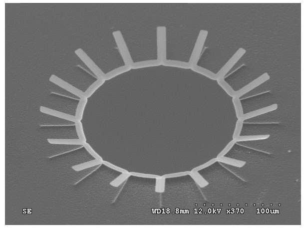
Oxide thin films processing:

some examples on how to take advantage of perovskite properties into devices

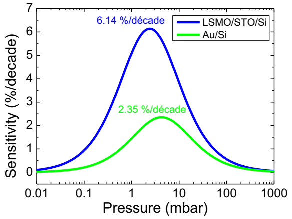
Workshop OSEPI
Villa Clythia - Fréjus
du 13 au 17 mai 2024

G. Agnus
T. Maroutian, S. Matzen, P. Aubert,
F. Pesty, Ph. Lecoeur

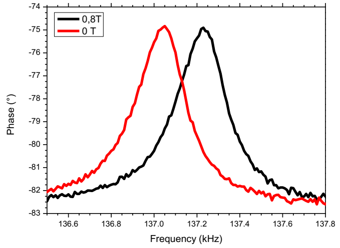
1. **Patterning perovskites into devices**
   - Motivations & approaches
   - Focus on MEMS devices
   - Material review for Si integration



2. **Thermal based devices**
   - Bolometer
   - Pressure sensor
   - Thermoelectricity



3. **Strain based devices**
   - Focus on the link magnetism/strain
   - Results on magnetic field sensing
   - Flexoelectricity



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Conclusion & discussion

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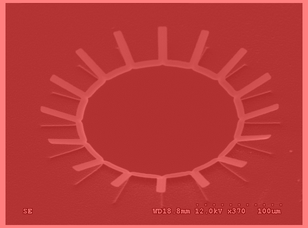
- Motivations & approaches
- Focus on MEMS devices
- Material review for Si integration



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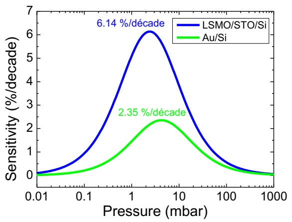

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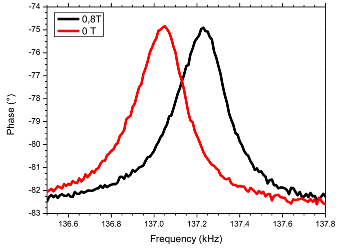
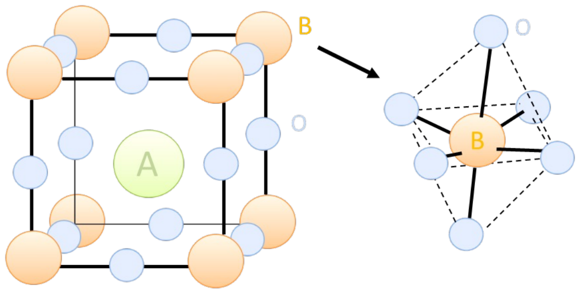


Conclusion & discussion

Perovskite oxides structure

Type de cation B	Cu, Co, Ti, Zn	Cu, Bi	Ti, Zr, Nb, Ta	Nb, Ta	Mn, Co, Fe, Ni
Exemple de composé	(La,Sr)CoO_3	(Ba,K)BiO_3	(Ba,Sr)TiO_3	$KNbO_3$	(La,Sr)MnO_3
Propriété remarquable	Conductivité	Supraconductivité	Ferroélectricité	Réponse optique non-linéaire	Magnétisme

