



energie atomique • énergies alternatives

# Best practice on S&T parks The Grenoble area Impact on large scale RIs

Amal CHABLI



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- Introduction
- Grenoble eco-system and Large-scale facilities
- Highlights of industrial R&D technological cases
- Technical limitations & mitigations
- Operational limitations & related initiatives

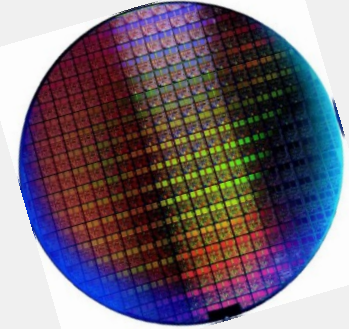
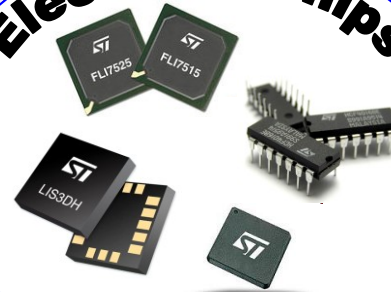
**How to leverage for industry  
the outcomes of basic research  
performed in RIs?**

## Usages



**Health  
Energy  
Communication**

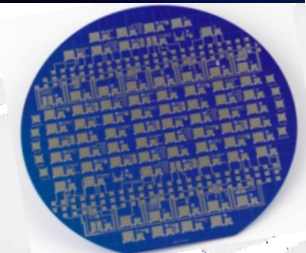
## Electronic chips



**Integrated devices**

**Nanomaterials**

## Batteries





- Applied technologies
- Transverse competencies
- Co-located industries



### Upstream research



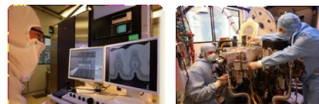
### Technology management



### Large scale facilities



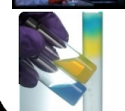
### Micro-nanotechnologies



### New technologies for energy



### Biology & biotechnologies



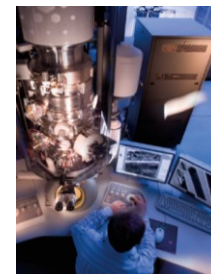
30 000 workers  
40 companies





Transfer of MNT  
from research  
to industry

Nanocharacterisation



leti  
liten

INDC  
INSTITUT NANOSCIENCES  
ET CRYOGENIE

Grenoble



300 mm

10 000 m<sup>2</sup>



200 mm



[www.minatec.com](http://www.minatec.com)

4000 actors  
300 patents  
1200 publications

## Comprehensive expertise

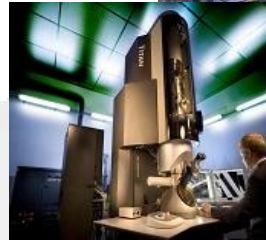
- Electron Microscopy
- Ion Beam Analysis
- Nuclear Magnetic Resonance
- Mechanical Tests
- Optical Techniques
- Sample Preparation
- Scanning Probe Microscopy
- Surface Analysis
- Trace & Contamination Analysis
- X ray Analysis
- Clean Room Metrology

## Multidisciplinary skills

- Biotechnology
- Material Science
- Micro & Nano systems
- Nanoelectronics
- Nanotoxicology
- Photovoltaic
- Optoelectronics

## Serving

- Choice and improvement of materials
- Analysis of scaling effects and correction or use
- Design of integration processes and validation



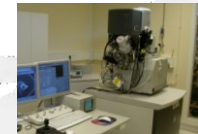
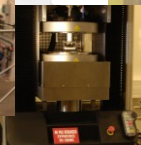
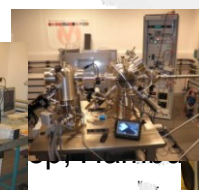
40 tools  
80 researchers

