ESIEE PARIS

L'ÉCOLE DE L'INNOVATION TECHNOLOGIQUE

Labex MMCD et EquipEx Sense-City - L'apport des micro-capteurs et des microtechnologies dans l'étude des matériaux pour la construction duable

Tarik BOUROUINA, ESIEE Paris

4. Les projets de recherche du PIA

Laboratoires d'excellence - LABEX

• 5 portés par UPE (+ 11 impliquant des équipes d'UPE)

Futurs Urbains / Urban Futures — MMCD — Labex BÉZOUT — LabEx Sciences, Innovations et TEchniques en Société — VRI Institut de Recherche sur le Vaccin — Investissements d'Avenir

gérés par le PRES — géré par une FCS

NB. : succès pour les SHS + "Futurs urbains" interdisciplinaire

Infrastructures de recherche

✓ *« Santé et Biotechnologies »*

- Cohortes : « *Psy-Coh* » portée par UPE + 2 impliquant UPE
- 4 projets impliquant UPE (bioinformatique, bioressources)

✓ Equipement d'excellence - EQUIPEX

- « *Sense-City* » à la Cité Descartes (site du CSTB)

✓ Au delà du PIA : tirer parti des autres infrastructures

- *ex.* salles blanches d'ESIEE Paris

Institut pour l'aménagement et la ville durable efficacity

plate-forme de recherche privé-public, sur financement IEED

UNIVERSITÉ PARIS-EST

LABEX MMCD

Source : Philippe COUSSOT

« Modélisation et Expérimentation Multi-échelles des Matériaux pour la Construction Durable »

5 axes scientifiques : THEMES

- T1 : **Multi-physique des matériaux nano-structurés**
  - Q.C. He (MSME)
- T2 : **Micromécanique, imagerie multi-échelle et modélisation**
  - M. Bornert (Navier) – J. Yvonnet (MSME)
- T3 : **Rhéologie des matériaux et fluides complexes**
  - J.N. Roux (Navier)
- T4 : **Modélisation stochastique, quantification et propagation d'incertitude pour les modèles mécaniques multi-échelles**
  - T. Lelièvre (CERMICS)
- T5 : **Applications en Génie Civil, Environnement et Energie**
  - P. Faure (Navier) – J.M. Pereira (Navier)

- **CERMICS (ENPC)**

Centre d'Enseignement et de Recherche en Mathématiques et Calcul Scientifique

Maths. Appliquées - Méca flu - Optimisation - Simul. moléculaire - Probabilité

- **ICMPE (UPEC, CNRS)**

Institut de Chimie et des Matériaux Paris-Est

Eco-matériaux, Matériaux avancés, Réactions multicomposants, Métallurgie structurale et fonctionnelle

- **MSME (UPEM, UPEC, CNRS)**

Modélisation et Simulation Multi-Echelle

Mécanique – Chimie théorique – Energétique – Biomécanique

- **Navier (ENPC, IFSTTAR, CNRS)**

Structures – Multi-échelles – Géotechnique – Rhéophysique - Micromécanique

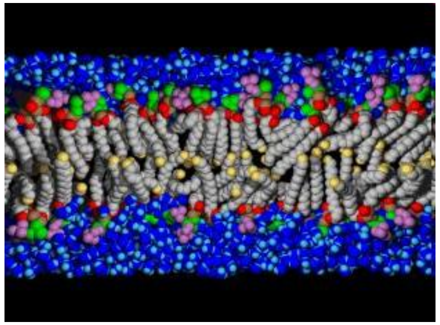
- **ESYCOM-CMM (ESIEE, UPEM, CNAM)**

Equipe « Capteurs et Microsystèmes de Mesure »

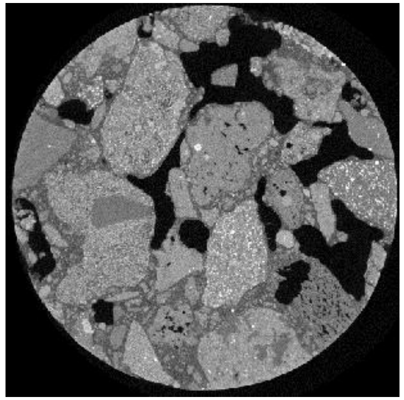
Capteurs, Matériaux modèles micro-structurés, Analyse physico-chimique sur puce

Source : Philippe COUSSOT

Développement d'une science « avancée » de ces matériaux

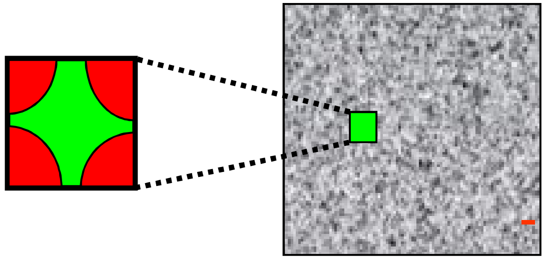


Simulation moléculaire

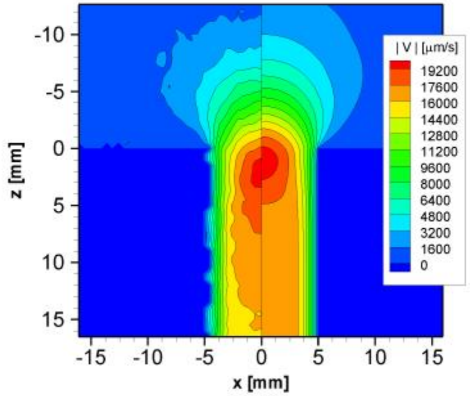


Microtomographie X

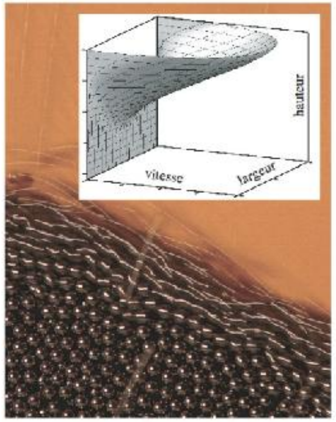
- Distinction des phénomènes essentiels aux échelles appropriées
- Etude de matériaux modèles
- Techniques modernes d'observations internes
- Techniques de simulation à toutes les échelles
- Recherches interdisciplinaires



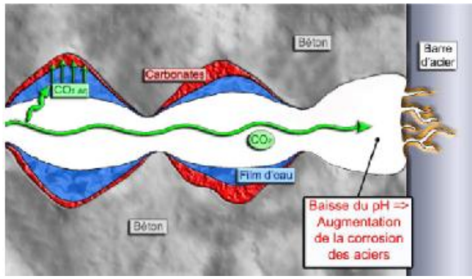
Micromécanique



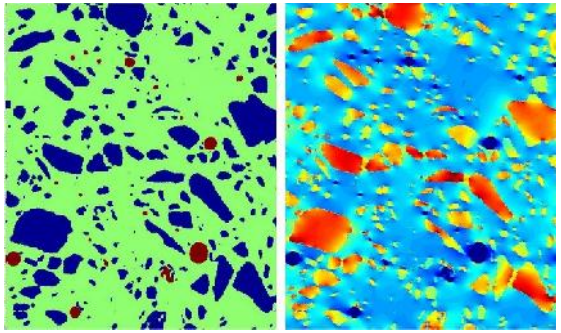
IRM



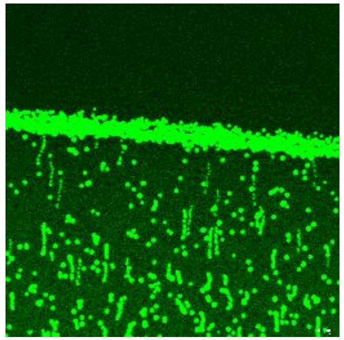
Matériaux modèles



Modélisations couplées



Simulations Milieux Hétérogènes



Microscopie confocale

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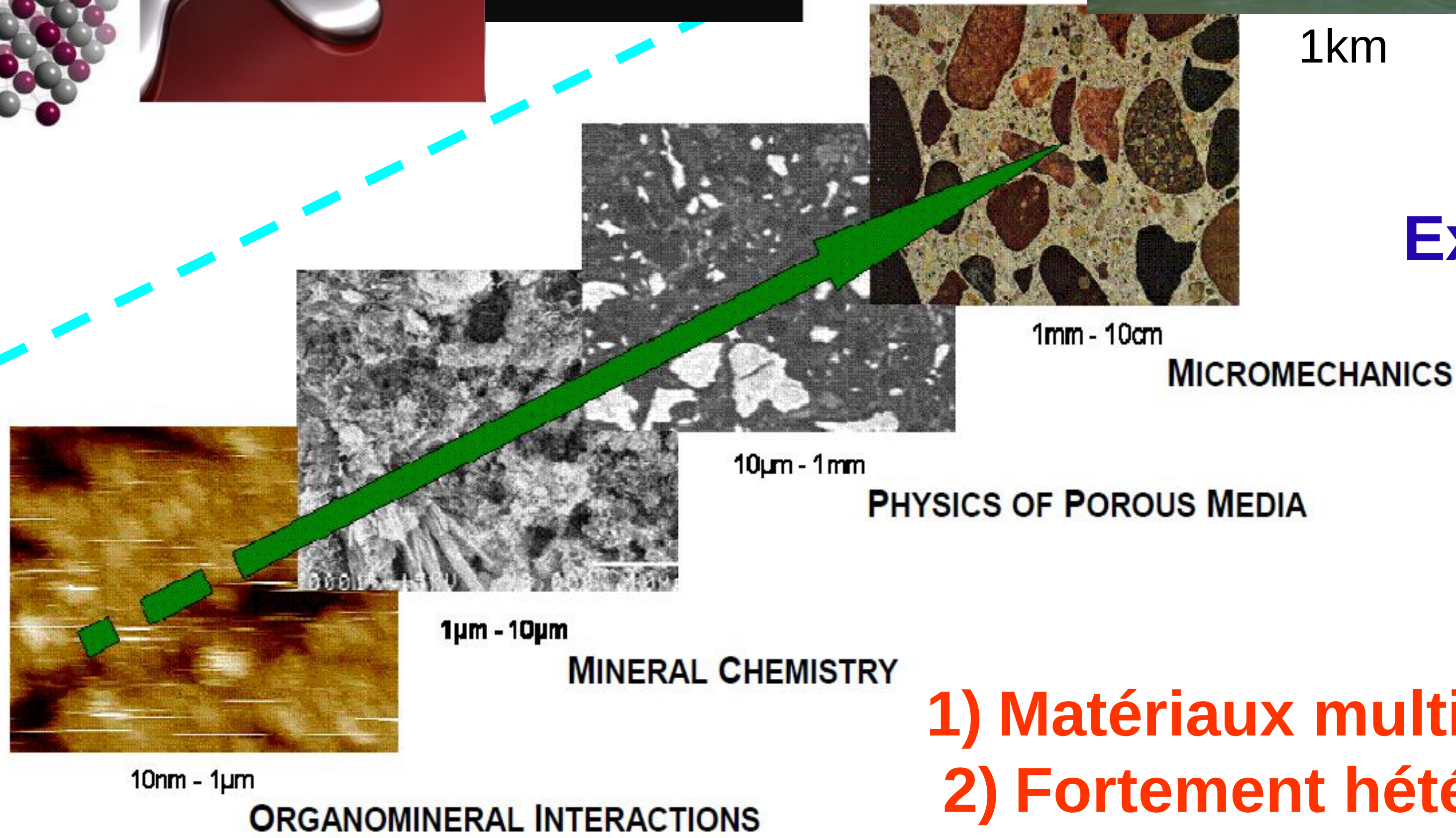
LABEX MMCD

Source : Philippe COUSSOT

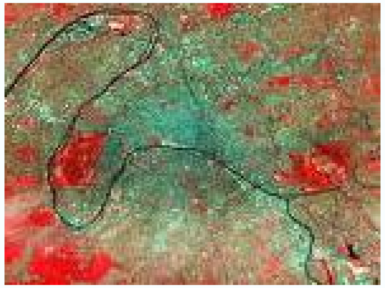
1km

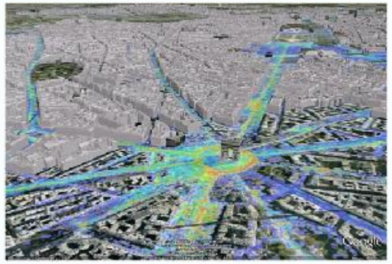


Ex: Béton

1) Matériaux multi-échelles
2) Fortement hétérogènes

Sense-City : Echelle de la Ville et de ses composantes





- **Ville (et réseaux)**

EQUIPEX « Sense-City »
Capteurs pour la Ville Durable

- **Quartier**
- **Bâtiment**
- **Personne**

  - AIR, EAU: Pollutions chimique, biologique, particulaire,
  - Réseaux
  - Sols et sous-sols,
  - Santé structurale des ouvrages
  - Performances énergétiques

- **Matériaux**

LABEX « Matériaux Microstructurés pour la Construction Durable »

- *SCIENCE: Une multitude de sujets d'études*
- *SOCIETE et ECONOMIE: Opportunités de nouveaux services*

basés sur le déploiement de réseaux de capteurs

Impacts socio-économiques : environnement, santé, énergie, durabilité

Convergence entre Monde Numérique et Monde naturel –ou presque : L'urbain

- **EquipEx Sense-City:** Echelle de la Ville, du Quartier, du Bâtiment, du Réseau:
  - Numérisation des espaces urbains au moyen de réseaux de capteurs
  - Finalité : **Compréhension du « métabolisme » de la Ville**
- **Labex MMCD :** Echelle du Matériau hétérogène et complexe:
  - Numérisation par techniques tomographiques,
  - Finalité : **Compréhension des lois comportementales des matériaux**

- ❑ **Dualité expérience–modélisation** : données mesurées / simulées
- ❑ **Rôle fondamental du capteur :** interface entre la nature et le monde numérique
- ❑ **Micro-capteurs** pour **matériaux structurés** et *vice versa*
- ❑ **Matériaux modèles :** solides/multi-phasiques (aérés, émulsions, colloïdes)

Applications (Outdoor) sur des Grands Espaces
Exemple du monitoring des espaces verts

- Surveillance de l'état de la végétation sur les bâtiments à l'aide d'un réseau de capteurs sans fils (Température, humidité, …).
- Remonter les informations des capteurs et les afficher sur une Interface Homme Machine.

Cartographie des polluants chimiques des sols: Application à l'agriculture pour une utilisation « mesurée » des engrais et des pesticides

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- Dutch Sprouts, a group of Dutch companies devise innovative and cost effective methods for **assessing soil and crops**.
- SoilCares is developing a **small, portable, NIR optical analyzer, which uses Si-Ware-System's spectrometer 'NeoSpectra',**
- *"Enabling farmers to make real time chemical measurements of soil samples in the field for helping to create better and more sustainable harvests"*

the soil cares initiative

Si-WARE SYSTEMS


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Home » More Spectroscopy Articles » Si-Ware MEMS sensors chosen for spectrometer-based soil analysis

Si-Ware MEMS sensors chosen for spectrometer-based soil analysis

02/20/2015
Posted by Gail Overton
Senior Editor

Integrated circuit (IC) and MEMS-based solutions provider Si-Ware Systems (SWS; Cairo, Egypt and La Canada, CA) has had its Prism-award-winning MEMS spectral sensors chosen by Dutch Sprouts (Wageningen, Netherlands) for use in its near-infrared (NIR) portable spectrum analyzers that farmers can use for *in-situ* soil analysis.