Broad range of physical properties

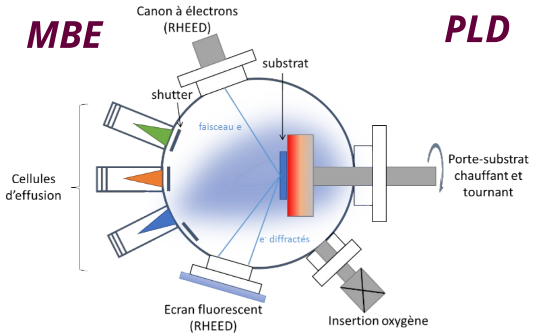


Perovskite ABO_3 structure

cristalline quality ⇔ physical properties

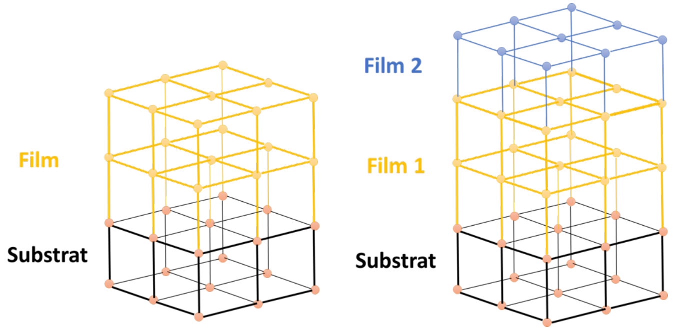
=> Epitaxial growth required

MBE



PLD

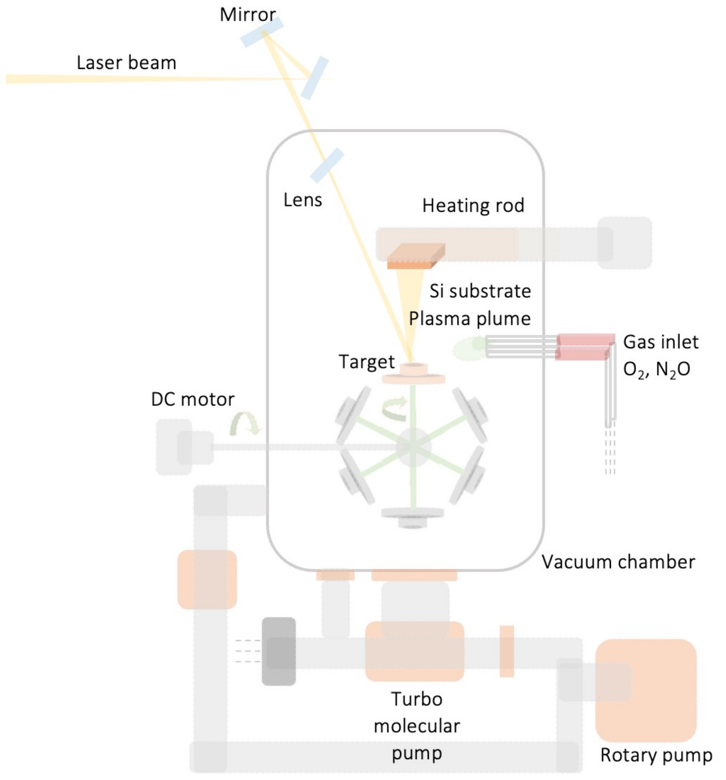
Mature epitaxial growth techniques

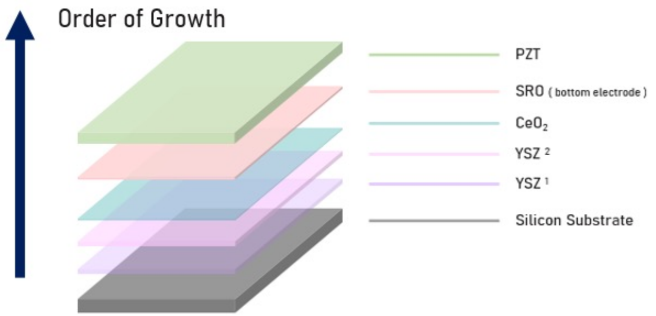


Heterostructures growth allow to couple physical properties

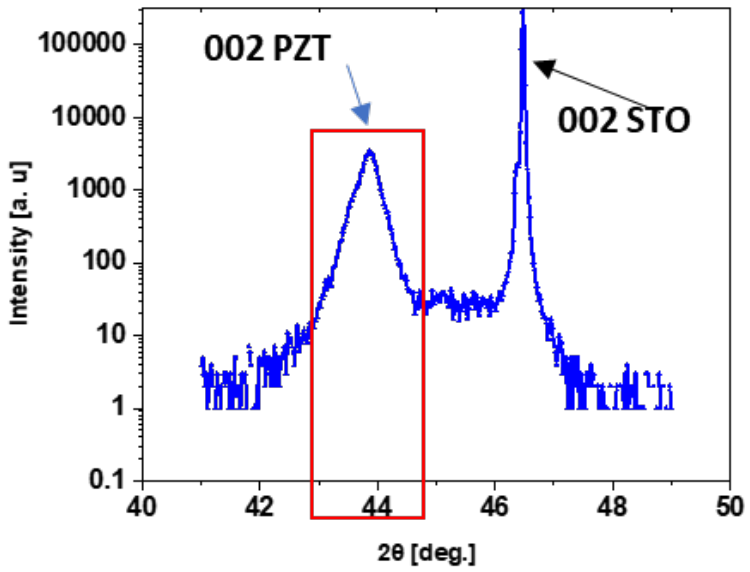
Growth and structural properties

PLD

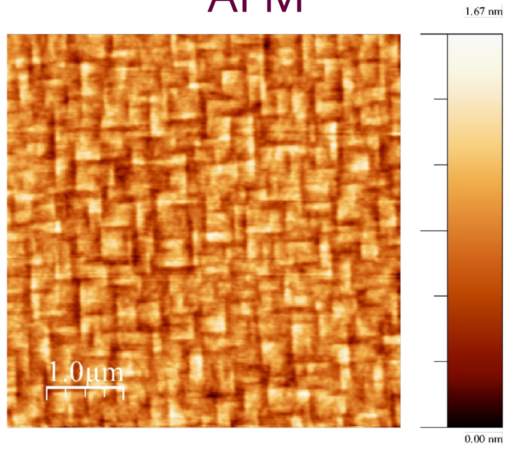




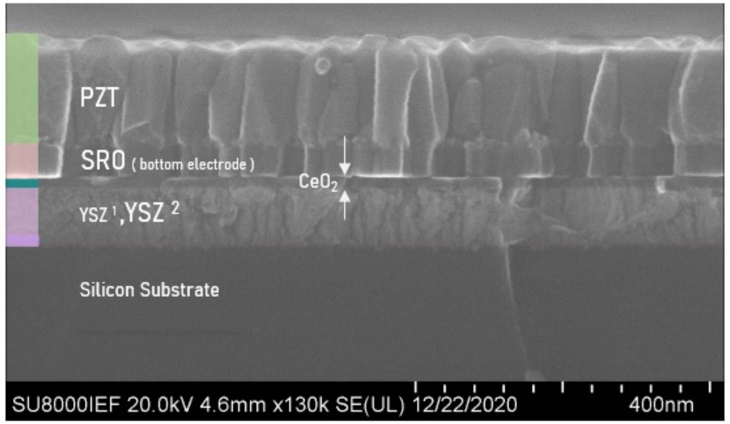
XRD



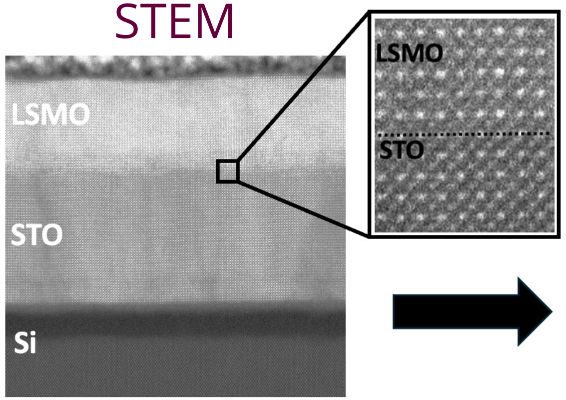
AFM



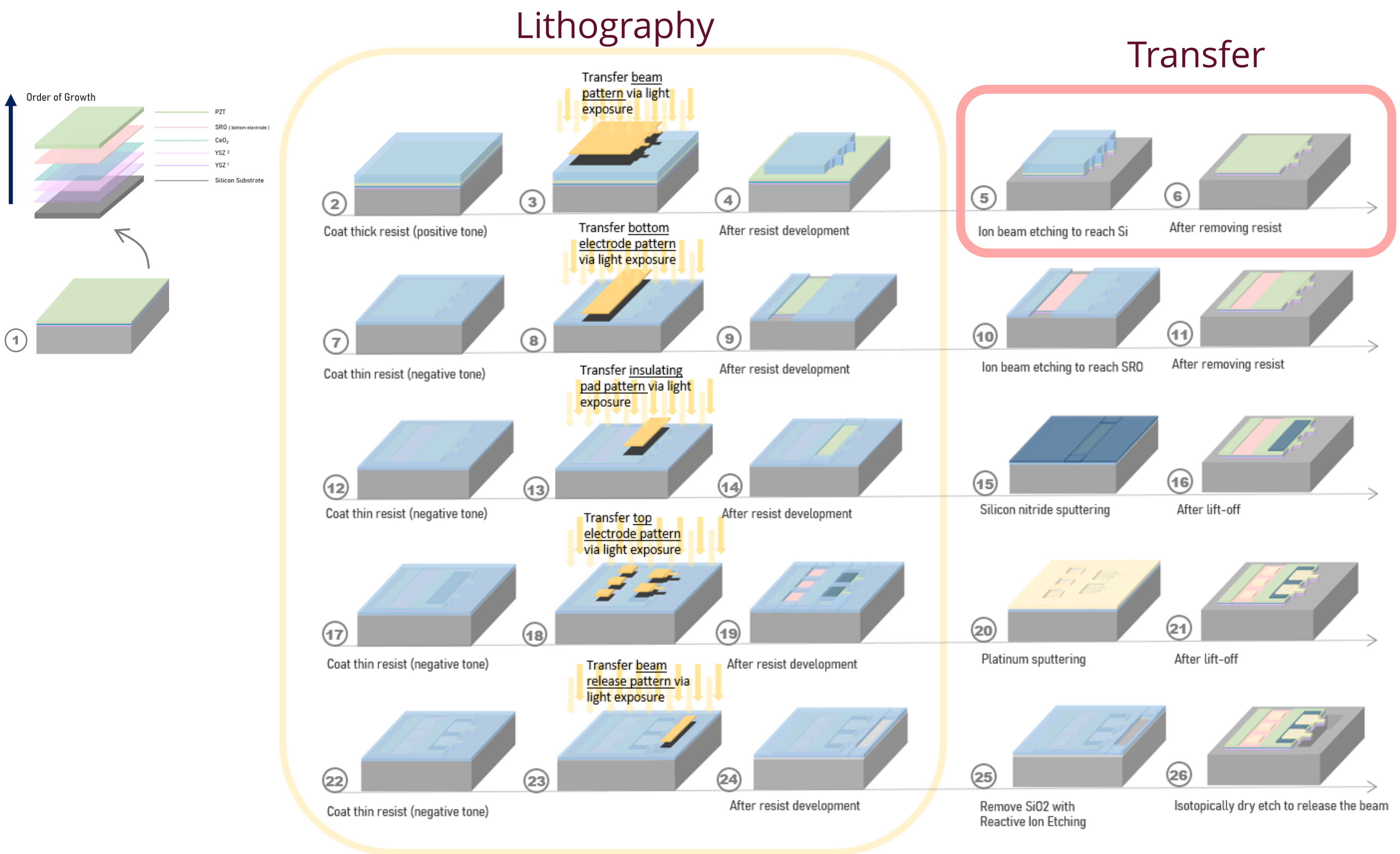
SEM



STEM



Perovskite oxides processing



I. Patterning - etching

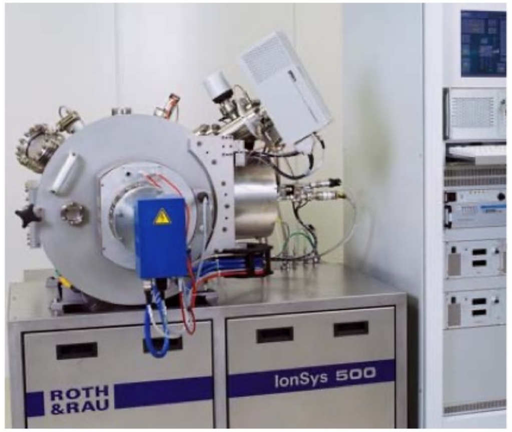
II. Open bottom electrode

III. Deposit contact

IV. Deposit top electrode

V. Beam release

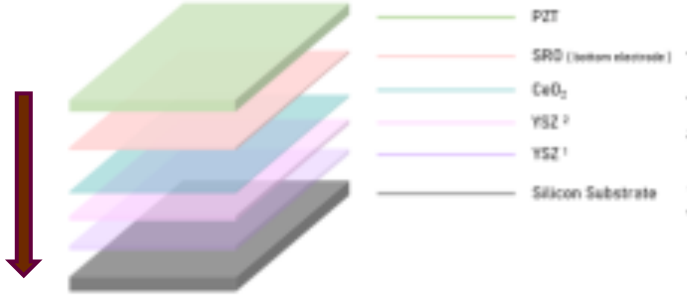
Perovskite oxides processing

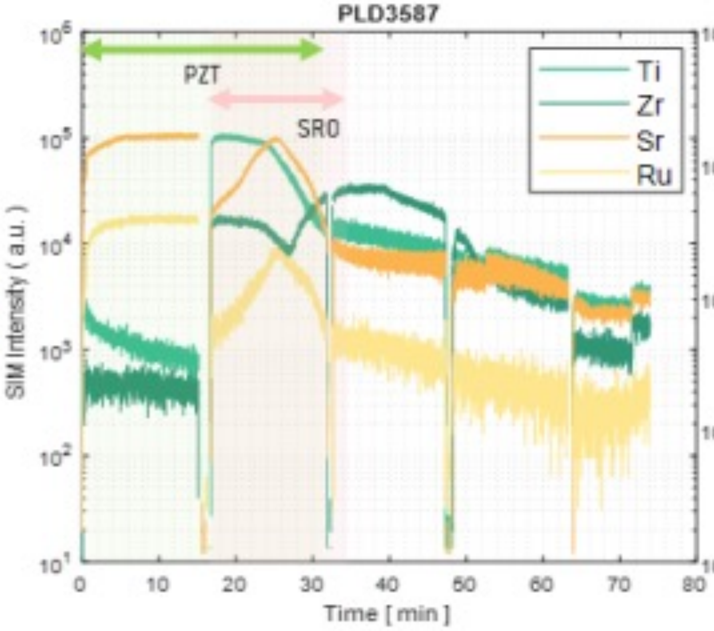


A critical step => etching

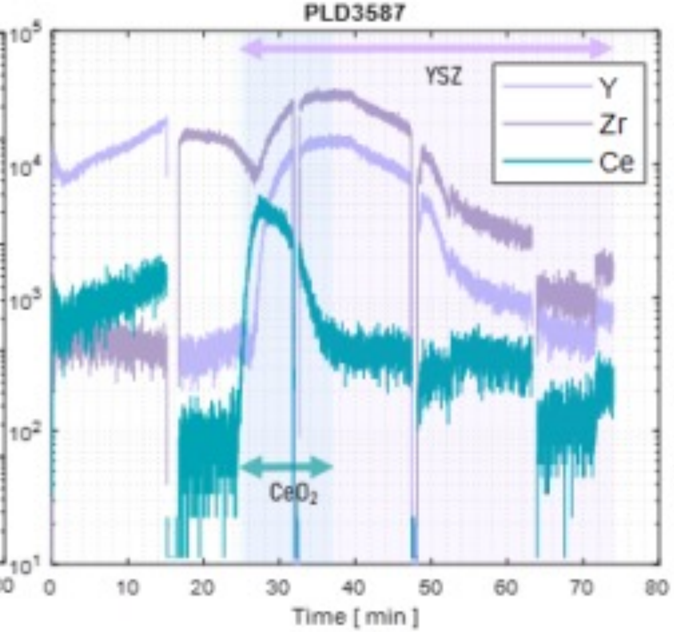
- Large Young Modulus
- High chemical stability

Etching down to Si substrate





Etching Time



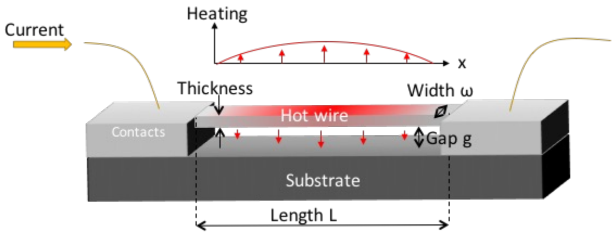
Etching Time

Exalted amplitude of physical effects

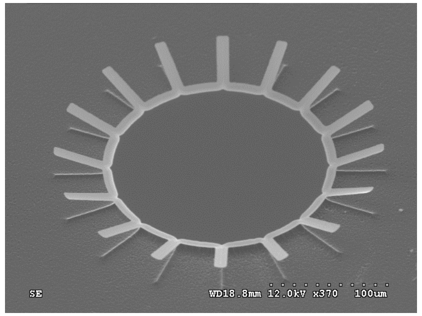
Thermal effects

- Bolometers
- Pressure and flux sensors
- Thermoelectricity

Thermal gradient due to lower thermal dissipation



Strain effects



High and complex strain effect due to low clamping

- Magnetisation control
- Magnetism Sensing
- Resonating sensors
- Strain gradient and flexoelectricity

Perovskite oxides MEMS

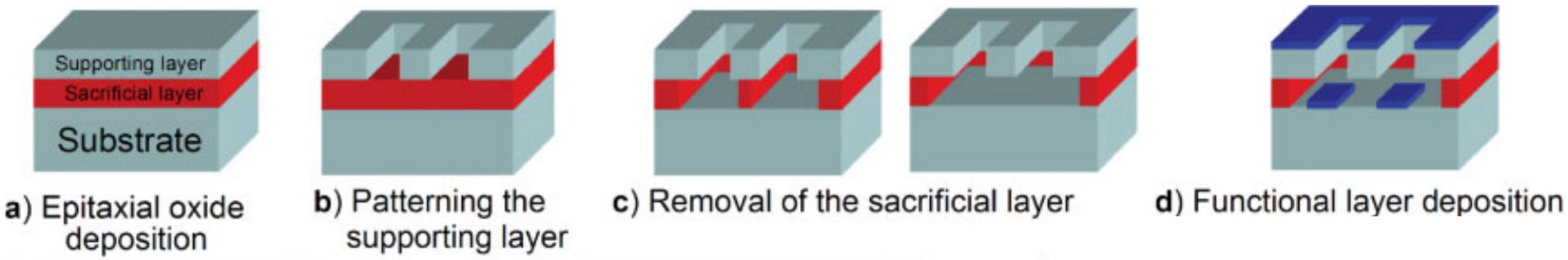
→ Full oxide approach

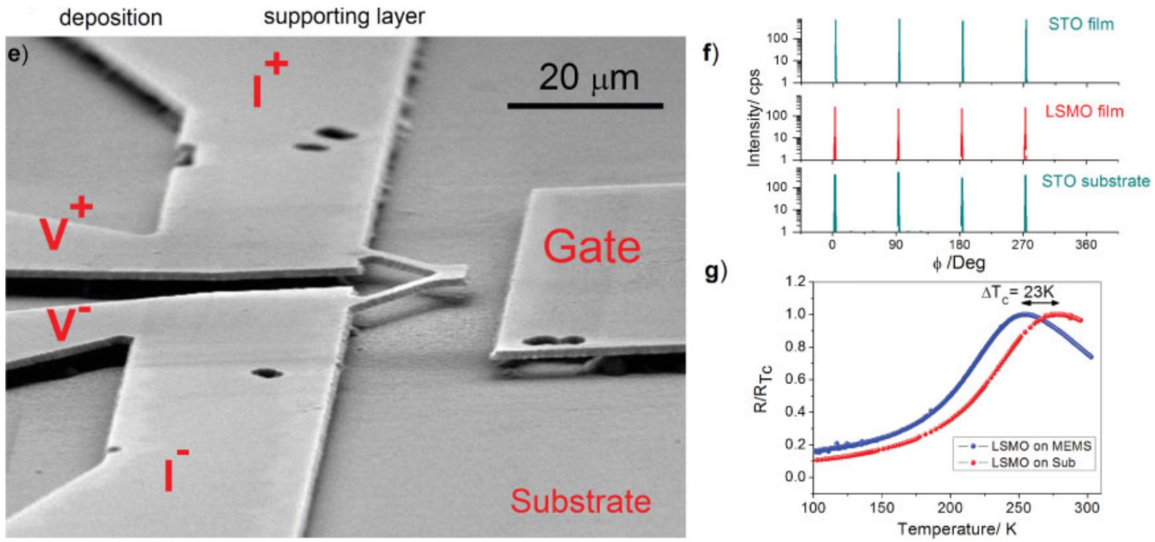
2009 ADVANCED MATERIALS

www.advmat.de

All-Oxide Crystalline Microelectromechanical Systems: Bending the Functionalities of Transition-Metal Oxide Thin Films

By *Luca Pellegrino,* Michele Biasotti, Emilio Bellingeri, Cristina Bernini, Antonio Sergio Siri,* and *Daniele Marré*





Pros:
- High quality films
- Low complexity of the freestanding stack

Cons:
- PLD on freestanding (fragile) substrate

Opens for films transfer of perovskites (requirement of wafer scale deposition technique !)