## Experts

- Material science
- Technological research
- Tool suppliers



Complementarily to the large-scale facilities  
like ESRF & ILL

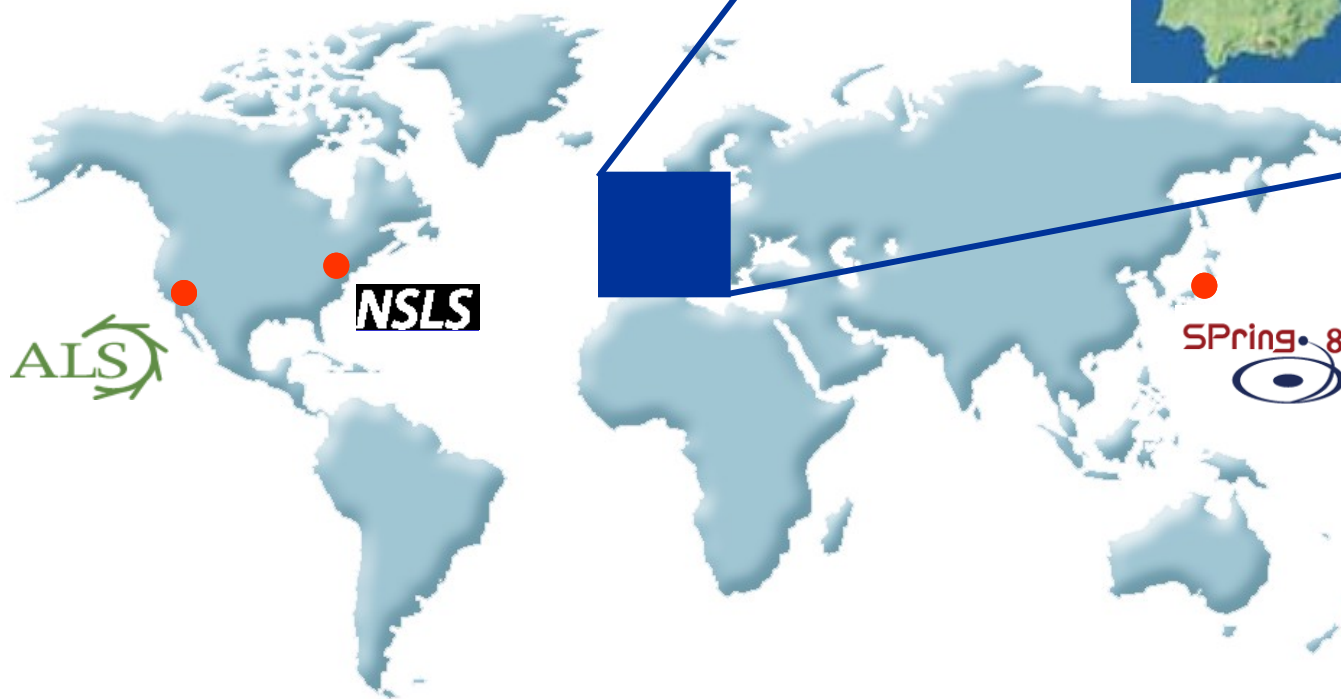




## Micro- & nano-technologies

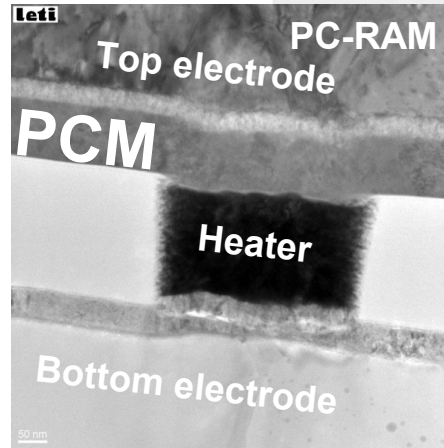
Key access figures  
2006-2011

- Annual proposal number      **15-20**
- Allocated shifts per year      **~100**
- Accepted proposals              **30%**
- Beam time purchase (occasional)



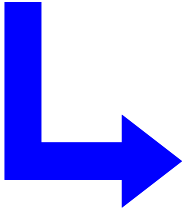
- Brilliance
- High flux
- High stability
- Spectroscopy
- Multimode analysis
- Tunable excitation
- Pulsed excitation
- In depth analysis

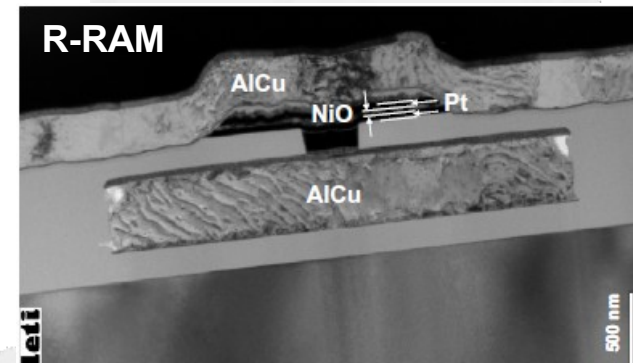
- Energy storage
- Photovoltaics
- MEMS-NEMS on Si
- Biotechnologies on Si
- Photonics on Si
- Non Volatile Memories



## SR X-ray & neutrons

- Diffraction
- Spectroscopy
  - Fluorescence
  - Absorption
  - Diffusion
  - Photoelectron
- Reflection
- Imaging
- Tomography
- ....

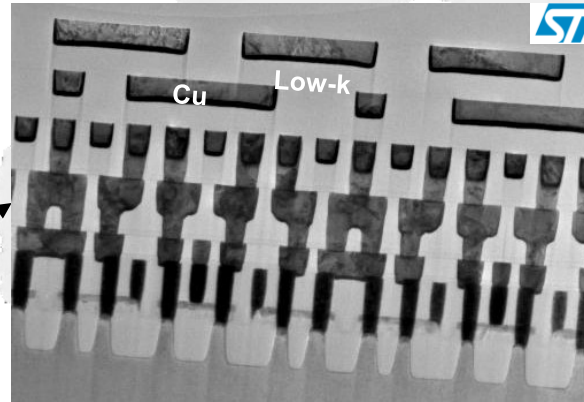
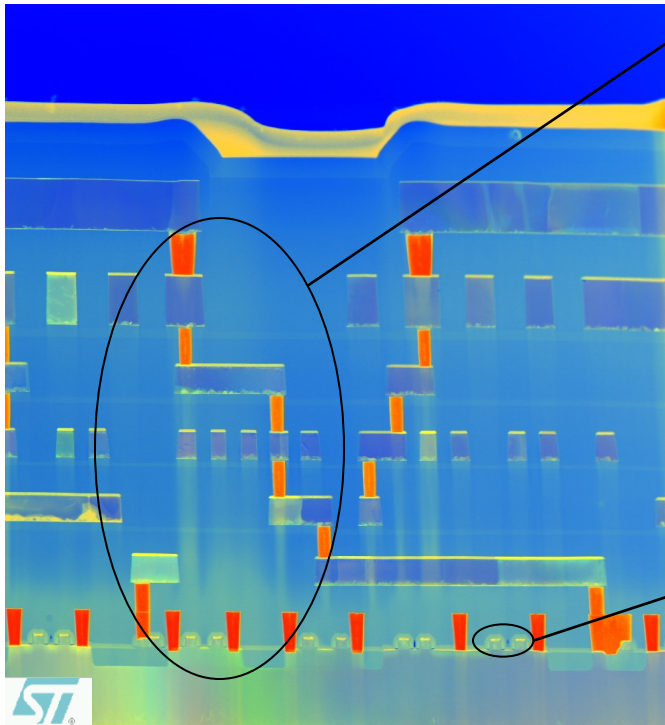
- 
- Local chemical composition
  - Local chemical bonding
  - Local crystalline structure
  - Nanoscale morphology
  - Interface properties
  - ...





