Laboratoire de l'Intégration, du Matériau au Système
(IMS-UMR5218)

Des micropoutres piézoélectriques en couches épaisses sérigraphiées

Screen-printed thick-films of piezoelectric cantilevers

Claude **LUCAT**, *Directeur de Recherches CNRS*
Hélène **DEBEDA**, *Maître de Conférence Bordeaux 1*

cnrs dépasser les frontières
UNIVERSITÉ DE BORDEAUX
UNIVERSITÉ BORDEAUX 1 Sciences Technologies
IPB INSTITUT POLYTECHNIQUE BORDEAUX
ENITA de Bordeaux

Position in the IMS Lab

- Pole 'from material to component'
  - 'Microsystems' Group
    - MDA Team (Acoustic wave based Detection Microsystems)
    - **MMM Team (Microassembled Materials for Microsystems)**

MMM Staff (january 2011)

- 6 (+1) Permanent positions

  1 senior researcher,

  2 professors,

  1 junior researcher +1 junior researcher for 2 years

  2 associate professors
- 1 Post-doc + 1 temporary engineer
- 6 PhD students

MMM research

Aim

- Innovative technological processes for MEMS
- New devices for chemical detection
- Original characterization methods of materials properties

Scientific actions

- Microcantilever based sensors
- MEMS polymer for detection
- **Thick-film components and microsystems**

This presentation

Microstructuration processes

Silicon

(nm → µm)

LIGA

….

Microstereolithography

MEMS

Thick-films

(µm → mm)

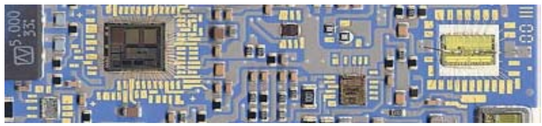
- Electrochemical deposition
- LTCC and HTCC
- Ink-jet
- Screen-printing ?
- …

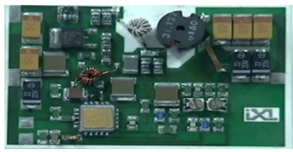
Standard screen-printed thick-films

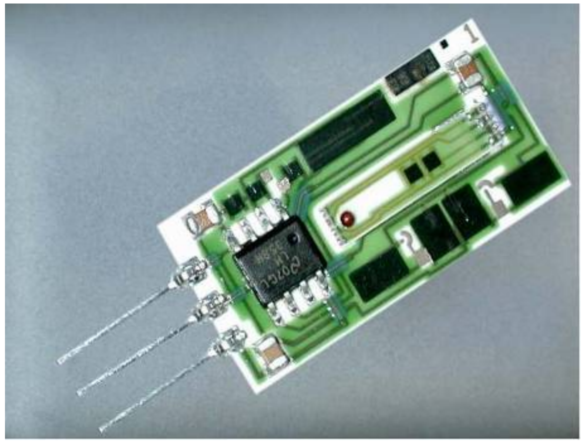
Electronic applications

- **Already used before World War II for electronics**
  - ➢ 1943 : mass-produced hybrid circuits for detonators

- **Today: packaging applications (SMT soldering, conductors, passive components) and sensors**







Context

Standard screen-printed thick-films for **SENSORS**

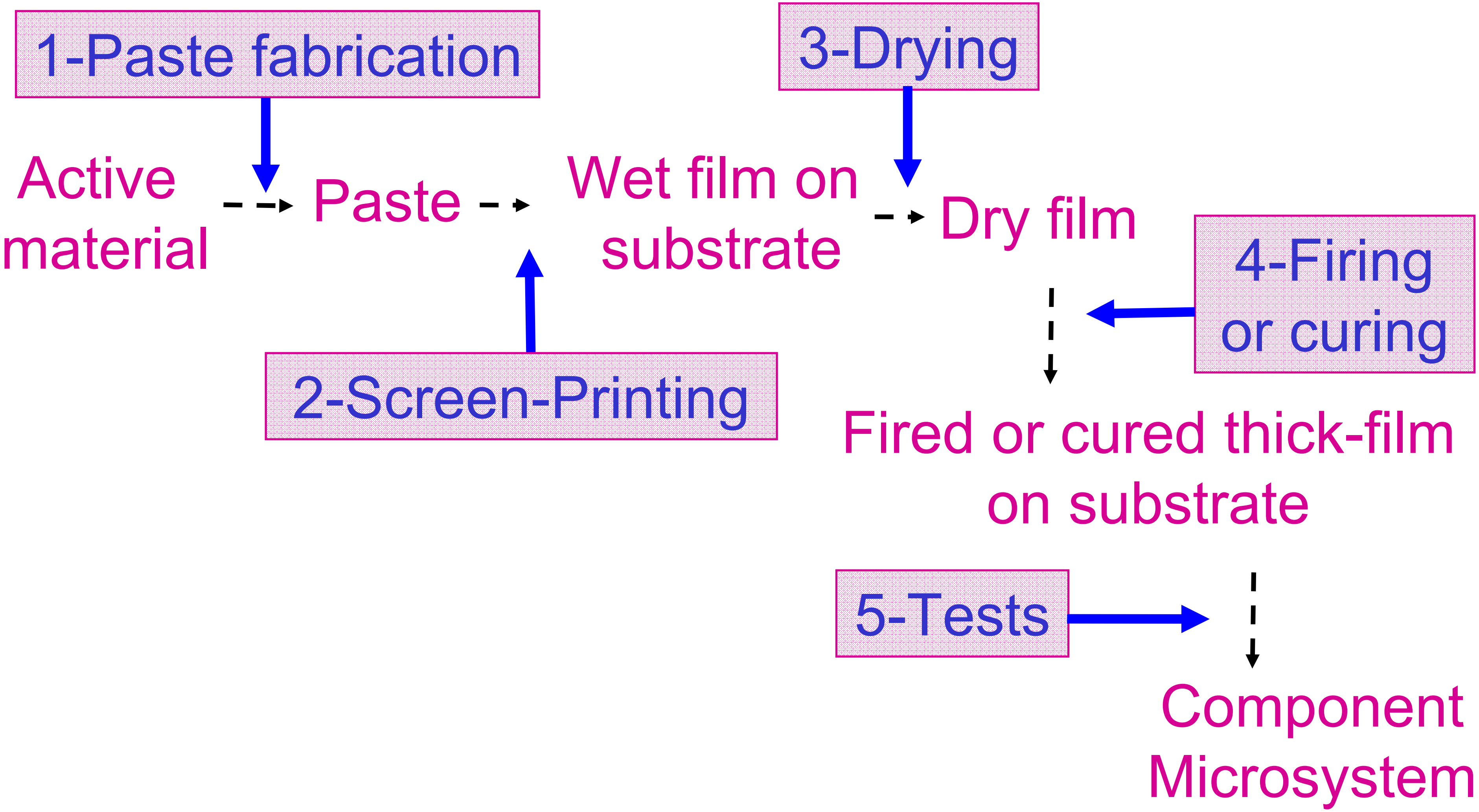
Free-standing thick films?