Integration on Si(100) : Material review

J. W. Reiner et al., *Adv. Mater.* **2010**, *22*, 2919

Lattice parameter compatible
Removing native silicate layer

Increasing cristalline quality

Al_2O_3 H. M. Manasevit et al., *J. Appl. Phys.* **1964**, *35*, 1349

$Mg_2Al_2O_4$ (MGA) M.Ihara et al., *J. Electrochem*, **1982** *129*, 2569

Y_2O_3, ZrO_2, YSZ, CeO_2 H. Fukumoto et al., *Jpn. J. Appl. Phys.* **1988**, *27*, L1404

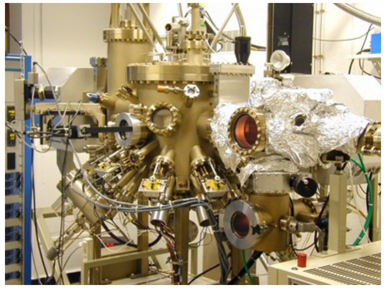
MgO D. K. Fork et al., *Appl. Phys. Lett.* **1991**, *58*, 2294

Perovskites:

- **SrO / $SrTiO_3$** R. McKee et al., *Phys. Rev. Lett.* **1998**, *81*, 3014
- **$CaTiO_3$** R. A. McKee and F. J.Walker, U.S. patent 5,830,270 (1998)
- **BaO / $BaTiO_3$** V. Vaithyanathan et al., *J. Appl. Phys.* **2006**, *100*, 024108

PLD

MBE

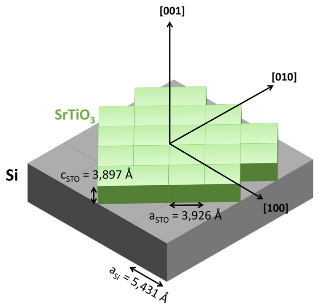


Direct integration of $SrTiO_3$ (STO) by molecular beam epitaxy

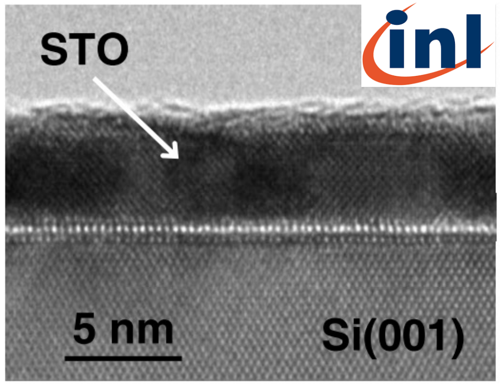
R. McKee et al., *Phys. Rev. Lett.* **1998**, *81*, 3014

Strategy :

1. Removing the amorphous silicate layer
2. Deposition of a thin poorly crystallized layer of STO that act as oxygen barrier
3. Deposition of $SrTiO_3$ at high temperature/oxygen pressure



1.7% of lattice mismatch with Si (cell rotation of 45°)



G. Niu et al., *Appl. Phys. Lett.* **2009**, *95*, 2009

Growth manage by a few groups in the worl:

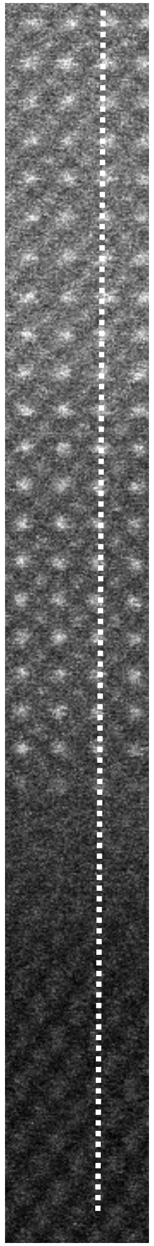
Etats-Unis:
C.B. Eom (*Wisconsin*)
D. Schlom (*Cornell*)
Yale, Texas Univ.
…

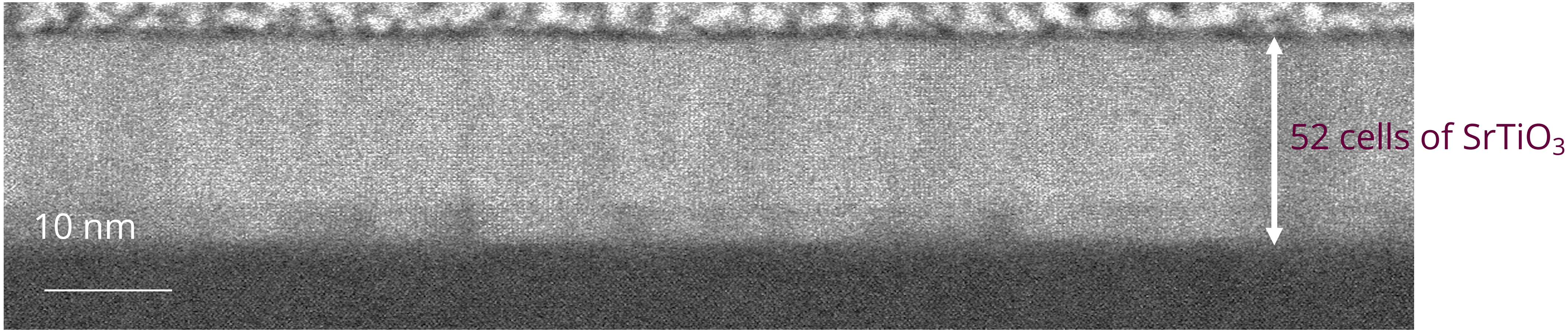
Europe:
G. Saint Girons (*INL, France*)
J. Fompeyrine (*IBM Zurich, Suisse)*
…

Critical steps

- preventing the interfacial silicate layer during the growth at high temperature/ oxygen pressure
- Stichometry difficult to control (Oxygen, Sr/Ti ratio)

STO/Si: TEM analysis





STEM @ Saragosse

- Coherent relationship between silicon and $SrTiO_3$
- Different contrasts between interfacial $SrTiO_3$ (10's of cells)) et the top $SrTiO_3$
- Several types of defects with different sizes

Complex structure

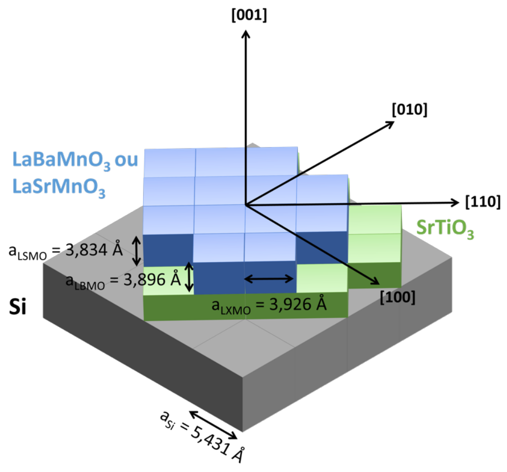
Changes induces by the PLD growth of active films

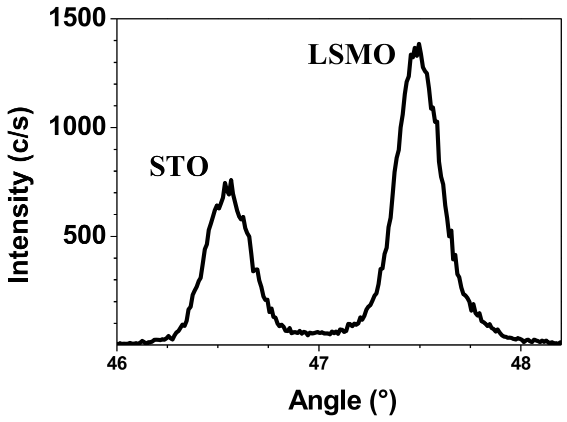
Homogeneity

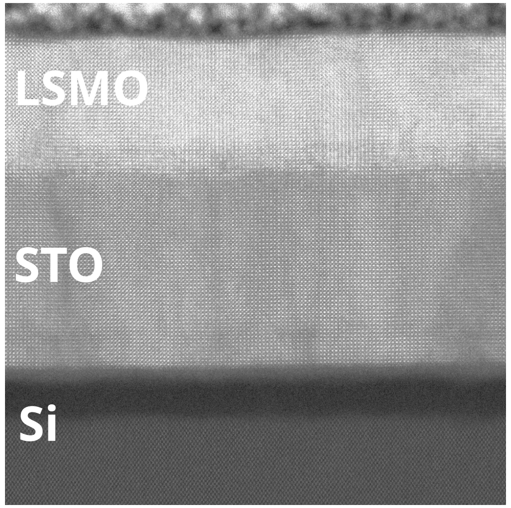
- **Growth of an interfacial SiO_2 layer**
- **Modification of the crystallinity of $SrTiO_3$?**
- **Strain ?**

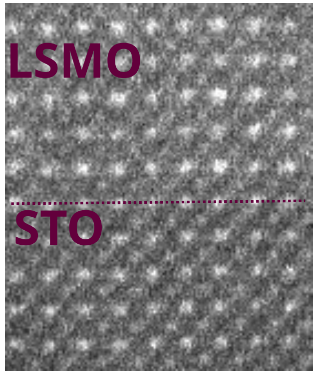
Manganites on STO/Si

Epitaxy of manganites: $La_{0.67}Sr_{0.33}MnO_3$



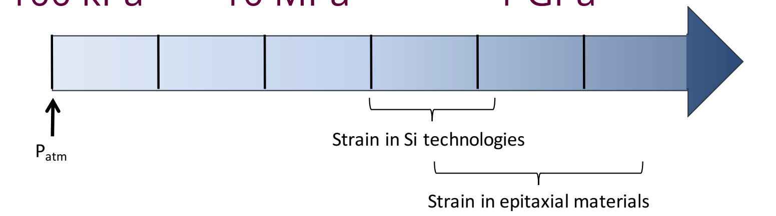






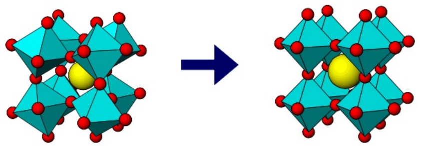
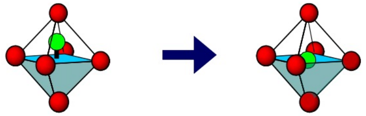
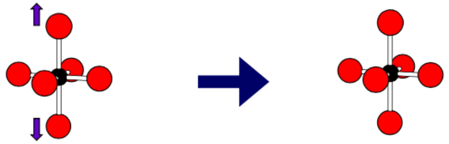
	STO	LSMO
a // plan	3,925	3,925
c ⊥ plan	3,89	3,84
bulk	3,905	3,866

- **Interfacial SiO_2 layer**
- **STO relaxed** with respect to Si 110 (3,839 Å)
- Coherent epitaxy between LSMO and STO
- **LSMO under tensile strain => no relaxation**
- **STO under tensile strain** in plane
  => Stoichiometry

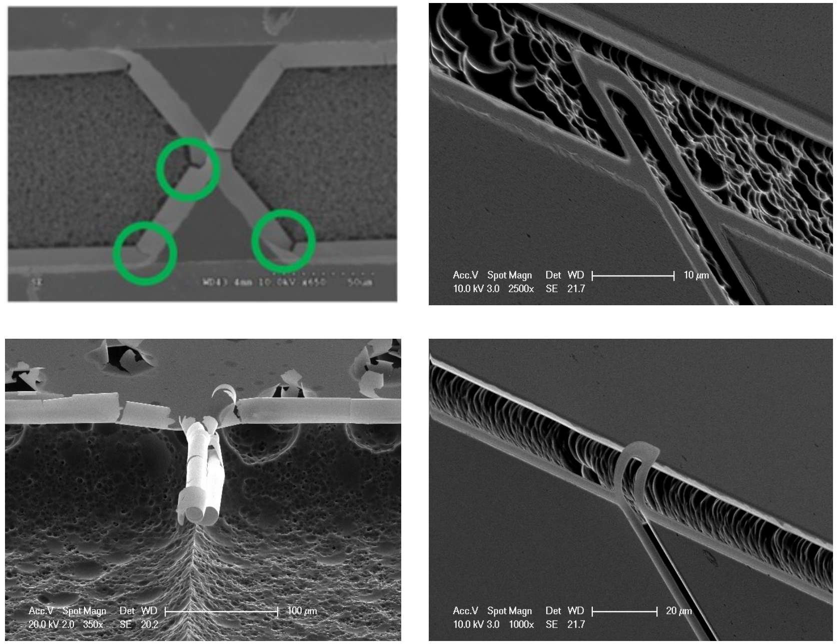


High level of stress in those epitaxial thin films

Modify the physical properties

- Octahedral tilting 
- Cation displacement 
- Octahedral distortio (Jahn-Teller) 

Modify the shape of MEMS structures
Reduce the yield of production



Strain in perovskites on Si

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Deneke *et al. Nanoscale Research Letters* 2011, **6**:621
http://www.nanoscalereslett.com/content/6/1/621

Nanoscale Research Letters
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NANO EXPRESS **Open Access**

Rolled-up tubes and cantilevers by releasing $SrRuO_3$-$Pr_{0.7}Ca_{0.3}MnO_3$ nanomembranes