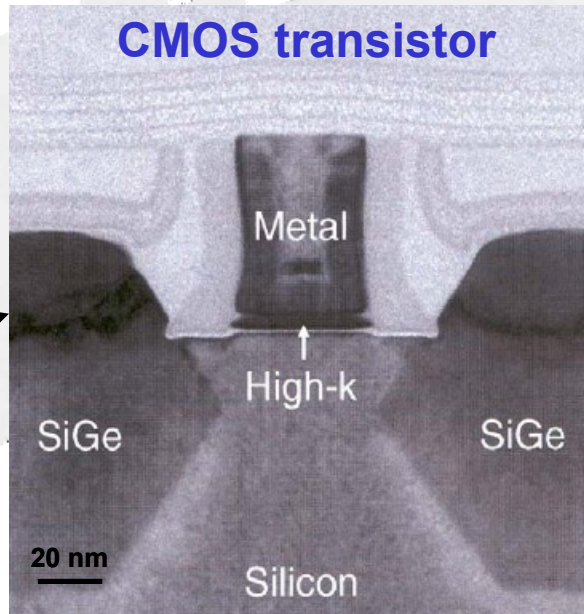
### Cu interconnections

### Transmission Electron Microscopy



- Cu polycrystalline properties
- Cu strain & stress
- Low-k material porosity
- Inter-diffusion
- Stability

### CMOS transistor

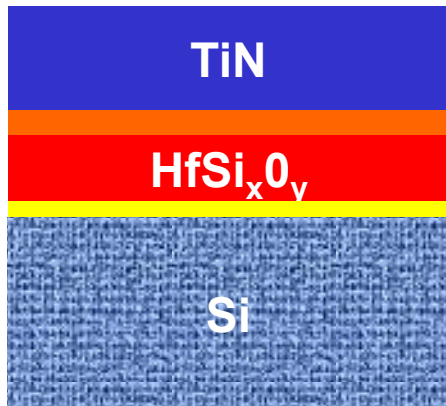


- Crystallography
- Composition
- Chemical bonds
- Dopant site & coordination
- Inter-diffusion
- Interface properties
- Stability

K. Kuhn (Intel), *SSDM*, Japan, 2009

ERF Workshop, Hamburg, May 31 – June 1, 2012

Metal Gate and High k dielectric  
for power consumption reduction



LaO<sub>x</sub>

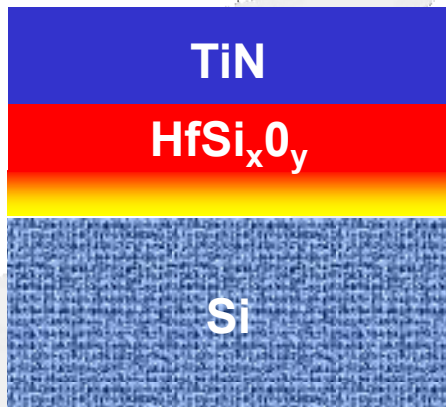
SiO<sub>2</sub>

Si

HAXPES

Without  
annealing

Diffusion of La

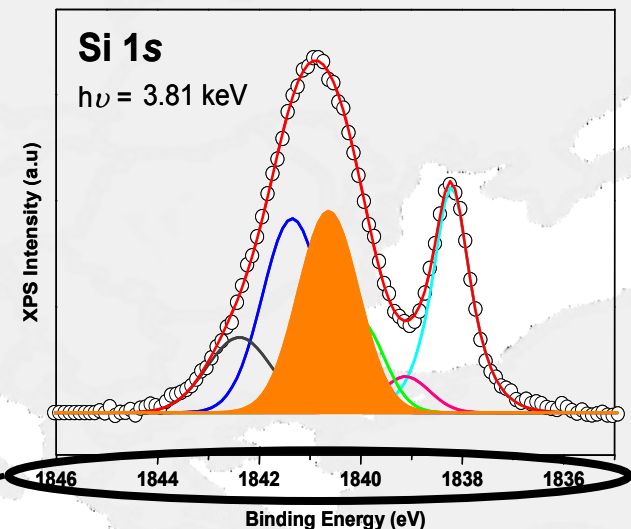
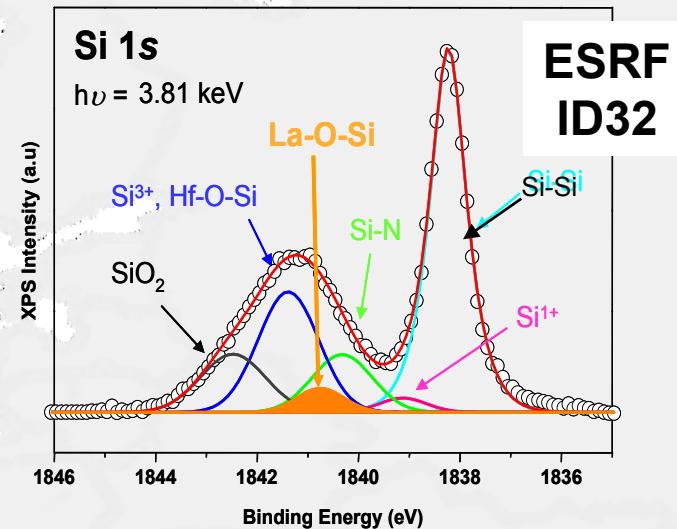