LE PLUS PETIT SPECTROMETRE OPTIQUE DU MONDE
L'ACCES A LA CHIMIE ANALYTIQUE SUR PUCE

www.si-ware-systems.com

HAMAMATSU Photonics

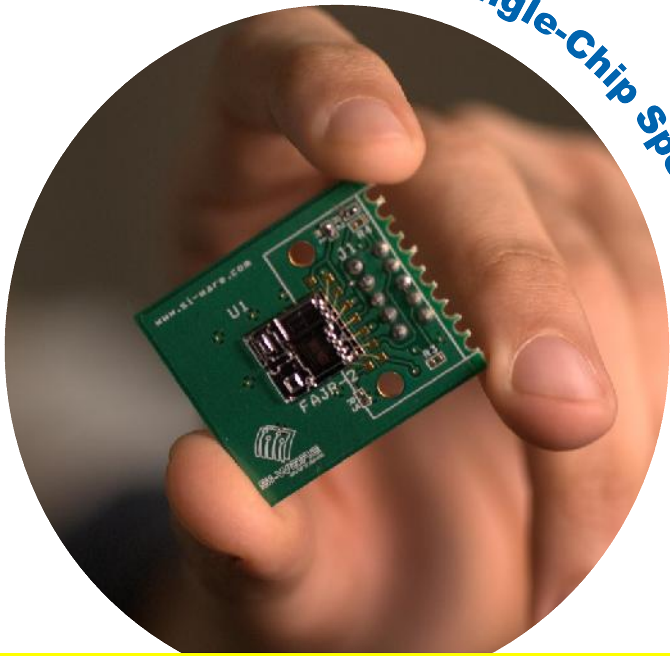
SI-WARE SYSTEMS

ESIEE PARIS

SiMOST

Conventional Spectrometer

SWS-OMTD Single-Chip Spectrometer

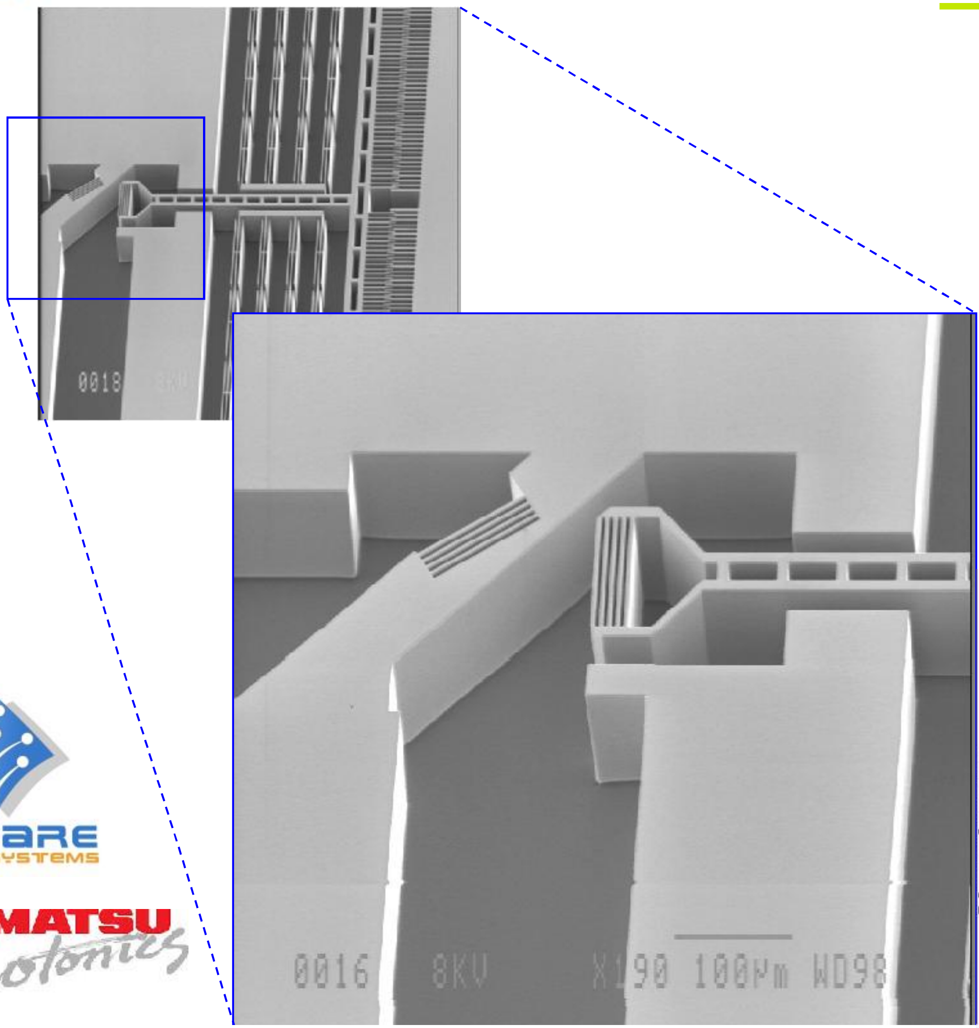


Thèse ESIEE 2007, Startup –Si Ware Systems

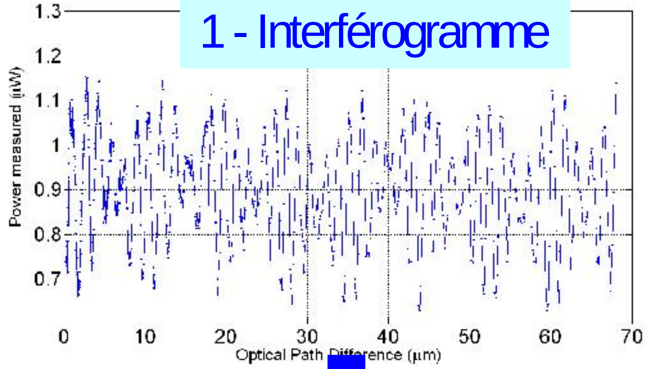
Prix du Meilleur Produit du Japon en 2013

Prix du Prism Award of Photonics Innovation, San Francisco, USA, 5 février 2014

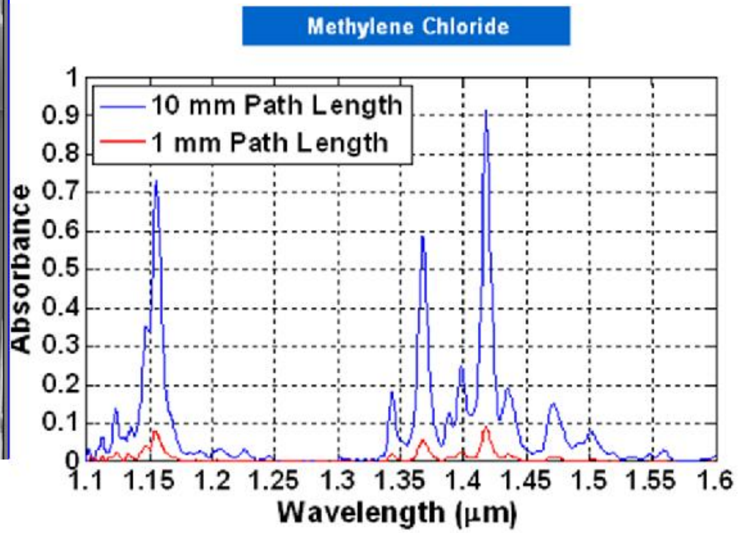
Principe du Spectromètre Optique sur Puce MEMS



1 - Interférogramme



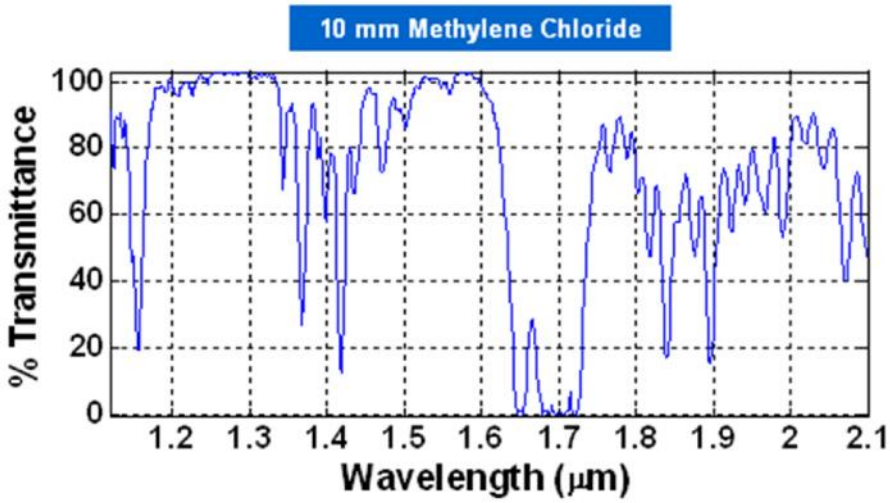
2 - Sa FFT donne le spectre

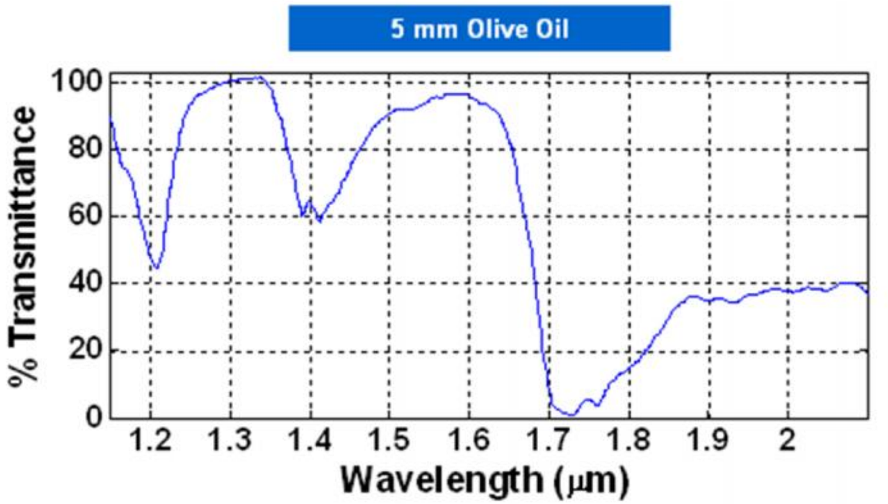


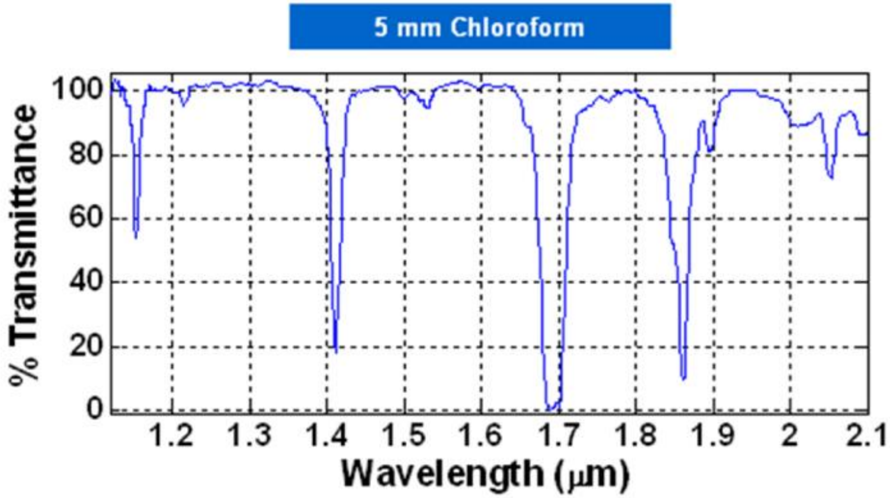
B. Saadany, T. Bourouina, et al. US Patent 648018.
B. Saadany et al., IEEE Optical MEMS and Nanophotonics 2009
Y. Nada et al, IEEE MEMS 2012

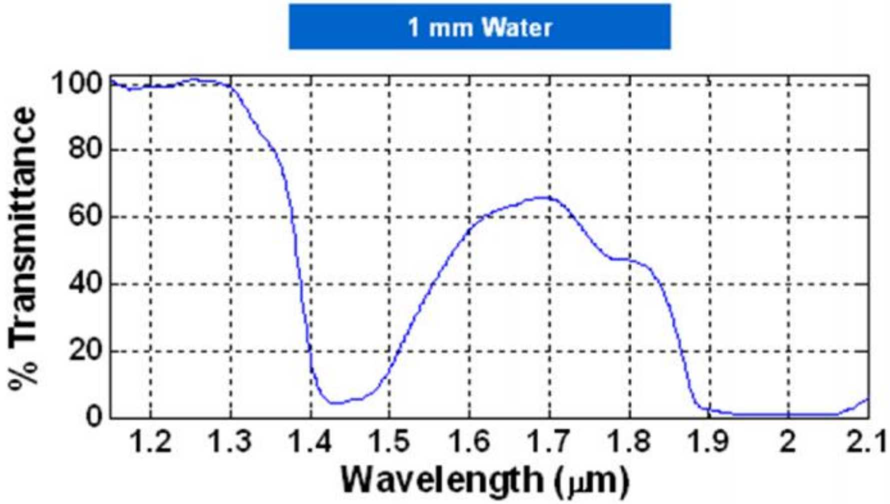
Résolution spectrale de 3 nm

Measurements of typical transmission spectra Using the MEMS FTIR Spectrometer

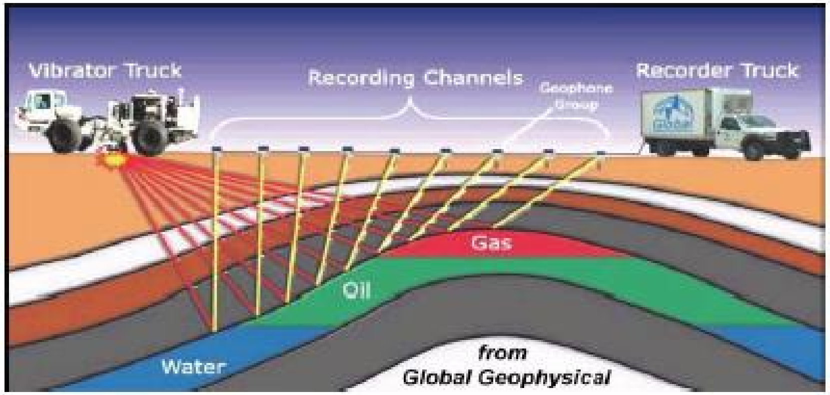


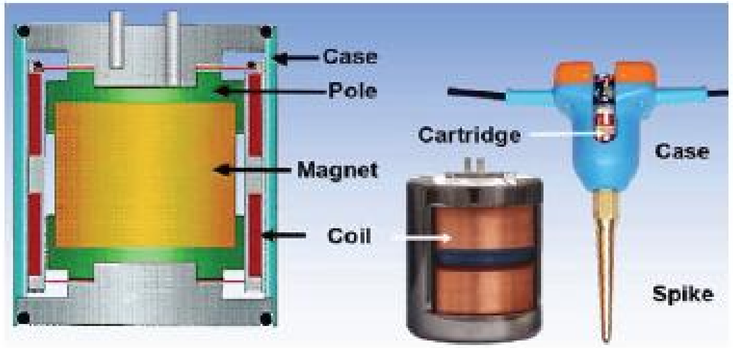






Numérisation des sous-sols par voie sismique au moyen d'un réseau de géophones





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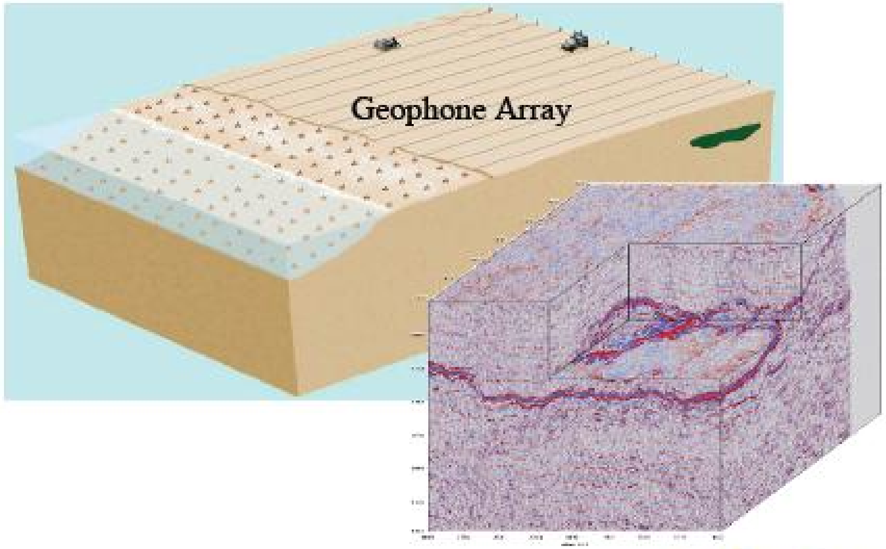


Schlumberger

Smartphones as Seismic Sensors

Smartphones Could Help Cities Create Real-Time Urban Seismic Networks

Tiny sensor used in smart phones could create urban seismic network

Bulletin of the Seismological Society of America, October issue

SAN FRANCISCO – A tiny chip used in smart phones to adjust the orientation of the screen could serve to create a real-time urban seismic network, easily increasing the amount of strong motion data collected during a large earthquake, according to a new study published by the *Bulletin of the Seismological Society of America* (*BSSA*).

Micro-Electro-Mechanical System (MEMS) accelerometers measure the rate of acceleration of ground motion and vibration of cars, buildings and installations. In the 1990s MEMS accelerometers revolutionized the automotive airbag industry and are found in many devices used daily, including smart phones, video games and laptops.

Antonino D'Alessandro and Giuseppe D'Anna, both seismologists at Istituto Nazionale di Geosifica e Vulcanologia in Italy, tested whether inexpensive MEMS accelerometers could reliably and accurately detect ground motion caused by earthquakes. They tested the LIS331DLH MEMS accelerometer installed in the iPhone mobile phone, comparing it to the earthquake sensor EpiSensor ES-T force balance accelerometer produced by Kinemetrics Inc.

