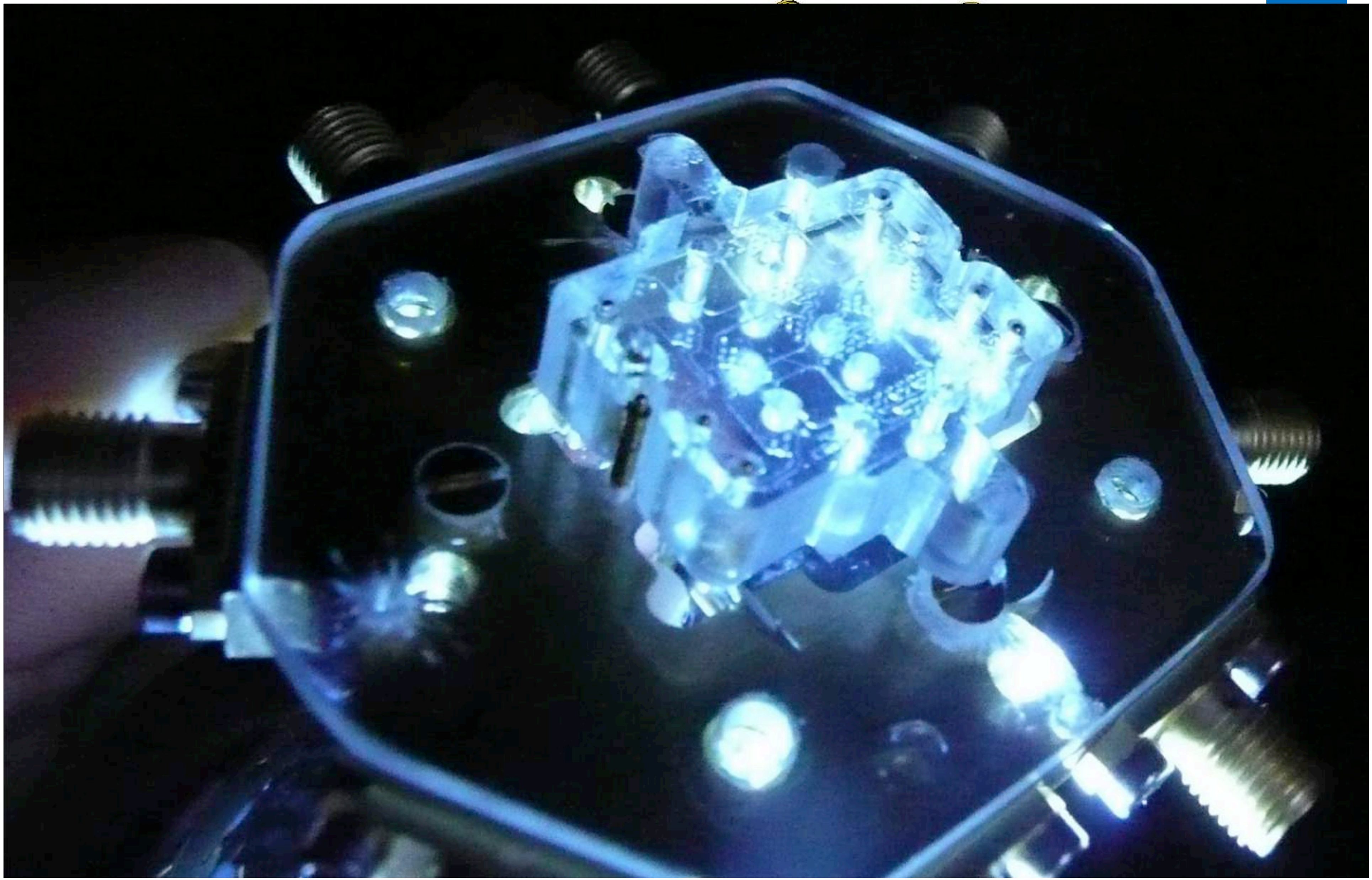
Biological Micro organisms detection



Biological Micro organisms detection

- Toxins (shellfish Quality)
- Bacteria *Escherichia Coli*
- Heavy metals
- Cancer biomarkers : nucleosides

Target species

◆***Bacteria***

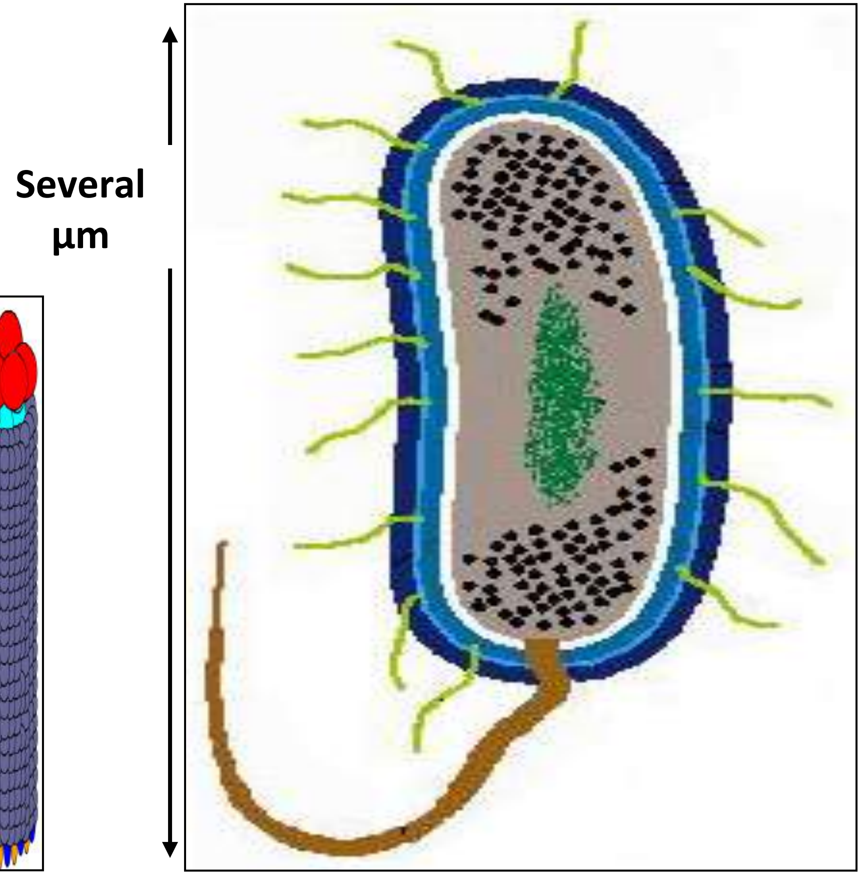
Unicellular micro-organismes, 1 à 5 mm
Simple structure with no nucleus
Self-reproduction by cell division
***ex. : coliforms, streptococci,* bacilli**

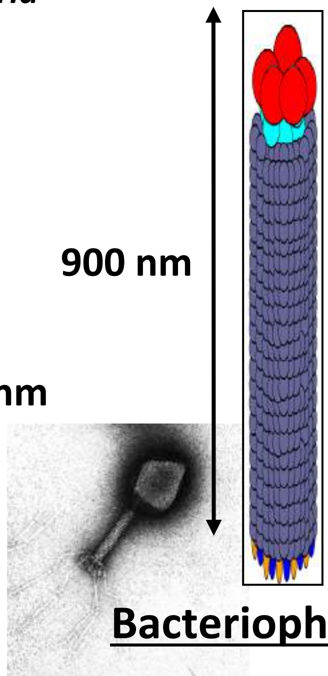
◆***Virus***

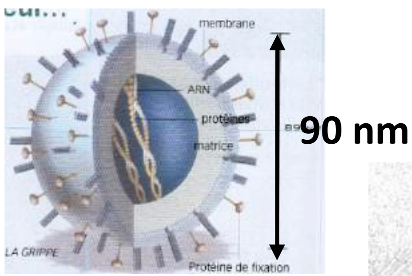
Intracellular parasitic micro-organisms, 20 to 300 nm
Bacteriophage : virus infecting bacteria

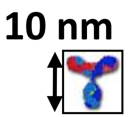
◆***Small molecules***

Hormones, toxins, proteins, …
Mass 1/1000 à 1 that of an antibody









Antibody **Virus** **Bacteriophage** **Bacteria**

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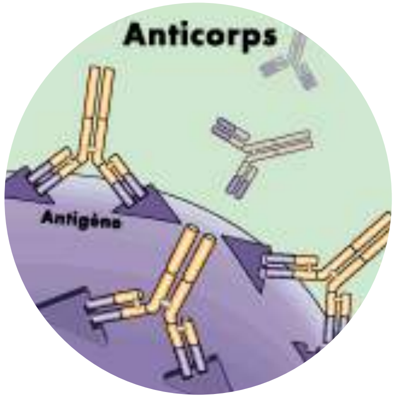
Current methods of biosensing

- Physico-chemical : extraction, separation, detection

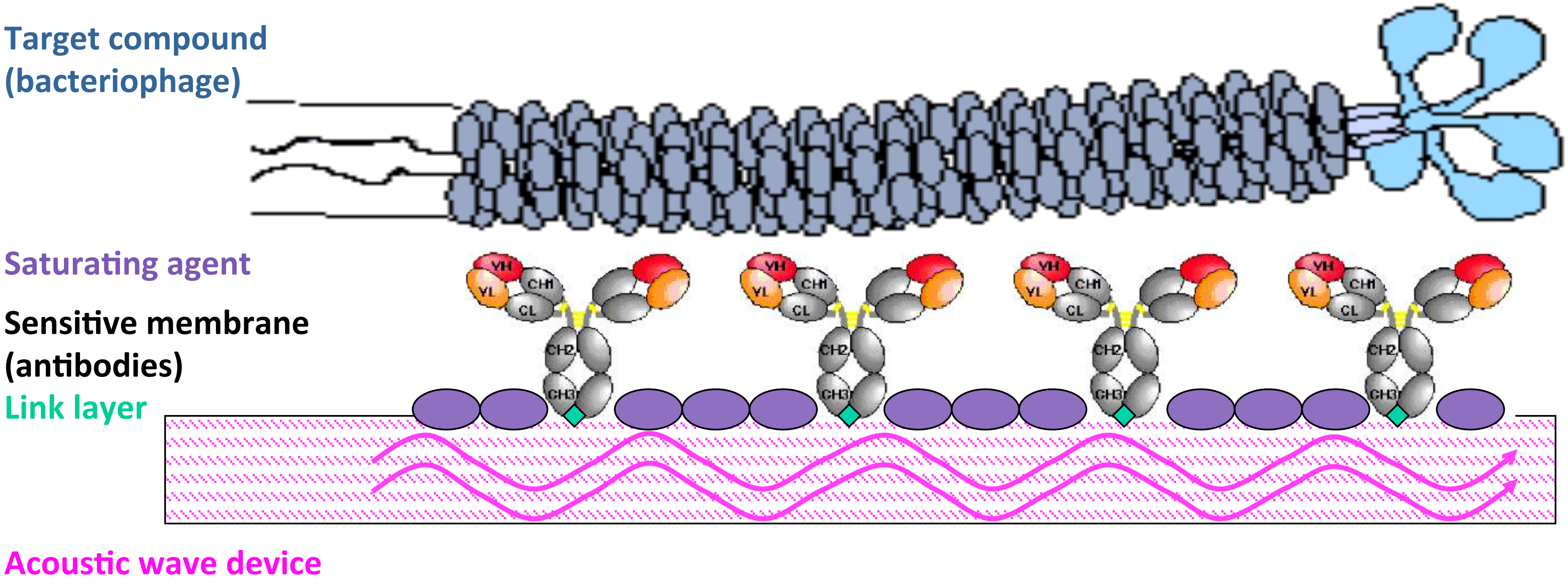
  → *long, expensive, low sampling*

- Immunological : antigen – antibody reaction
  - Antigen
    - immunogenic
    - antigenic
  - Antibody (Immunoglobulin, IgG)
    - synthesized by the immune system
    - specific antibody-antigen binding, neutralization
    - Generic shape: fixed part, variable ends