SiLa<sub>x</sub>O<sub>y</sub>

Si

After annealing

Inter-diffusion control  
for electrical characteristics monitoring



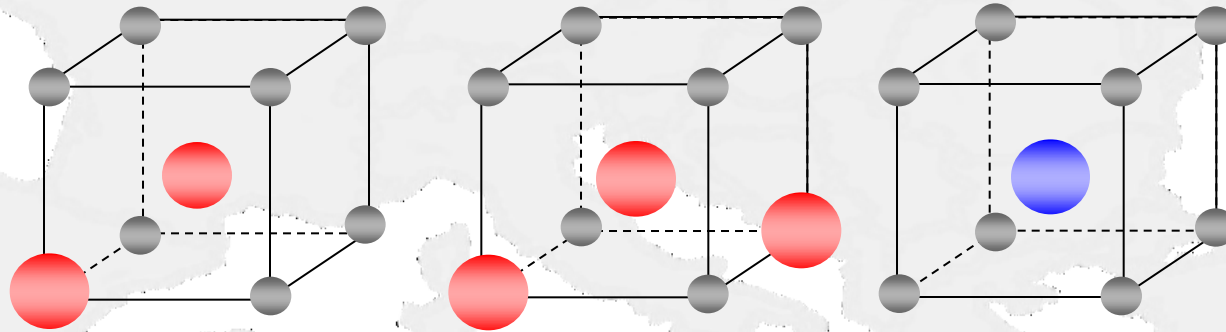
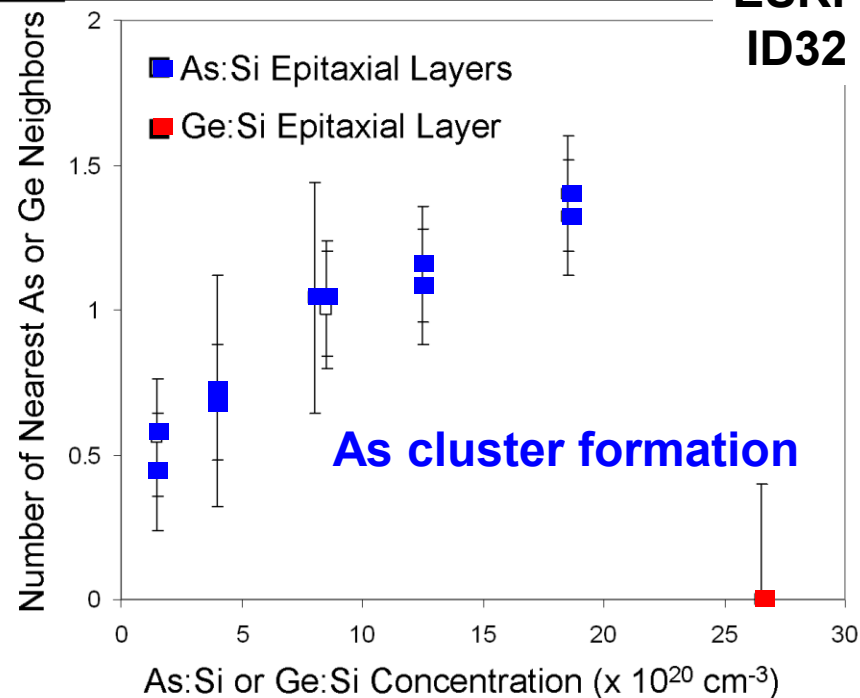
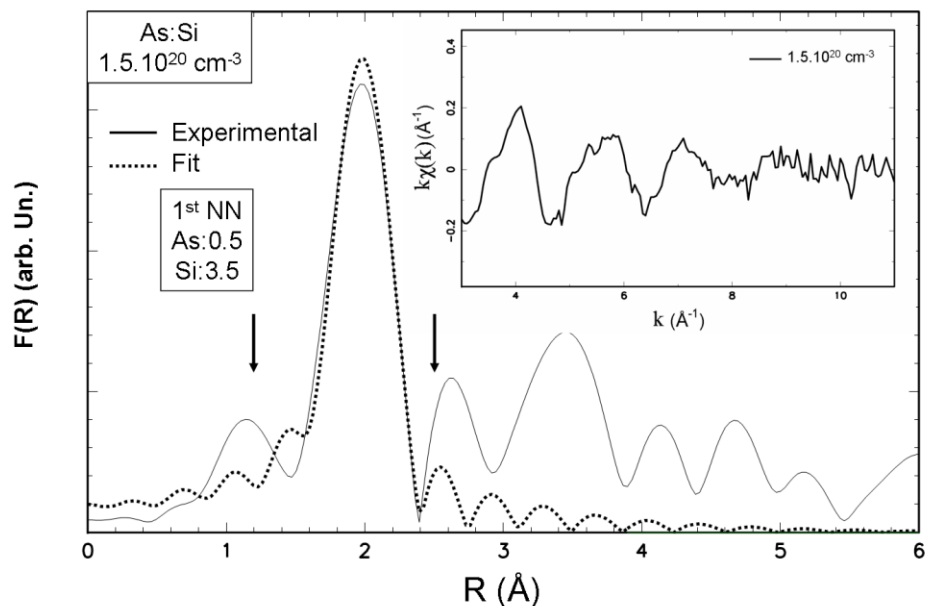
R. Boujamaa et al. JAP 111, 054110 (2012)