Christoph Deneke[1,2*], Elisabeth Wild[2], Ksenia Boldyreva[3], Stefan Baunack[2], Peter Cendula[2], Ingolf Mönch[2], Markus Simon[4], Angelo Malachias[5], Kathrin Dörr[3,6] and Oliver G Schmidt[2]

- ouvertures par IBE
- Attaque chimique humide sélective du $PrCaMnO_3$ avec la solution ($HF/HNO_3/H_2O$)

Volonté d'utiliser cette relaxation de contrainte @

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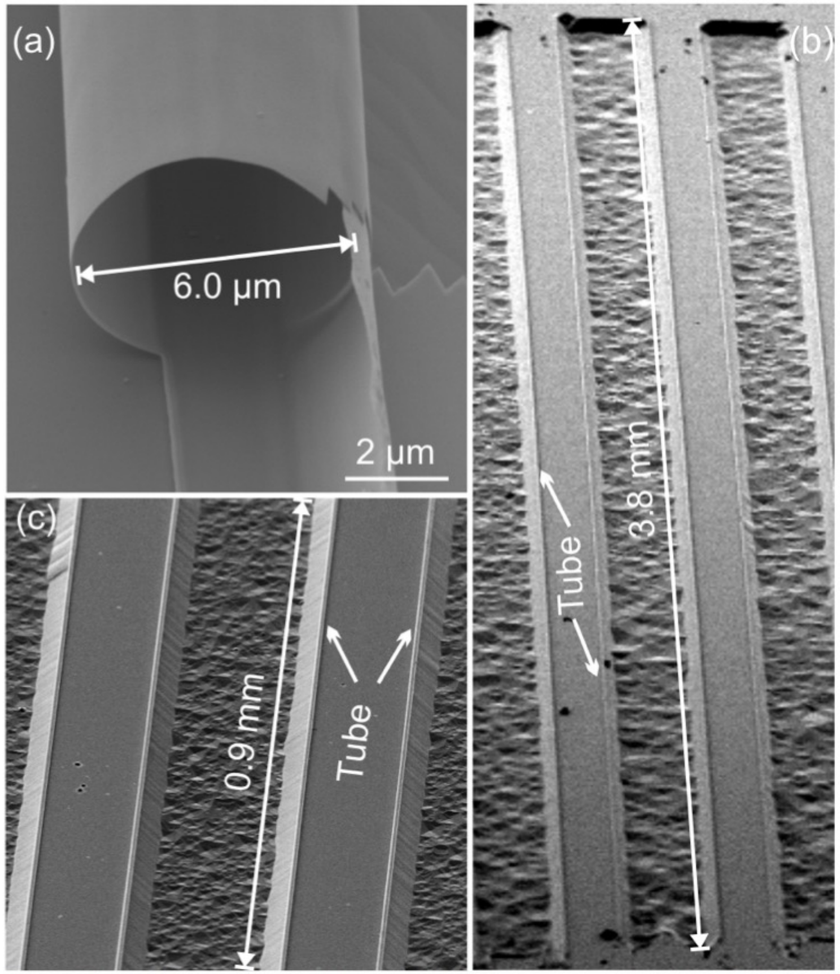

Figure 2 Rolled-up SRO/PCMO/SRO microtubes. (**a**) Rolled-up SRO/PCMO/SRO microtube with a diameter of 6.0 μm. (**b**, **c**) Positioned microtubes obtained from <100> -oriented trenches defined by optical lithography. The tubes in (b) exhibit an aspect ratio of nearly 1:700.

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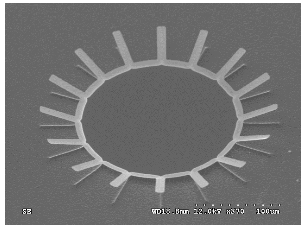

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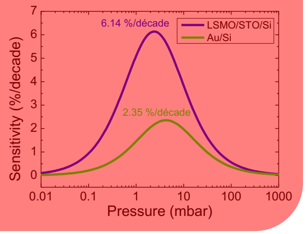

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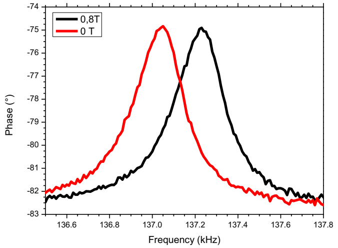

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Conclusion & discussion

Main idea:
generate heat and measure it through TCR

$$TCR = \frac{1}{R(T)}\frac{dR(T)}{dT}$$

The way you generate/lose heat makes your sensor

Photon => Bolometer

Air =>

- *Pressure sensor*
- *Gas flow sensor*
- *Accelerometer*

2 families of oxides materials

Low temperature

Superconductivity transition T°

Pro:
Very high sensitivity

Con:
low temperature (higher than for Metals $T°_{superC}$)

Room temperature

Matériau	TCR (10^{-3} K^{-1})
Platine	3,9
Silicium dopé p	9,0
LSMO/STO/Si	13,4
LBMO/STO/Si	-10,9

Pros :
High sensitivity (vs metals)
Low noise
Chemical and thermal stability

Cons :
Low sensitivity (vs T_{superC})

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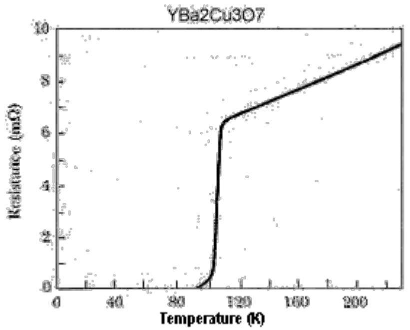

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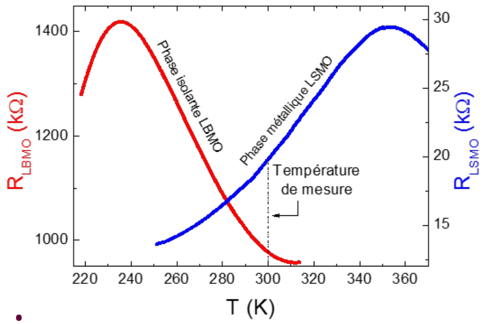
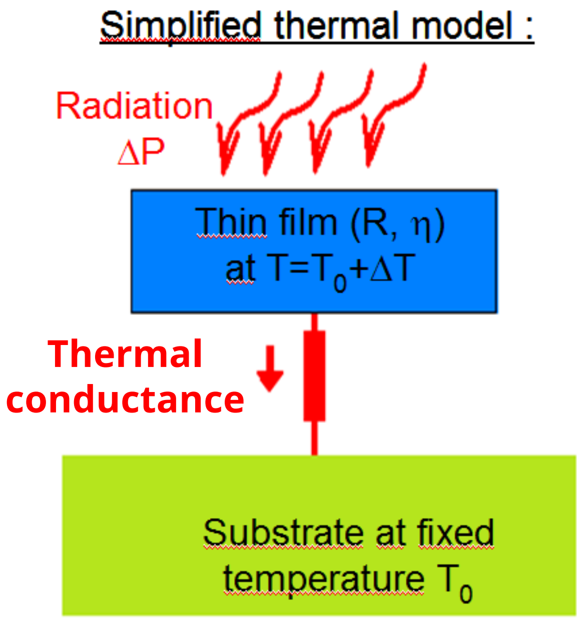


Pros :
High sensitivity (vs metals)
Low noise
Chemical and thermal stability

Cons :
Low sensitivity (vs T_{superC})

Microbolometer sensitivity:

$$S_V(\omega) = \frac{\eta \times R \times I}{G(1+j\omega\tau)} \times \frac{1}{R}\frac{dR}{dT}$$



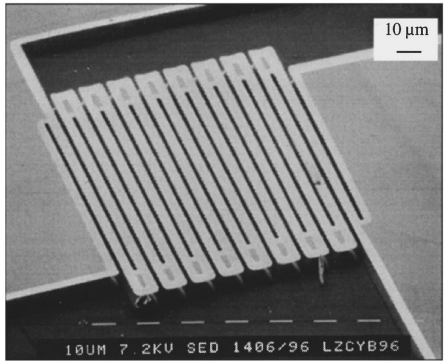
For fast and sensitive bolometer, one need:

- High TCR material
  - **Superconductors YBCO close to the supra-métal**
  - **Manganites LSMO à la transition métal-isolant**

- des membranes pour réduire la conductance thermique G et le temps de réponse **t = C/G**

Suspended epitaxial YBaCuO microbolometers fabricated by silicon micromachining: Modeling and measurements

Laurence Méchin[a)] and Jean-Claude Villégier
DRFMC/SPSMS/LCP-CEA Grenoble, 17 rue des Martyrs, 38054 Grenoble cedex 9, France
Daniel Bloyet
GREYC (URA CNRS 1526)-ISMRA, 6 boulevard Maréchal Juin, 14050 Caen cedex, France



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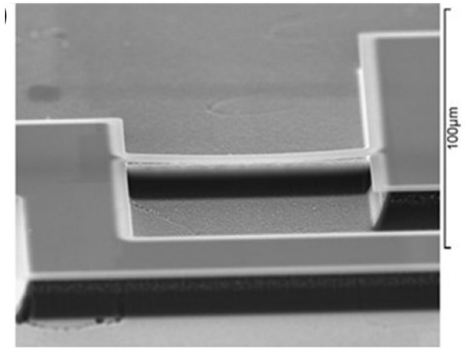
Microelectronic Engineering
journal homepage: www.elsevier.com/locate/mee
ELSEVIER

$La_{0.7}Sr_{0.3}MnO_3$ suspended microbridges for uncooled bolometers made using reactive ion etching of the silicon substrates

S. Liu[a], B. Guillet[a], A. Aryan[a], C. Adamo[b], C. Fur[a], J.-M. Routoure[a], F. Lemarié[c], D.G. Schlom[b,d], L. Méchin[a,*]

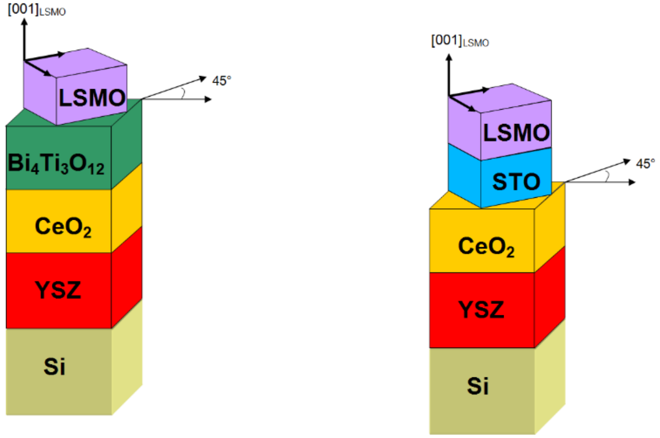
S. Liu et al. Microelec. Eng. (2012)
S. Liu, thèse univ. Caen (2013)

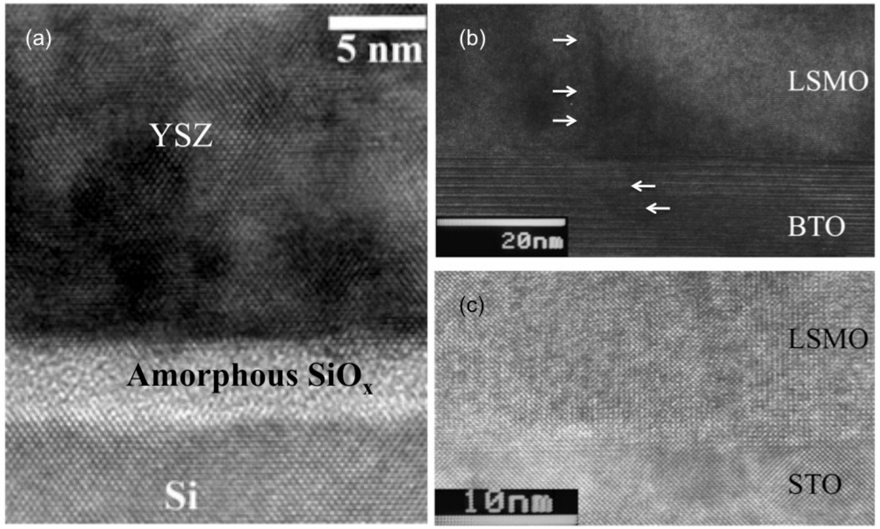


GREYC

Free-standing bolometers $La_{0.7}Sr_{0.3}MnO_3$ (LSMO)