MEMS:

sensors , **actuators**

Screen-printing process

Standard process



Screen-printing process

Paste fabrication


Weighting


Planetary ball-mill


Hand mixing


Mixer


Viscosity, printability

- **Polymer** : Curing time 20 min at 120<T(°C)<150.

  Structural material (Ag, C, dielectric powder,…)

  + epoxy or polyester, phenolic or….

- **Mineral** : Firing temperature 400 < T(°C)< 1100

  Active material (powders Au, Pt, Cu, AgPd, PtAu, Ni, dielectric ($BaTiO_3$+ glass), resistors (RuO_2 + glass + …)

  + organic vehicle (solvants+binding polymer)

  + mineral binders (glass, oxides, eutectic, …)

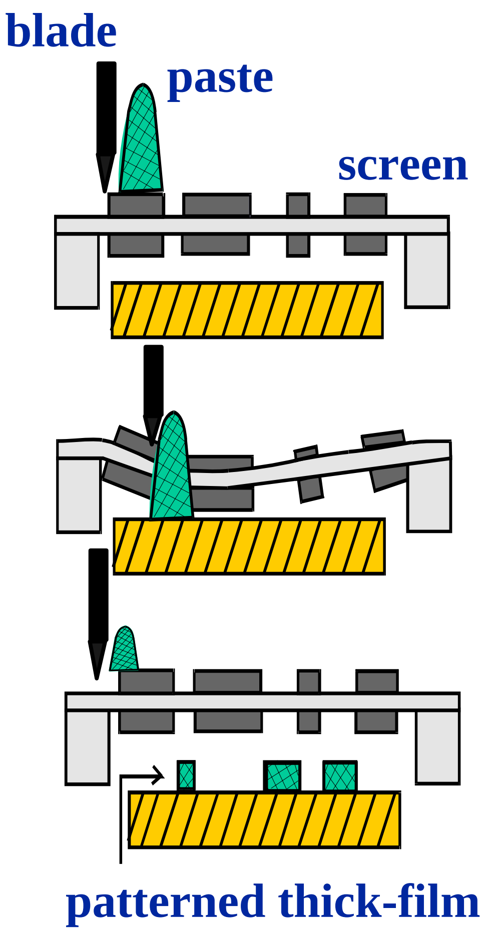
Screen-printing process

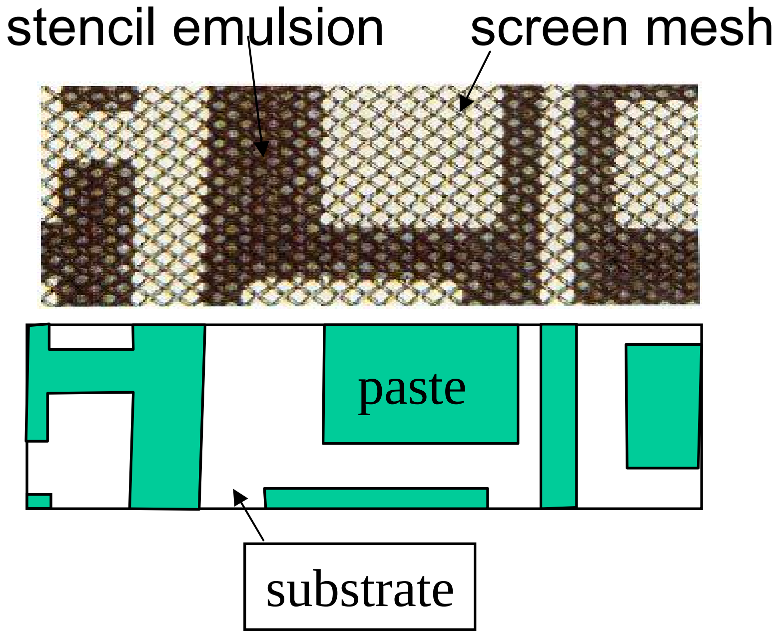
Substrate ⟷ Application

- Alumina
  - ➢ Doctor blade tape casting process
  - ➢ 4% SiO_2/MgO promotes the adhesion of the film
    - ✓ reaction with the mineral binder
    - ✓ reaction with the active material itself
- Aluminium nitride, Silicon carbide, AlSiC
  - ➢ Better thermal conductivity
  - ➢ Pre-oxidation often required
  - ➢ Intermetallic compounds with metallic pastes ?
- Yttrium stabilized zirconia, Silicon, Glass, Metal, Polymer, PCB, …

Screen-printing process

Screen-printing





Screens

Stainless steel mesh

Polyester mesh

Metallic or plastic stencil

- Monolayer or multilayers

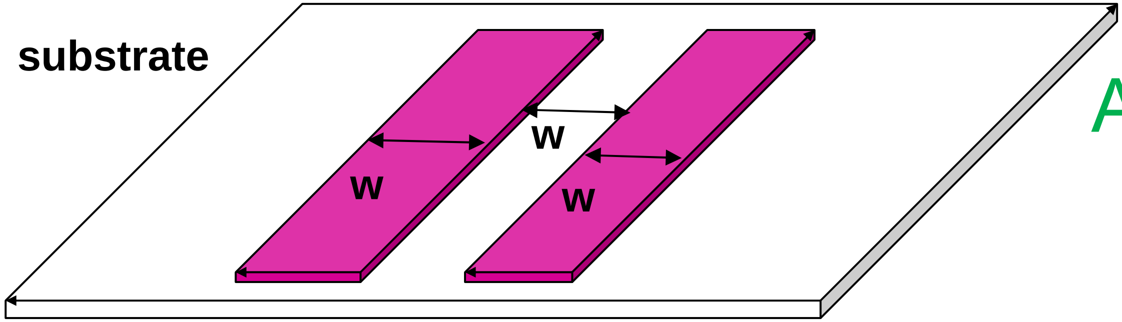
Screen-printing process

Dimensions

Thickness depends on:

- screen-printing parameters (equipment settings, screens mesh, emulsion…)
- paste viscosity

Standard process → Surface > 100µm×100µm, 5 <thickness (µm)< 20



Aspect ratio ≅ 0,1- 0,2 (silicon > 50)

Combined with lithography or with UV sensitive ink → Surface > 30µm×30µm, thickness ~7-8µm