- ▶ Hard x-ray: analysis of full stack
- ▶ Si 1s: not observable with lab source

## Trends towards high level doping of the transistor channel

EXAFS

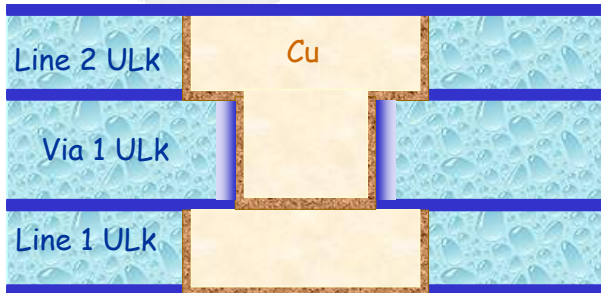
G. Servanton. PhD research, Grenoble (2010)



▶ **Opposite results to the objectives**

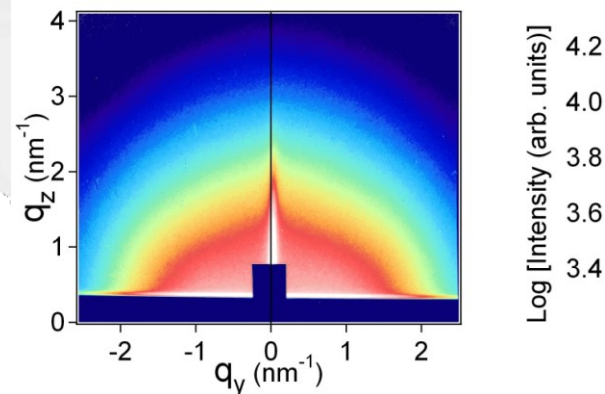
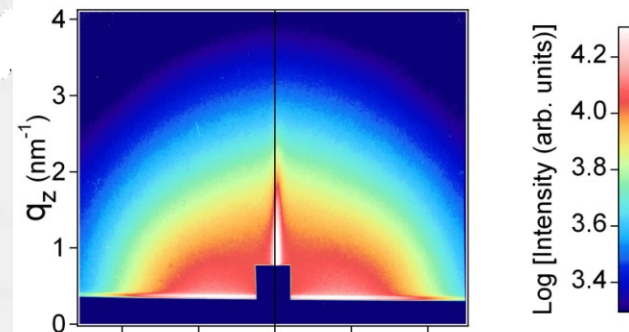
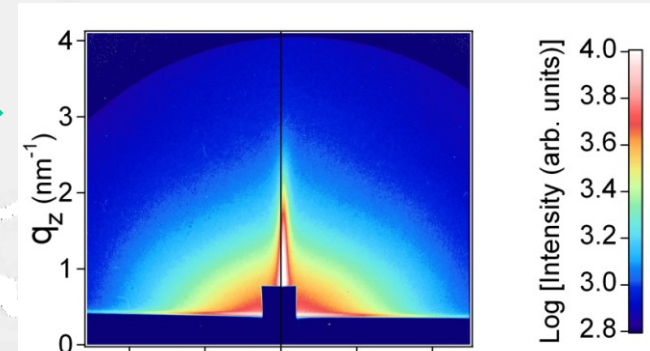
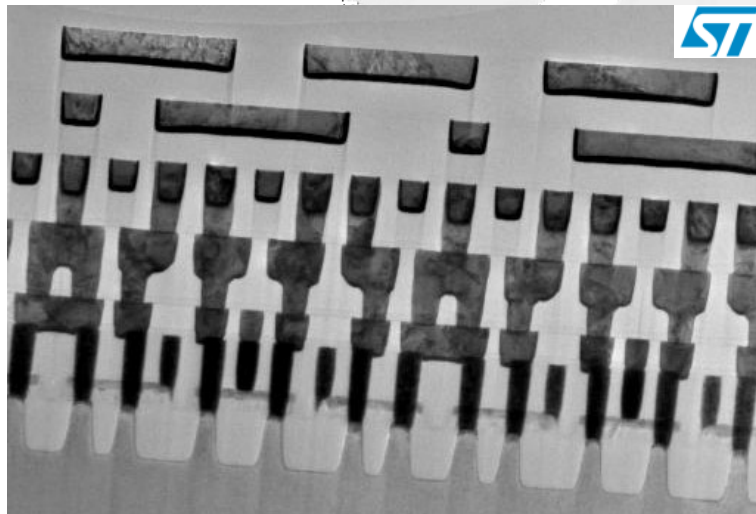