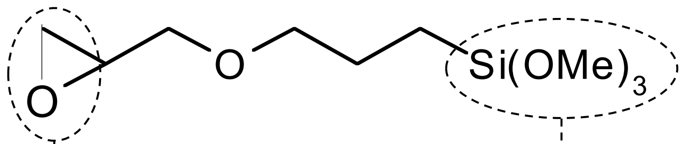
→ *specific, sensitive, reproducible, parallelism*

Surface Acoustic Wave Biosensors for Real Time Immunodetection



Surface Functionalization

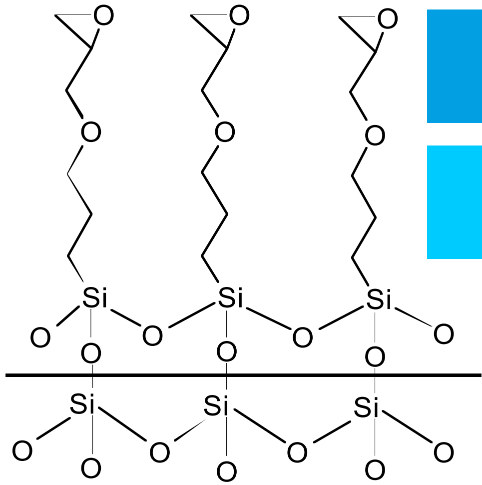
(3-glycidoxypropyl)trimethoxysilane (GPTS)



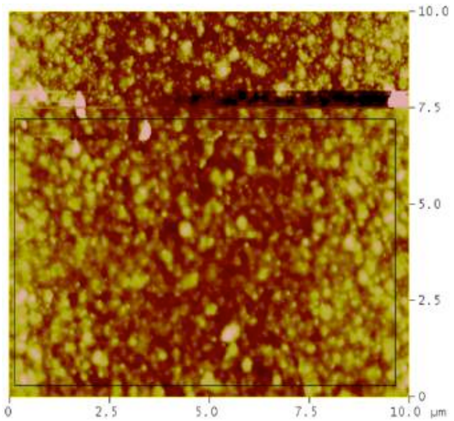
Epoxy end: link with amine functions of antibodies

Hydrolyzable End: bonding with silanol on the sensor surface

Monomolecular film

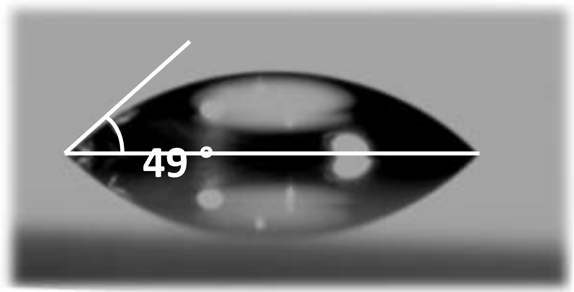


Substrate



AFM image of the sensor surface (SiO_2 – PECVD deposited)

Roughness : Rq ~ 8nm (~ antibodies)



Contact angle 49°

Current: new nanostructured materials, new silylated coupling agents, surface characterizations

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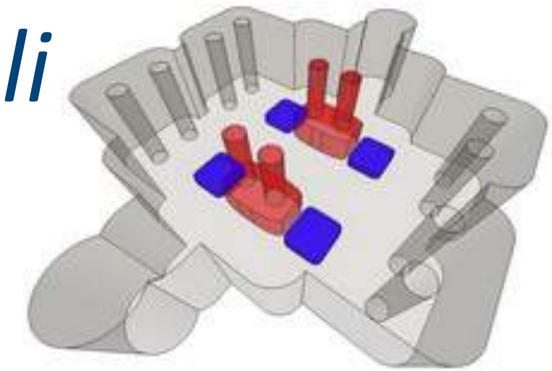
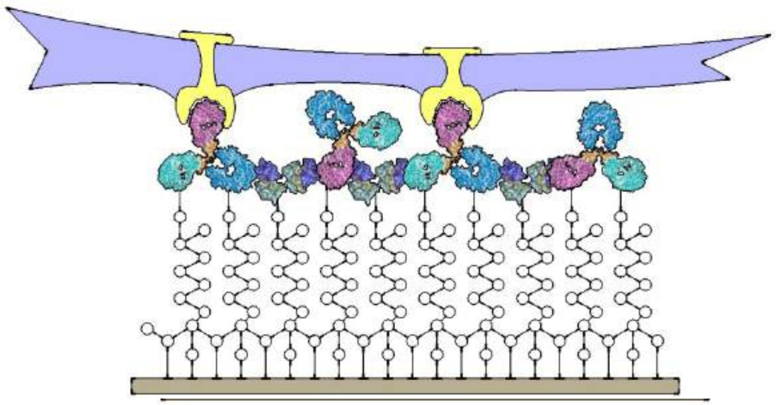
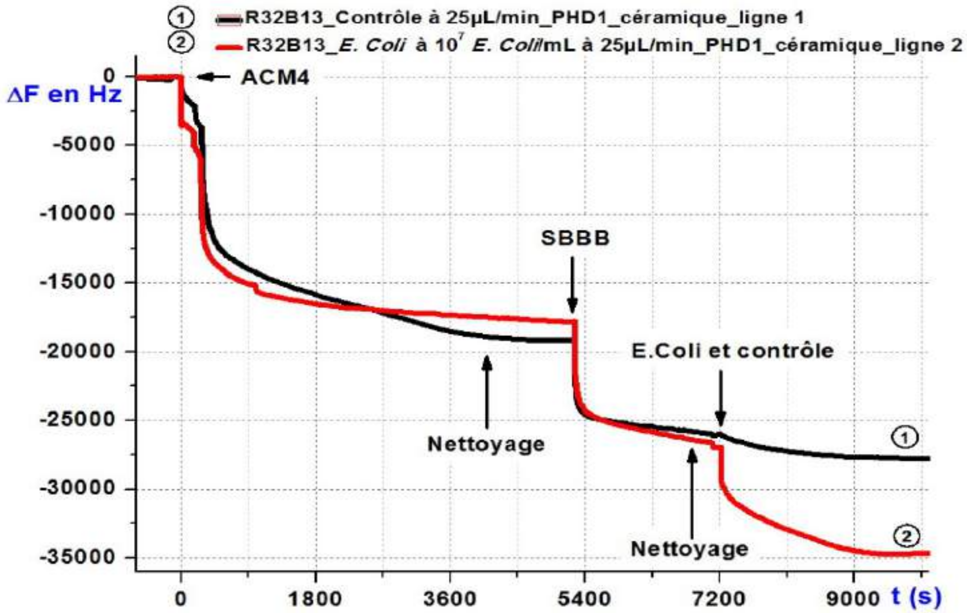
Immunodetection : *E. Coli*

Thèse H.Tarbague 2011







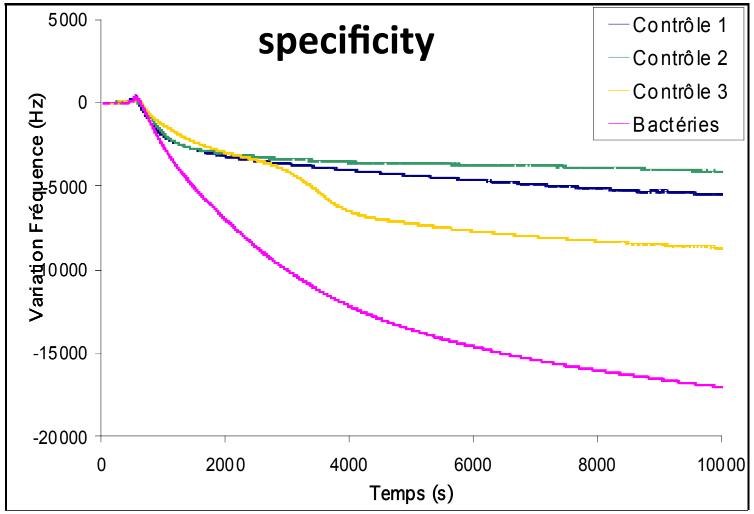
Biofunctionalization

Bioreceptive antibodies

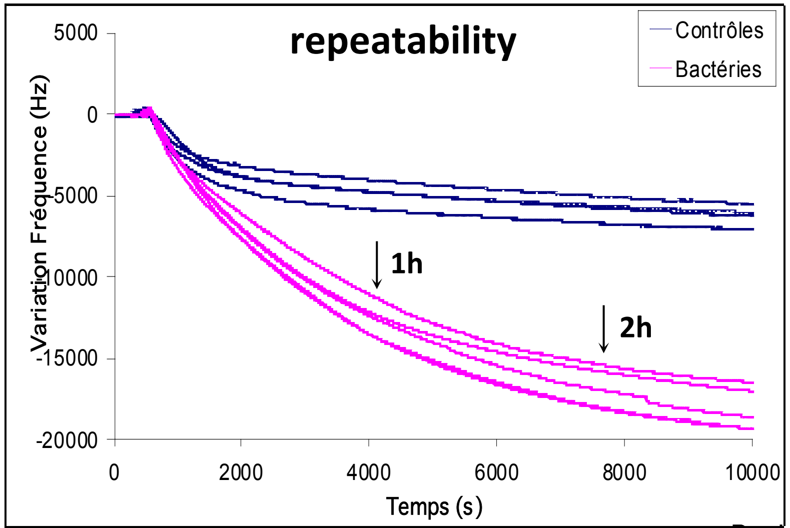
Saturating agent

Application to detection of bacteria
Evaluation of characteristics

- **Specificity**



Controls :
1 : Without bacteria
2 : Nonspecific antibody
3 : Nonspecific bacteria



- **Repeatability**
- **Duration of detection**
  - ✓ **Total duration: 3h**
  - ✓ **Significant duration : 1h**
- **Detection threshold**
  - ✓ **Estimated at 10^6 bactéries/mL**