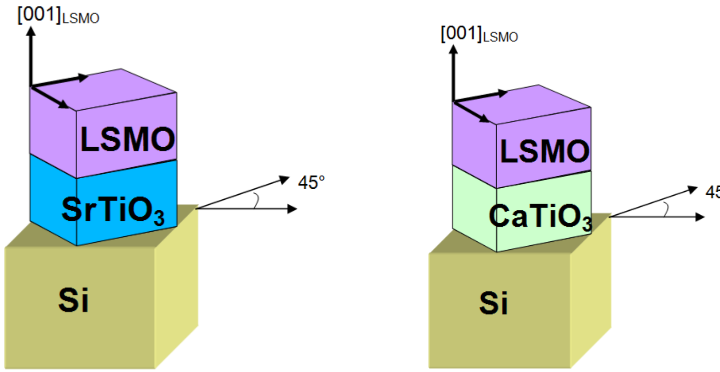
LSMO/YSZ-based buffered Si par PLD

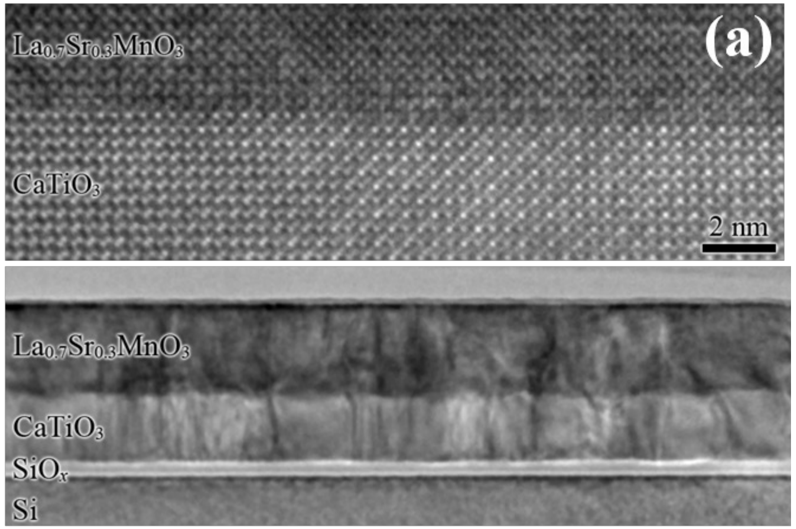




L. Méchin et al. M. Science Eng. B (2007)
P. Perna et al. J. Phys. Condens. Matter (2009)

LSMO/STO-CTO/Si par MBE puis PLD

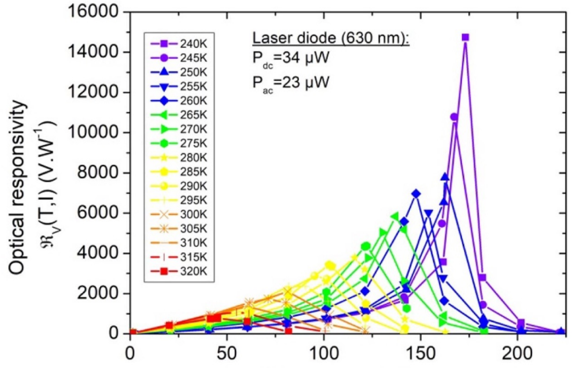




L. Méchin et al. PSSA (2012)
Coll. D.G. Schlom, Univ. Cornell

Cornell University

=> Light sensitivity

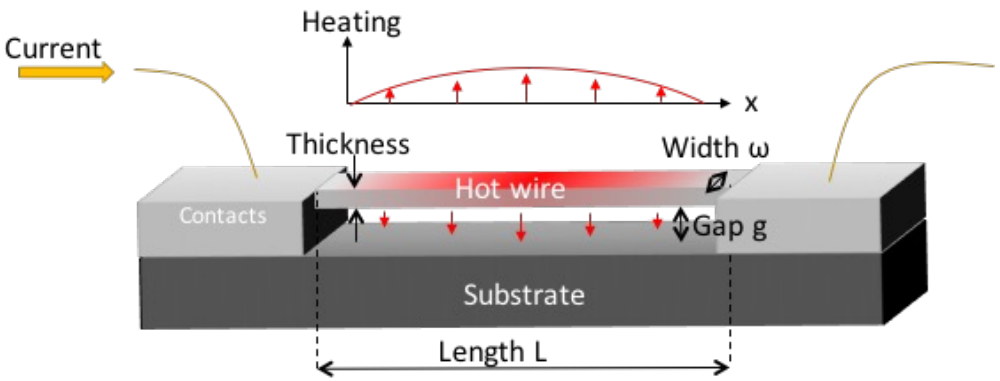


IOP Publishing
J. Phys. D: Appl. Phys. **54** (2021) 055301 (7pp)

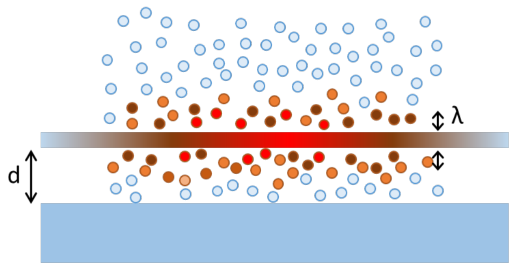
Thermal based devices: free-standing pressure gauge

D. Le Bourdais PhD

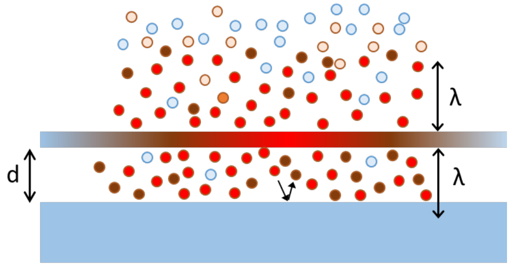
D. Le Bourdais et al.,
J. Appl. Phys. **2015**, *118*, 124509



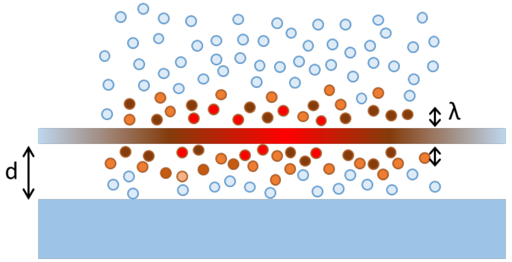
<u>Low pressure</u>:
Low thermal dissipation
Bridge temperature: High



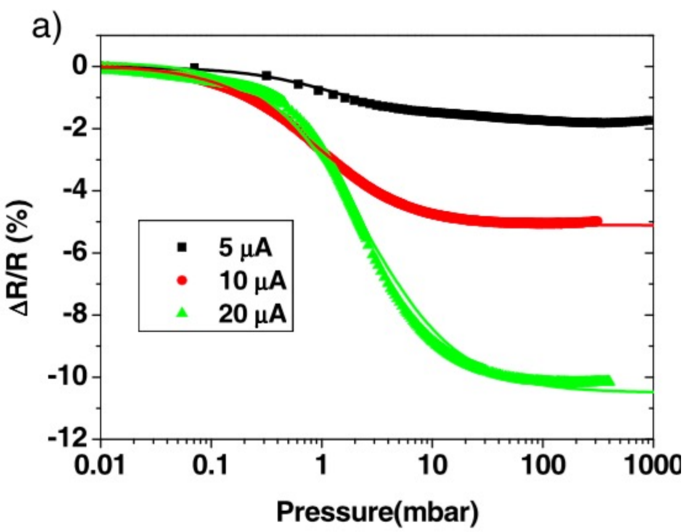
<u>Pressure of sensitivity:</u>
Thermal dissipation
Bridge temperature: depends on P

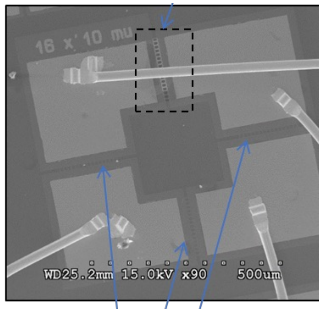


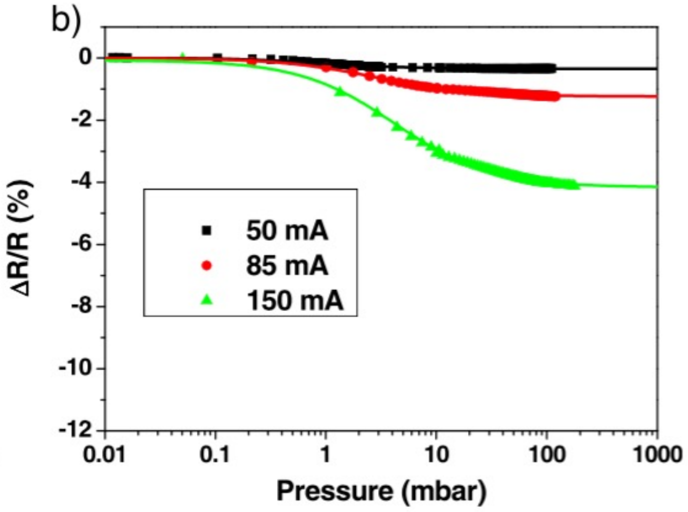
<u>High pressure:</u>
Maximal thermal dissipation
Lowest bridge temperature

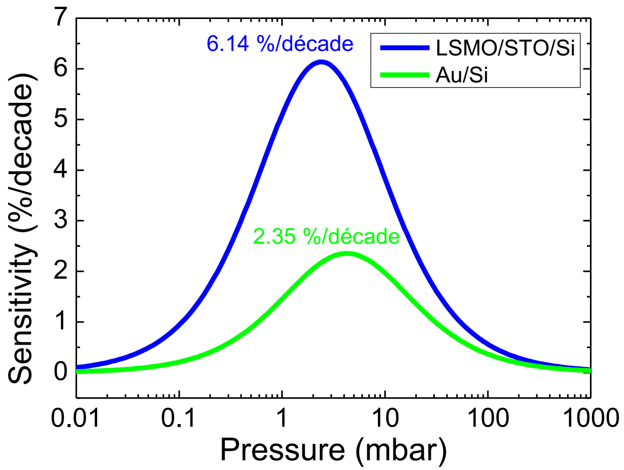


Thermal based devices: free-standing pressure gauge









Sensitivity increased by a factor of 3

D. Le Bourdais et al.,
J. Appl. Phys. **2015**, *118*, 124509

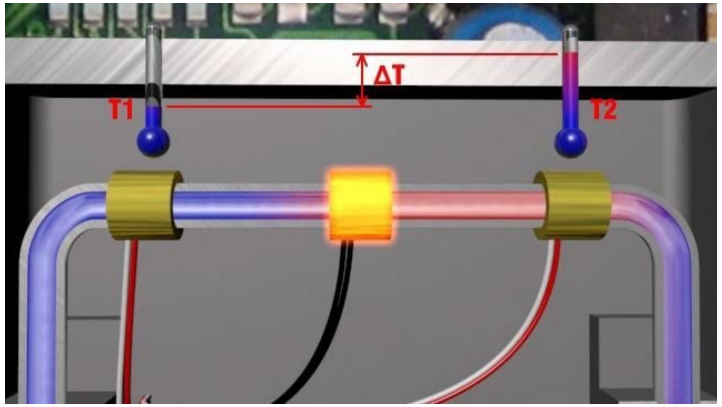
Power consumption reduced by <u>3 orders of magnitude</u>

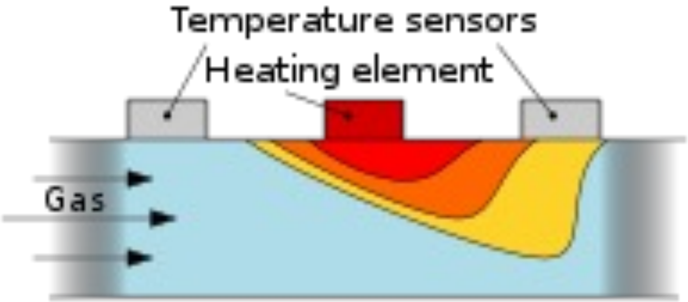
- **Manganites chemical stability**
- **Low intrinsic noise**

A. Lisauskas et al., Appl. Phys. Lett. **77**, 756 (2000)

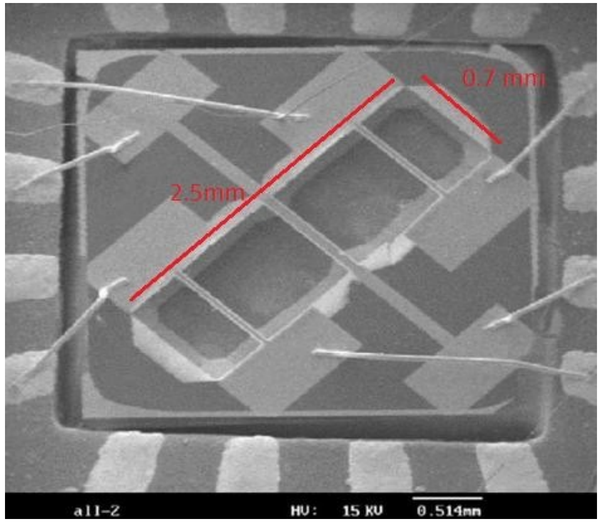
Thermal based devices: other sensors ?