## Porous materials for dielectric isolation of Cu interconnections

**GISAXS**

**Pores**

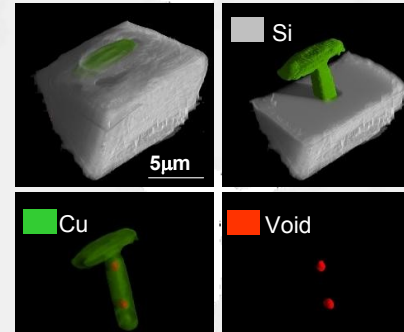
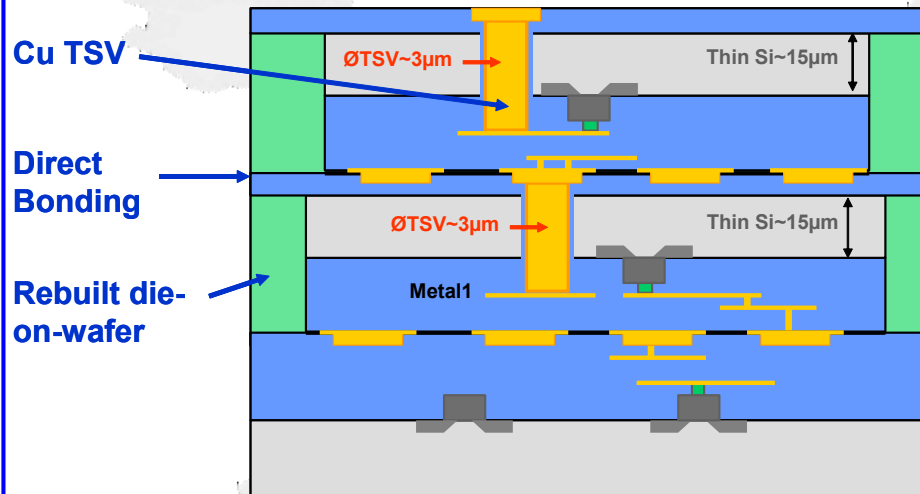
- Size & shape
- Density
- Stability during process

**Selection of the ULK material and integration process optimization for production of the 65-nm node and below**

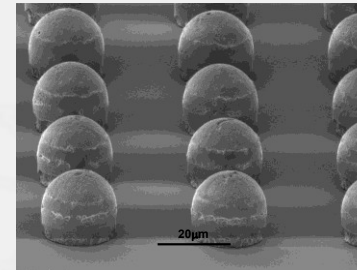
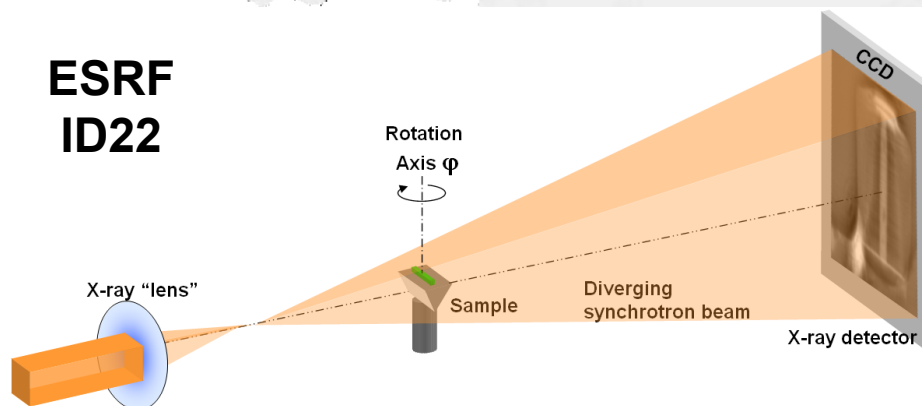