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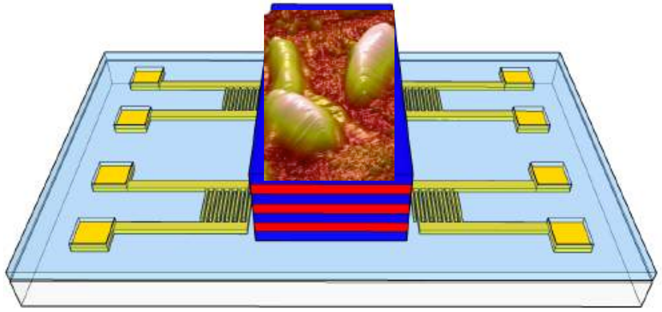
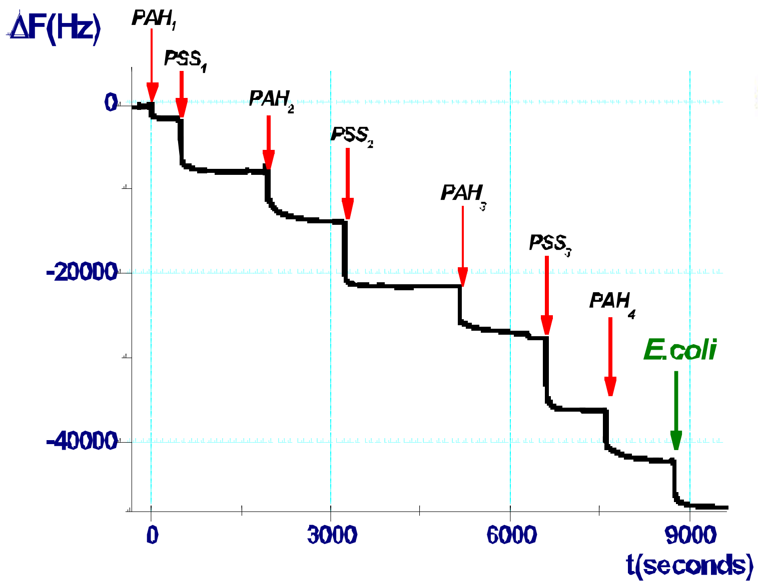
Application : Heavy metals



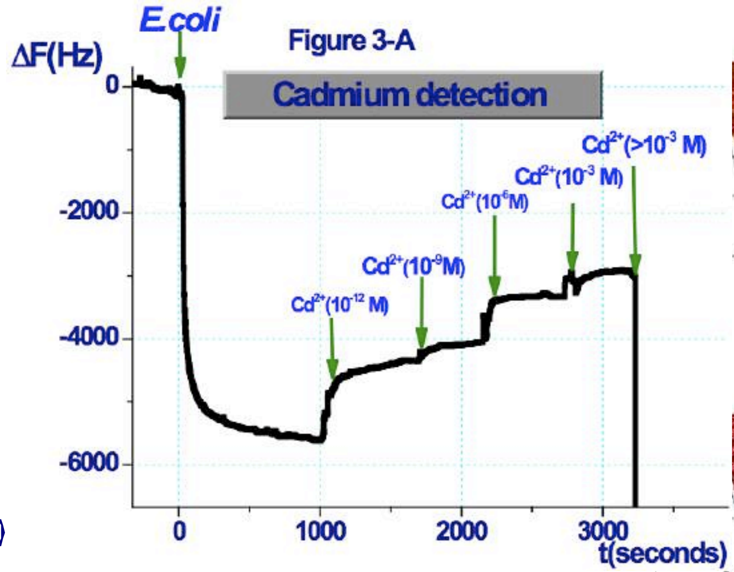
((PAH-PSS)3-PAH) + E.Coli

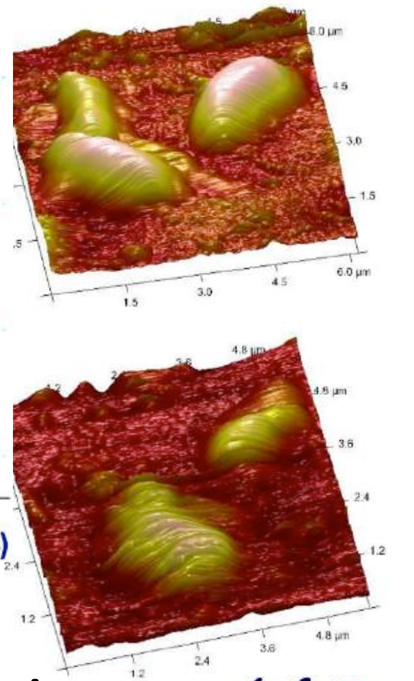
Whole cell-based biosensor

I.Gammoudi, L.Blanc – Collaboration 2011



Trapping of *E. coli* bacteria





Cd^{2+} detection

- Perturbation of viscoelastic properties due to modifications of bacterial metabolism
- Detection threshold lower than 10^{-12} M (Cd, Hg)

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LOMA UMR5798 Laboratoire Ondes et Matière d'Aquitaine

LMTA Laboratoire Méthodes et Techniques d'Analyse

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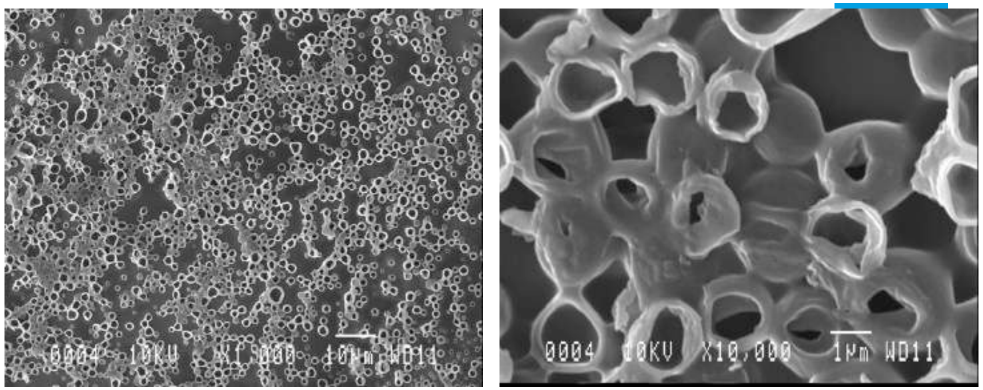
Cancer Biomarkers detection (nucleosides)

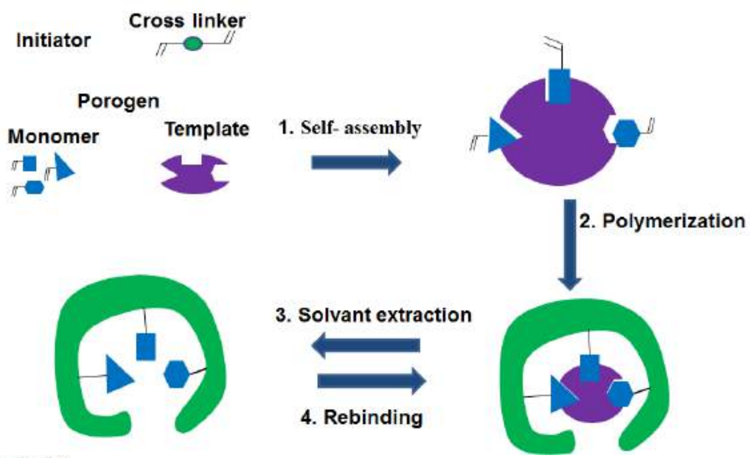
- Molecularly Imprinted Polymer (MIP)
  - Thin film, Spin-coated
  - Compatible with acoustic propagation
- Thin film characterization
  - Profilometry, SEM
  - Sensor response to vapor sorption (toluene, ethanol)
- Target species detection
  - Static tests
  - Dynamic with microfluidic handling
- Adaptation to various target biomarkers

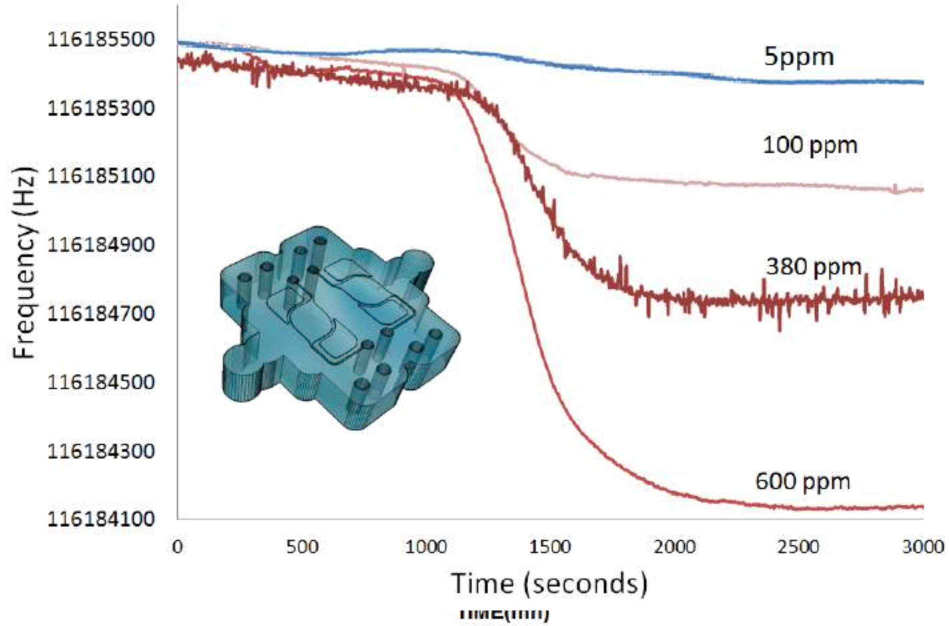
N. Omar-Aouled et N. Lebal - PhD



SEM pictures : MIP layer morphology
Thickness layer : 500 nm to 1µm

Molecular imprinting





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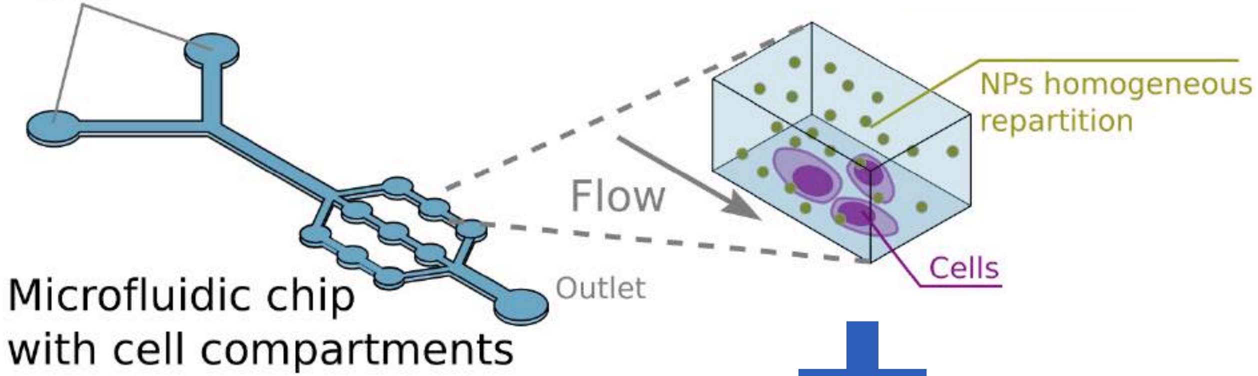
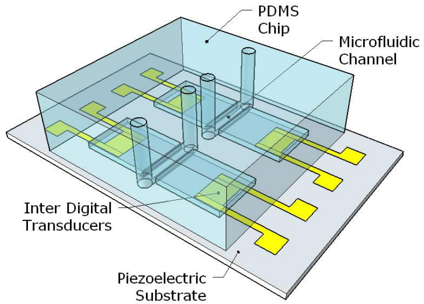
Now and then…