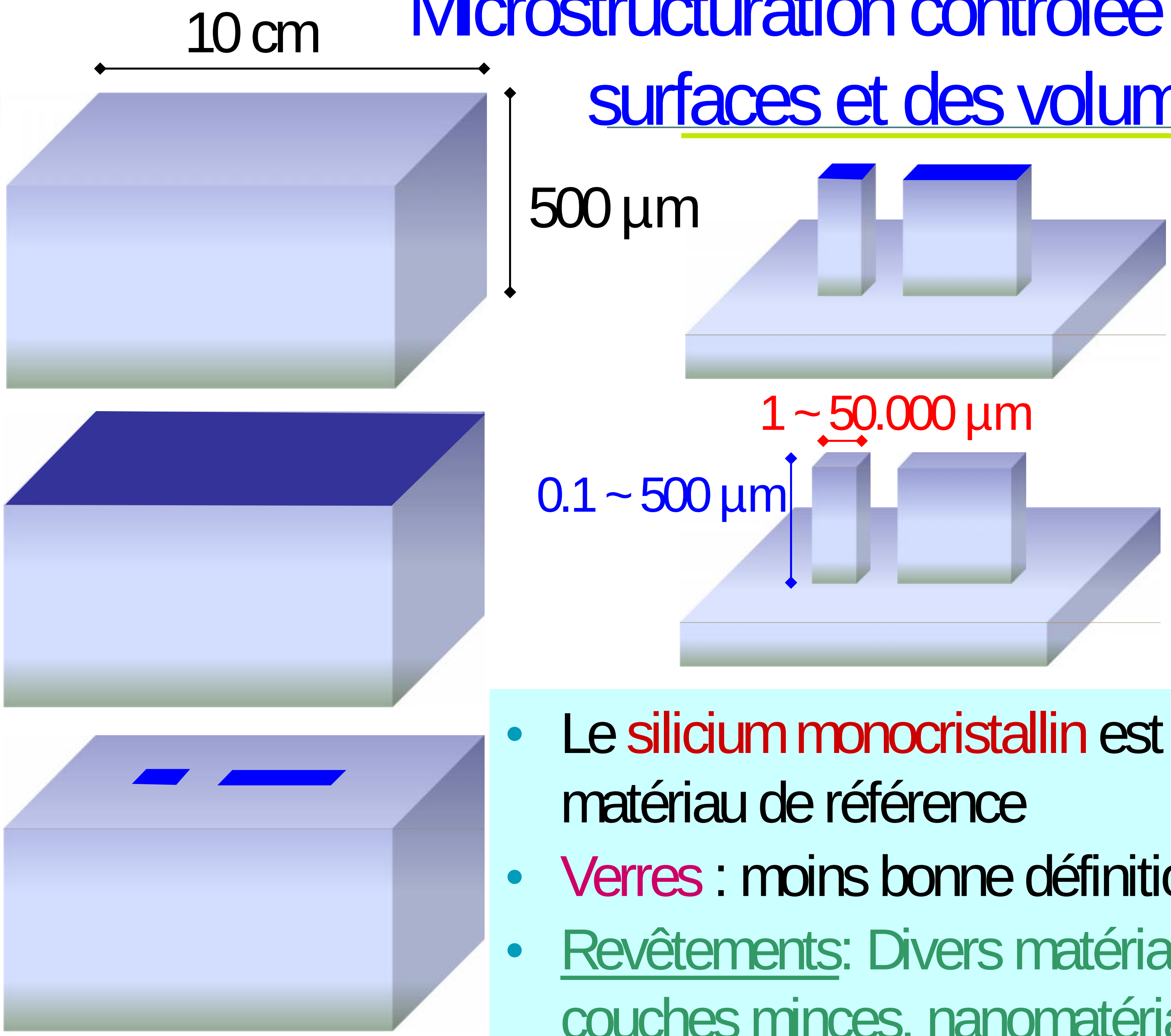
SEISMOLOGICAL SOCIETY OF AMERICA
400 EVELYN AVENUE, SUITE 201
ALBANY, CALIFORNIA 94706
(510) 525-5474 • FAX (510) 525-7204

I - Métamatériaux et Matériaux Modèles

Mouillabilité, Membranes poreuses et Fluides Complexes

Dan ANGELESCU, Frédéric MARTY, Tarik BOUROUINA (ESIEE),
Mathilde REYSSAT (ESPCI),, Hélène BERTHET (Schlumberger)
David QUERE (ESPCI), Ptraick TABELING (ESPCI), Yong CHEN (ENS)

Microstructuration contrôlée de surfaces et des volumes

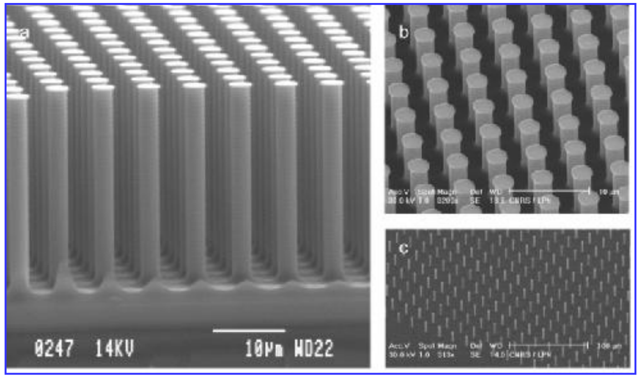


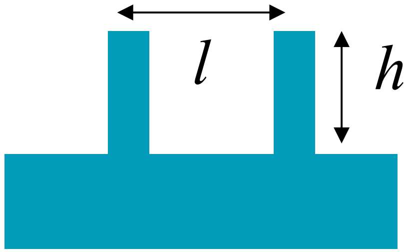
- Le silicium monocristallin est le matériau de référence
- Verres : moins bonne définition
- Revêtements: Divers matériaux en couches minces, nanomatériaux

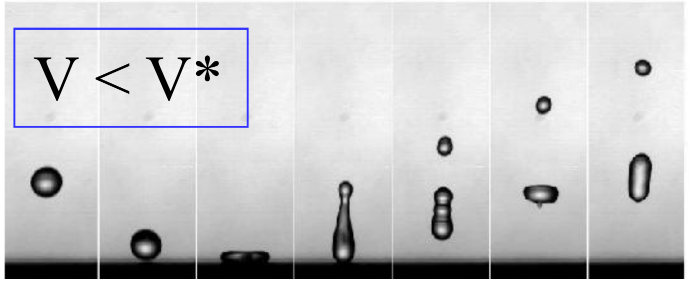
METAMATERIALS 1: Pillar array with controlled pitch

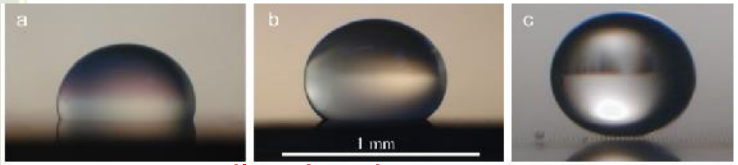
Water-Repellent (Superhydrophobic) Surfaces

Control of contact angle and droplet impact behavior



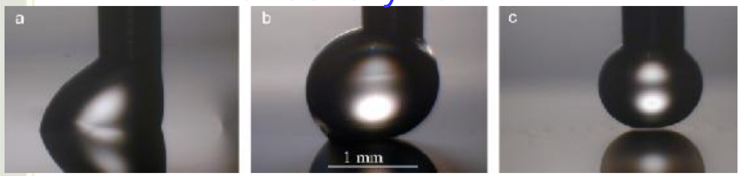


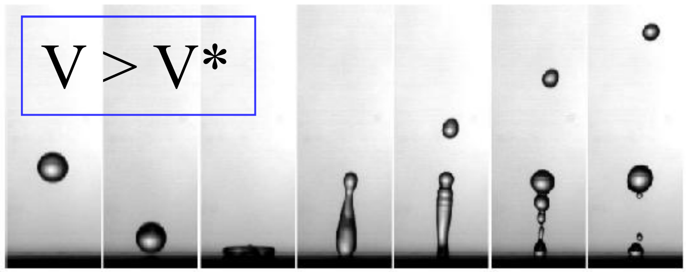




Flat surface — Pilar density 26% — Pilar density 10% — Pilar density 1%

$$V^* = \sqrt{\frac{h}{l^2}}$$ *Threshold pinning transition speed*



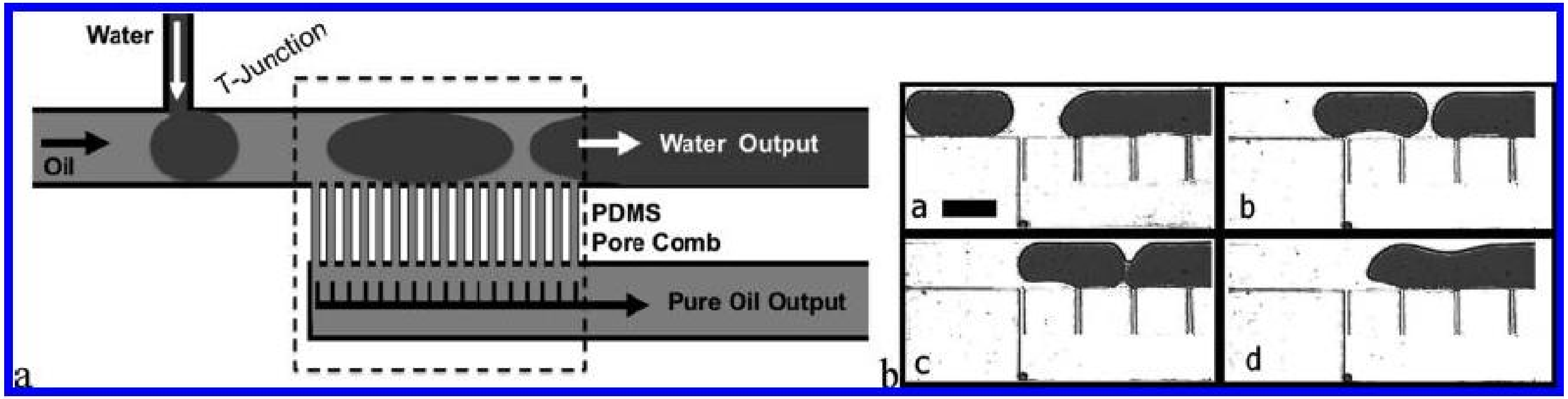


M. Reyssat et al, Euro Phys. J., 2006

Collaboration Mathilde Reyssat, David Quéré (ESPCI) Yong Chen (ENS)

Matériaux modèles (2): Membranes poreuses

- Membrane capillaire constituée de canaux microfluidiques
- Utilisée ici pour la séparation de phases (huile et eau)

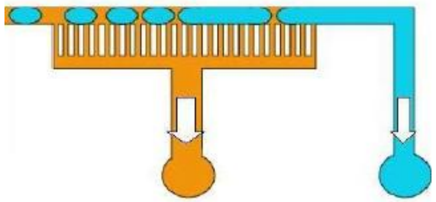


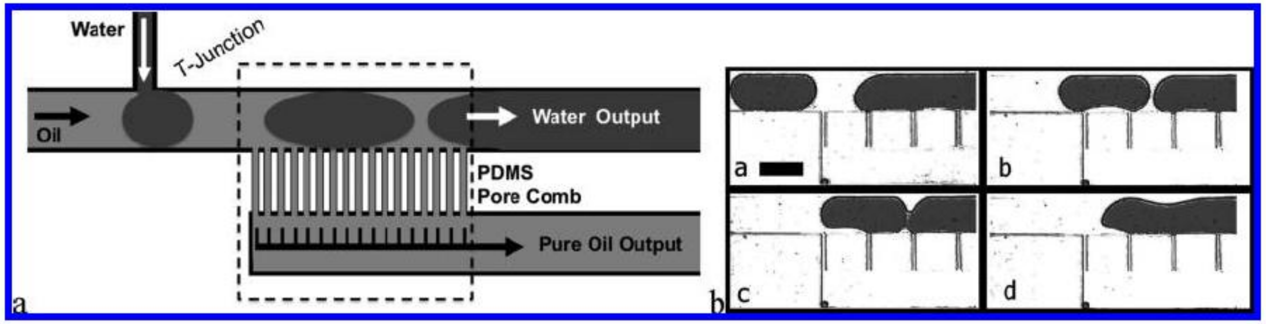
- Autres utilisations : Gels articficiels, filtres, colonnes de séparation chromatographique

MRS Fall Meeting 20

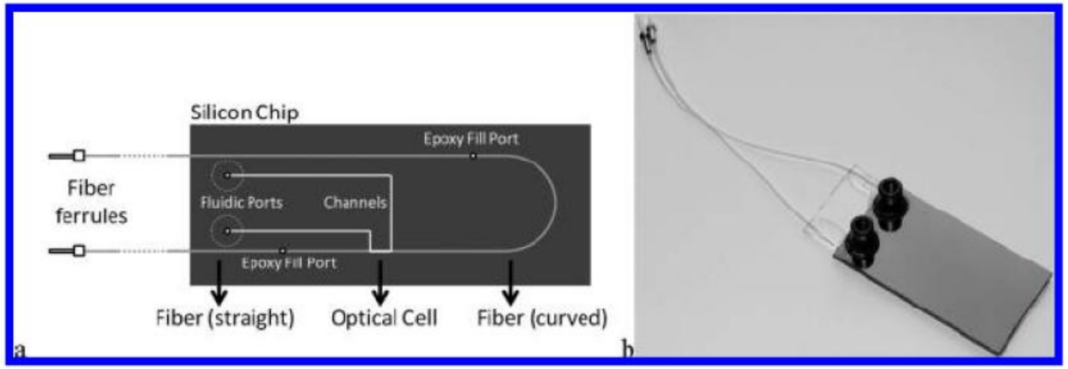
Optical Spectroscopic Analysis of Oil from a Multi-phasic Mixture

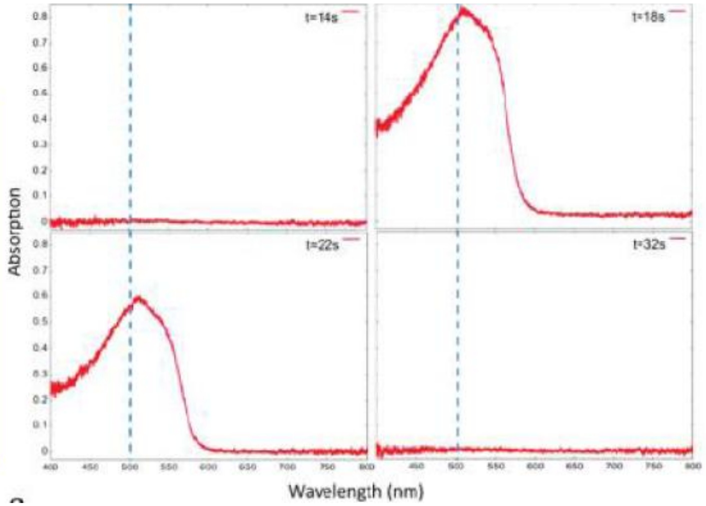
- Microfluidic capillary separation used to separetate oil from water





- Optofluidic cell for monitoring red-died oil





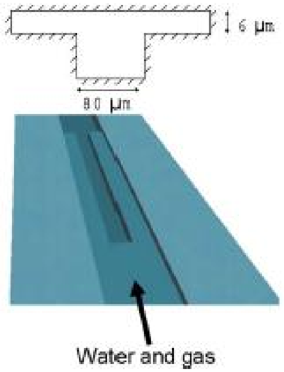
D.E. Angelescu, B. Mercier D. Siess and R. Schroeder, Analytical Chemistry, 2010

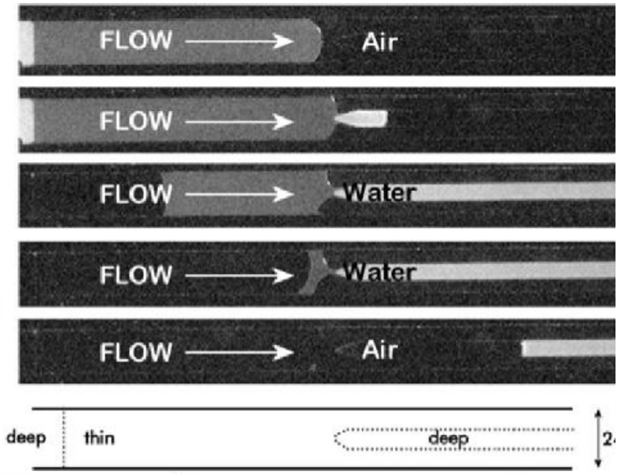
Optofluidic Cell for *In-line* measurement of CO2 concentration in Natural Gas

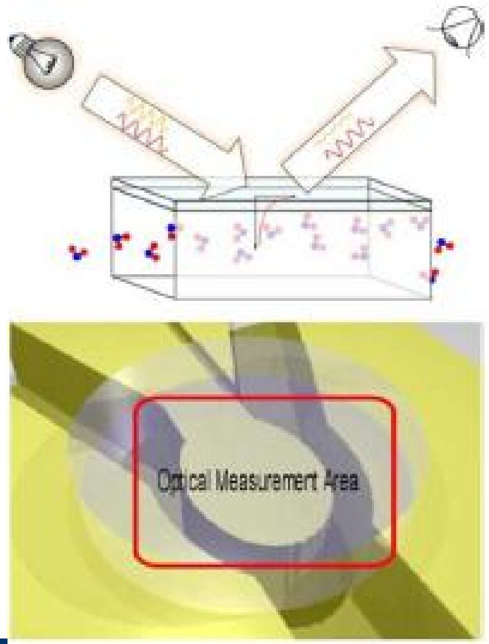
ESIEE PARIS
UNIVERSITÉ PARIS-EST

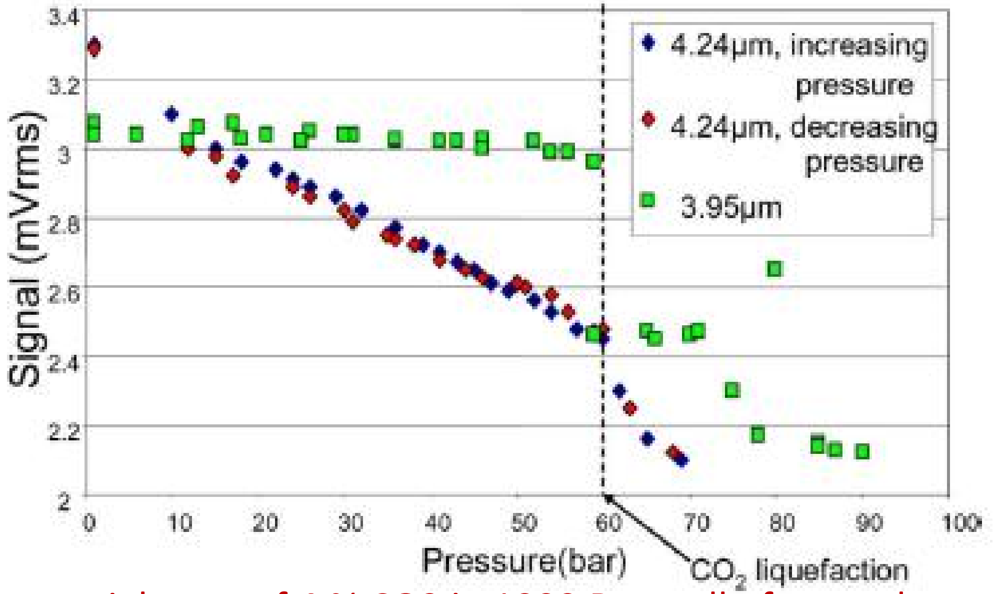
Collaboration P. Tabeling (ESPCI) and E. Donzier (Schlumberger)

- Capillary phase separation followed by optical absorption of the gas phase
  Operation at fixed λ=4,26 µm (absorption peak) with reference @ λ=3,95 µm