Screen-printing process

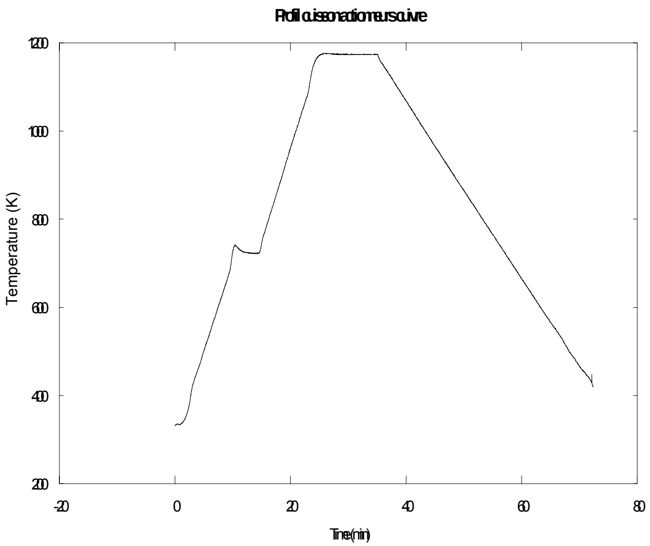
Thermal treatment

- **Drying step**: ~20min at 120°C

  - solvents evaporation

oven

- **Firing or curing process**: 15min to some hours at T_{Max}= 120 to 1000°C



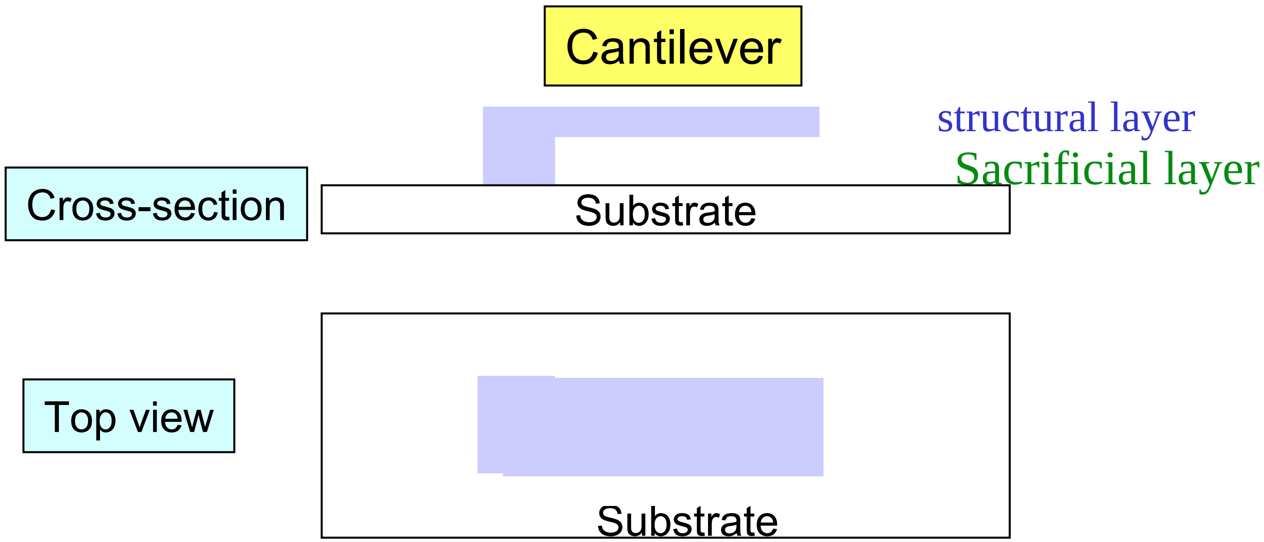
Belt furnace

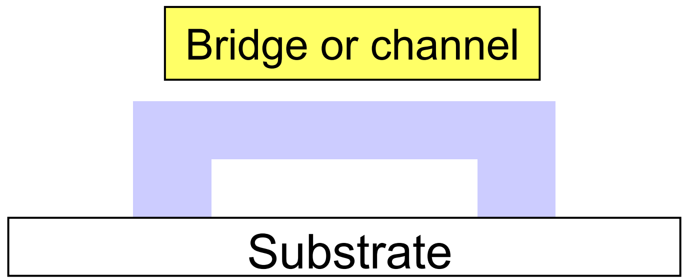
Controlled temperature profile

- **Possibility of cofiring**

Free-standing thick-films

New releasing process of thick-films*





- ✓ **Sacrificial layer deposition**
- ✓ **Consolidation**
- ✓ **Structural layer deposition**
- ✓ **Final firing**
- ✓ **Removal of sacrificial layer**

***CNRS/UnivBx1/IMS Patent**
WO 2007077397, EP 1968885, FR 2895986

Free-standing thick-films

Key parameters of the process

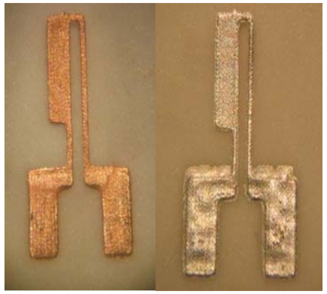
Sacrificial layer ≡ temporary stable support

- ✓ material choice
- ✓ paste fabrication
- ✓ firing conditions
- ✓ removal of the sacrificial layer
  (without any change of the sample properties)

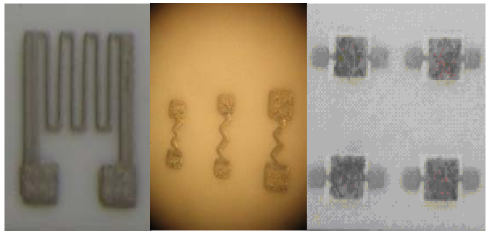
Sacrificial layer = epoxy + $SrCO_3$

Free-standing thick-films

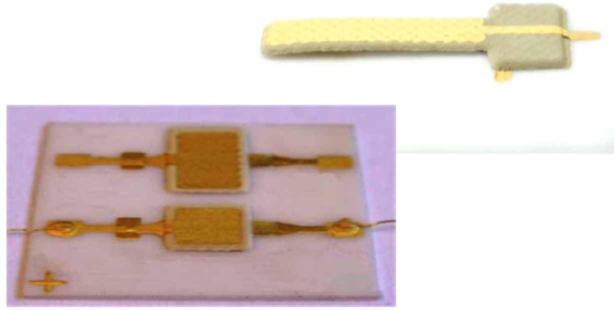
Process capabilities



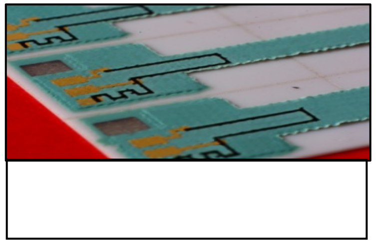
Electrothermal microatuator



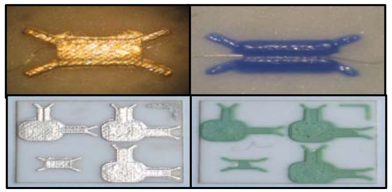
Heating resistors: bridge and cantilever