- **Detection applications**
- **Mechanical characterization of the medium at the near interface**
- **A new issue… as a realistic way for *in vitro* assays**



The real-time monitoring of the effect of innovative nanodrugs

- **Therapeutic effect**
- **Toxicity**



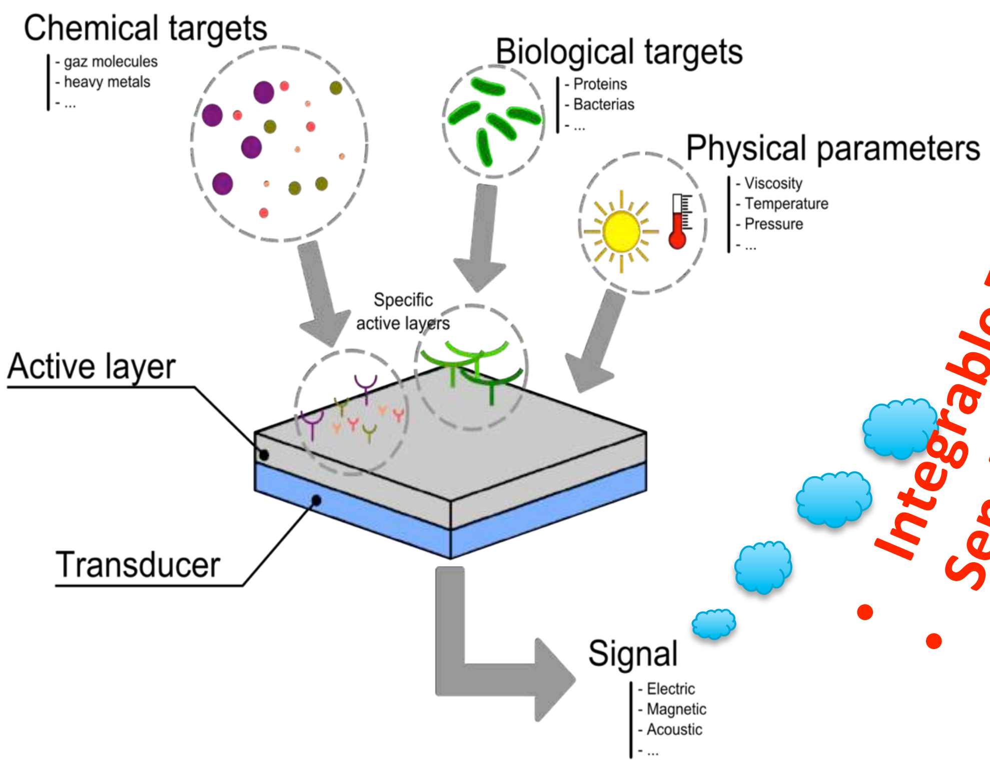
C.Dejous, H.Hallil, V.Raimbault, R.Rukkumani, J.V. Yakhmi, Using Microsensors to Promote Development of Innovative Therapeutic Nanostructuresm in Therapeutic Nanostructures – Vol.I: Novel Approaches, Elsevier, A.M. Grumezescu, 2016, adapted from (Mahto et al., 2010)

Environmental and health related applications

- Introduction: Background, **Principle of Wave-based Resonant Microsensors**
- Focus on Acoustic Love Wave Devices
  - Principle
  - Examples of Gas sensing applications
  - Introduction of microfluidics and examples of Liquid-phase sensing applications
- Other Resonant Devices and Applications
  - Flexible imprinted Electromagnetic Devices & Carbon-based Sensitive films
  - Polymer Optical Microring Resonators & Digital Microfluidics
- Conclusion, perspectives

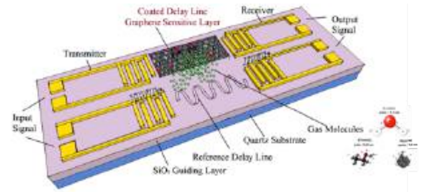
Ultrasensitive Resonant Transducers :
Application for gas and bio sensing

Gas or Biosensor principle

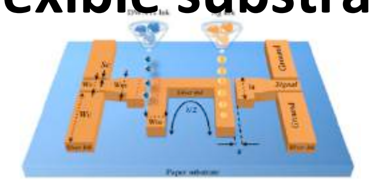


- Integrable Technologies
- Sensitivity, Selectivity
- Sustainability

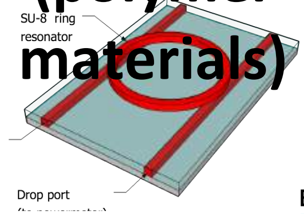
Acoustic platform (piezoelectric substrate)



Electromagnetic platform (flexible substrate)



Optical platform (polymer materials)



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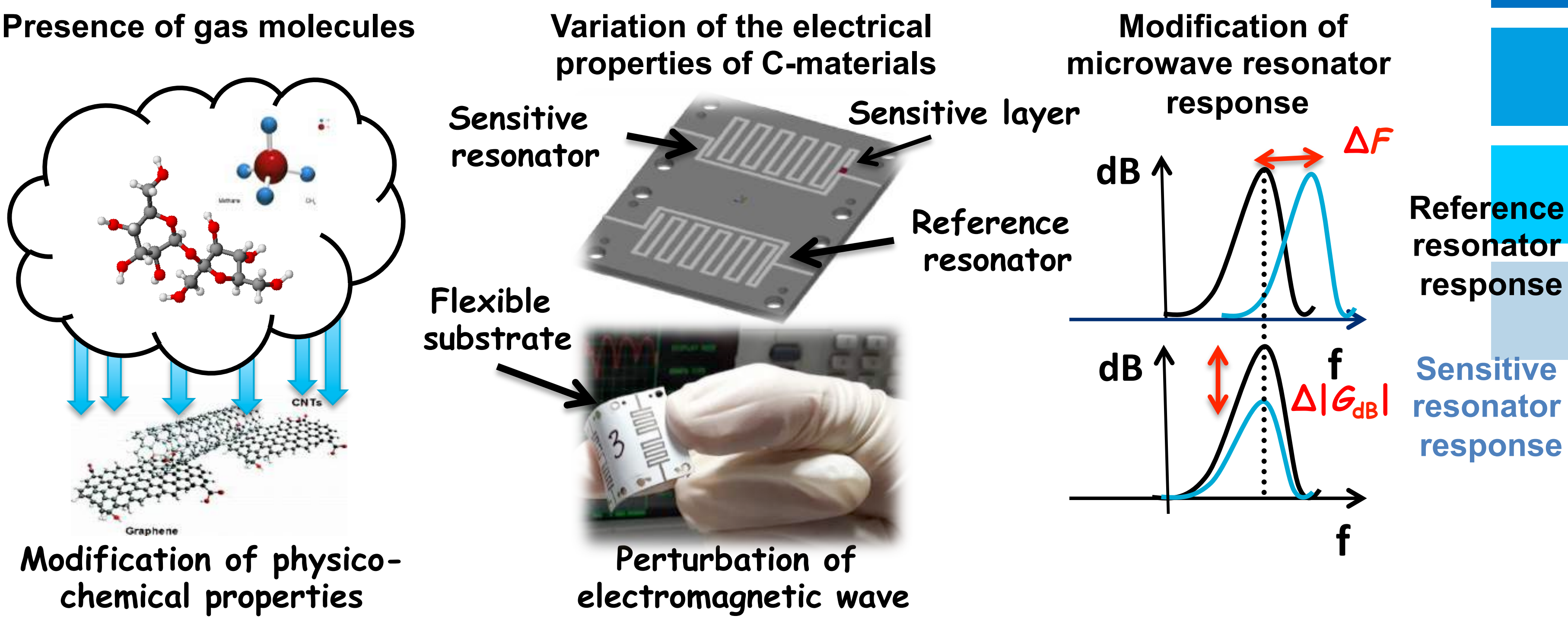
CArbon and Microwave-based Ultrasensitive gas Sensors (CAMUS)

Electromagnetic Sensing Principle



Advantages

Carbon materials

- High sensitivity
- Totally integrated
- Room temperature

Inkjet Printing Technologies

- Low cost
- Planar circuits

Microwave Resonator device

- Differential detection
- Wireless Communication
- Autonomy (Passive)
- Network of sensors & Easy integration for IoT applications

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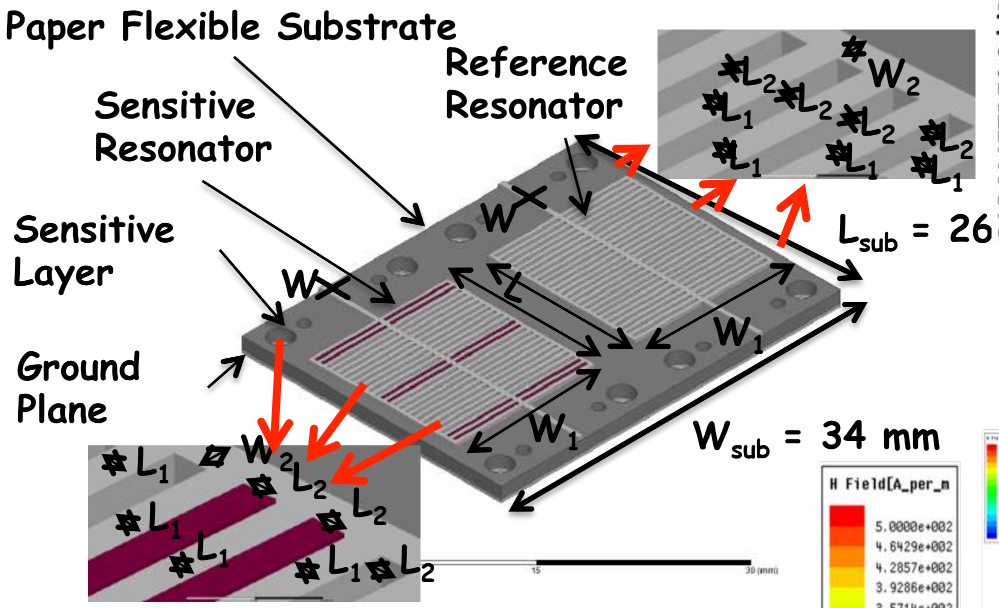
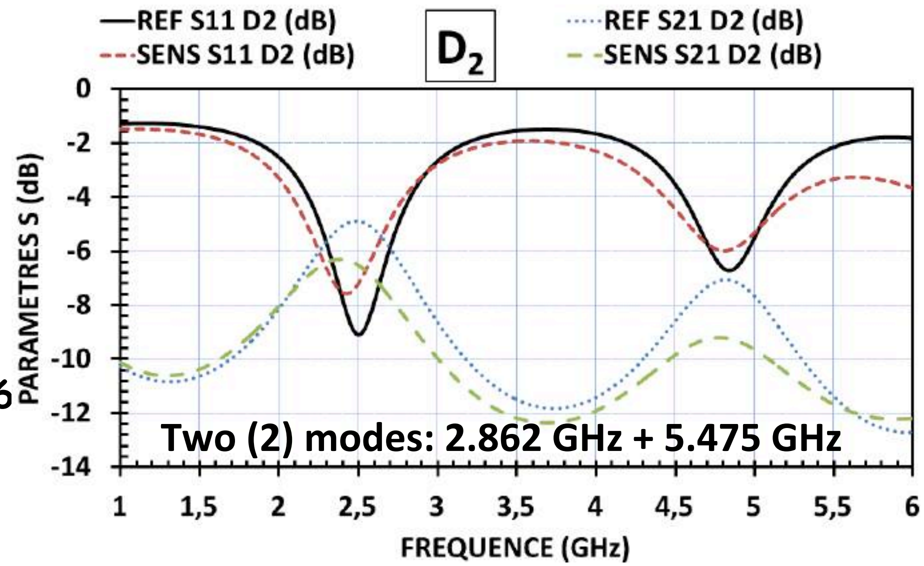
CArbon and Microwave-based Ultrasensitive gas Sensors (CAMUS): Application for gas sensing