Schlumberger

57 Bar ~ partial pres. of 4 % CO2 in 1000 Bar well of natural gas

E. Tavernier, J. Sellier, F. Marty, P. Tabeling, T. Bourouina, IEEE Sensor Journal, 2008

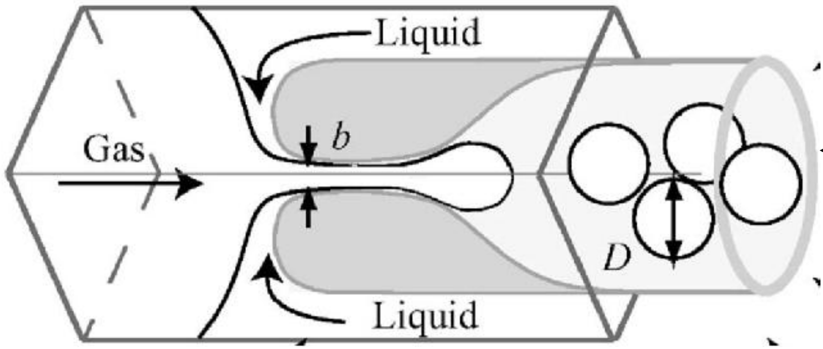
II - Rhéologie sur Puce MEMS

Fluides Complexes Modèles et Mesure in situ des Propriétés des fluides à échelle micro- nano-

Y. Cheung Sang, E. Lorenceau, S. Cohen-Addad, R. Höhler, Langmuir 2008

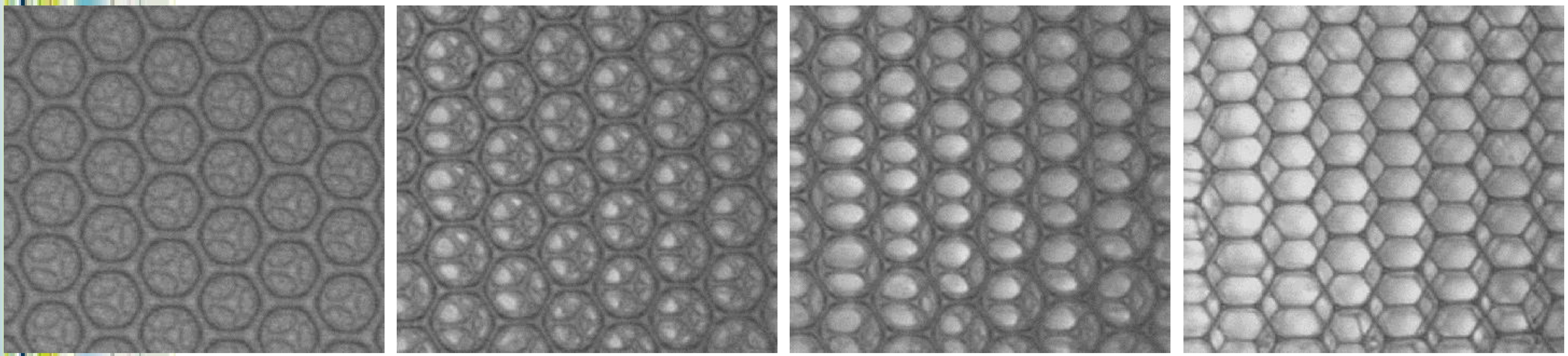
Dryer foam

Conventional Method : Production of monodisperse foams via flow focusing



Bubble

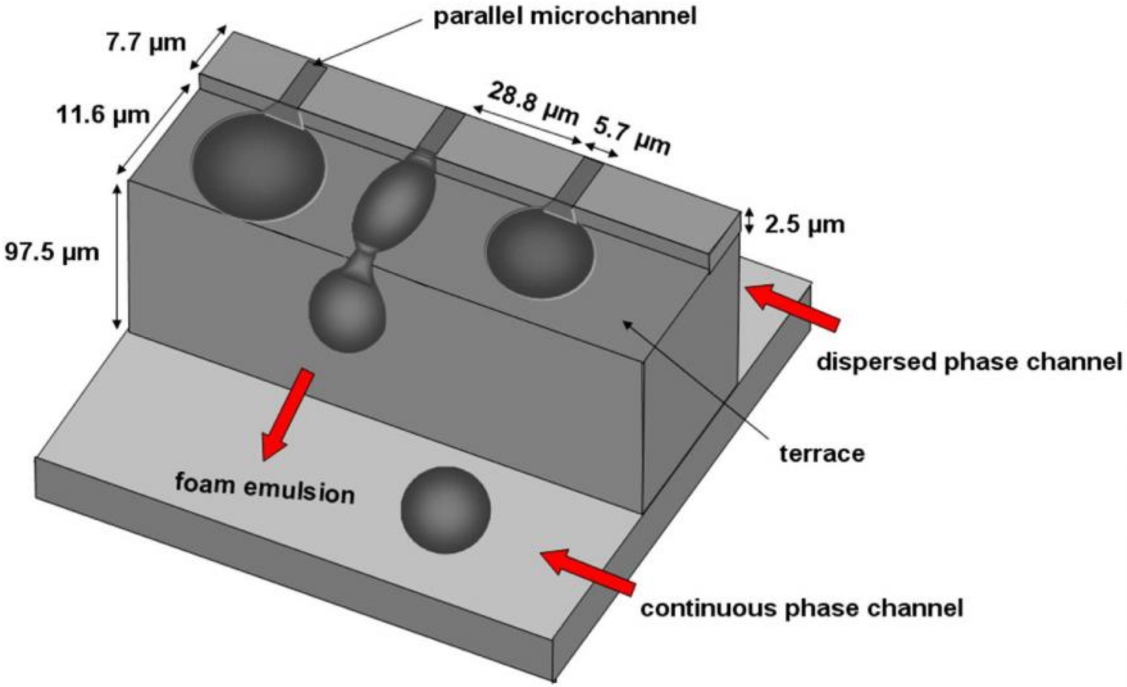
Structural transition in foam Crystal

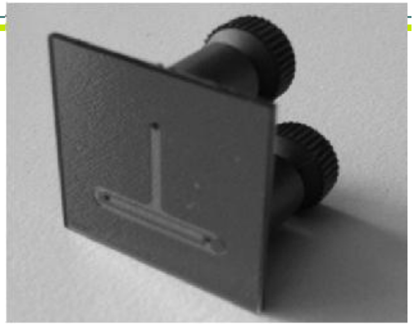


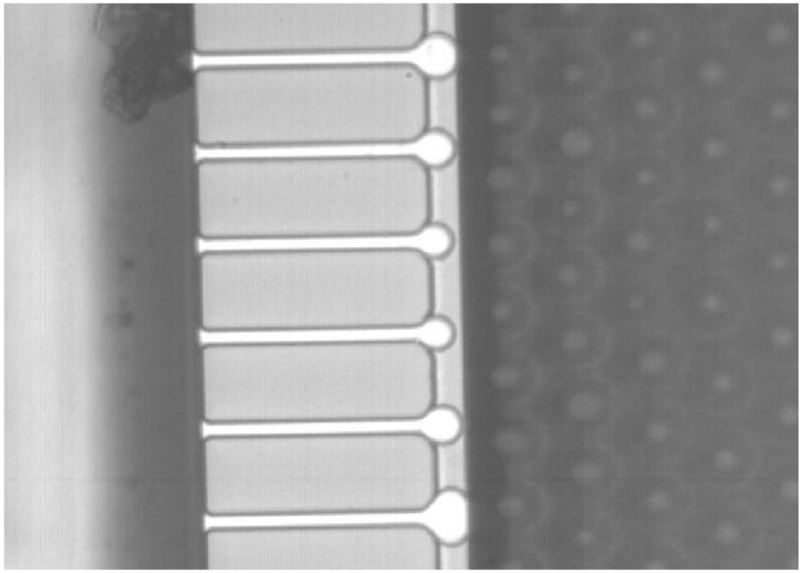
Dryer foam

Y. Cheung Sang, E. Lorenceau, S. Cohen-Addad, R. Höhler, Langmuir 2008

On-Chip Production of Monodisperse foams by topological transition





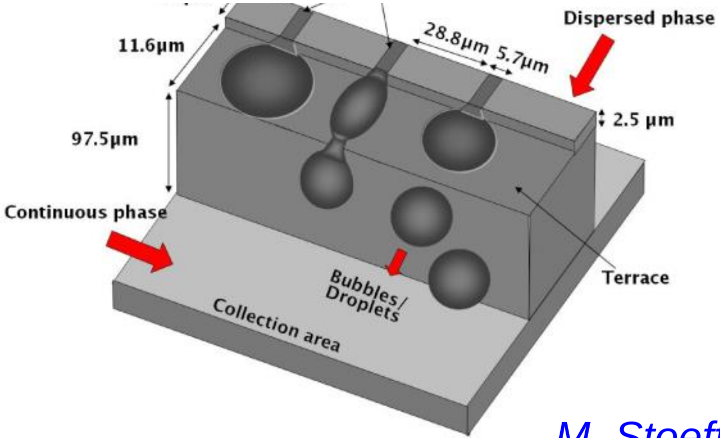


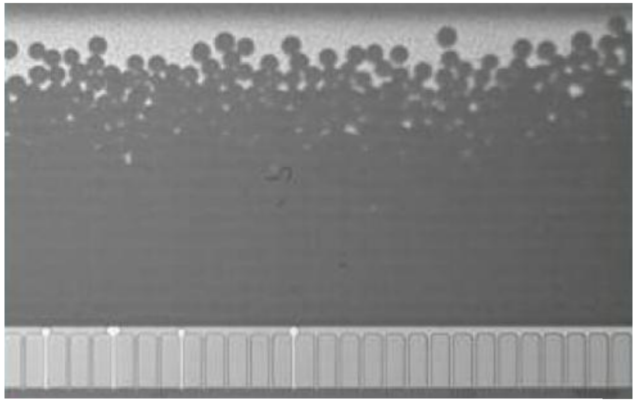
- Strong 2D to 3D transition leads to abrupt break-off
- Complex physics: pinch-off depends non-trivially on fluid viscosities, contact angles, and device geometry

M. Stoeffel, D.E. Angelescu, Physical Review Letters, 2012

Fluides complexes modèles

- Monodisperse Bubble/Droplet Generation and Characterization

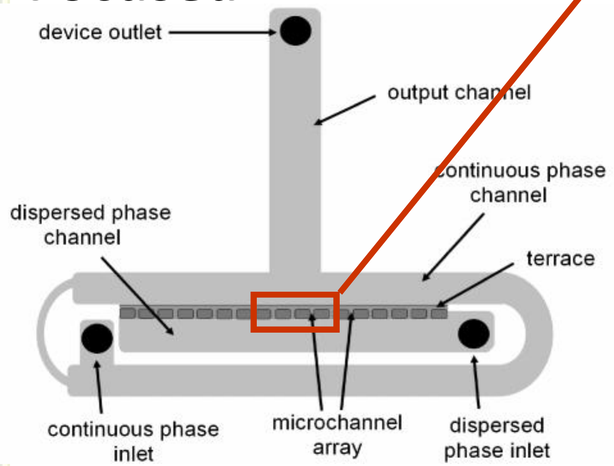




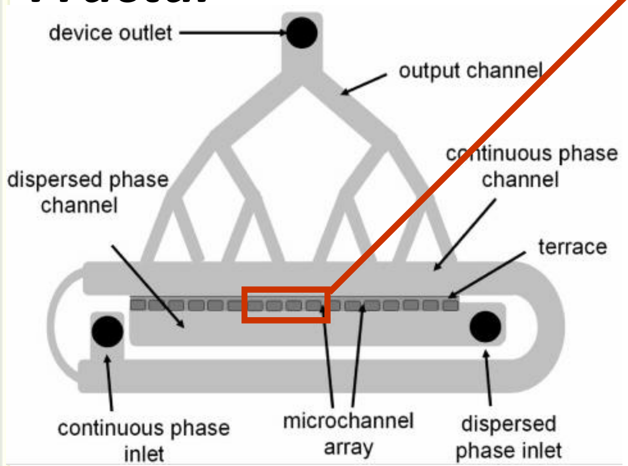
M. Stoeffel et al, Physical Review Letters, 2012

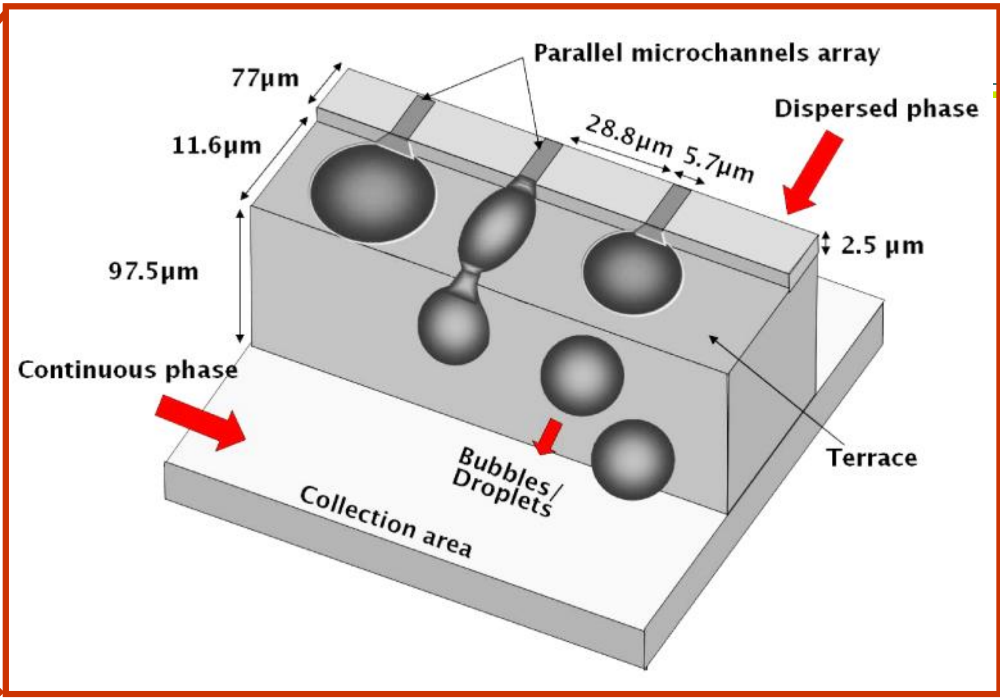
Monodisperse foams by topological transition

Focused



Fractal





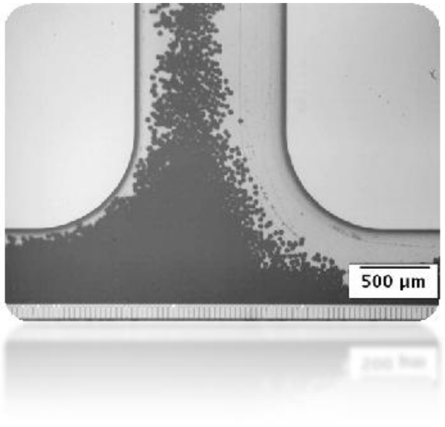
Principle of foam/emulsion generation on a production site

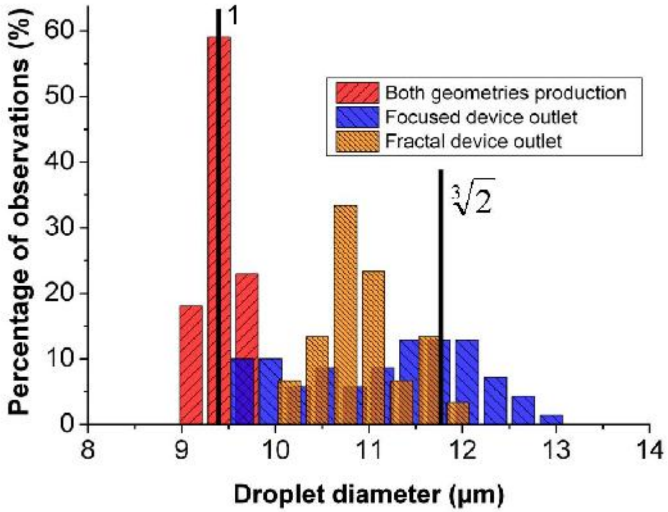
- Collection of the dispersions ***within*** the device
- Observation of the dispersions behaviour ***along the entire device.***
- Production of either ***foams or emulsions.***

M. Stoeffel, D.E. Angelescu, PRL, 2012

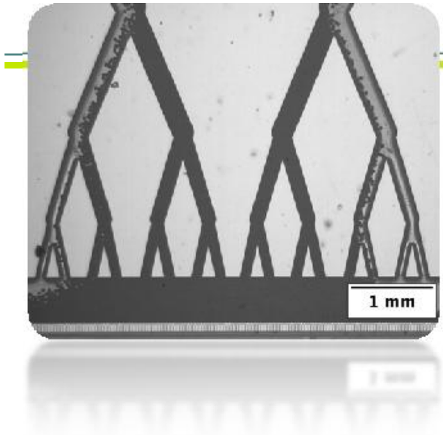
Assumption: Fractal geometry = ***better evacuation?***

Emulsion production with both geometries

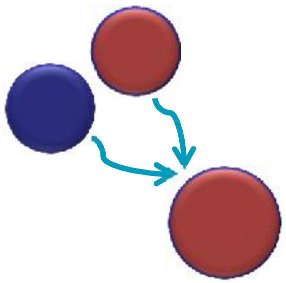




DI Water-SDS 1 wt % at 5µl/min and C_{16} flowrate of 0.1µl/min



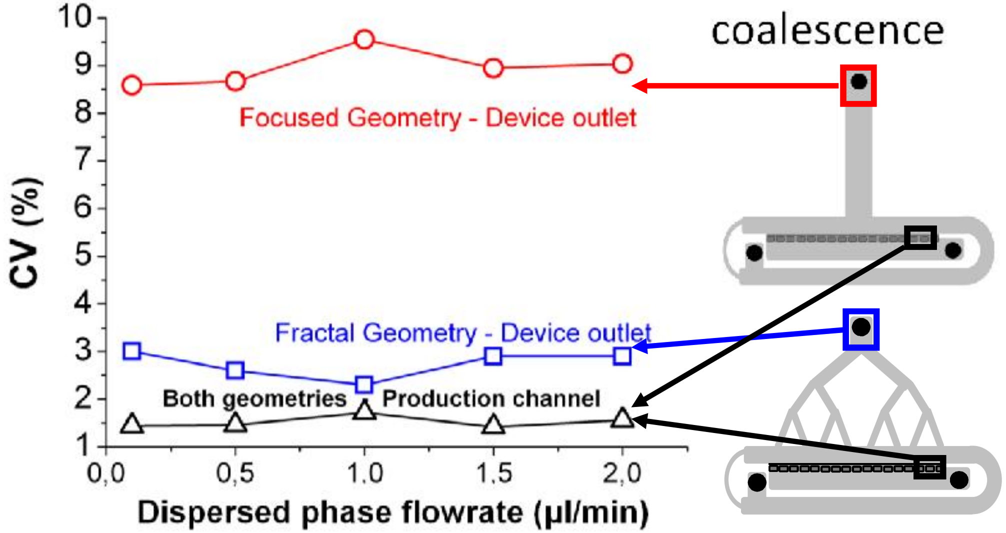
- ***Focused geometry:*** coalescence between two droplets.