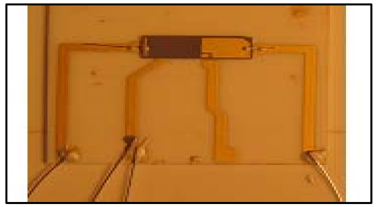
Piezoelectric microsystems: bridge and cantilever



Force sensor



Microchannels

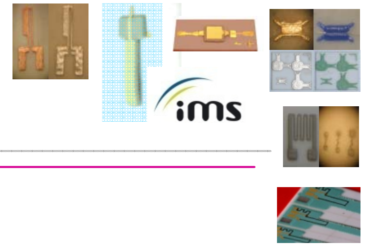


Piezoelectric transformer

Applications:

- Sensors
- Actuators
- Transducers
- Microfluidic
- Microreactors
- Micro fuel-cells

Piezoelectric microsystems



• Faisabilité de composants piézoélectriques libérés à base de PZT

• Amélioration des propriétés du composant à base PZT

• Applications capteur, actionneurs, transducteurs et récupération d'énergie,

• Etude de matériaux sans plomb:BZT

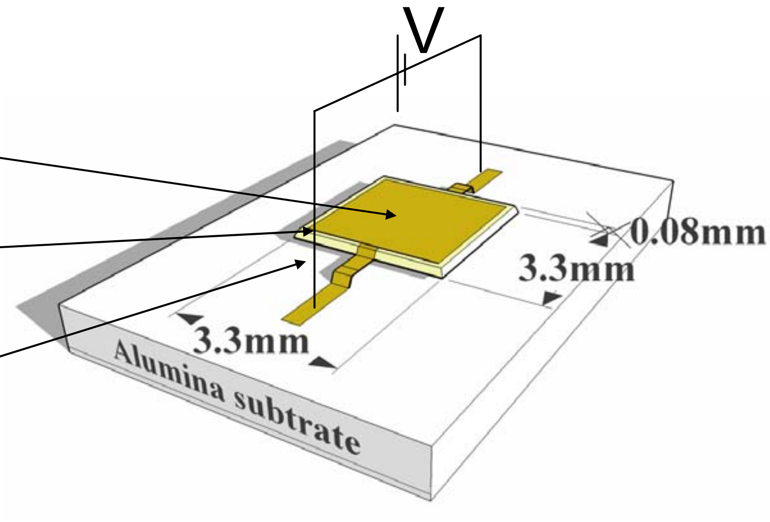
Piezoelectric microsystems

Bridge type

Gold electrodes
≈12µm

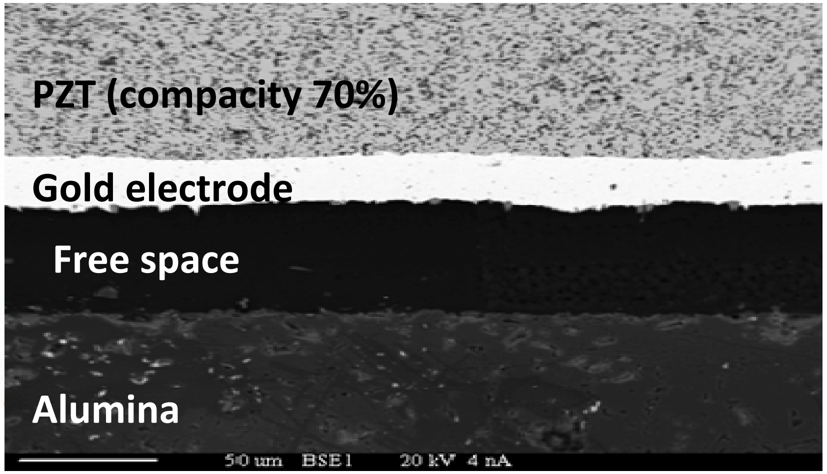
PZT(Zr/Ti = 52/48 + 4%PbO/PbF_2)
≈80µm

Sacrificial layer
($SrCO_3$+epoxy) ≈30µm



Stages

- ✓ Firing 20 min at 920°C
- ✓ Isostatic pressure (1kBar)
- ✓ Elimination
- ✓ Poling (270°C, 500V)



Piezoelectric microsystems

Bridge type

Sample design → expect two kinds of resonance mode

<table>
<tr><td>Mechanical vibrations</td><td>Out of plane
(parallel to the poling axis)</td><td>In-plane
(perpendicular to the poling axis)</td></tr>
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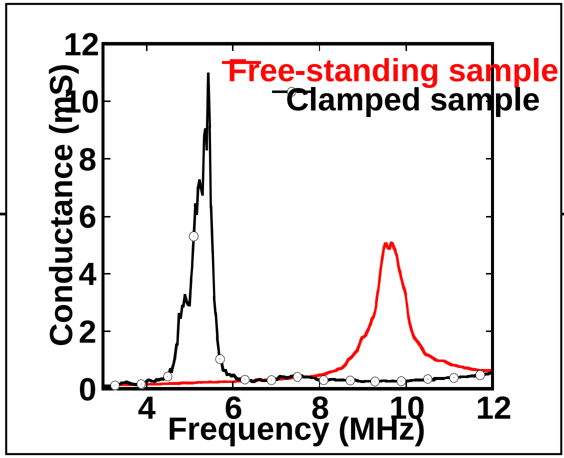
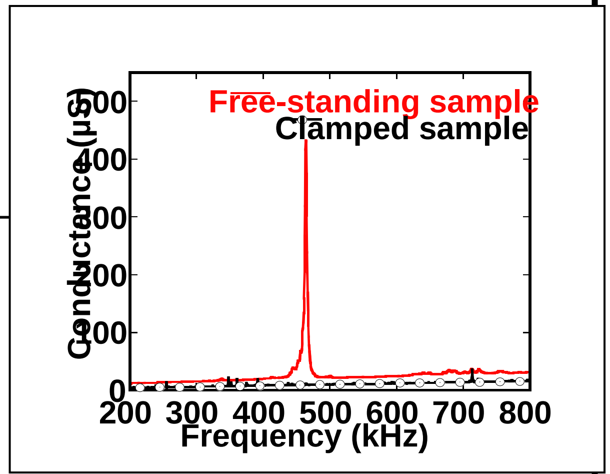
Clamped</td><td rowspan="2">
</td><td rowspan="2">
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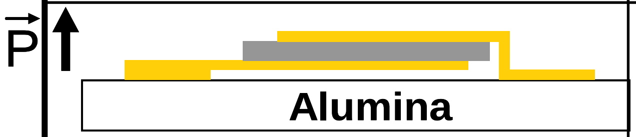
<tr><td>