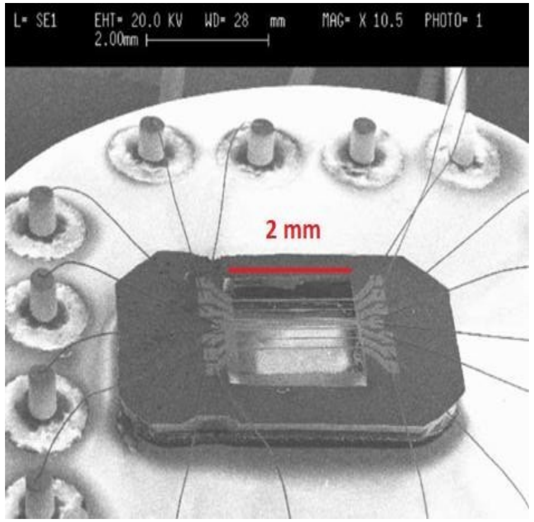
Flux sensor





Accelerometer

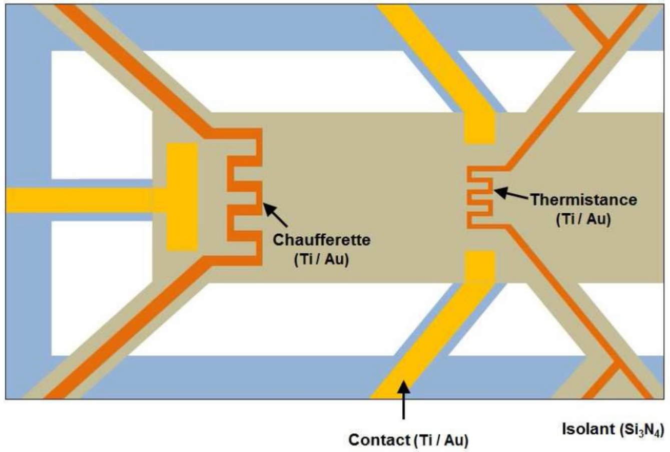


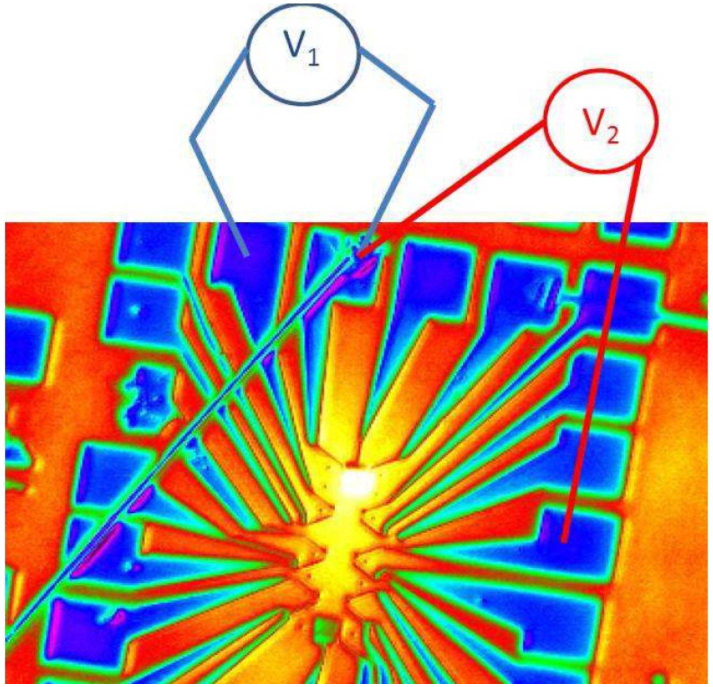


Thermal based devices: probing other properties

Thermoelectricity

=> Superlattices $SrTO_3$/Nb:$SrTiO_3$ for Si integrated thermoelectrcity



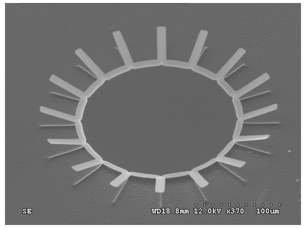


Thèse Yann Apertet (2013)

Critical point => managing strain relaxation during the release

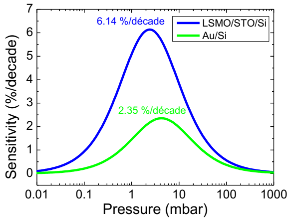
1. Patterning perovskites into devices

- Motivations & approaches
- Focus on MEMS devices
- Material review for Si integration



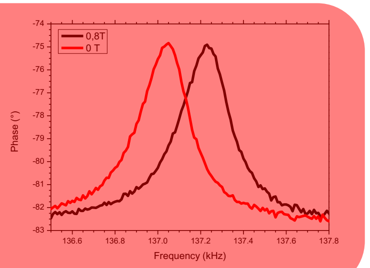
2. Thermal based devices

- Bolometer
- Pressure sensor
- Thermoelectricity



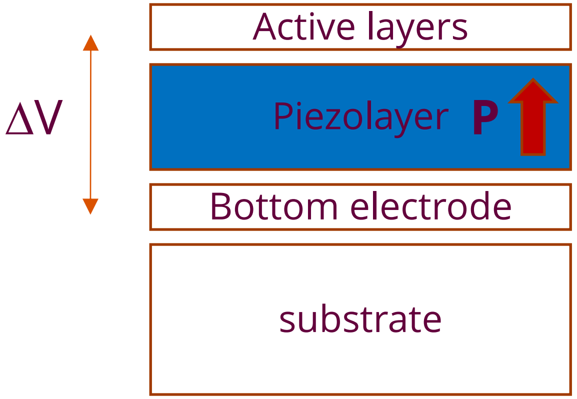
3. Strain based devices

- Focus on the link magnetism/strain
- Results on magnetic field sensing
- Flexoelectricity



Conclusion & discussion

Main idea:
Additional degree of freedom thanks to substrate realease of piezoelectric films



For perpendicular geometry: $\frac{\Delta l}{l} = d_{33}^{eff} . \Delta V$

$$d_{33}^{eff} = d_{33} - \frac{2d_{31}(s_{13}^{E} + {}^{\upsilon}/_{Y})}{s_{11}^{E} . s_{12}^{E}}$$

S_{ij} are the elastic compliances of the film at constant electric field

υ

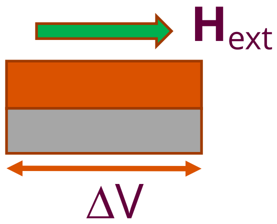
ν : Poisson's ratio of the substrate

Y : Young's modulus of the substrate

K. Lefki and G.M. Dormans, J. Appl. Phys. 76, 1764 (1994)

Freestanding devices => release of substrate clamping $\boldsymbol{d_{33}^{eff} = d_{33}}$

Extrinsic Magnetoelectric effect (ME): coupled magnetic and electrical phenomenon via elastic interaction



$$ME_H = \frac{\text{magnetic}}{\text{mechanical}} \times \frac{\text{mechanical}}{\text{electric}}$$

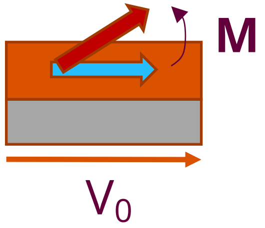
Direct magnetoelectric effect

Magnetism H — Magnetostriction → Mechanical T — Direct piezoelectric effect → Polarisation P

Magnetism H ← Villari effect — Mechanical T ← Inverse piezoelectric effect — Polarisation P

Converse magnetoelectric effect

$$ME_E = \frac{\text{electric}}{\text{mechanical}} \times \frac{\text{mechanical}}{\text{magnetic}}$$



Some reviews:
C-Wen Nam, JAP (2008)
G. Srinivasan, An. Rev. of Mat. Res. (2010)

ME direct and converse are highly non-linear mechanisms

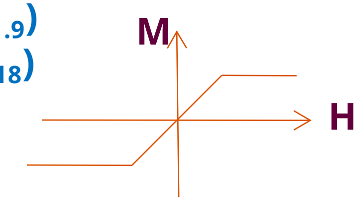
T Tran Nguyen, JAP 2011

Focus on the link magnetism/strain

Concerned materials:
=> Strong link between selected materials and desired device application

Direct effect: $\alpha = \frac{\delta \vec{P}}{\delta \vec{H}}$

Linear and high magnetostrictive ferromagnet
Terfenol-D ($Tb_{0.3}Dy_{0.7}Fe_{1.9}$)
Metglass ($Fe_{40}Ni_{28}Mo_{4}B_{18}$)



Example of magnetoelectric sensors
$\Rightarrow$ passive detection
$\Rightarrow$ 50pT sensibility achieved with glued layers

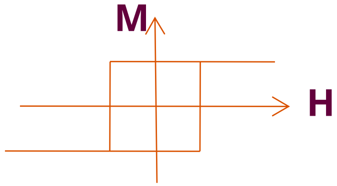
Huang Giang D.T., Sensors and Actuators A (2009)

Energy harvesting…

Converse effect: $\alpha = \frac{\delta \vec{M}}{\delta \vec{E}}$

Remnant state and low saturation field in the ferromagnetic layer are needed
CoFeB
FeGaB

Lebedev G.A., JAP 2012



Tunable RF devices
Lou J., APL 2009
Laur, V. IEEE Trans. on Mag 2013

Spin electronic devices
=> Control of the magnetization by electric field

Piezoelectric layers: $PbZrTiO_3$ (PZT) or $PbMgNbO_3$-$PbTiO_3$ (PMN-PT) (large piezoelectric response)
$BaTiO_3$ (using domain engineering; c to c/a2 multi-domain state S. Gepрägs PRB 88 (2013))

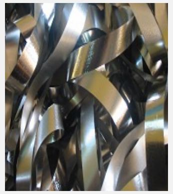
Magnetoelectric sensors

N. Nguyen PhD 2018

S. Hem PhD 2023

Macro-sensors

Bulk materials **Glue => Reduce mech. Coupling** **Large coupling surface**

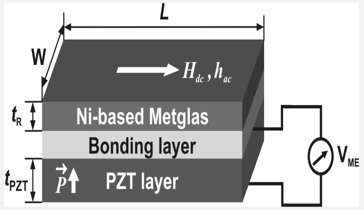


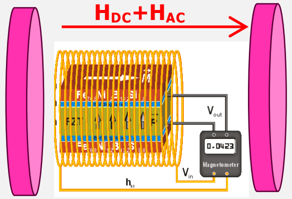
Metglas

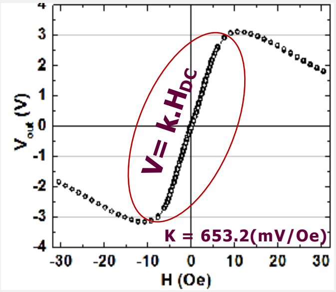
+ (Glue)

PZT

=







D.T Giang, SNA, 2012

⇩

Micro-sensors

Mobile applications : Expected market growth of 30% / year

Sensors for Wearable Electronic & Mobile Healthcare, report, Yole Développement (2015)

Reducing:

Size

Power consumption

Price

Improving

Lifetime

Harsh environment

Electronic integration

Device:
Freestanding cantilever

Measurement:

Resonance frequency shift

Magnetoelectric sensors

N. Nguyen PhD 2018

S. Hem PhD 2023

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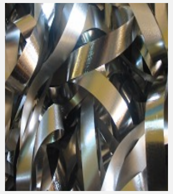
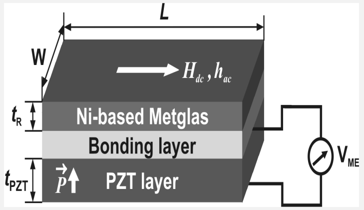


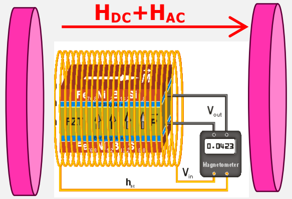
Metglas

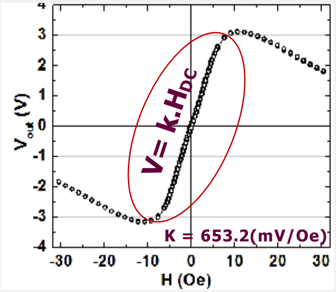
+ (Glue)

PZT

=







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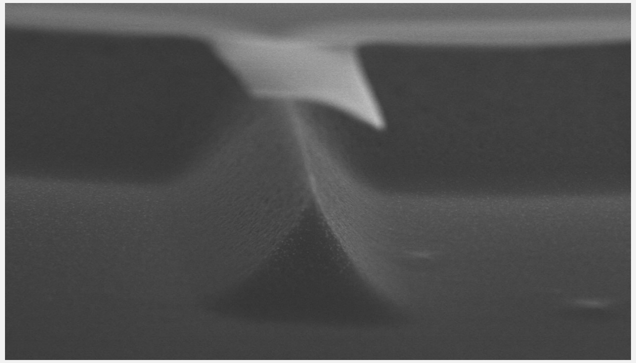
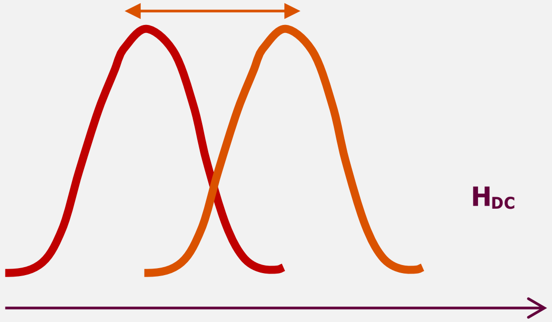


