Olivier Renault

CARAC 2020 7th edition

Lab-scale Hard X-ray Photoelectron spectroscopy (HAXPES) at the PFNC

23 novembre 2020

NANOELEC.

Surface Analysis at PFNC

- A state-of-the-art characterization platform
- 3 institutes of CEA-Grenoble share instruments & staff scientists
- 80 people
- 50 world-class equipments
- 3000 m² laboratories

PFNC

MINATEC

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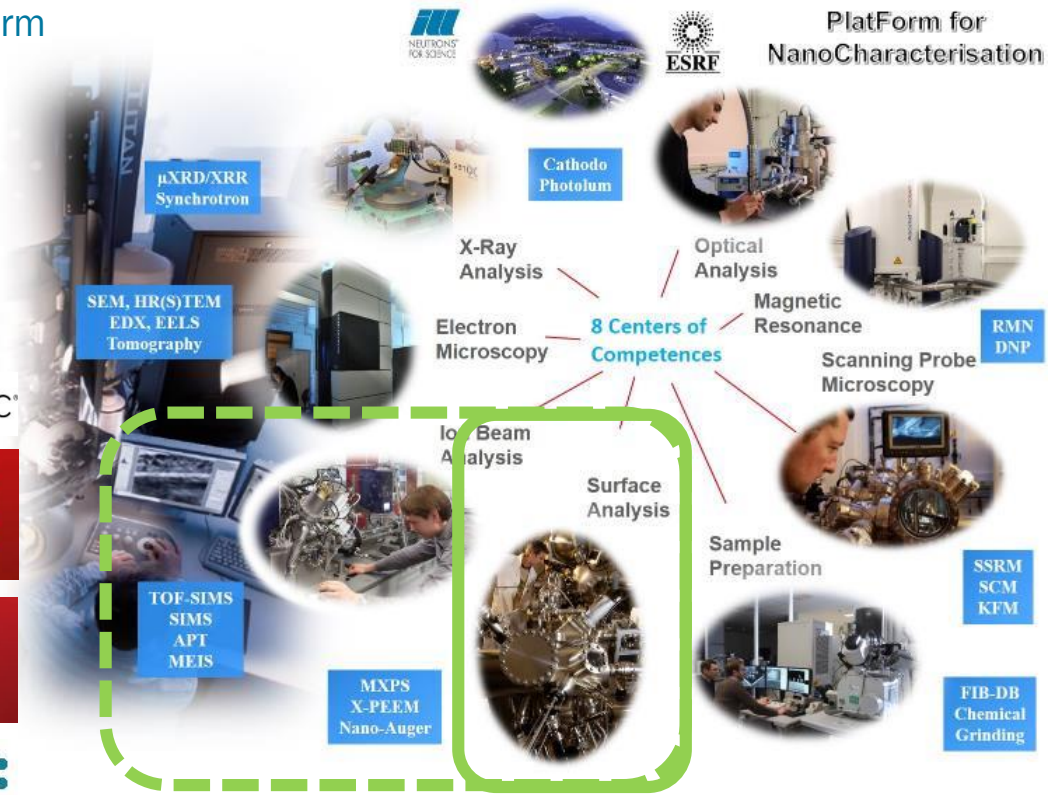
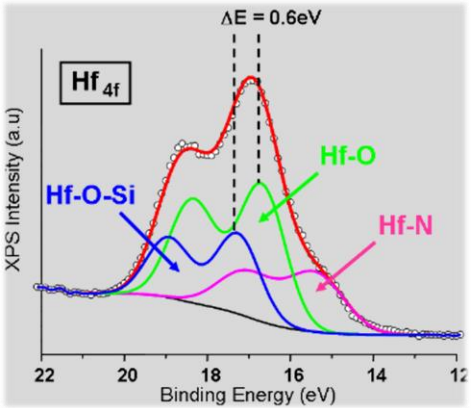
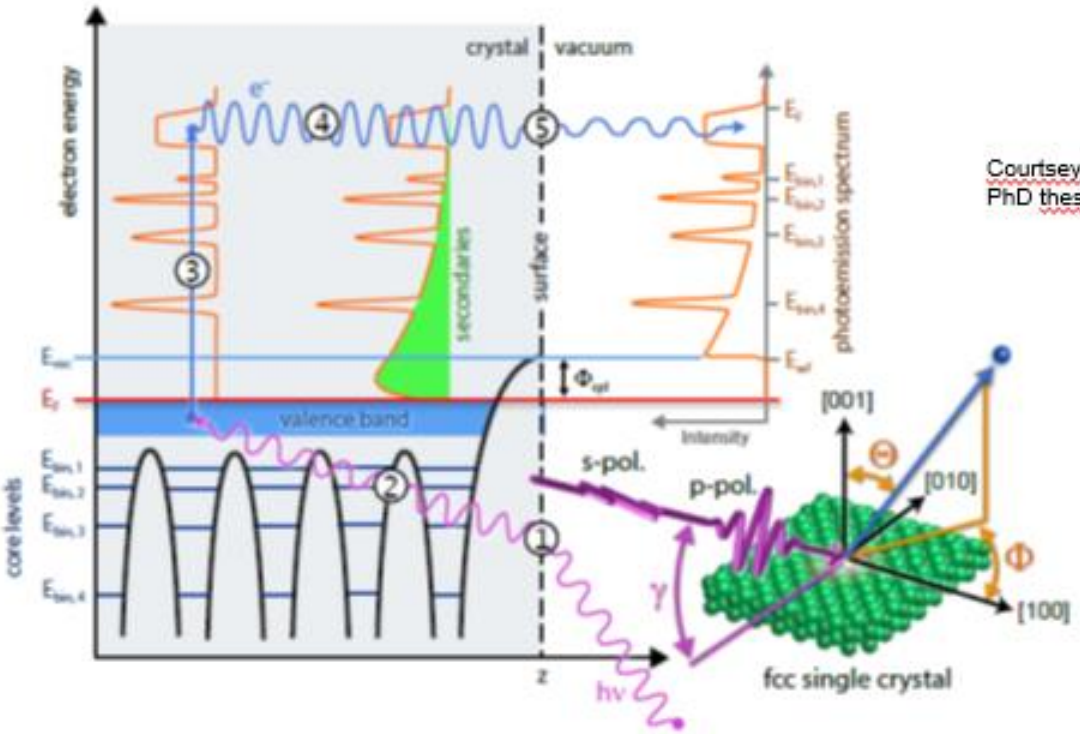