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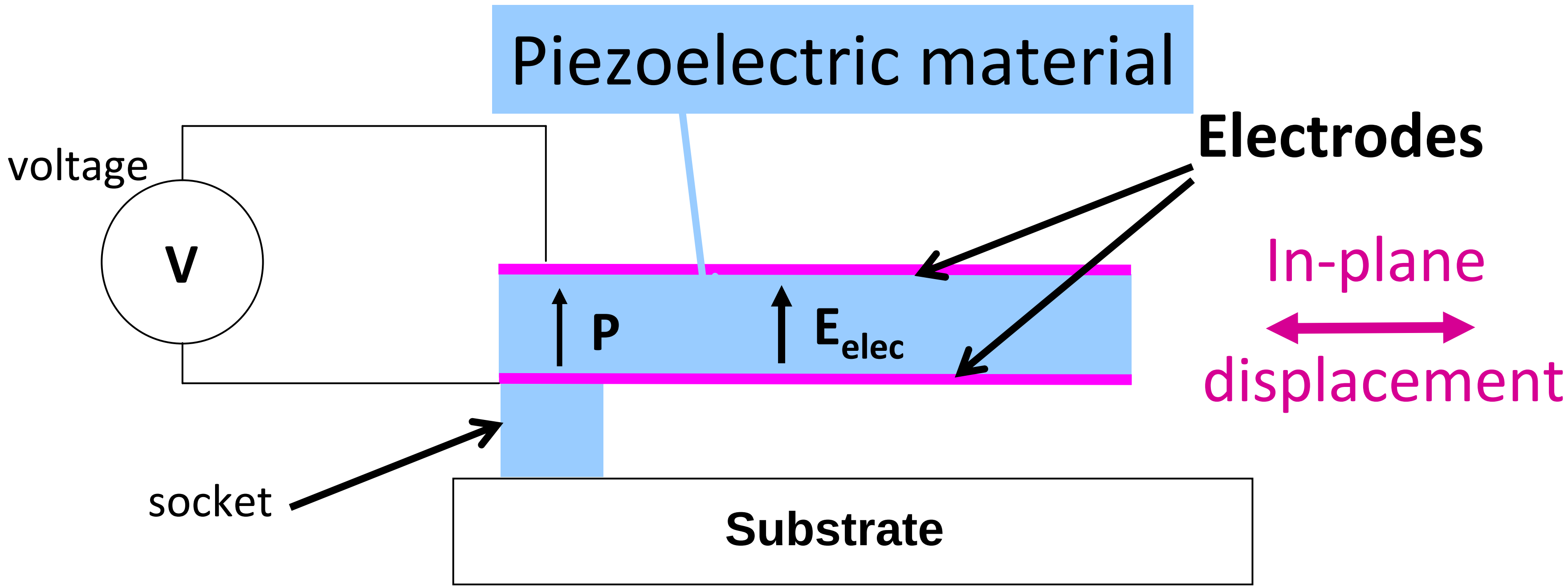
✓Admittance analysis (HP4194A)

Piezoelectric microsystems

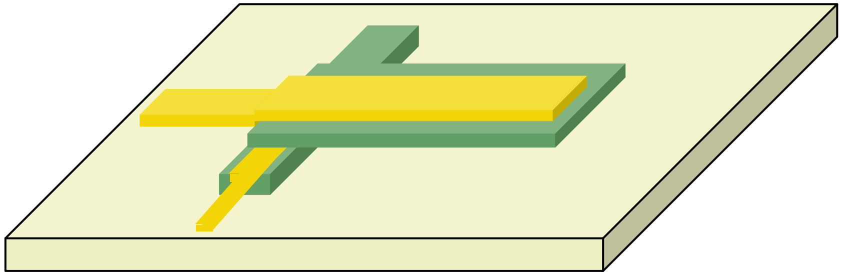
PZT cantilever

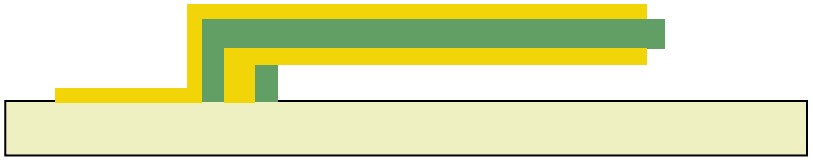


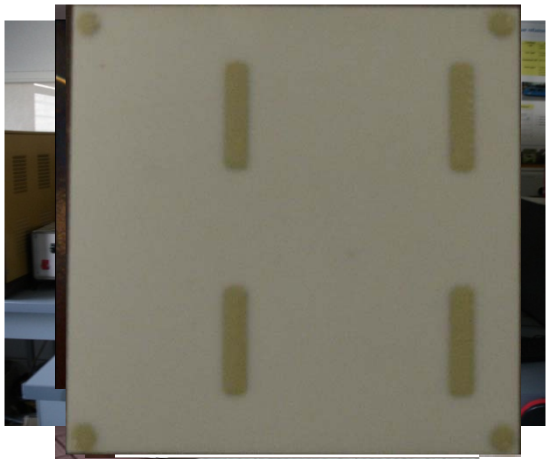
Symmetry of the microstructure: no flexural bending