Stop-band resonator, 2.8 GHz

Design



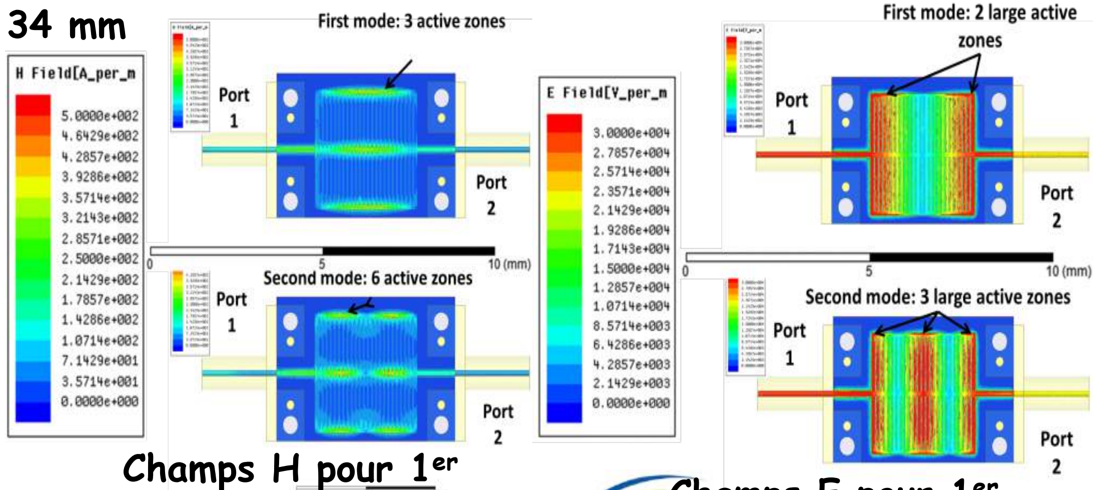


$L_1 = 0.4$ mm ; $L_2 = 0.3$ mm

$W = 0.5$ mm ; $W_1 = 14$ mm

$W_2 = L_1$; $L = (25 \times L_1) + (24 \times L_2)$

Simulation

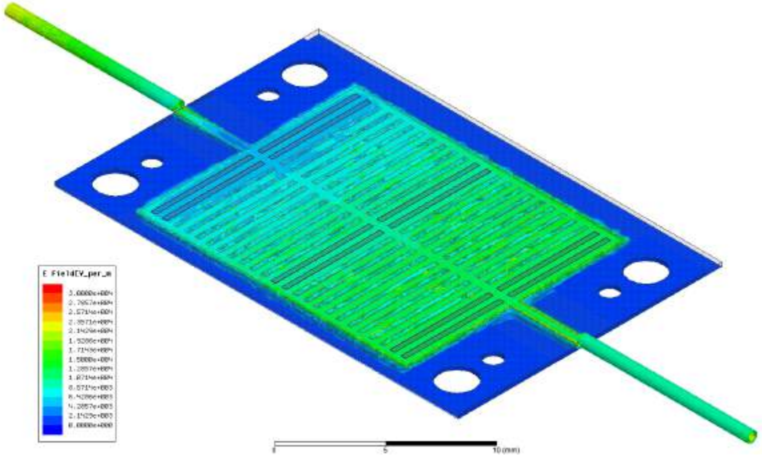


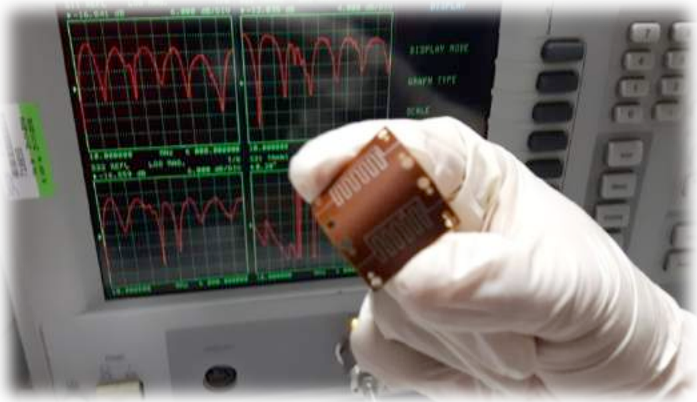
Champs H pour 1er et 2ème mode

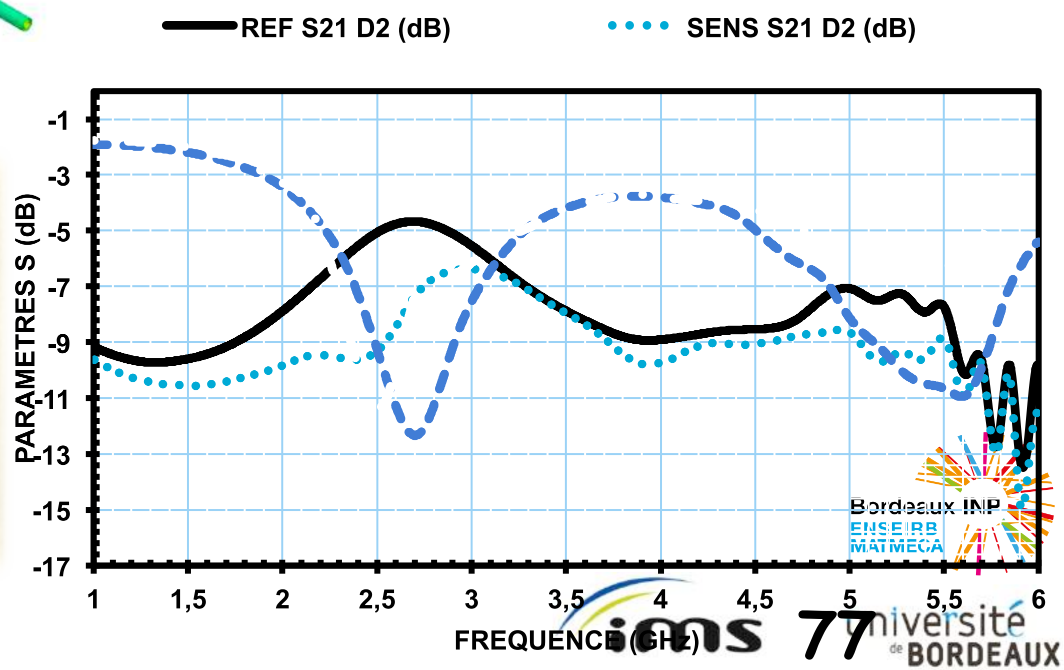
Champs E pour 1er et 2ème mode

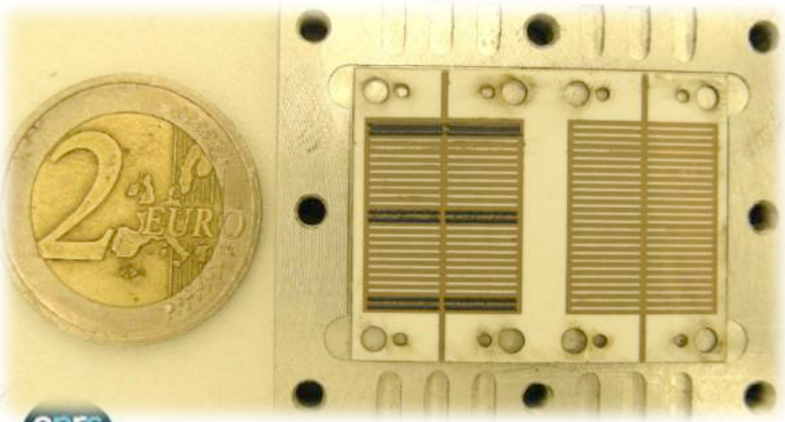
CArbon and Microwave-based Ultrasensitive gas Sensors (CAMUS): Application for gas sensing