Photoelectron spectroscopy

- A **photon-in/electron-out** technique for analyzing occupied electronic states in matter
- **Higher photon energies** generate **faster photoelectrons**
- **Chemical** fingerprinting

$$E_K = h\nu - E_B - e\Phi_s$$

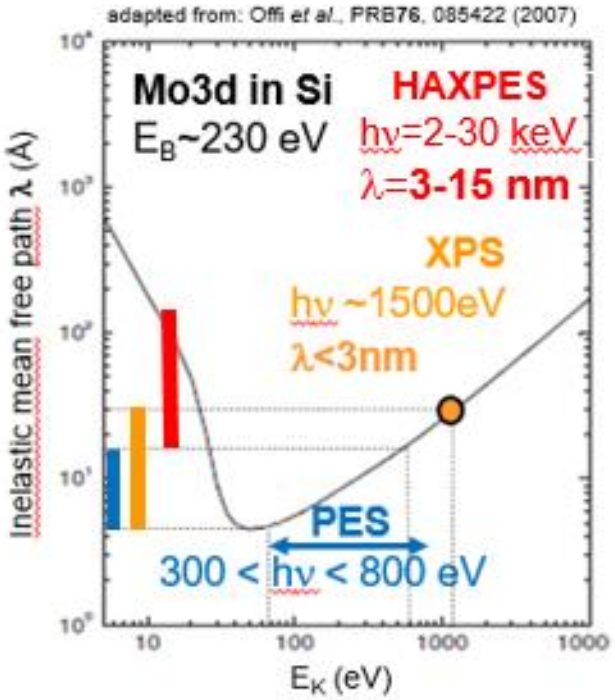


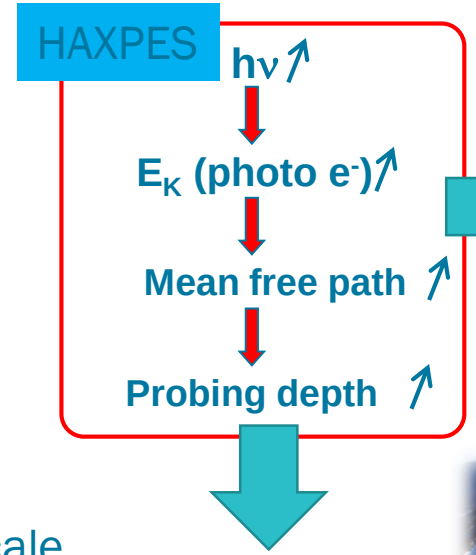


Courtsey M. Patt,
PhD thesis, Jülich, 2016

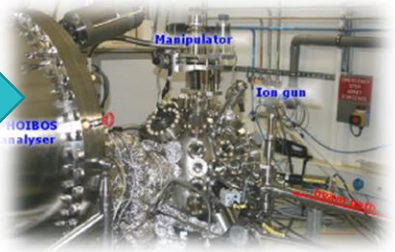
Hard X—ray Photoelectron spectroscopy (HAXPES)

- Photon energies < 10 keV
- Developped at synchrotron facilities
- Since 2018: commercial lab-scale instruments





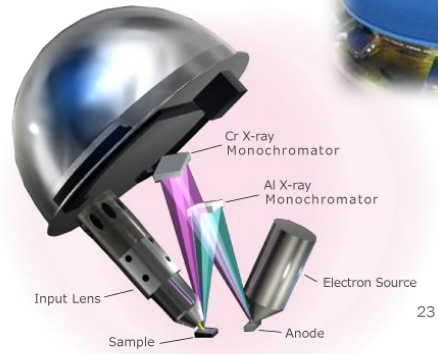
Synchrotrons



Lab-scale instruments

Al Kα (1486,6 eV)

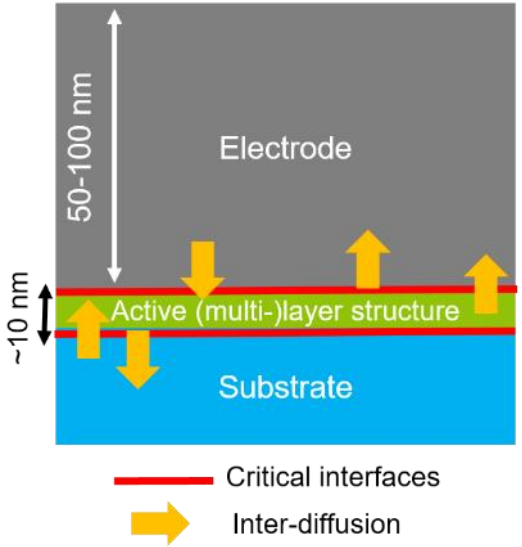
Cr Kα (5414,9 eV)
Ga Kα (5414,9 eV)



HAXPES for analyzing buried interfaces

- Access to sub-surface of materials (<50 nm)
- **Device technology:** access to buried interfaces

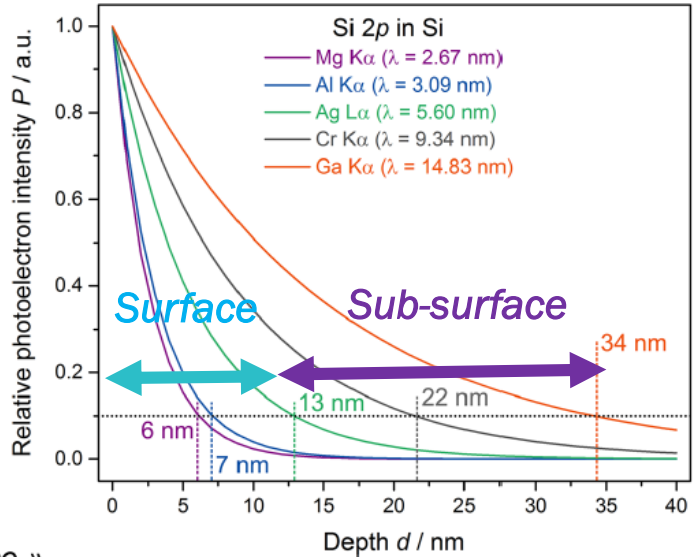
Multi- layered materials
In device technology



2000: *« The interface is the device »*
Herbert Kroemer, Nobel Lecture, dec. 8th, 2000