31-Longitudinal or in-plane resonant mode expected

Screen-printed PZT cantilever fabrication







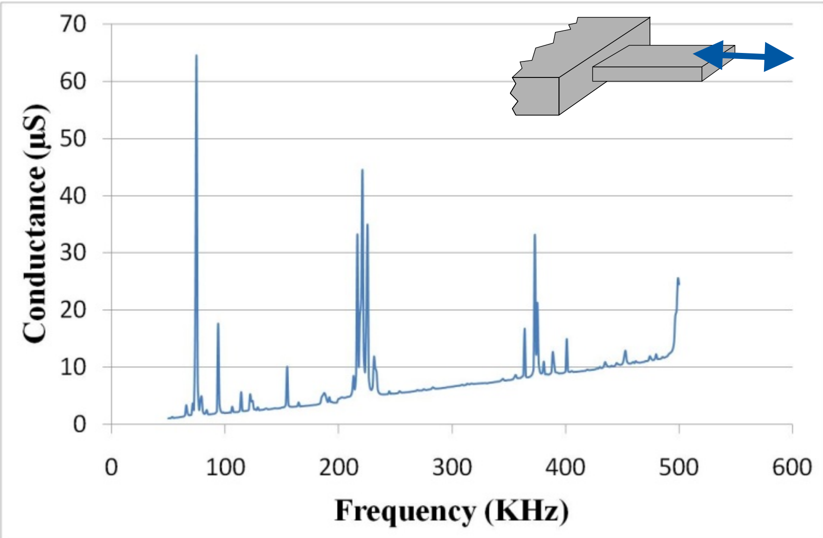
1. PZT based Pad, drying 20min 120°C
2. Sacrificial layer ($SrCO_3$), drying 20min 120°C
3. Bottom electrode (Au), drying 20min 120°C
4. PZT based beam, drying 20min 120°C
5. Top electrode (Au), drying 20min 120°C
6. Isostatic pressure (1kBar, 1min)
7. Cofiring 2h, 900°C → $8x2x0{,}08mm^3$
8. Sacrificial layer removal ($0.9 mole.l^{-1}$ H_3PO_4)
9. Poling (550K , $5kVcm^{-1}$) and characterisation

Piezoelectric microsystems

PZT cantilever

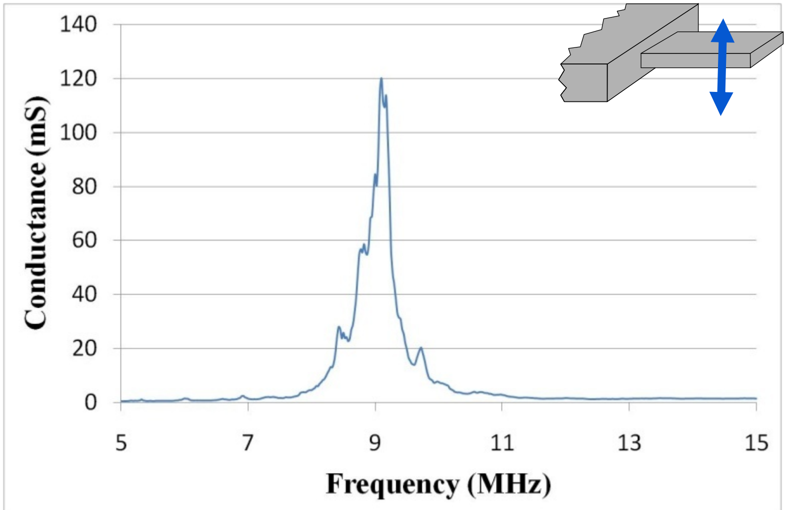
- Electrical characterization

In plane 31-Longitudinal mode



$60 < f_{res}(\text{kHz}) < 100$
$100 < Q < 400$

Out of plane 33-thickness mode

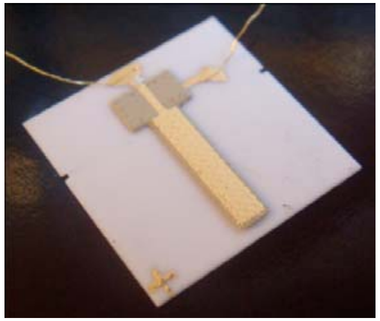


$8 < f_{res}(\text{MHz}) < 10$
$4 < Q < 20$

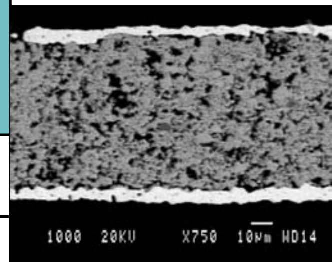
- Optical characterization : Displacement longitudinal ~1µm V=9V

Electrical characterization

A. Influence of the sintering temperature



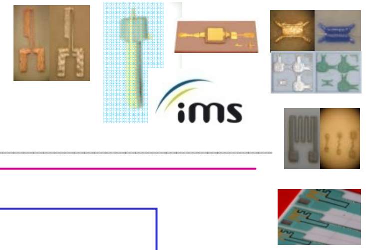
	T_{firing} (°C)	Quality factor (Q)			K_{3T}	$-d_{31}$ (pC/N)
		First 31 mode	Second 31 mode	33 mode		
Glass-frit	850	-	-	-	-	-
	900	320	770	16	343	85
	950	430	270	16	392	81
LBCu	850	242	317	23	333	94
	900	329	371	43	344	100
	950	327	336	32	265	76



- ❖ $300 < K_{3T} < 400$
- ❖ **$80 < -d_{31} < 100$pC/N**

(Previous study Samples with 7% borosilicate fired 15min at 850°C : 10 < -d31 < 30pC/N)

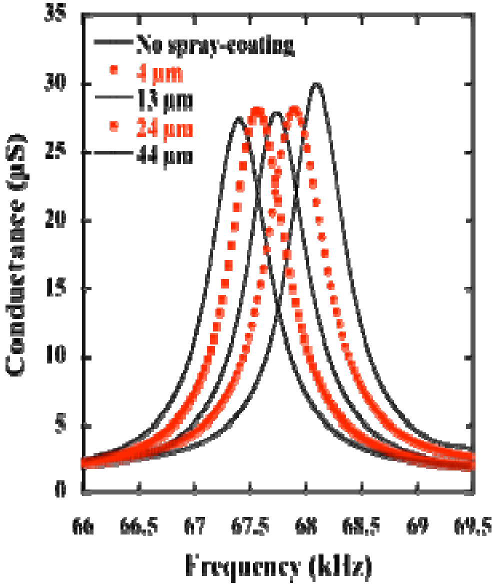
Piezoelectric microsystems



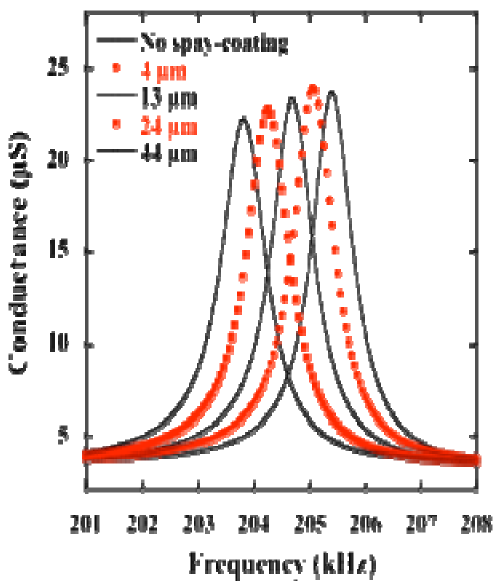
Application PZT cantilever: **gas sensors**