Realization & Electrical characterization

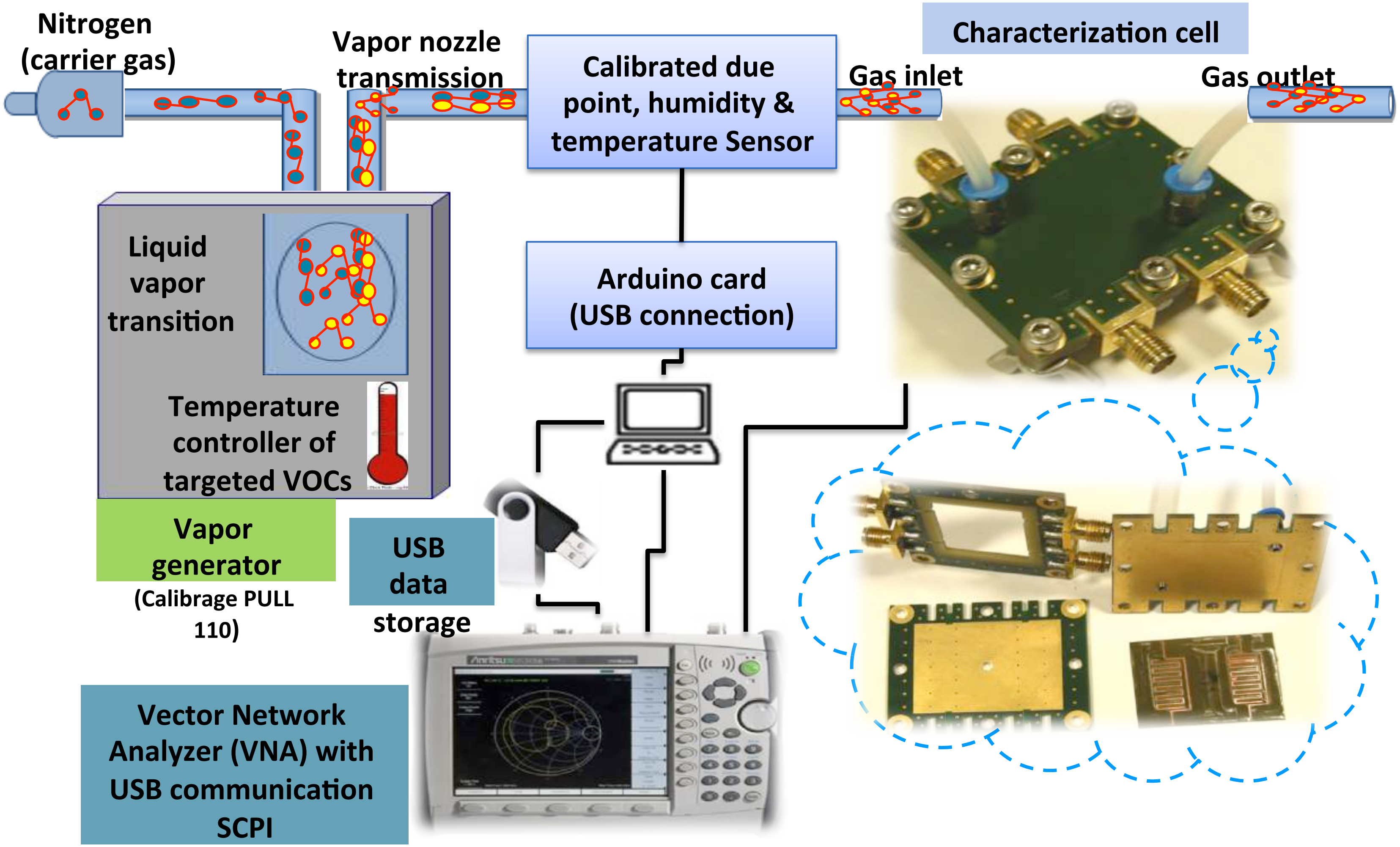








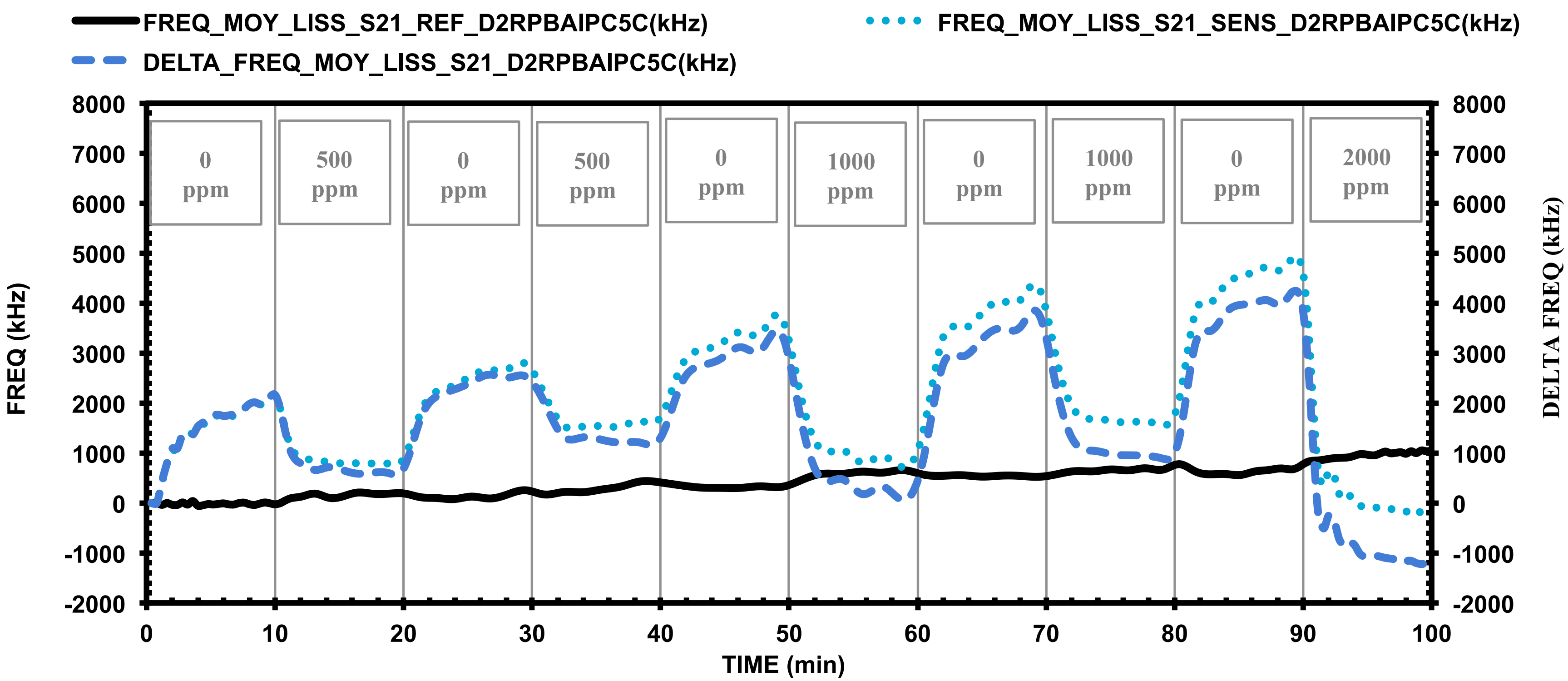
Experimental setup for detection of Ethanol & toluene vapors



- ✓ Dedicated low cost characterization cell (FR4-based)
- ✓ concentration range : 100-2000 ppm ethanol, 100-1200 ppm toluene

CArbon and Microwave-based Ultrasensitive gas Sensors (CAMUS): Application for gas sensing

Real-time characterization with ethanol



Frequency range 2 to 4 GHz: shift of the operating frequence (S21)

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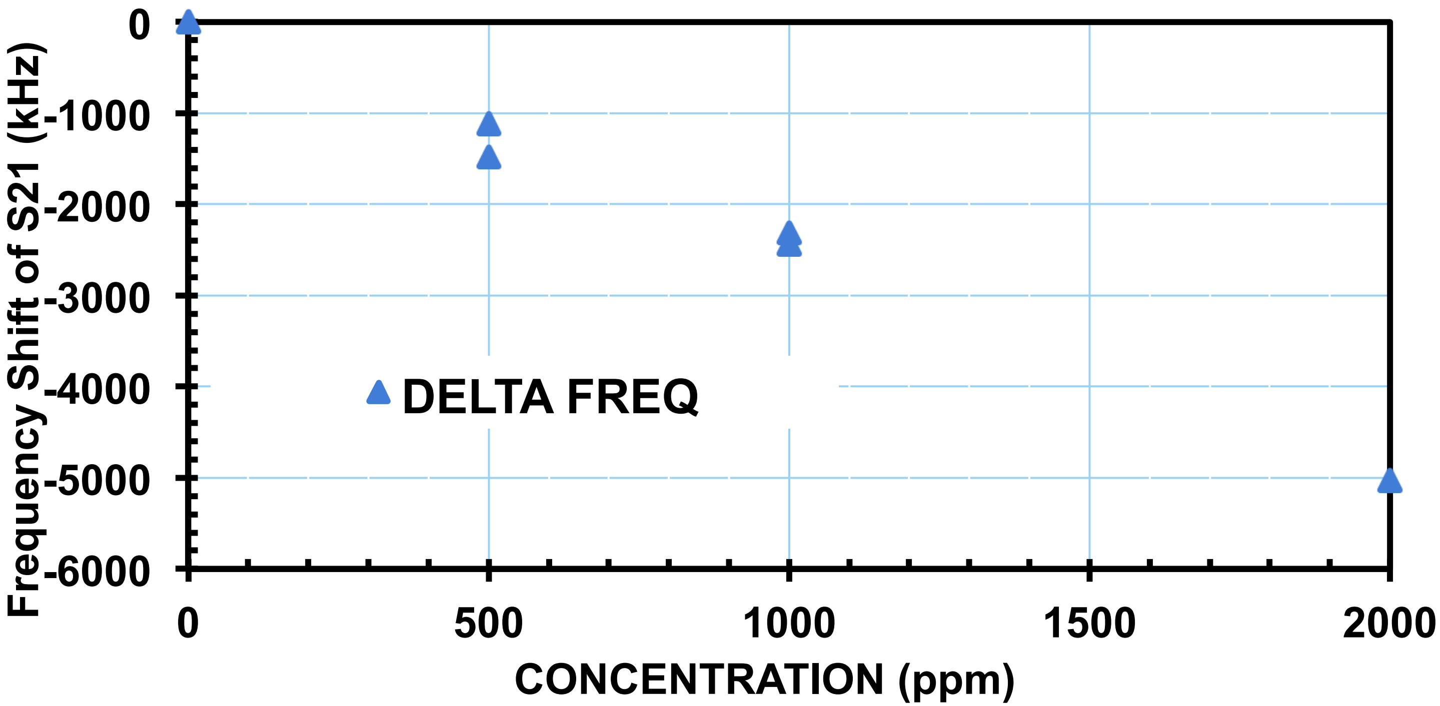
CArbon and Microwave-based Ultrasensitive gas Sensors (CAMUS): Application for gas sensing

Sensitivity to with ethanol



- ✓ **500 ppm ethanol:** frequency shift **1.118 MHz**
- ✓ **Proof of concept: electromagnetic sensor, ultrasensitive, wireless communication**

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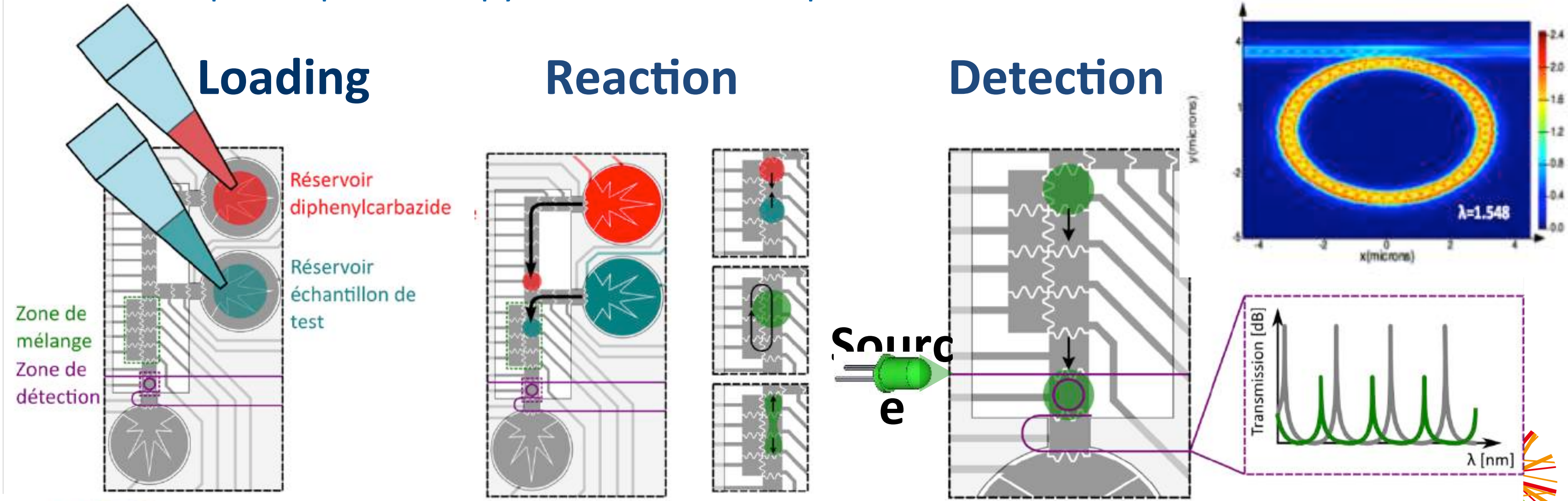
Polymer optical microring resonator (OMR)