Measurement:

Resonance frequency shift



université PARIS-SACLAY

Magnetoelectric sensors

N. Nguyen PhD 2018

S. Hem PhD 2023

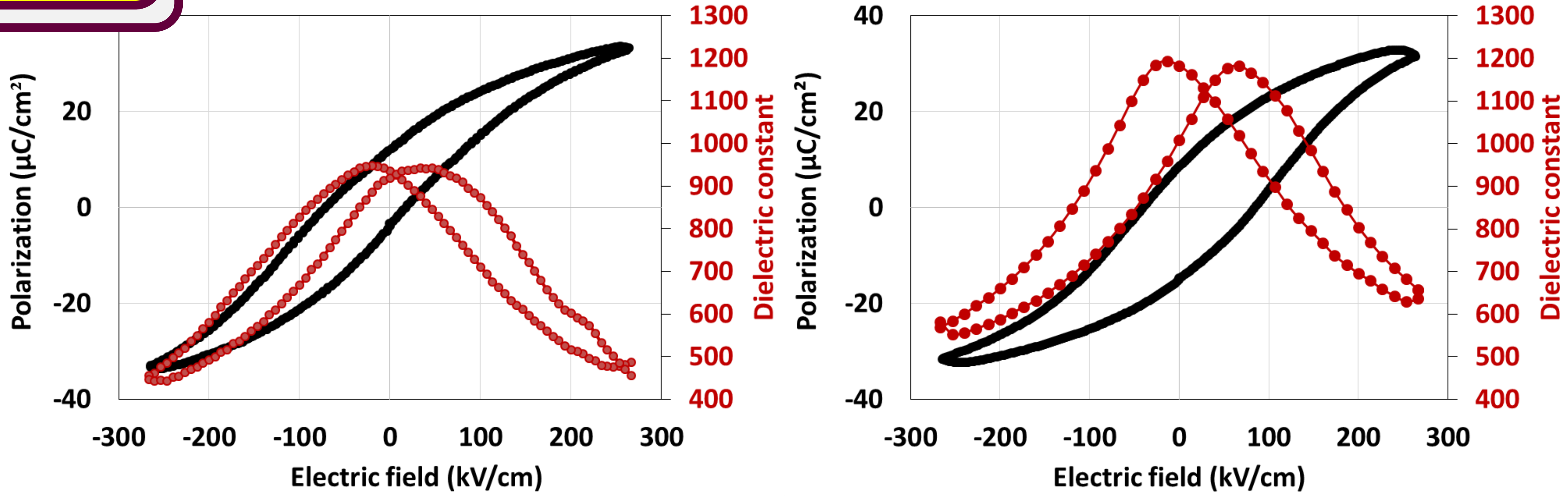
Sputtered TbFeCo

No bonding layer

Epitaxial PZT
52/48 Zr/Ti ratio

➢ **Structure and ferroelectric properties of cantilevers**

Before release **After release**



No major change in ferroelectric properties of the epitaxial PZT thin film polarization

Magnetoelectric sensors

N. Nguyen PhD 2018

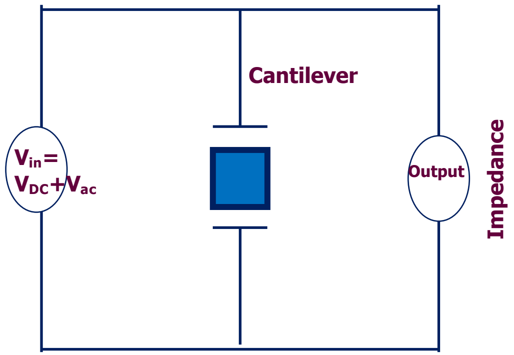
S. Hem PhD 2023

Sputtered TbFeCo

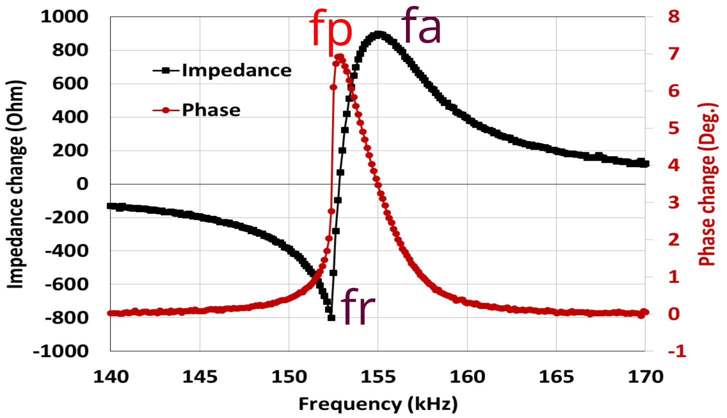
No bonding layer

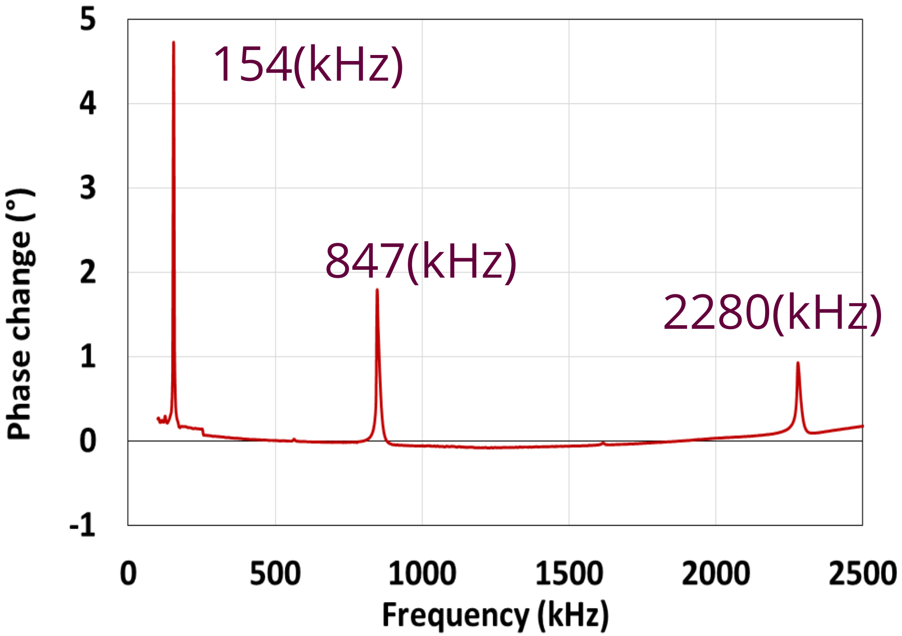
Epitaxial PZT
52/48 Zr/Ti ratio

➢ Dynamic response – Methods of study



fr: the resonant frequency, fa: the anti-resonant frequency





Theory: 1: 6.22 : 17.61

Impedance analyzer: 1: 5.5 : 14.8

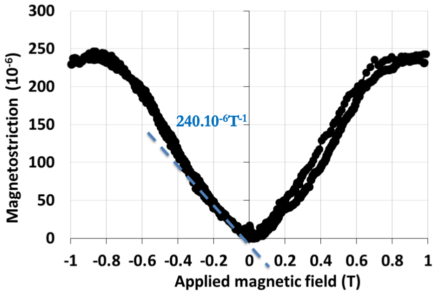
A GOOD agreement between theory and the experimental methods

Q-factor:
64 @ Athmosphere
1000 @ 1 mbar

Magnetoelectric sensors

N. Nguyen PhD 2018

S. Hem PhD 2023



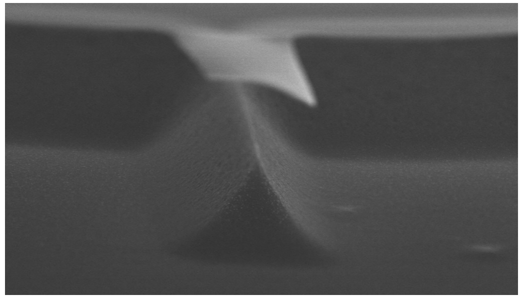
Sputtered TbFeCo

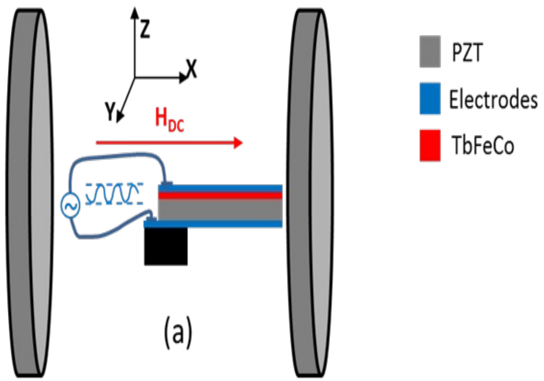
No bonding layer

Epitaxial PZT
52/48 Zr/Ti ratio

Addition of TbFeCo

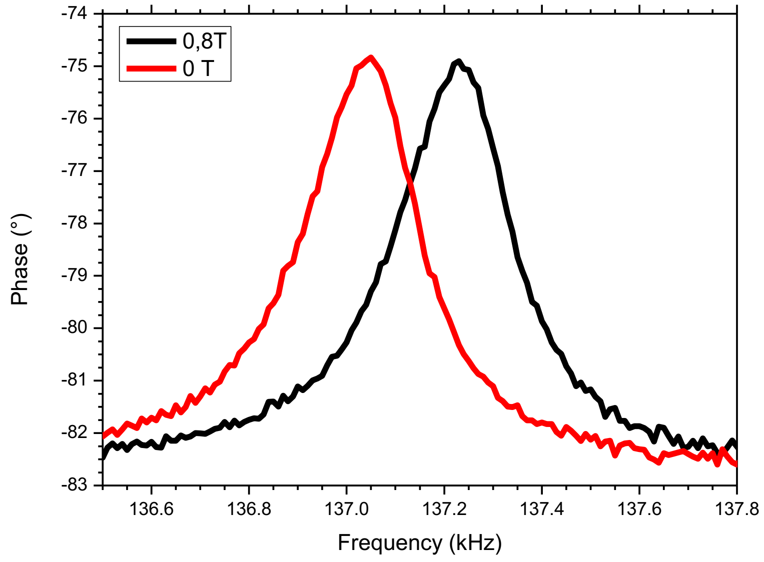
RF magneton sputtering





More details in Nguyen et al.; APL Mater. 9, 041103 (2021)

Resonant frequency depends on external magnetic field



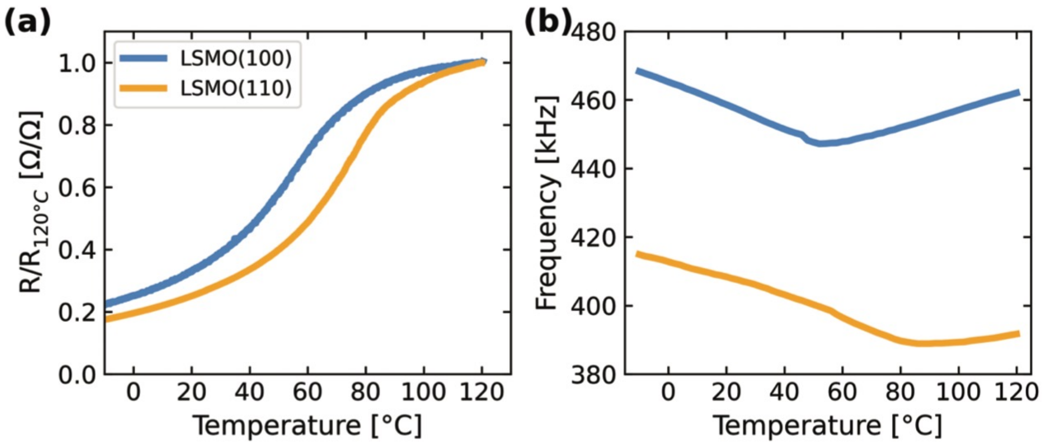
Optimizing sensitivity => optimize material quality