$V_{final}=2.V$

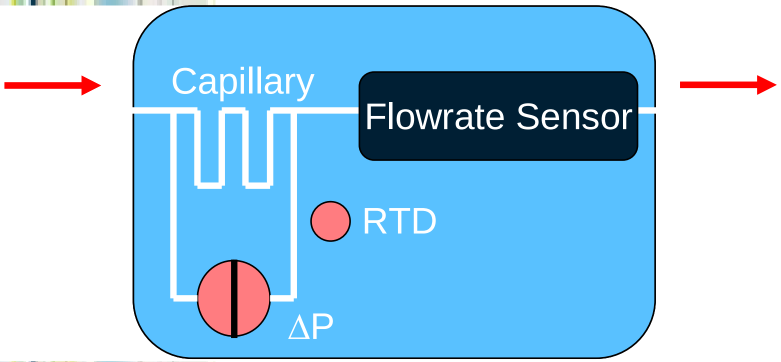
$\Rightarrow D_{final}= \sqrt[3]{2}\ D$

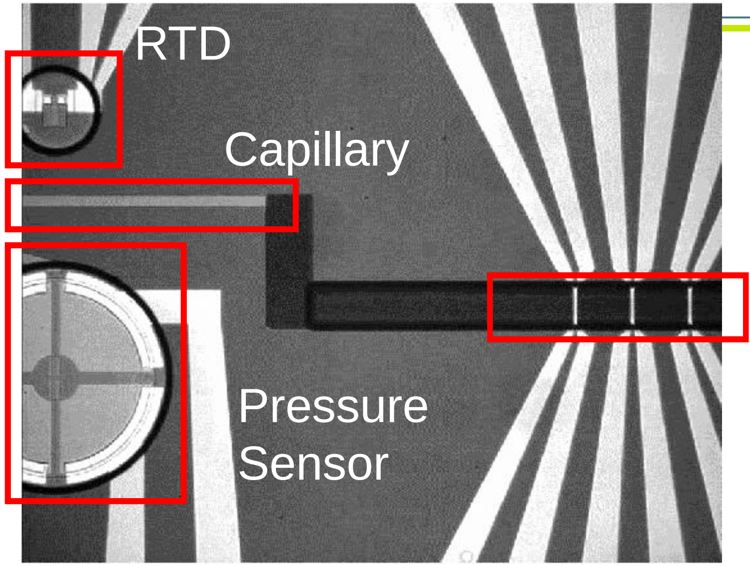
- ***Fractal geometry:*** reduce of the jamming-effect involving less coalescence

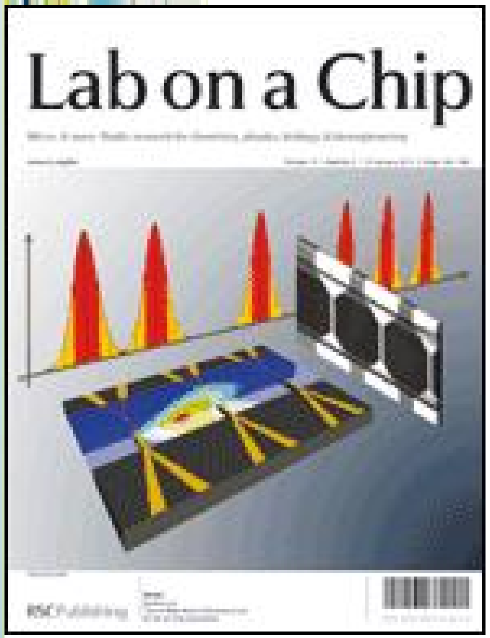


Better CV with the *fractal geometry* at the device outlet.

Rheology on Chip :
Prospects for *in situ* Analysis of Complex Fluids



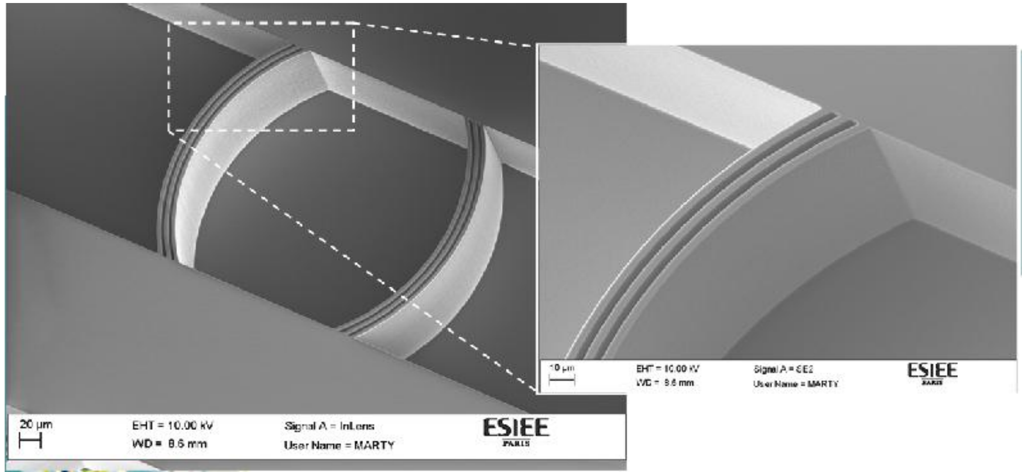




- Simultaneous Measurement of **flow rate** AND **differential pressure**. + Temperature monitoring
- Designed for Complex Fluid Rheology including multiphasic & non-Newtonian fluids
- Precise flowrate measurement according to thermal time of flight method based on stochastic analysis

H. Berthet, J. Jundt, J. Durivault, B. Mercier, D. Angelescu, Lab on Chip, 2011 (Cover Page).

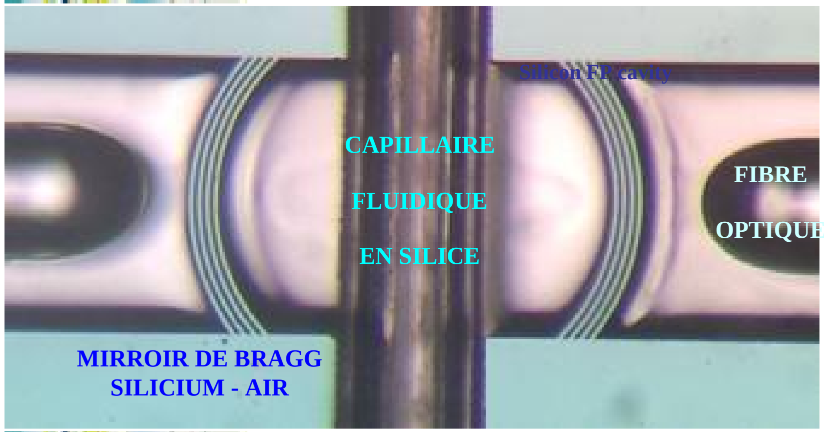
Colloids : The Optofluidic Fabry-Perot Cavity

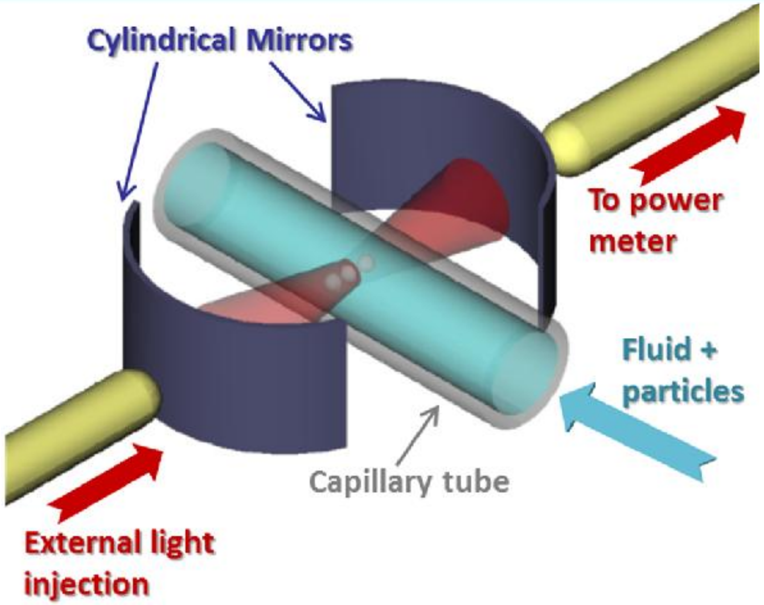


- **Optical Resonance**

Enhances the effective optical path n.L : refraction index measurement in liquids

- **Confinement:** intracavity electromagnetic field is enhanced Optical trapping of particles (including biological cells) before their analysis





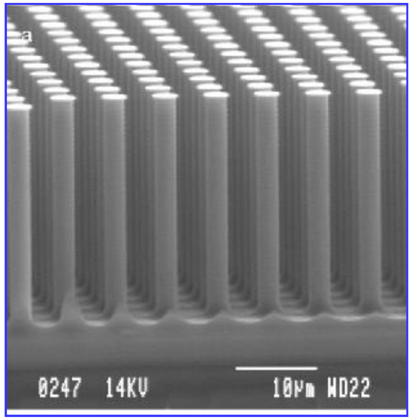
N. Gaber et al., OPTOFLUIDICS 2013, Best Paper Award.

N. Gaber et al. Lab on Chip, 2014

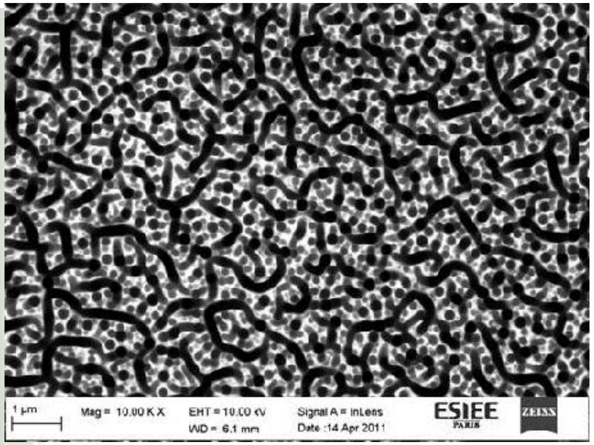
METAMATERIALS 2 :

Periodic Structures from Milli- to Nano- Scale

- Phononic and Photonic crystals

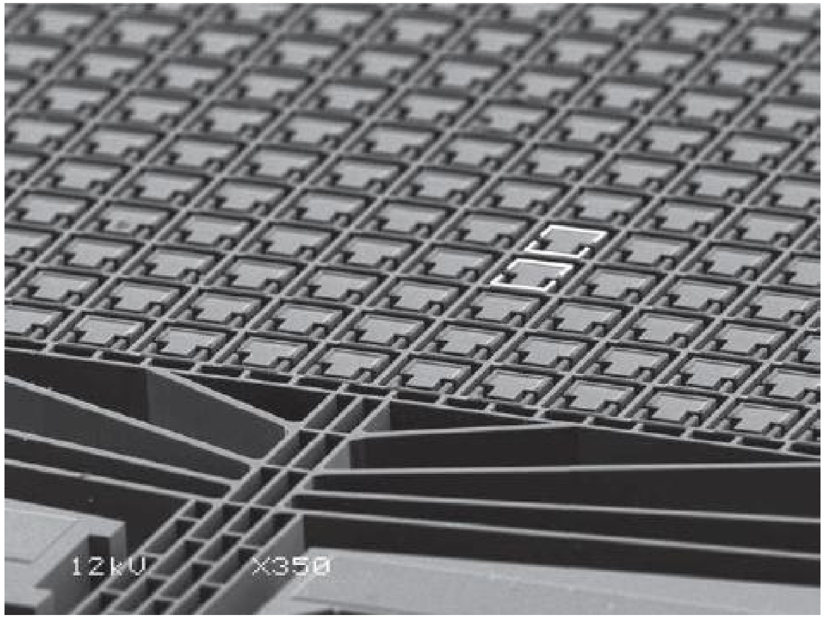


- Black Silicon



- MEMS Tunable Terrahertz Metamaterials

(Thesis of W.M. Zhu, 'UPE joint-supervision with NTU-Singapour, T. Bourouina, Ai-Qun LIU)



W.M. Zhu et al, Advanced Materials, 2011

W.M. Zhu et al, Nature Communications, 2012

III – Matériaux Micro-Structurés pour les Capteurs: Cas du Black Silicon

Etude des mécanismes de formation par Nanotomographie SEM - FIB et Application aux Micro-Capteurs

Thèses de David ABI-SAAB, Kim Ngoc NGUYEN, Sebatiain WAHL

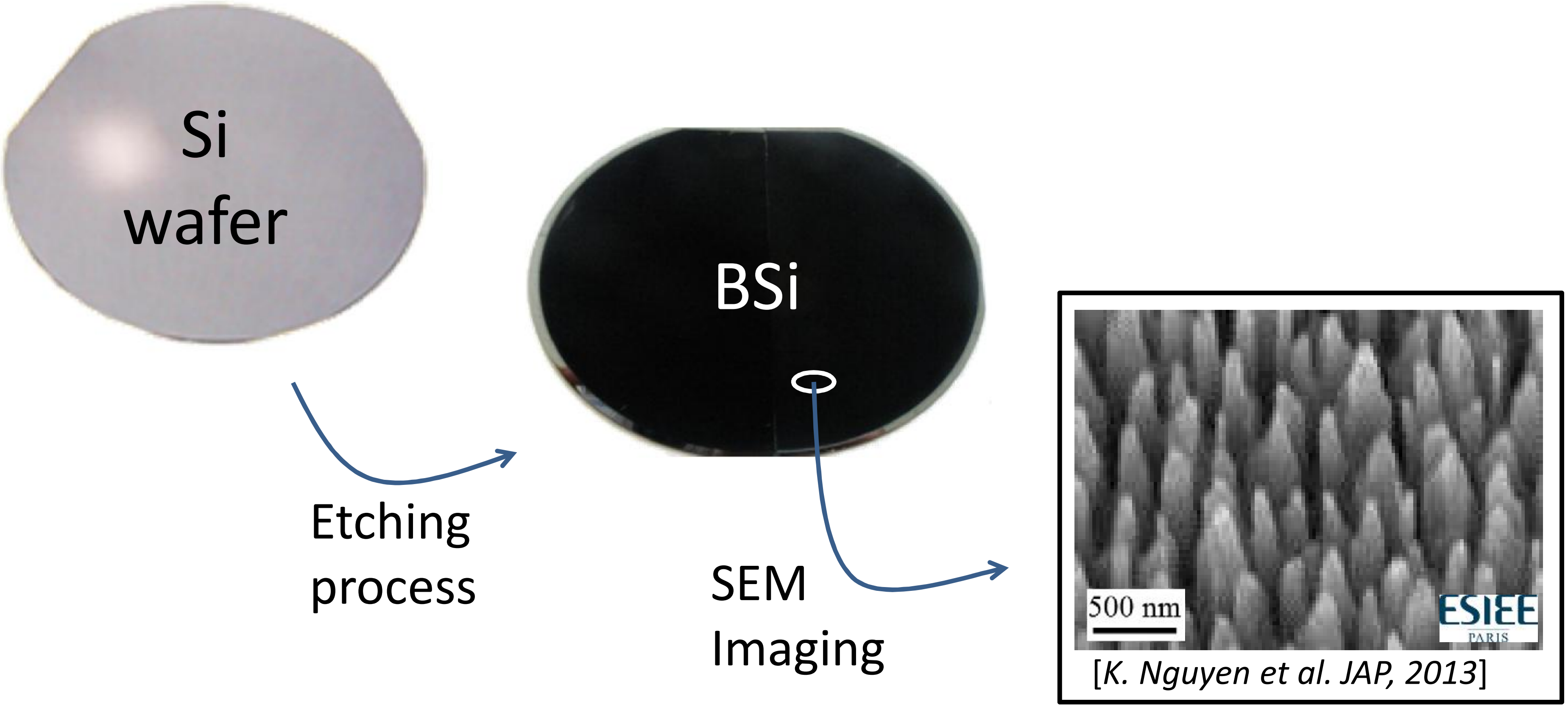
Co-encadrement : Dan ANGELESCU, Philippe BASSET, Tarik BOUROUINA (ESIEE), Elodie RICHALOT, Yamin LEPRINCE (UPEM)

ESYCOM

LABORATOIRE ELECTRONIQUE, SYSTÈMES DE COMMUNICATION ET MICROSYSTÈMES

What is Black Silicon (BSi)?