Application for environmental detection

Metal ions sensor integrating polymeric optical microresonator (OMR) and digital microfluidics (EWOD*) for environmental applications

Atomic absorption spectroscopy: common technique for Chromiun detection in water



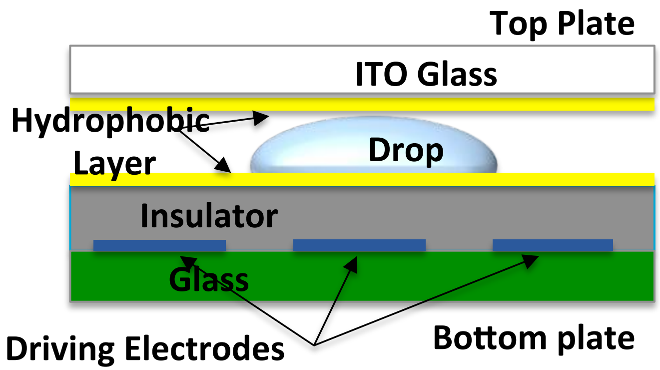
*** ElectroWetting On Dielectric**

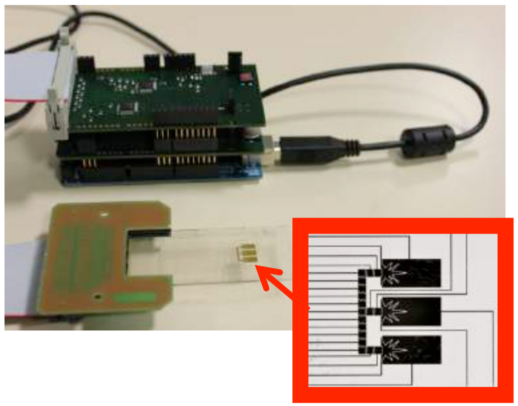
PhD F.Meziane, 2016

Polymer optical microring resonator (OMR)

Application for environmental detection

ElectroWetting On Dielectric (EWOD)



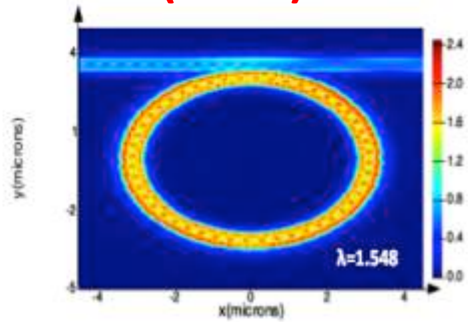


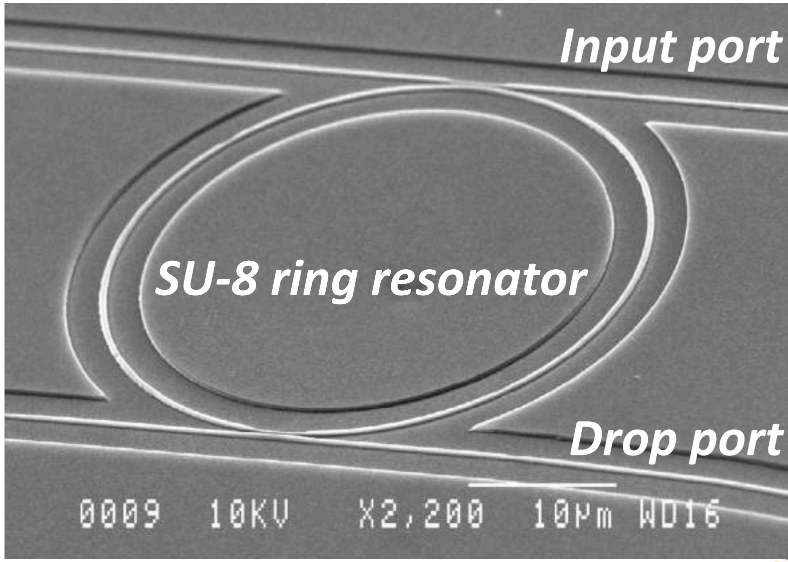
Optical Microring Resonator (OMR)

- ✓ **On glass**
- ✓ **Visible domain**

PhD F.Meziane, 2016

PhD M.Diez Garcia, on-going





Coll. LAAS-CNRS, Tecnalia Research & Innovation, Spain

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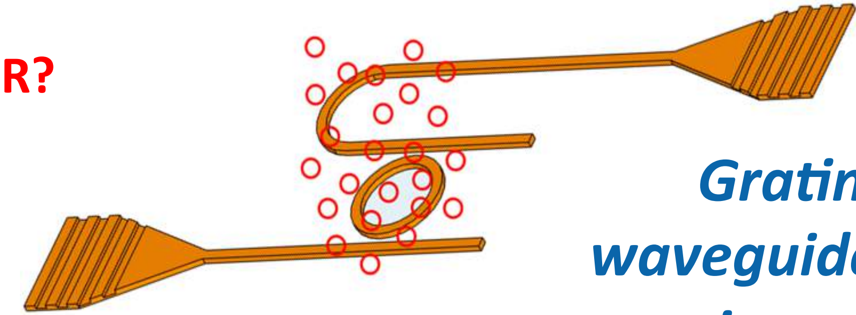
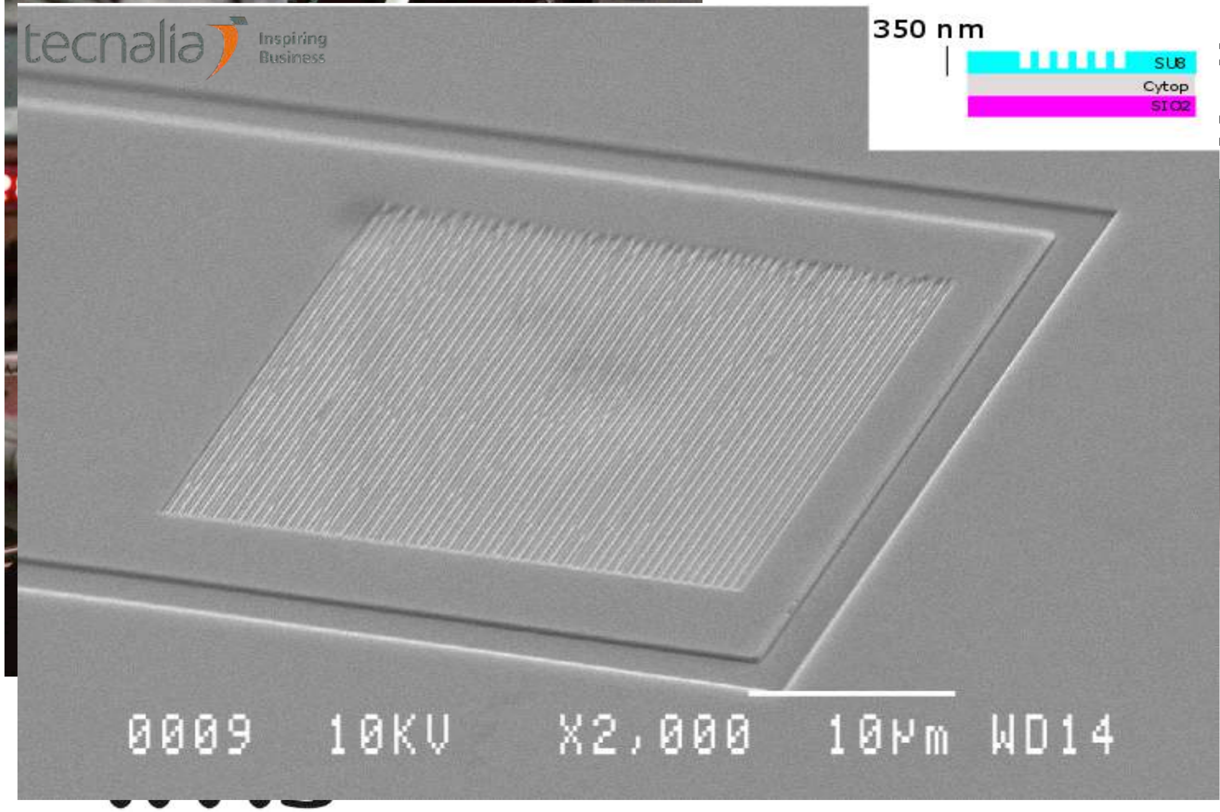
Polymer optical microring resonator (OMR)

Application for environmental detection

Light coupling to OMR?