**ESRF  
BM32**

*V. Jousseume et al.,  
Appl. Surf. Science,  
254 (2007) 473*

## ► Non destructive detection of voids

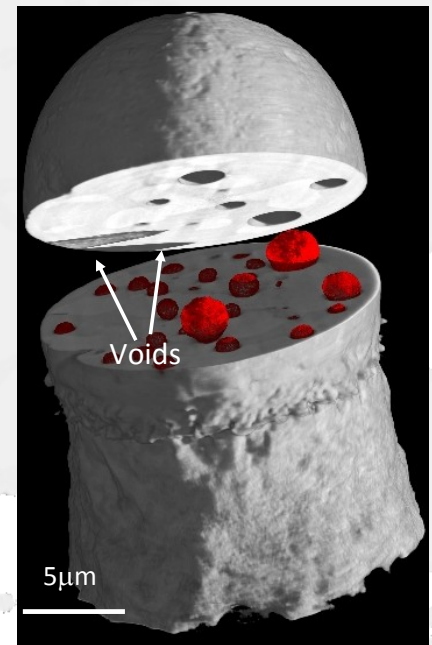


## X-ray tomography



Cu

*P. Bleuet et al.,  
Rev. Sci. Inst.,  
80, 056101-3 (2009)*



**Up-grade of beam lines towards 10-nm spatial resolution**

## White beam $\mu$ Diff: Cu interconnect lines

P. Gergaud et al., *AIP Conf. Proc.* 817, p. 205 (2006)



BM32

## HAXPES: CMOS gate stacks

E. Martinez et al., *J. of Vac. Sci. & Technol. B* 25, 86 (2007)

J. Rubio-Zuazo et al., *AIP Conf. Proc.* 931, p. 329 (2007)

C. Gaumer et al., *AIP Conf. Proc.* 1173, p. 40 (2009)



ID32

## Neutron reflectometry: Low k dielectric for Cu interconnects

D. Rebiscoul et al., *Microelectronic Eng.*, 85, p. 2089 (2008)



D17

## Soft x-ray XPS: CMOS Gate dielectric

L. Q. Zhu et al., *J. of Appl. Phys.* 105, 024102 (2009)



TEMPO

## X-ray total scattering: Doping of GeTe for PC-RAM

G.E. Ghezzi et al., *Appl. Phys. Lett.*, 99 151906 (2011)



CRYSTAL

## EXAFS: HgCdTe for IR detectors

P. Ballet et al., *J. Electron. Mater.* 38, p. 1726 (2009)



BM29

## EXAFS: GeSbTe for PC-RAM

X. Biquard et al., *Appl. Phys. Lett.* 98, 231907 (2011).



BL01B1

Large-scale RIs are  
key component  
in the innovation cycle  
and industrial R&D



## At the technical level

- Shutdown of regularly used beam lines
  - case of **ESRF-ID32 ?**
- Need for improved spatial resolution
- Need for time resolved experiments


## At the operational level

- Special requirements of industrial development cycle
  - Speed and frequency of access
- Specific constraints of industrial competitiveness
  - IP management and confidentiality
- Cost of beam time



**Several initiatives**

- Focused bilateral agreements with LSFs
  - Demonstration of  $\mu$ Tomography capabilities for MNT
    - ◆ Dedicated postdoctoral position (ANR MiDiFaBI, 2006)
  - Nano-pencil beam diffraction instrumentation developments
    - ◆ Dedicated postdoctoral position (ANR RTB programme, 2010)
  
- Contribution to French CRG lines up-grade
  - $\mu$ Diffraction instrumentation developments (CRG-BM32)
    - ◆ Dedicated national project (ANR MiDiFaBI, 2006)
  - Energy range extension of beam line (CRG-BM1)
    - ◆ Dedicated investment and postdoctoral position (ANR RTB programme, 2011)



**R&D requirements of MNT  
taken into account in the  
upgrade of the LSFs**

- Development of XPS microscopy (XPEEM)
  - High photon flux on sample to Improves lateral resolution(x5) in core-level imaging
  - NanoESCA XPEEM acceptance tests
    - ◆ Dedicated national project (ANR XPEEM, 2005)



ID08

## Direct Quantification of Gold along a Single Si Nanowire

A. Bailly,<sup>†</sup> O. Renault,<sup>\*†</sup> N. Barrett,<sup>‡</sup> L. F. Zagonel,<sup>‡</sup> P. Gentile,<sup>§</sup> N. Pauc,<sup>§</sup>  
F. Dhalluin,<sup>||</sup> T. Baron,<sup>||</sup> A. Chabli,<sup>†</sup> J. C. Cezar,<sup>⊥</sup> and N. B. Brookes<sup>⊥</sup>

NANO  
LETTERS2008  
Vol. 8, No. 11  
3709-3714

- Application of XPEEM to scientific cases
  - Specific proposals submission
  - BESSYII, ELETTRA (2008, 6 weeks)
  - SOLEIL (2009, 3 weeks)



TEMPO



CIPO



UE52-PGM1

K. Huang et al., *J. Phys. Chem.*, 113, p. 21389 (2009)

K. Huang et al., *ACS Nano*, 4, p.4799 (2010)

C. Mathieu et al., *Phys. Rev. B* 83, 235436 (2011)

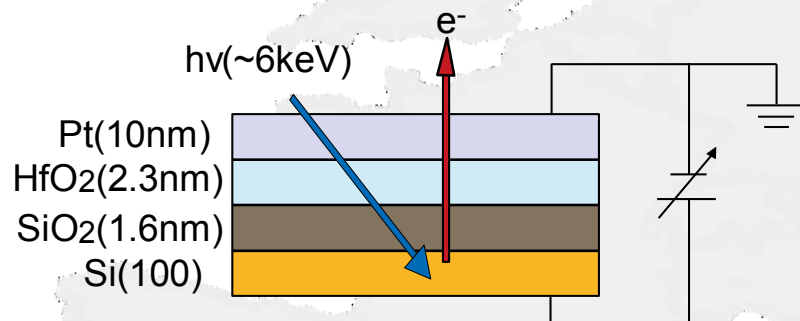
► **Impact on industrial R&D @ long term**



## ■ LTP for Innovation (LTPI)

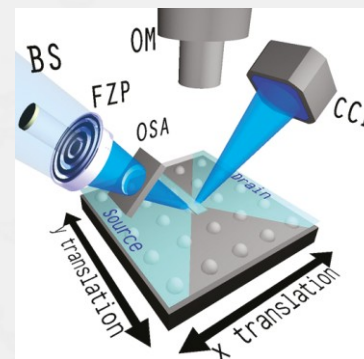
- To address a specific challenge of the industrial R&D
  - ◆ Example : In-operando characterisation for MNT (CMOS, MNV, NEMS)
    - Time and spatial resolution
    - Device non-destructive
    - Design of specific devices
- To induce breakthrough in LSFs instrumental developments