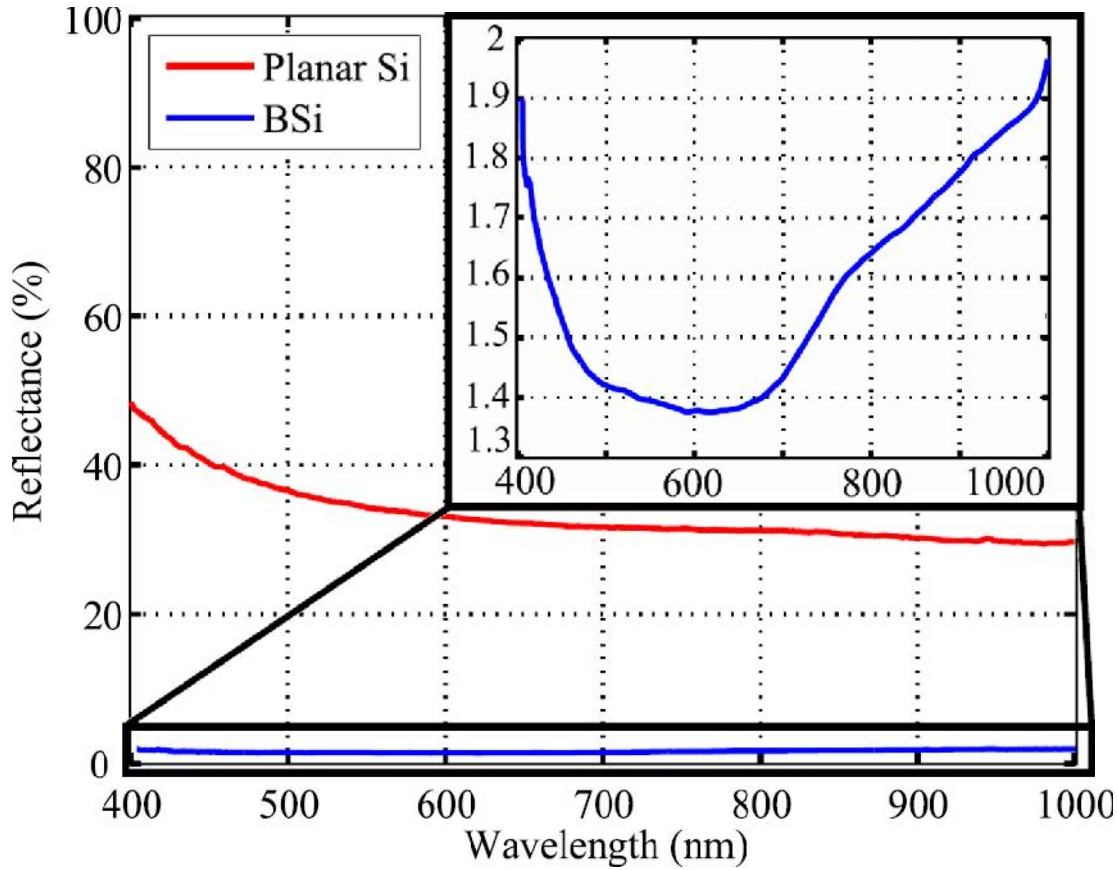
- It is a Si surface that becomes dark because of its three-dimensional micro/nanostructuration



[*K. Nguyen et al. JAP, 2013*]

BSi properties and applications (1)

- Decrease of Si total reflectance (~30%) down to less than 2%



Ex. of BSi applications: PV cells

- BSi can improve the power conversion efficiency of PV cells:

Source	Efficiency without microstructures	Efficiency with microstructures
Burgers et al. ECN, 1998	11.6%	13%
Schnell et al. PSC, 2000	11.3%	12.2%
Oh et al. 2012	13.1%	18.2%

- But it does not yet outperform conventional PV cells with antireflection coating (~24%)

BSi properties and applications (2)

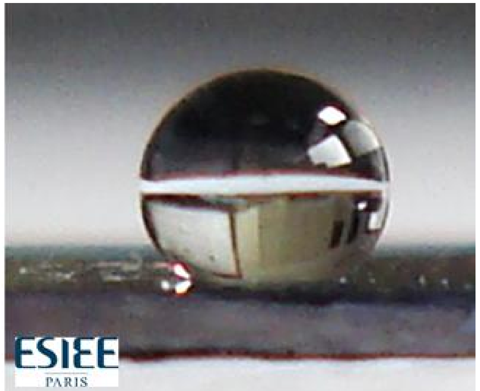
- Decrease of Si total reflectance (~30%) down to less than 2%

- Superhydrophobic (water repelling) or Superhydrophilic (water sticking) surfaces.

BSi + perfluorinated polymer layer ⟹ Superhydrophobic surface

BSi + SiO_2 layer ⟹ Superhydrophilic surface

- **Potential applications in the fabrication of self-cleaning PV cells.**



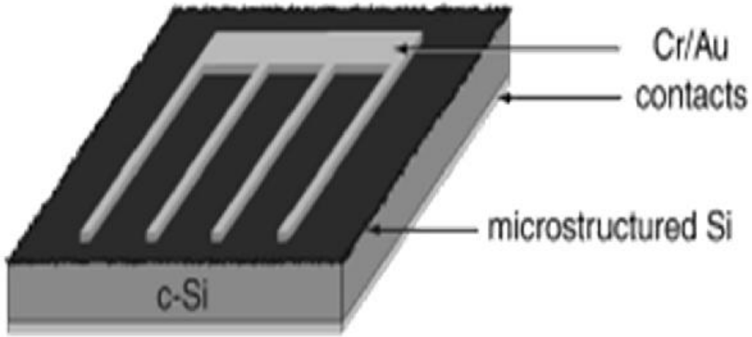
[N. Gaber et al. LoC, 2013]

BSi properties and applications (3)

- Decrease of Si total reflectance (~30%) down to less than 2%
- Superhydrophobic (water repelling) or Superhydrophilic (water sticking) surfaces.
- Increase of surface area to more than 10 times with respect to a planar surface
  - Increase of catalytic activity in micro-reactors and fuel cells electrodes
  - Increased sensitivity in glucose biosensors

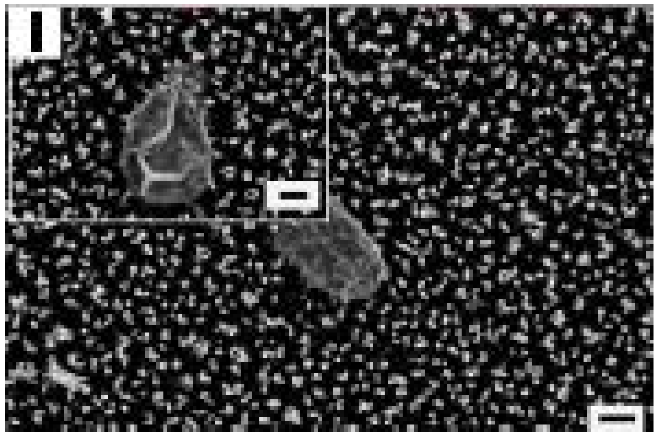
Ex. of other BSi applications

- Photodetectors



[J. Carey et al. APL, 2001]

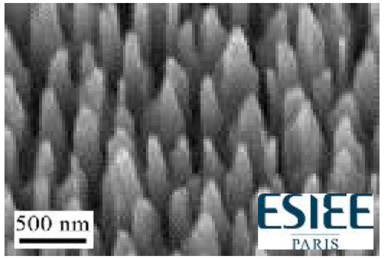
- Thermal absorbers
- Support for biological samples
- Bactericidal materials



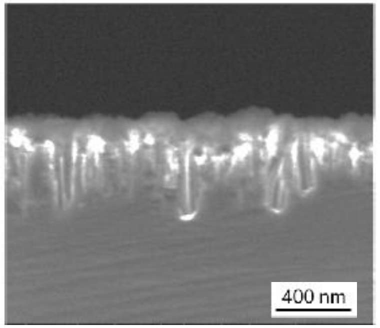
[E. P. Ivanova et al. Nat. Commun. 2013]

BSi fabrication methods

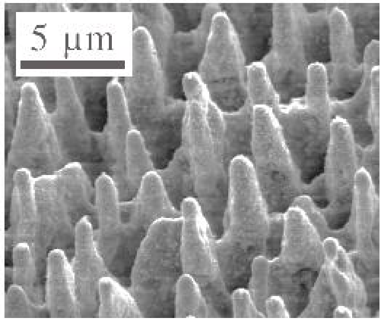
- Plasma etching (Maskless)
  - **Continuous cryogenic deep reactive ion etching (cryo-DRIE)**
  - Time-multiplexed deep reactive ion etching at room temperature (Bosch process)
- Wet etching (Nanomasking)
  - With prior nanometric particles deposition by evaporation or coating with solution containing nanometric particles.
- Laser-chemical etching (Maskless)
  - With femtosecond laser pulses in the presence of etching gases.



[K. Nguyen et al. JAP, 2013]



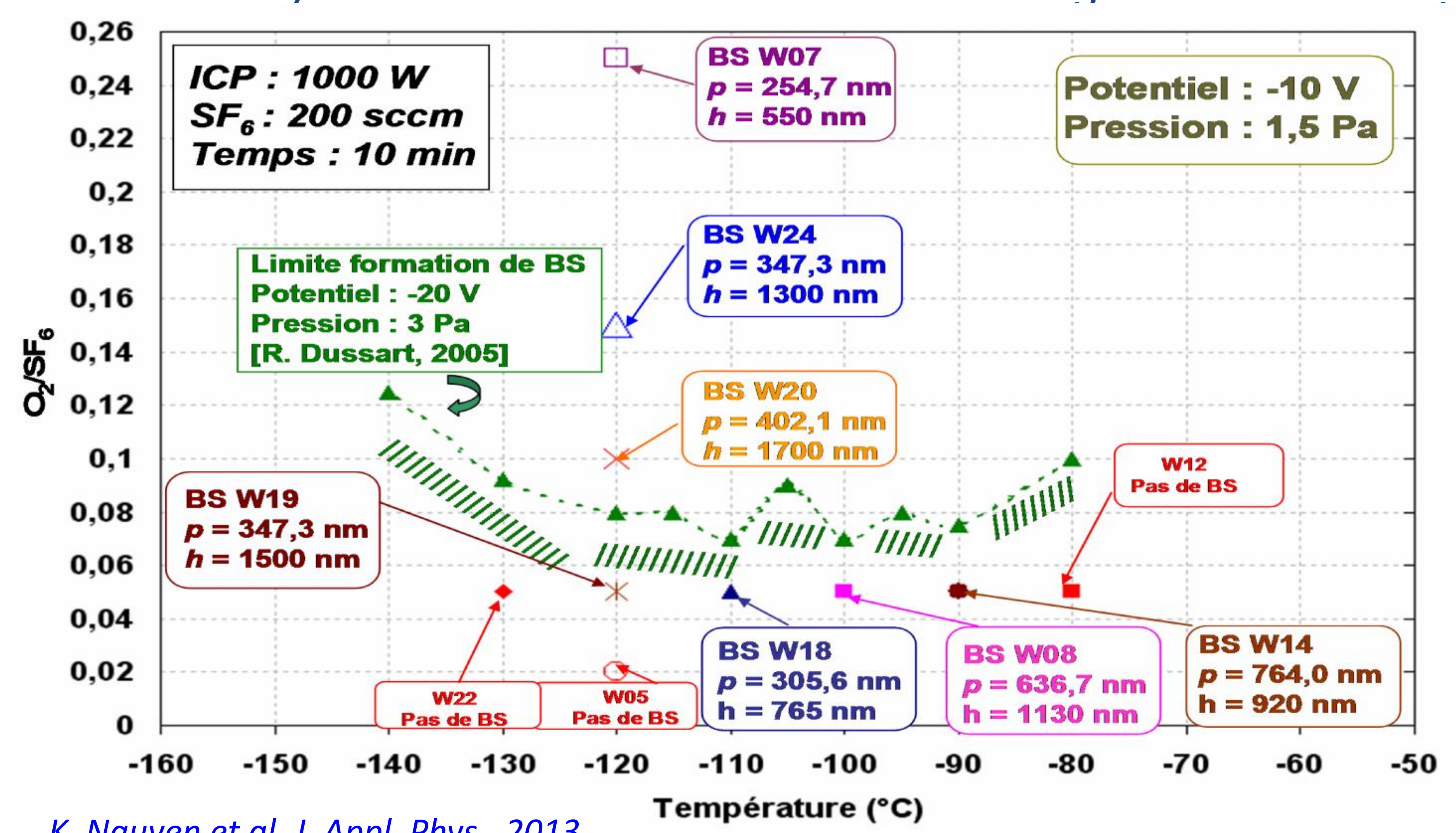
[H.-C. Yuan et al. PVSC, 2009]



[C.H. Crouch et al. Appl. Phys. A, 2004]

Bottom-up, « Random » Microstructuration

Study of Black Silicon obtained from Crogenic ICP Etching

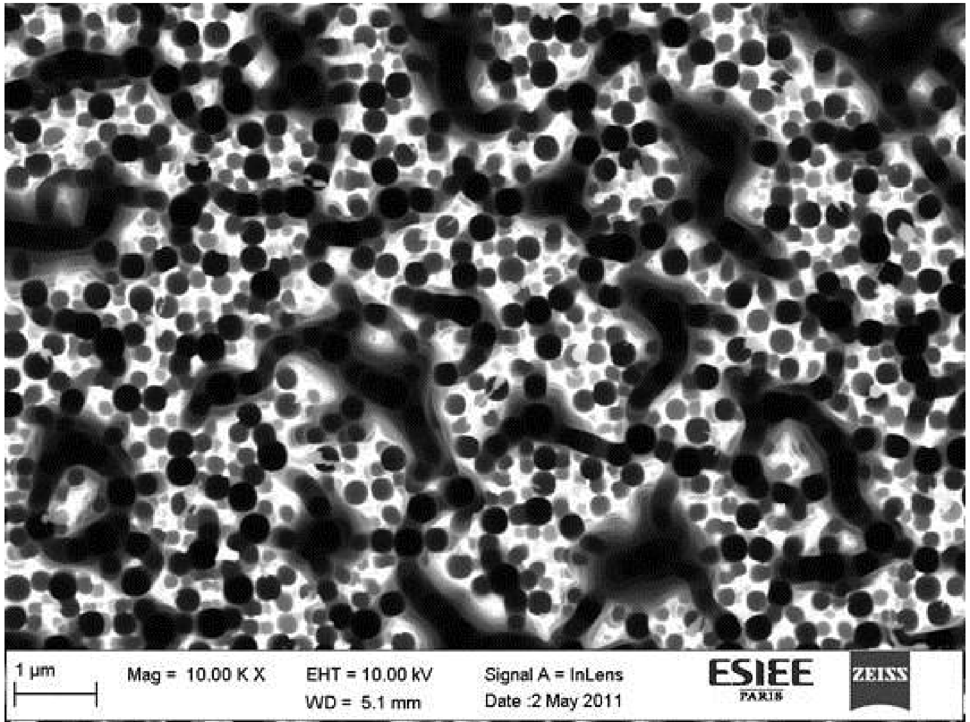


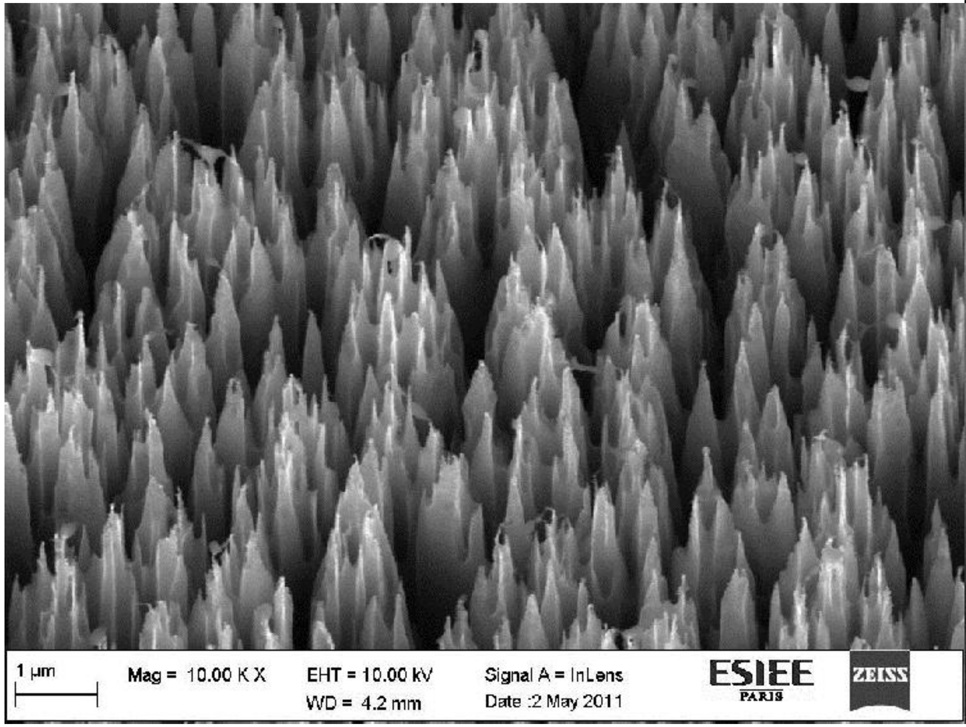
K. Nguyen et al. J. Appl. Phys., 2013

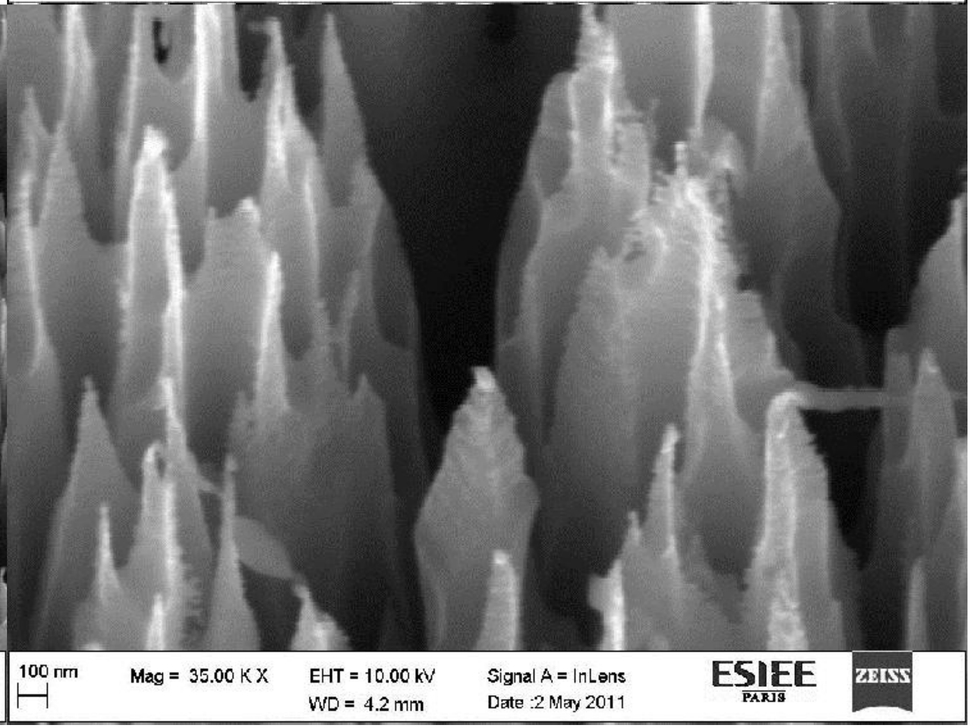
W15

O_2/SF_6	0.05
V Bias	-10 V
T°	-120°C
Durée	**10 min**

- Zone BS noire et homogène
- Structure avec des points et des groupes des points de taille µm sur lesquels il y a des très petits pics.
- **Hauteur :** grande structure : ~3 µm, moyenne : ~ 1.7 µm, petite : ~ 0.3 µm
- **Largeur :** grande structure : 1 – 1.4 µm, moyenne : 0.3 – 0.5 µm, petite : 0.1 – 0.2 µm
- **Périodicité moyenne :** 955,0 nm