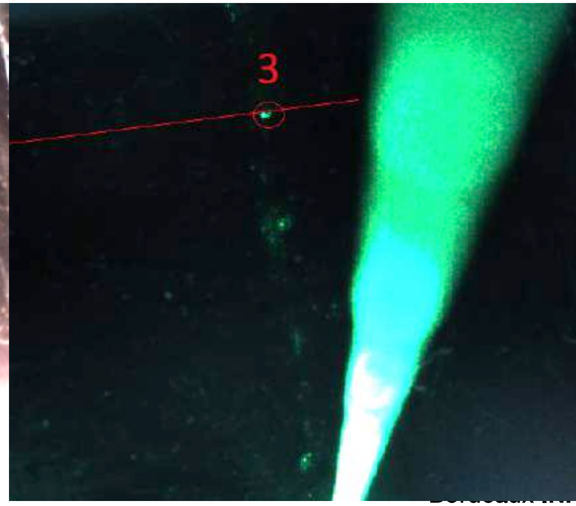


Gratings on waveguides coupled to ring resonators



les coupled to ring resonators

of optical coupling



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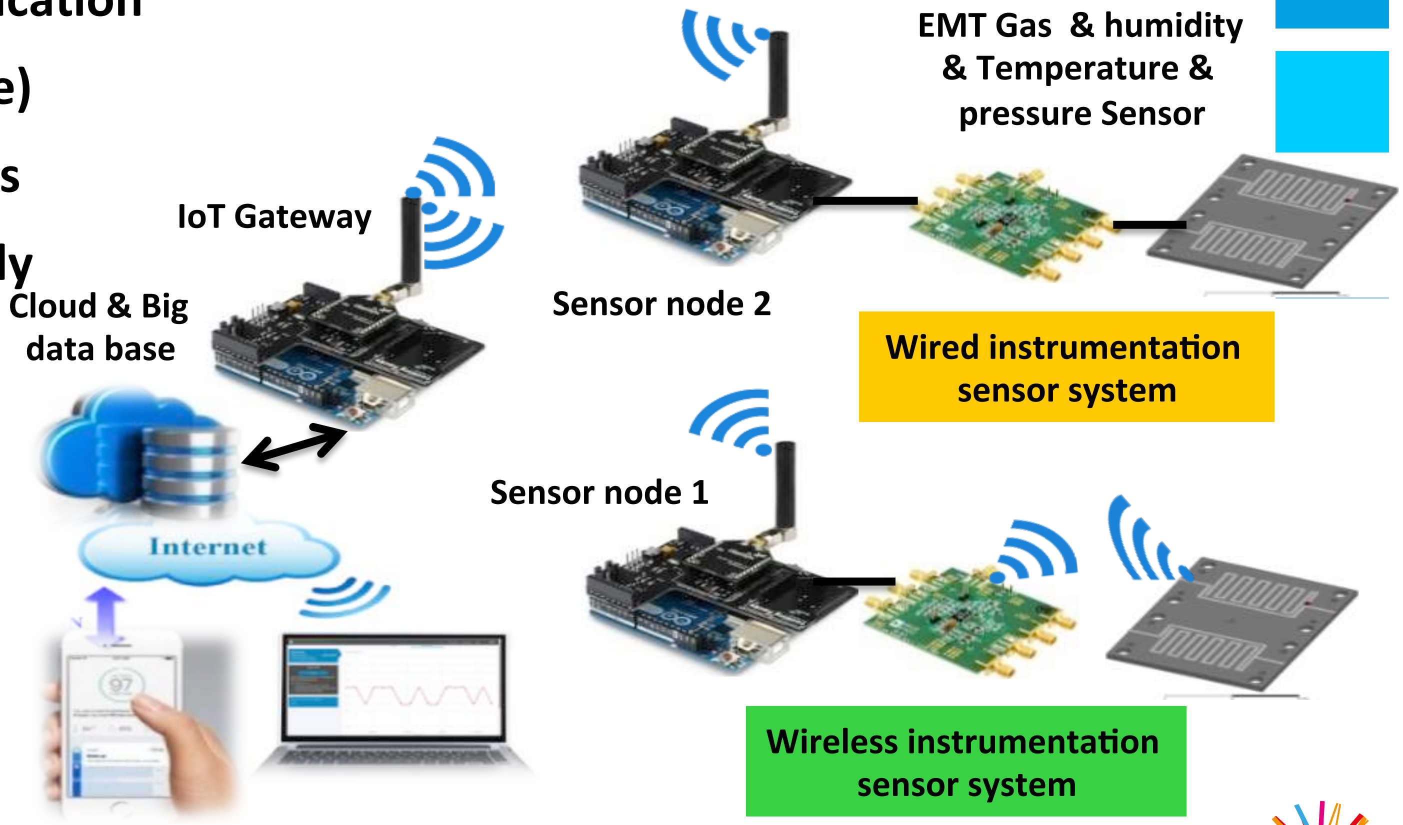
Prospects – a “*Graal*” ?

Integration of wireless sensor network in communicating systems and Internet of Things (IoT) applications

- Wireless Communication
- Autonomy (Passive)
- Network of sensors
- Integration-Friendly

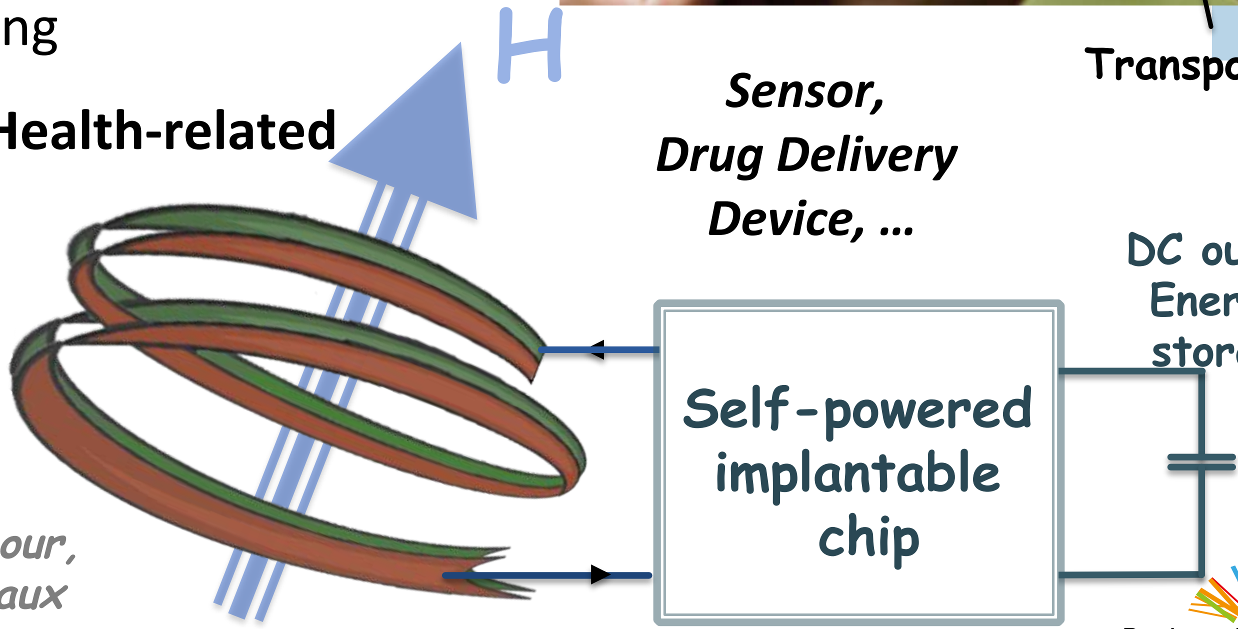


Ex. Population sensitivity and vulnerability to cyanobacteria in the Amazon

- Satellite data
- Need for sensor network *in situ*

Conclusion, perspectives

- **Resonant Wave-based Microsensors**
  - Acoustic, Electromagnetic, Photonic
  - …Sustainable Devices and Systems
- **Solution for Micro-energy**
  - Wireless implant communication
  - Energy harvesting
- **Environment and Health-related Applications**



Work leaded by S.Hemour, LabEx AMADEus Bordeaux

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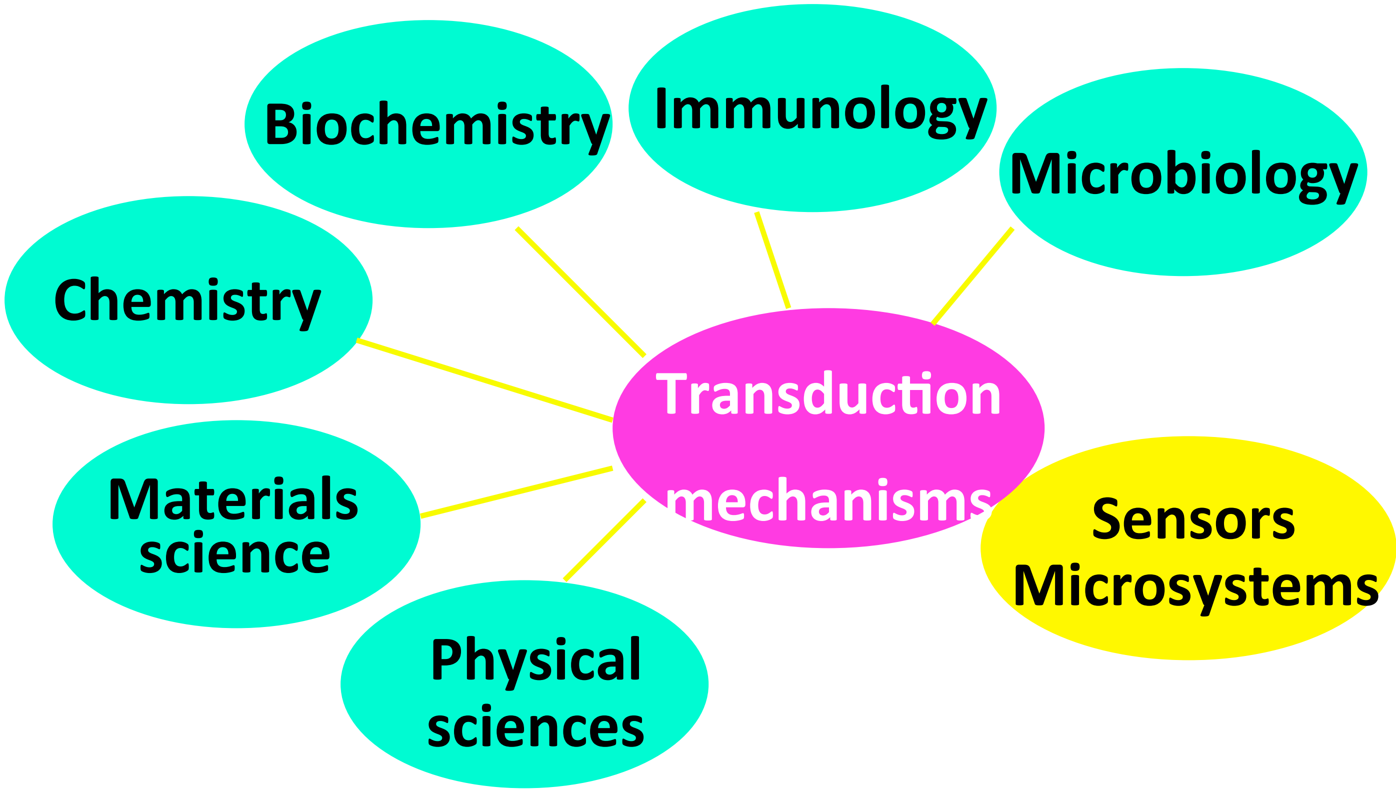
Conclusion



- **An interdisciplinary approach, A versatile platform**
- **Trends: “green” materials and technologies, for sustainable environment and environmental health**

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IMS Bordeaux and Sensors

Sensors… circuits and instrumentation, Students welcome !

Sensor-related scientific communities

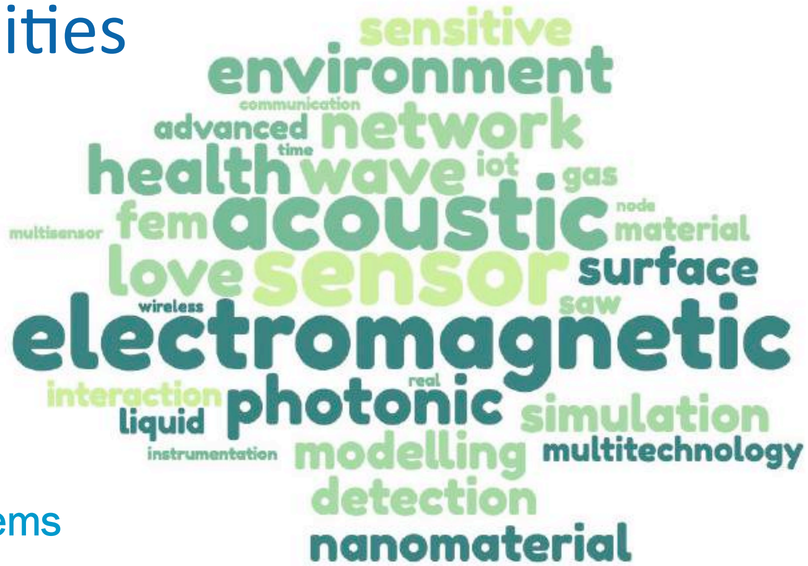
IEEE Sensors Council

IEEE INSTRUMENTATION & MEASUREMENT SOCIETY

BEE Branch

Bordeaux Electrical Engineering
IEEE Student Branch

CMC2



https://nuagedemots.co/

25th IEEE Intern. Conf. on Electronics Circuits and Systems
Bordeaux, France, 9-12 December 2018
https://www.ieee-icecs2018.org/

Large-Scale Distributed Digital Systems,
From Sensors to Decision Processes

- Digital ecological systems
- Smart campus
- Robotics and drones

RÉGION Nouvelle-Aquitaine


Thank you !
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Bordeaux, FRANCE
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Thanks for your attention !