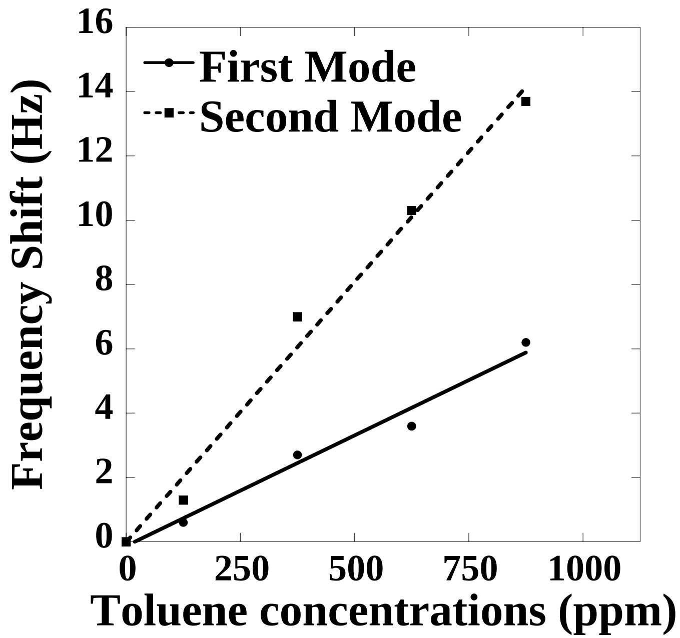
- Toluene detection with 31-Longitudinal mode

1st mode



2nd mode





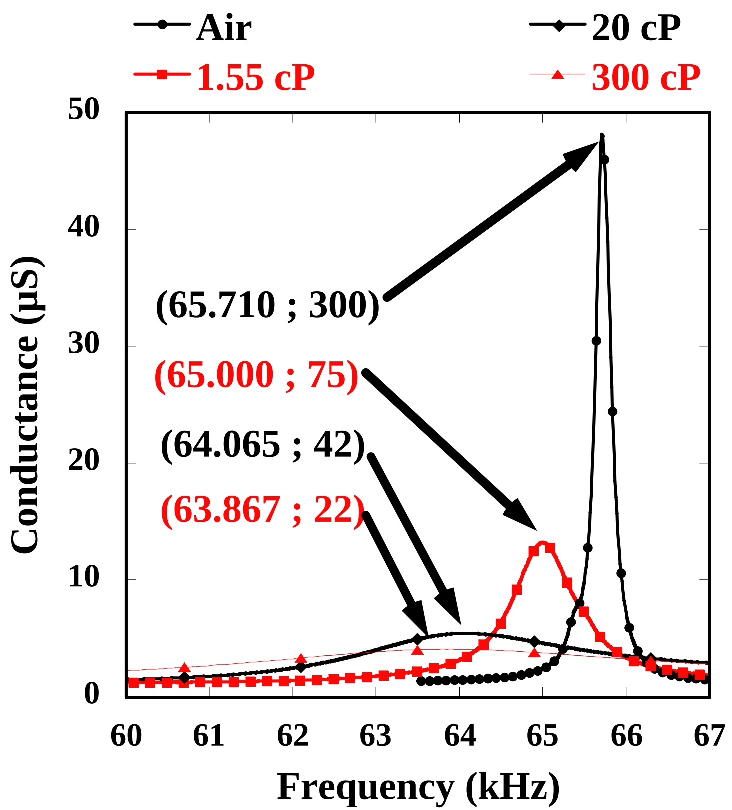
Mass effect with PEUT coating

Mass effect with toluene absorption

Piezoelectric microsystems

Application PZT cantilever: **liquid characterization or detection of species**

- 31-Longitudinal mode in liquid media



Resonant frequency:

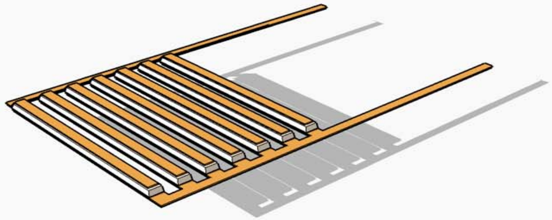
- 65.7 kHz in air
- 65 kHz with 1.55cP
- 64 kHz with 20cP
- 63.8 kHz with 300cP

Quality factor:

- 300 in air
- 75 with 1.55cP
- 42 with 20cP
- 22 with 300cP

- Small decrease of the resonant frequency in liquid
    - Relatively high quality factors in liquid

Résultats préliminaires :
patch piézoélectrique à base de PZT



Etapes:

1/ Cuisson à 850°C

2/ Elimination couche sacrificielle

3/ Polarisation à 200°C

4/ Enrobage et polymérisation

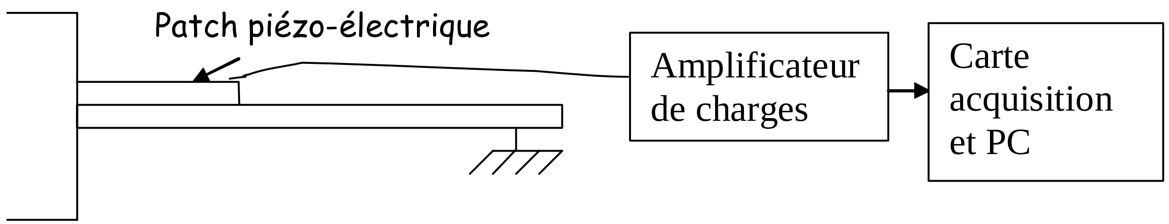
Photo d'un patch après libération et désolaridation du substrat

Photo d'un patch enrobé

Collab. ISAE

Résultats préliminaires :
validation du patch IMS en tant que capteur *…(ISAE,LGM)*

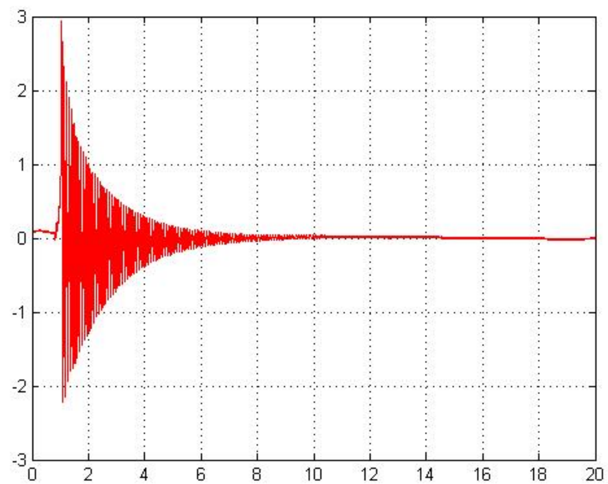
✓ **Description de l'essai :**



Essai de lâcher sur la poutre en flexion

=> excitation du premier mode

✓ **Résultat de la mesure :**



=> Patch OK

pour mesure vibrations

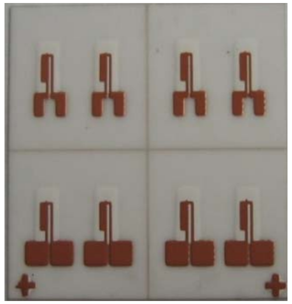
Conclusion

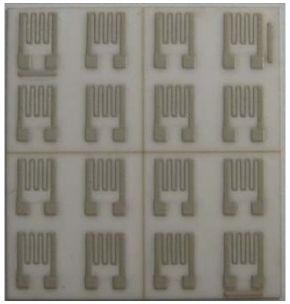
❑ Faisability of free-standing layers → MEMS