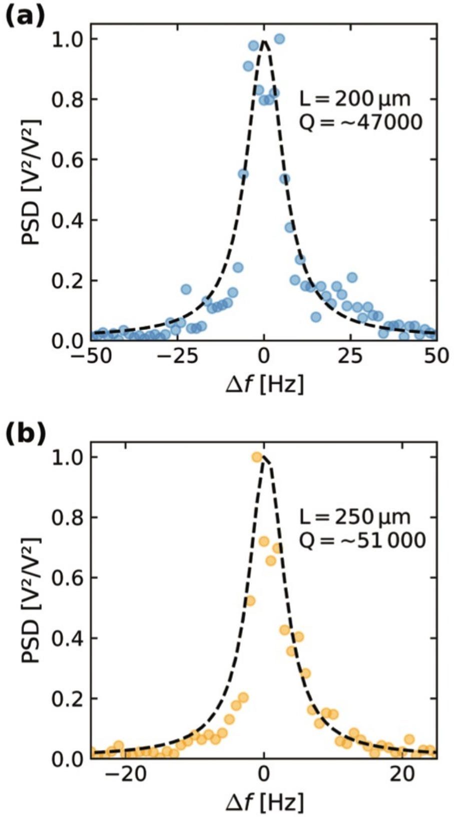
RESEARCH ARTICLE

NANO · MICRO small
www.small-journal.com

Stress Analysis and Q-Factor of Free-Standing (La,Sr)MnO_3 Oxide Resonators

Nicola Manca, Federico Remaggi, Alejandro E. Plaza, Lucia Varbaro, Cristina Bernini, Luca Pellegrino, and Daniele Marré*





Higher cristalline quality => Higher quality factor

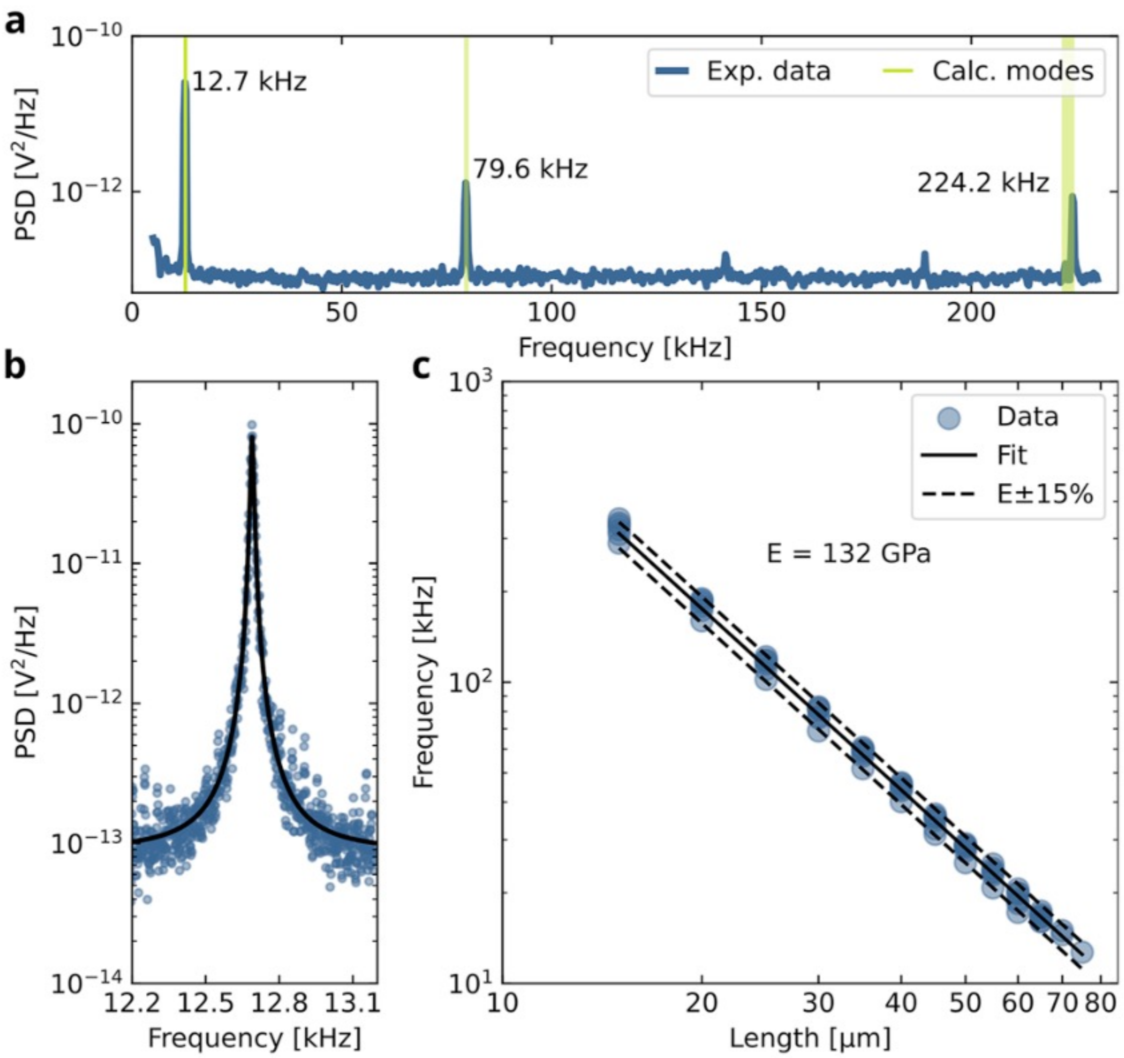
Optimizing sensitivity => optimize material quality

Strain, Young's modulus, and structural transition of $EuTiO_3$ thin films probed by micro-mechanical methods

Cite as: APL Mater. 11, 101107 (2023); doi: 10.1063/5.0166762
Submitted: 7 July 2023 • Accepted: 18 September 2023 •
Published Online: 6 October 2023

View Online Export Citation CrossMark

Nicola Manca,[1,a)] Gaia Tarsi,[2] Alexei Kalaboukhov,[3] Francesco Bisio,[1] Federico Caglieris,[1]
Floriana Lombardi,[3] Daniele Marré,[1,2] and Luca Pellegrino[1]



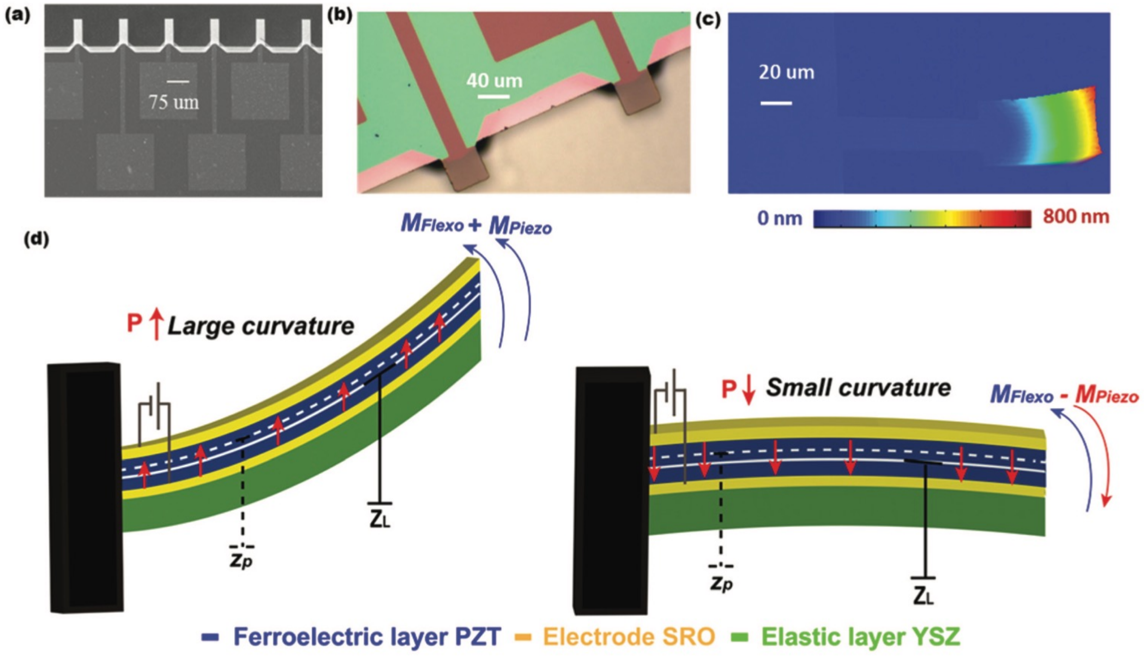
Single phase multiferroic EuTiO3

Opens for higher sensitivity

Other strain based devices

Energy harvesting through flexoelectricity ?

G. Catalan, *The emancipation of flexoelectricity*
J. Appl. Phys. 131, 020401 (2022)



Inkjet Pinting

G. Rjinders, *Epitaxial PZT films for MEMS printing applications*
MRS Bulletin 37 1030 (2012)

UNIVERSITY OF TWENTE.

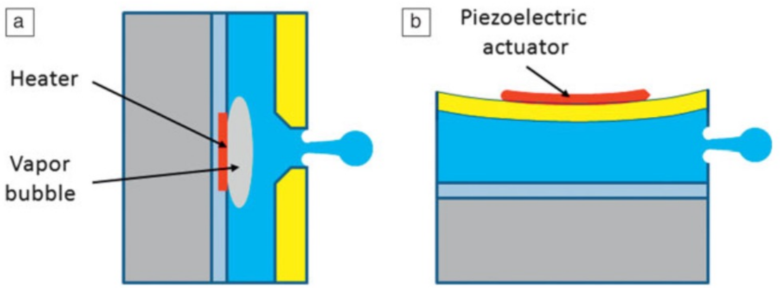


Figure 1. Principle of (a) a thermal bubble jet print head and (b) a piezoelectrically actuated inkjet print head. Thermal print heads have currently the major share in the microelectromechanical inkjet market for use in small office/home office printers, while piezoelectric print heads are an emerging product in the professional printing arena.

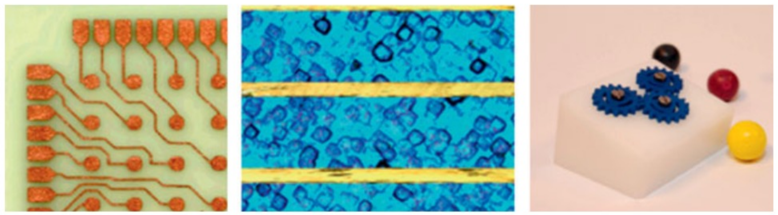


Figure 2. Examples (from Reference 84) of industrial printing applications of piezoelectric inkjet (left to right): printed circuit board inner layer, solar cell front side metallization, and 3D printed wheels.

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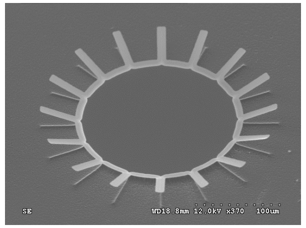
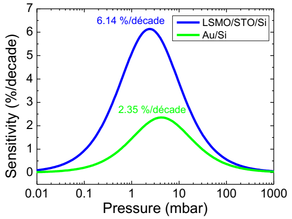
- Motivations & approaches
- Focus on MEMS devices
- Material review for Si integration



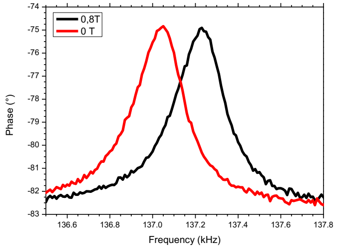
2. Thermal based devices

- Bolometer
- Pressure sensor
- Thermoelectricity



3. Strain based devices

- Focus on the link magnetism/strain
- Results on magnetic field sensing
- Flexoelectricity



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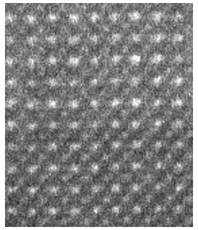
Conclusion & discussion

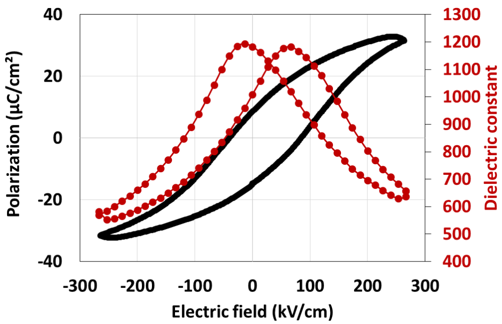
Remarques and discussions



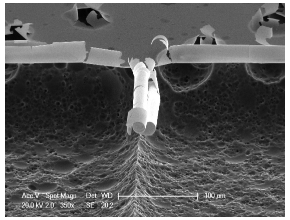
Good material quality is a challenge

Room for improvement compared to semiconductors

Level of quality depends on the aimed physical properties

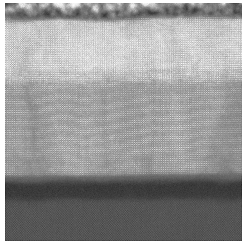


Difficult compromise



Managing strain is crucial for MEMS

Adaptation layers brings complexity



Silicon integration remains a challenge (Interfacial layers and SiOx)

Devices need to be thinked from thin film growth