### HAXPS @ SPRING8-NIMS beam-line



Y. Yamashita et al., *ECS Trans.*, 41, p.331 (2011)

### $\mu$ XRD @ ESRF-ID01



N. Hrauda et al., *Nano Letters* 11, 2875 (2011)

► **Technical challenges**

► **Funding issues**

Ref. No 29587

EUROPEAN SYNCHROTRON RADIATION FACILITY  
INSTALLATION EUROPÉENNE DE RAYONNEMENT SYNCHROTRON



# New

## Standard Application for Beam Time at the ESRF

### Proposal Title ( 175 chars maximum.)

Study of the local environment of Ag<sup>+</sup> ions in Ag-Ge-S glasses thin films for Conductive Bridging RAM devices

### Keywords

#1: CBRAM

#2: X-ray Absorption Spectroscopy

#3: Ag-Ge-S

#4:

### • This proposal is:

A new proposal A resubmission of A continuation of : 

### • This proposal is:

Fundamental Science  % 34Applied Science  % 33Industrial Science  % 33

### Research Area of the proposal

 CH - Chemistry MD - Medicine HE - Hard Condensed Matter - Electronic and Magnetic MI - Methods and Instrumentation

**Compatibility of  
Scientific excellence with  
Technological excellence**

Applied Science  % 33 Industrial Science  % 33

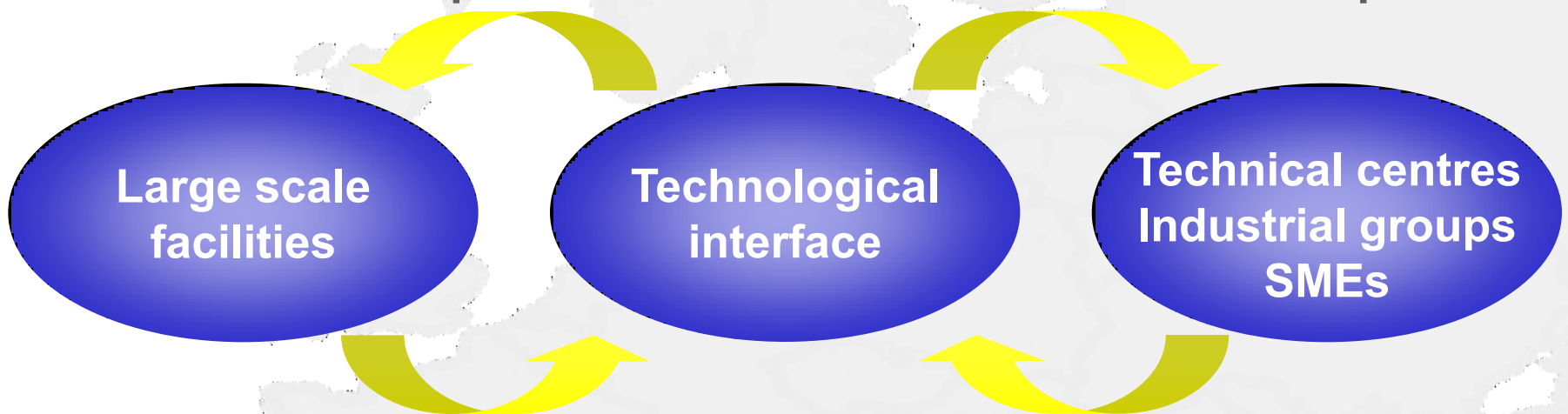


**What success rate ?**



- Beam line Scientists
- Characterisation experts

- Technology experts
- Material experts



- Experiment feasibility & commissioning
- Sample preparation
- Complementary characterization
- Data interpretation expertise
- Training & valorization

- ▶ **Investment in specific tools**
- ▶ **Funding projects for initiation and optimization through R&D programs (ANR IRT NanoElec)**



- Large-scale RIs are key component in the innovation cycle and industrial R&D
- Matching to industrial R&D requirements
- Technical limitations & mitigations
  - Specific cooperation agreements supported by dedicated funded projects to ensure specific beam line access
  - LTPI concept addressing industrial challenges together with coordinated breakthrough instrumental developments at LSFs
- Operational limitations & related initiatives
  - Enlarge scientific excellence to innovation
  - Offer a specific interface to industrial applications to optimize beam time costs
  - Fulfill IP constraints and confidentiality requirements by patent highlights introduction and post-patenting publications



- A. Bailly
- N. Barrett
- Cl. Gaumer
- G. Feuillet
- K. Huang
- B. Hyot
- V. Jousseume
- F. Martin

- C. Mathieu
- D. Rebiscoul
- I. C. Robin
- J.-Cl. Royer
- B. Stirling
- ...



- N. Bicais-Lepinay
- R. Boujamaa
- M. Gros-Jean
- R. Pantel
- A. Pakfar
- G. Servanton
- ...



- J. Beaucour
- N. Brookes
- E. Mitchell
- J. Susini
- ...



Thank you for your attention