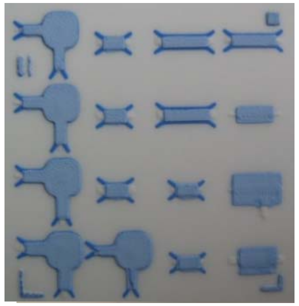
❑ Advantages of this new process

✓ simple, reproducible, fast, cost-effective

✓collective fabrication







✓ coupling with other technologies

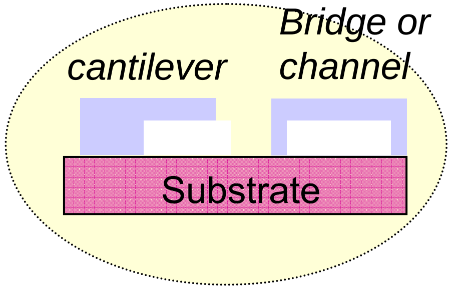
LTCC, Ink jet, …

Conclusion

✓ large choice of substrates and starting materials

✓volume of the layer
Larger forces and power

✓Integrated hybrid MEMS



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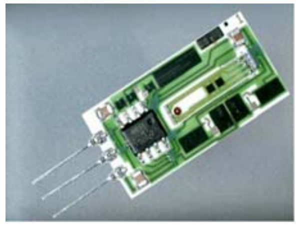

❑ Dimensions

✓ thickness > 1µm, up to hundreds of microns

✓ surface > 100x100 µm² up to hundreds of cm²

✓ various geometries

✓ distance from the substrate 0 < d < hundreds of microns

AKNOWLEDGMENTS

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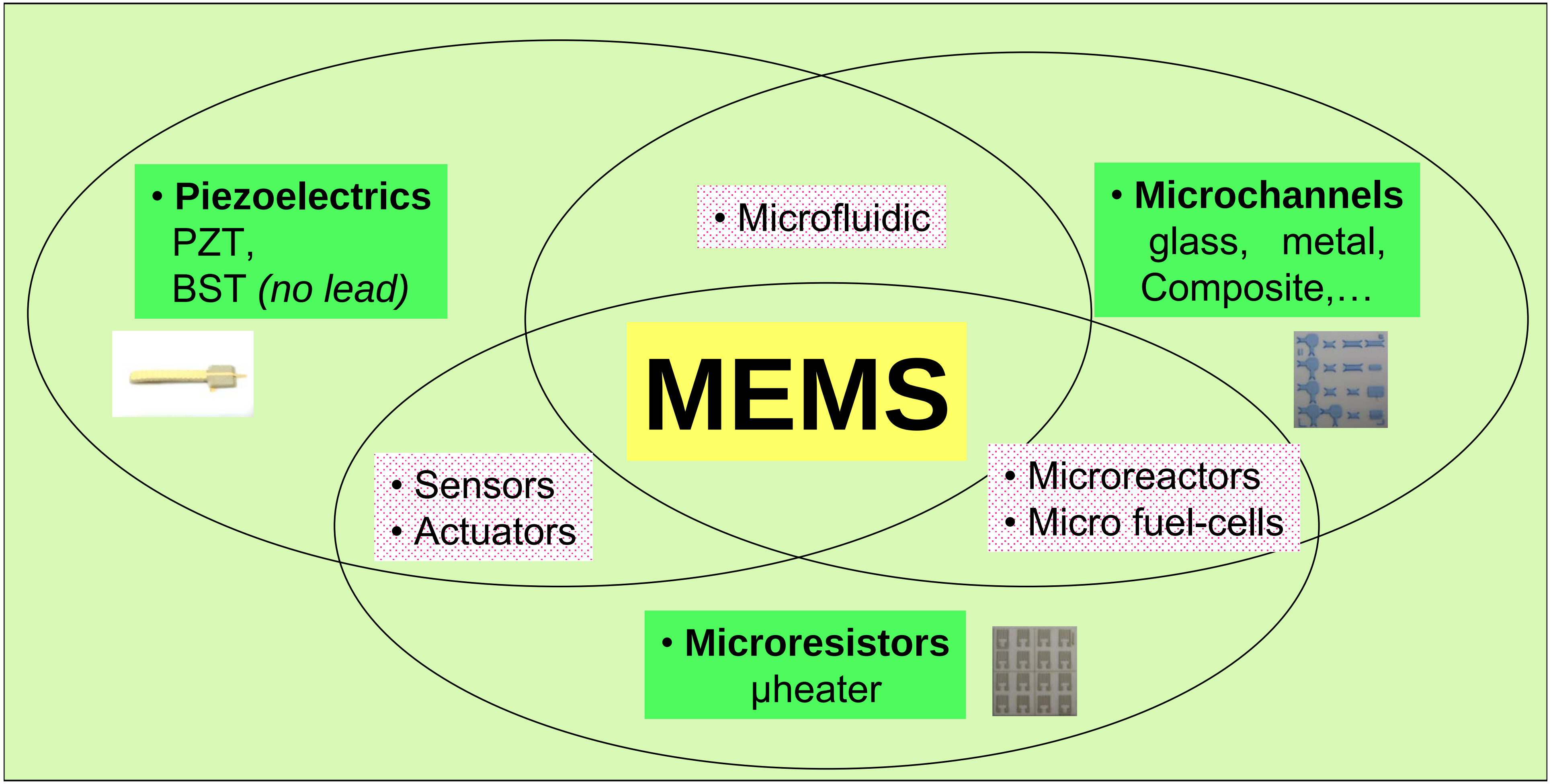
V. BUDINGER, Ass. Professor, ISAE, Toulouse

M. BUDINGER, Ass. Professor, LGET, Toulouse

THANK YOU


• Piezoelectrics
PZT,
BST (no lead)
• Microfluidic
• Microchannels
glass, metal,
Composite,…
MEMS
• Sensors
• Actuators
• Microreactors
• Micro fuel-cells
• Microresistors
µheater