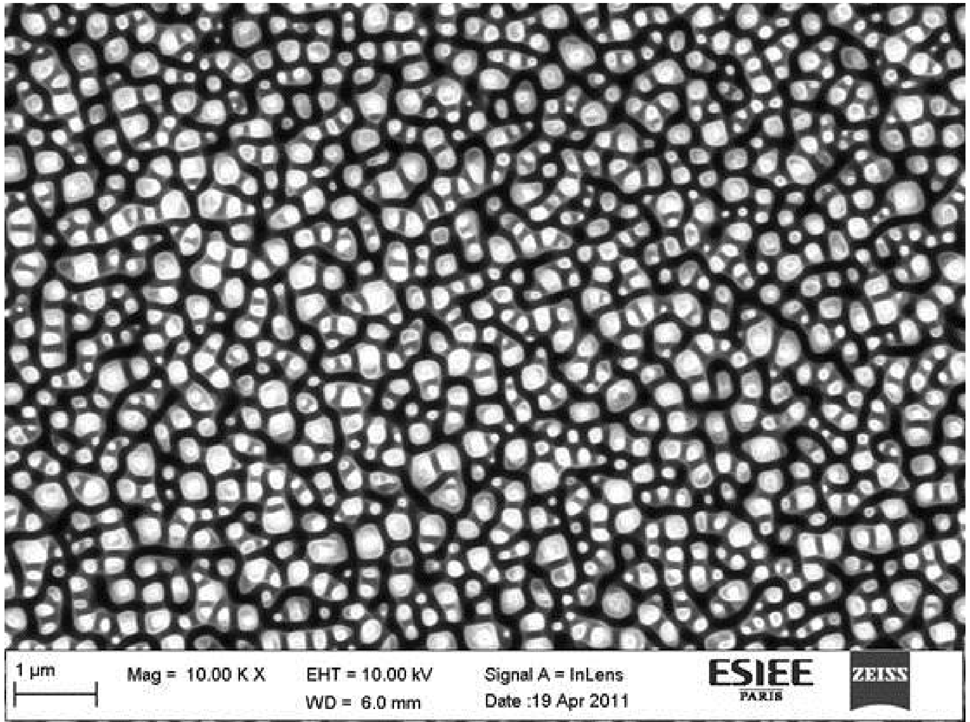




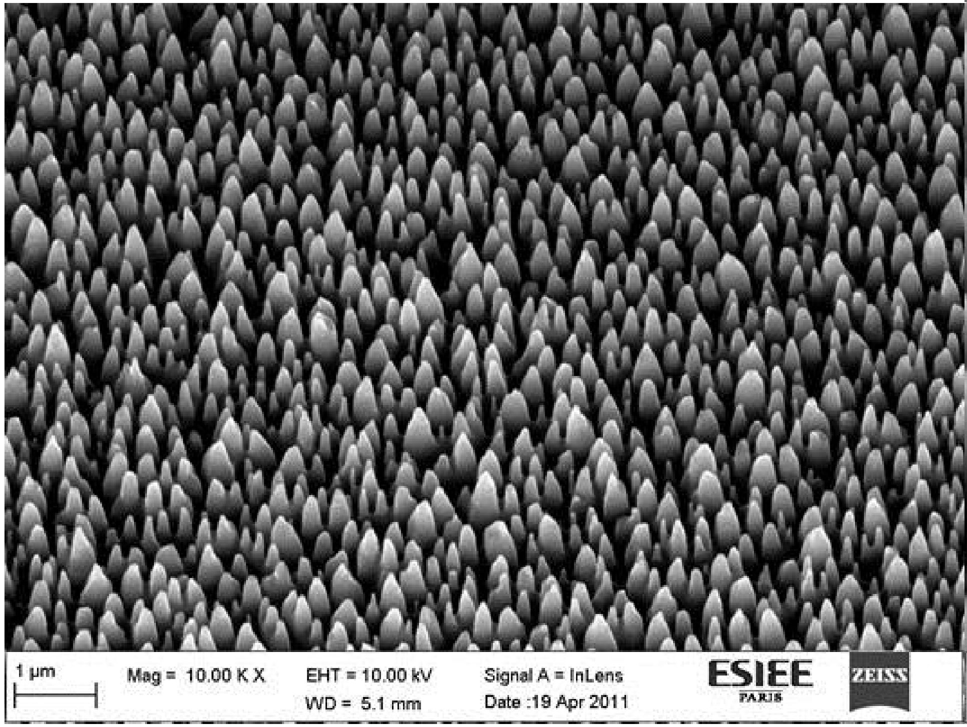


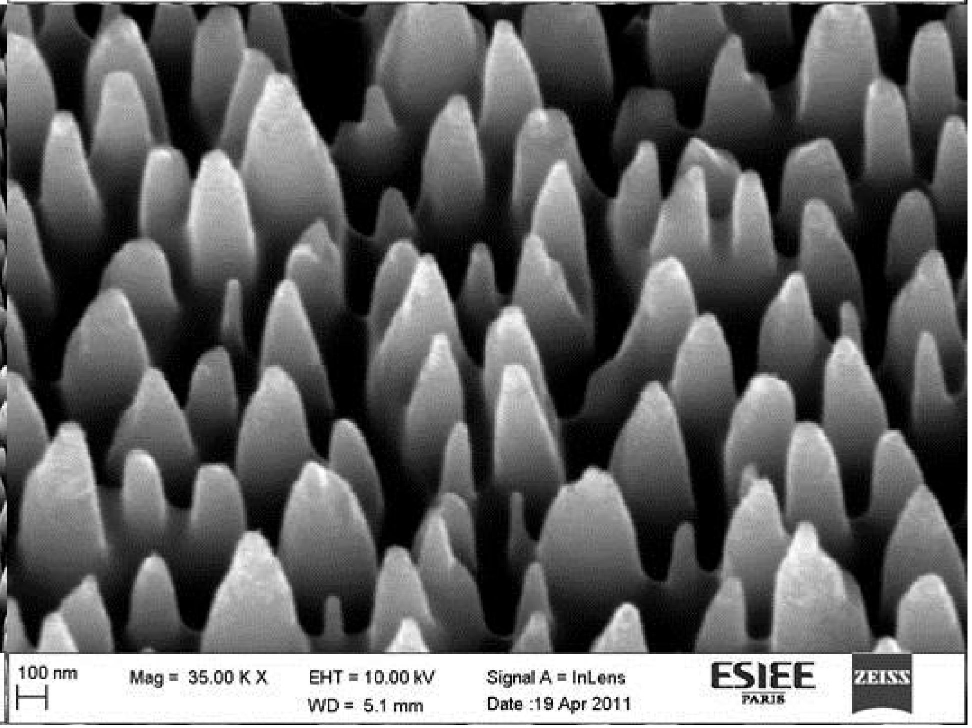
W10

O_2/SF_6	0.05
V Bias	-10 V
T°	-120°C
Durée	**5 min**



- Zone BS noire et homogène
- Surface pas très dense de 3 types: petits, moyens et grands pics pas pointus et « penguin-like » structure
- **Hauteur :** ~ 650 nm
- **Largeur :** 140 – 350 nm,
- **Périodicité moyenne :** 363,8 nm

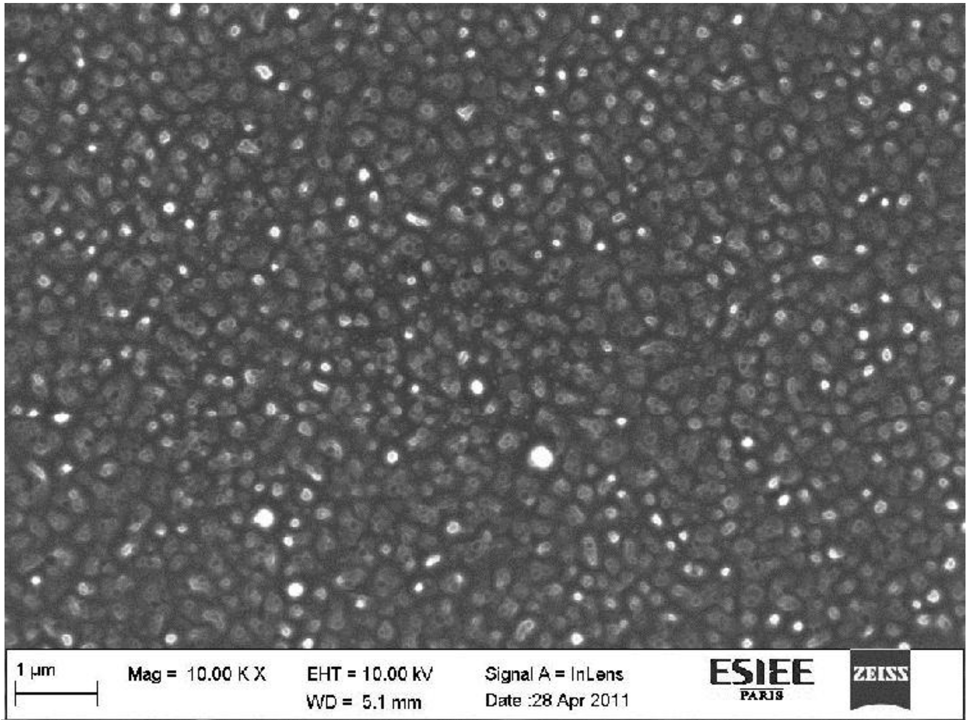


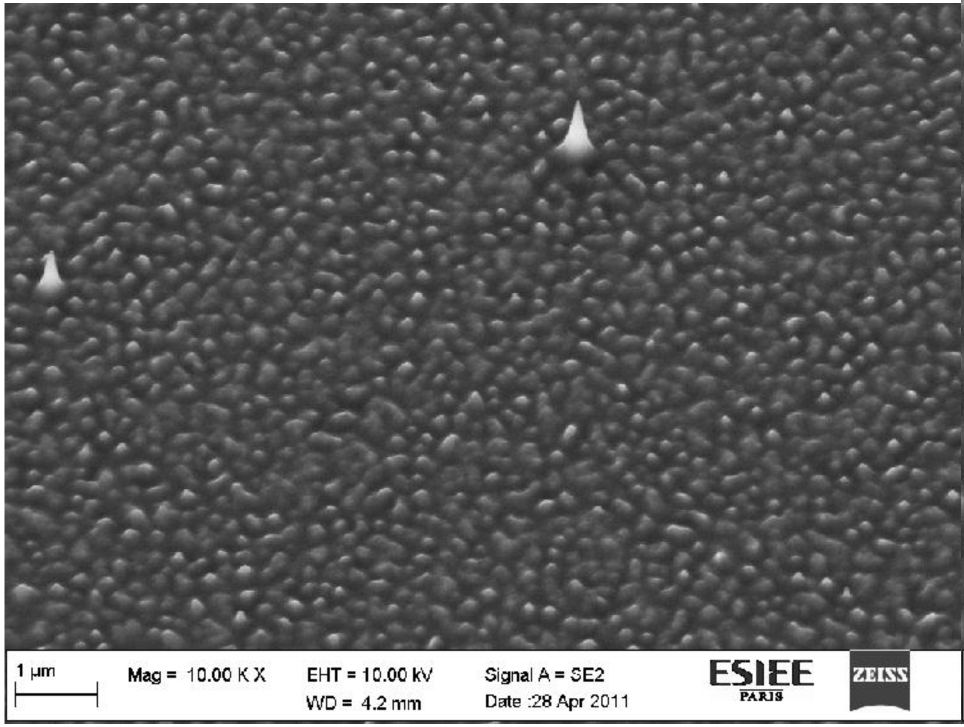


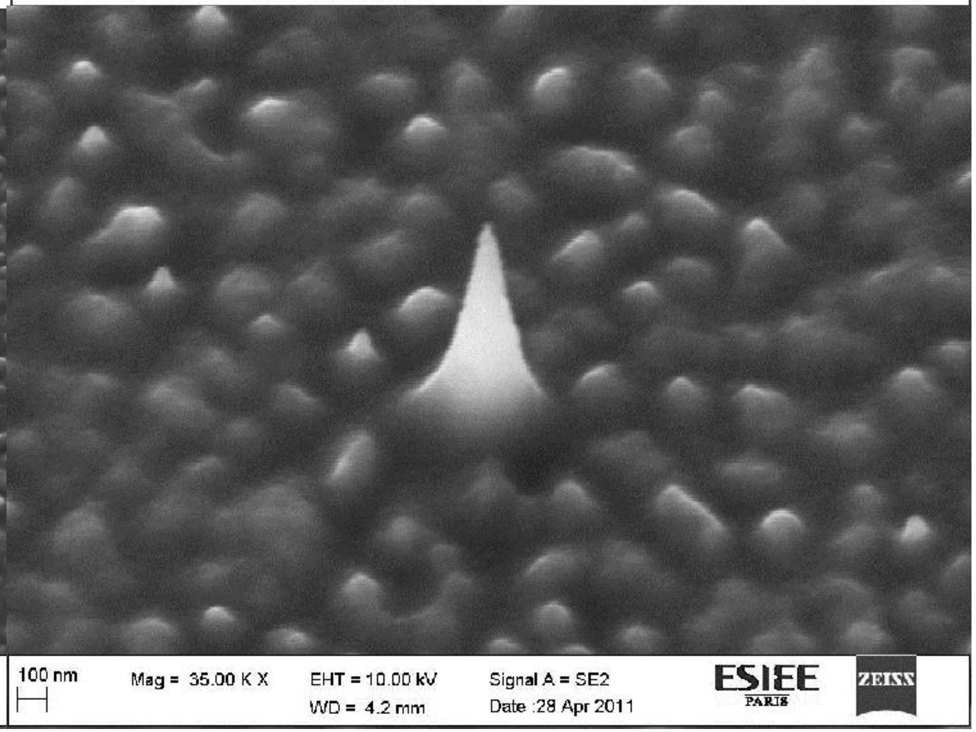
W22

O_2/SF_6	0.05
V Bias	-10 V
T°	**-130°C**
Durée	10 min

- **Zone 1: zone grise**
- La rugosité: ~ 100 nm
- Point plus grand: **hauteur :** ~ 600 nm, **largeur :** ~ 400 nm

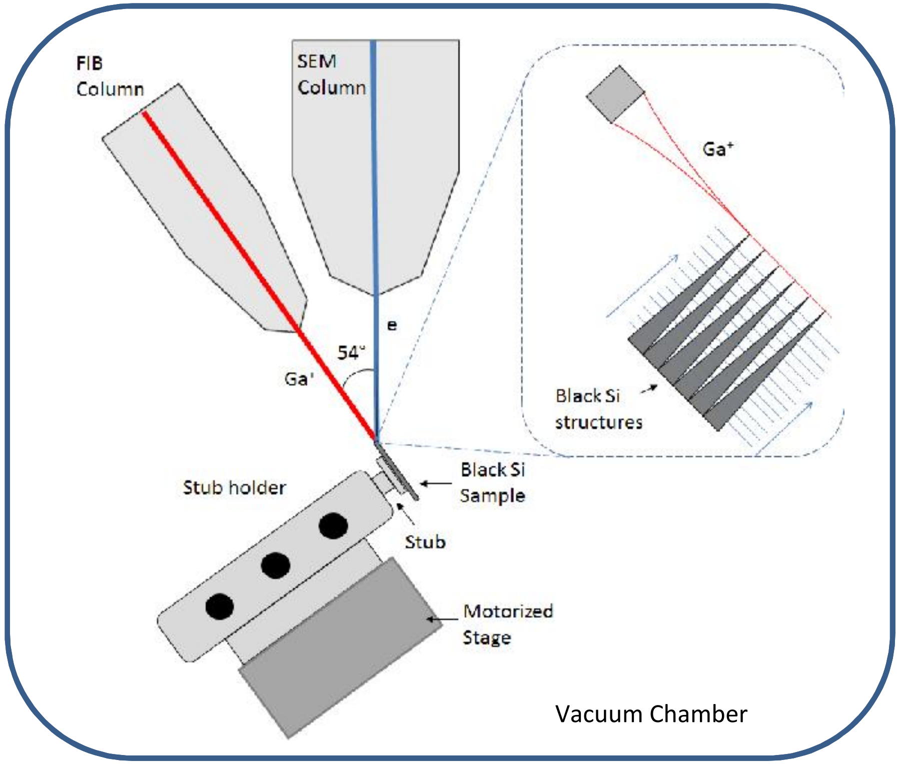






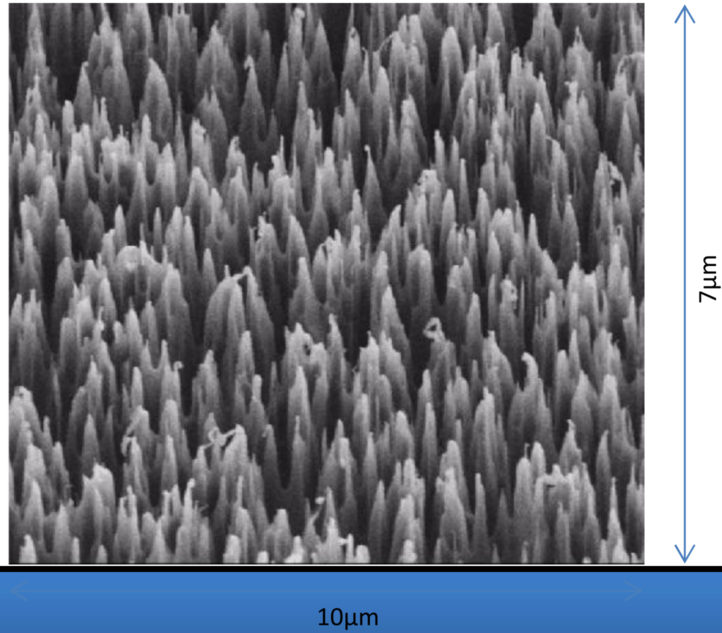
SEM-FIB Dual-Beam System Experiment:

Ion Beam Milling & SEM Imaging



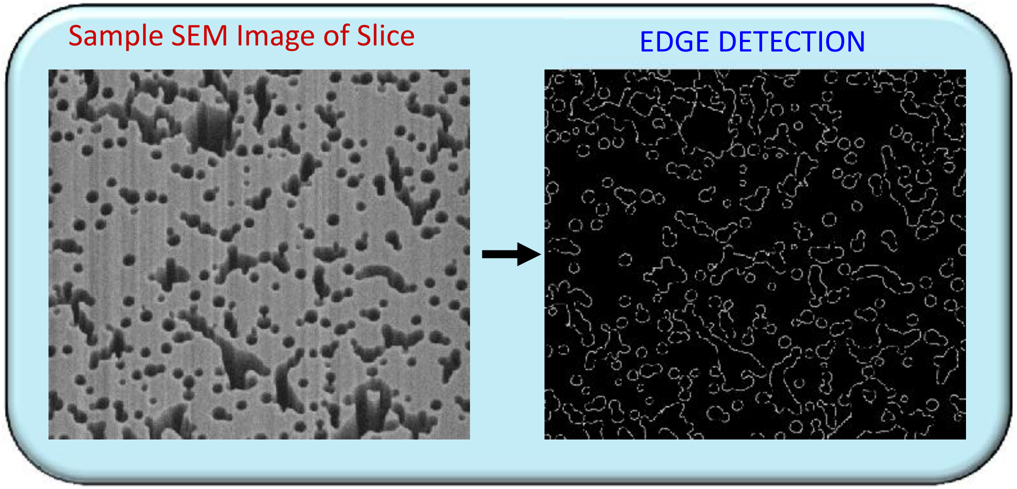
FIB-SEM Setup

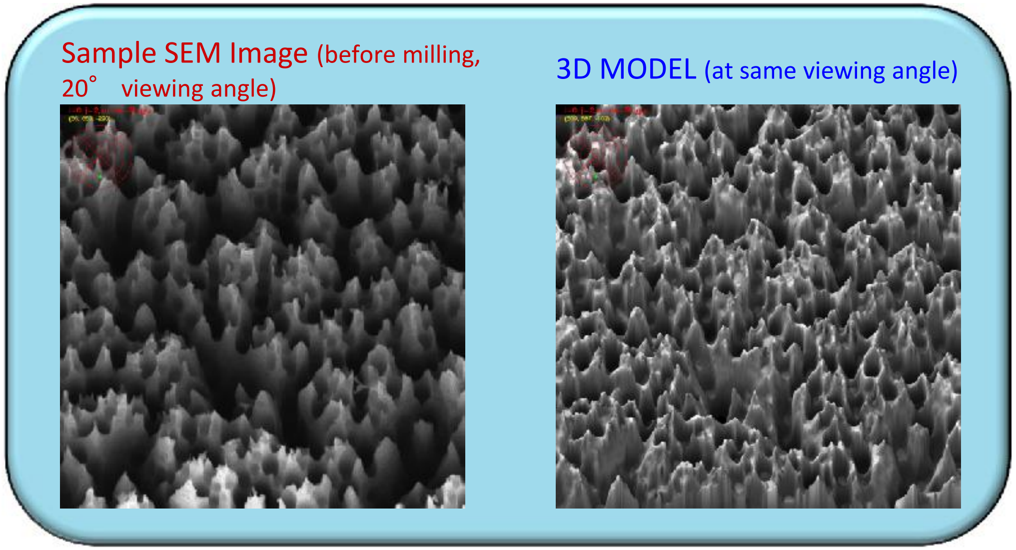
Black Si sample FIB milling & SEM imaging sequence



3D Reconstruction Algorithm Steps

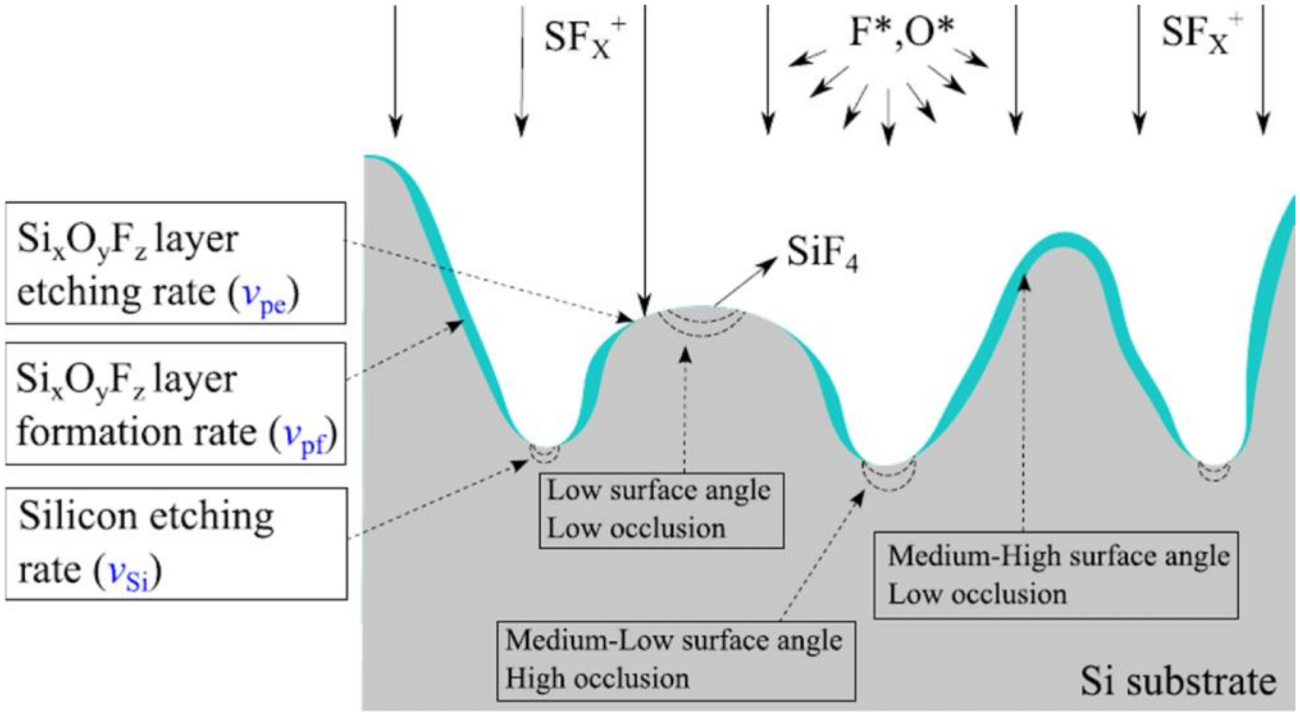
1. Slice SEM Image pre-treatment (registration and translation corrections)
2. Slice SEM Image edge detection.
3. 3D Reconstruction with edge data from each slice and translation measurements.
4. 3D scattered data interpolation.
5. 3D model qualitative comparison with previously taken SEM images of the sample at different angles.





BSi formation mechanism

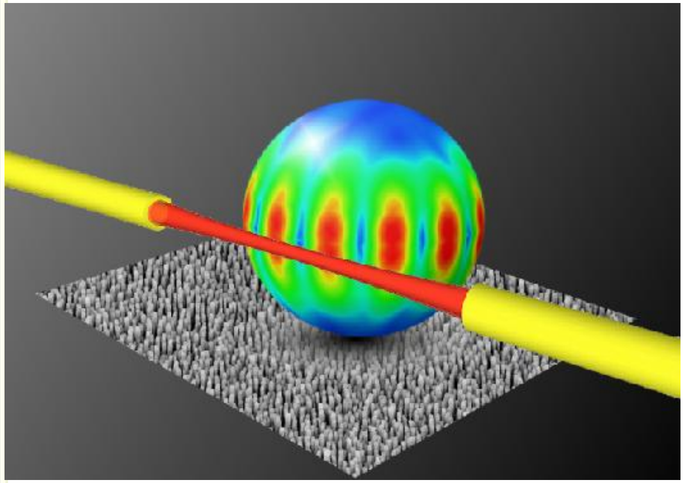
- Flat surface is unstable to holes formation.
- Passivation layer formation is hindered in bottom of holes due to high occlusion.
- Top of needles are overpassivated due to high exposure.

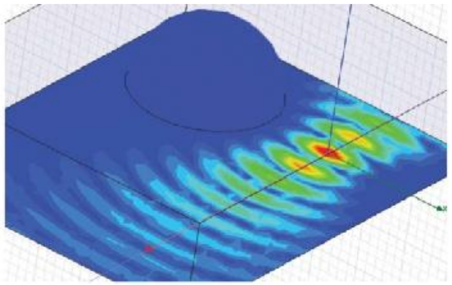


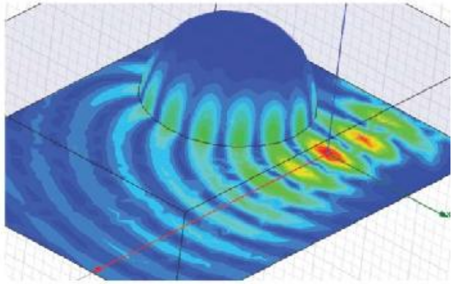
- Passivation formation and etching are balanced on saddle points.

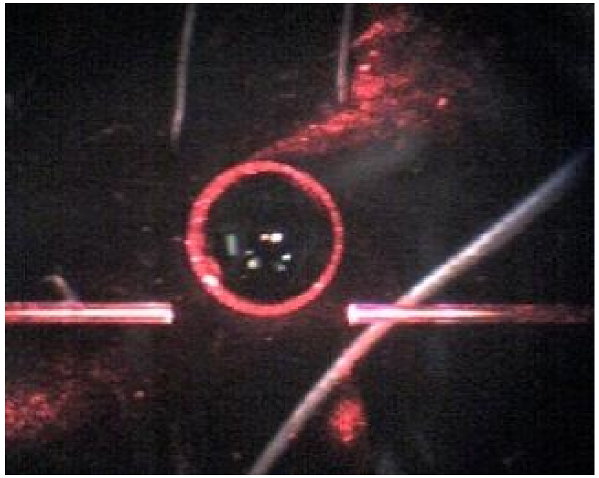
D. Abi-Saab et al. Phys. Rev. Lett, 2014

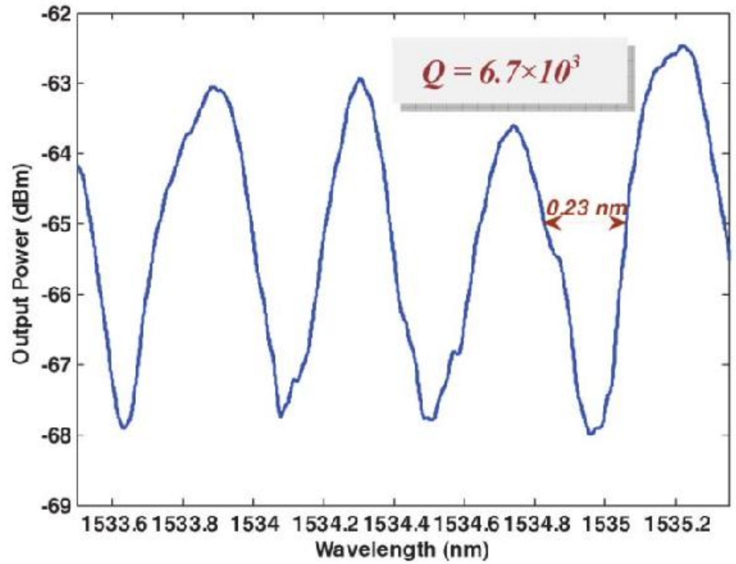
Application of BS : (1) Free-space Light coupling into liquid droplet resonator (Whispering Gallery Modes)









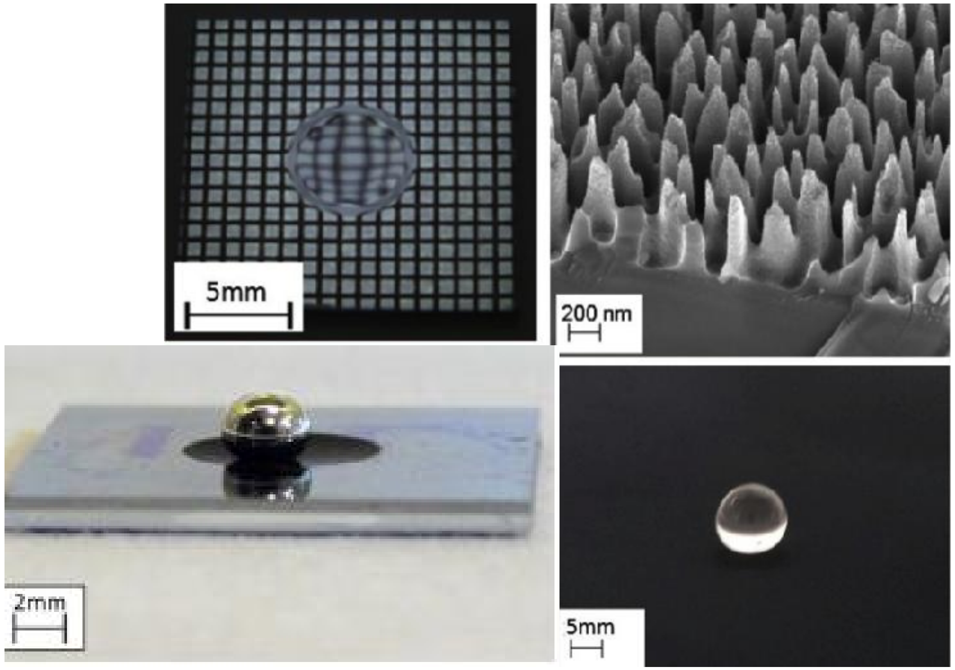


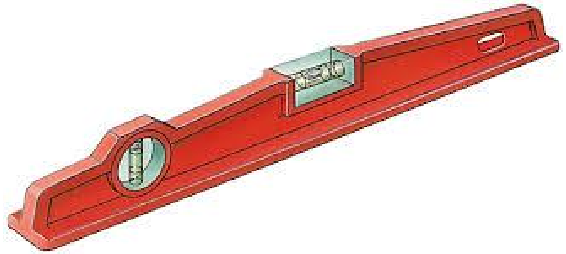
N. Gaber et al, Lab on Chip, 2013

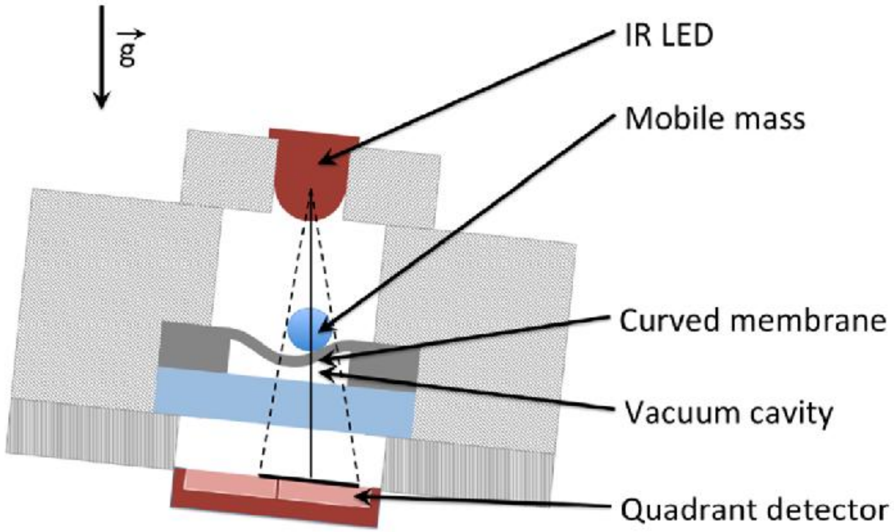
Application of BS : (2) Dual-Axis Inclinometer with sub-0,01° precision

- **VERTICALITY : A serious issue in Civil Engineering (Structural Health Monitoring)**
  - Optofluidic inclinometer

→ **Based on nanostructuration and functionnalization of curved membranes** (patent pending).
Provides the main advantage of reducing contact angle hysteresis







S. Wahl et al, IEEE MEMS'2014

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