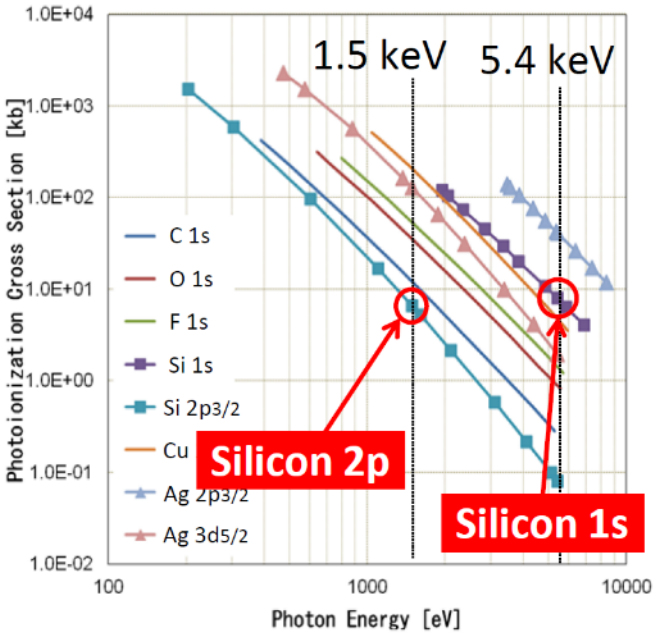
2012: *« The interface is **still** the device »*
Nat. Mater. ***11****, 91*

From: Regoutz *et al.*, *Rev. Sci. Instrum* **89**, 073105 (2018).

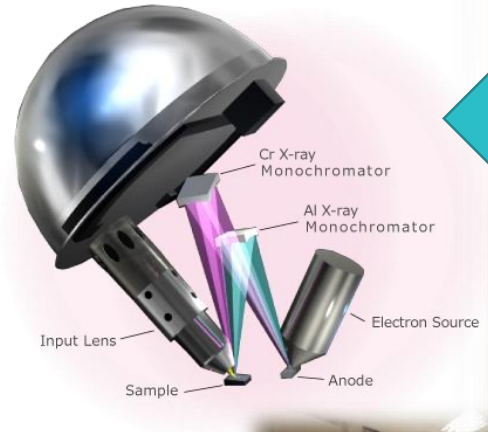


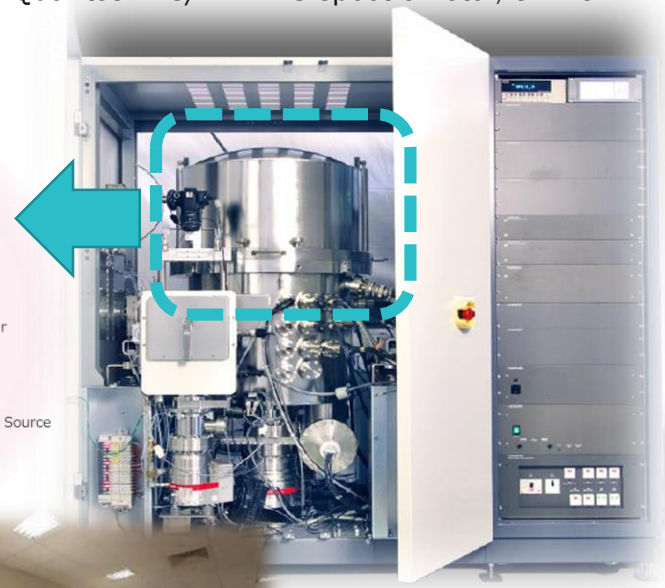
HAXPES with lab-scale Cr/Al sources

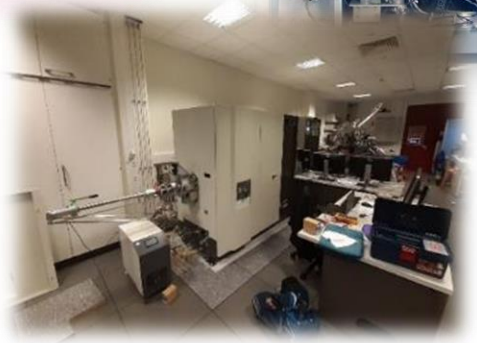
- Two confocal X-ray micro-sources
- **Device technology:** no sensitivity loss for Si-based systems



Quantes XPS/HAXPES spectrometer, ULVAC-PHI



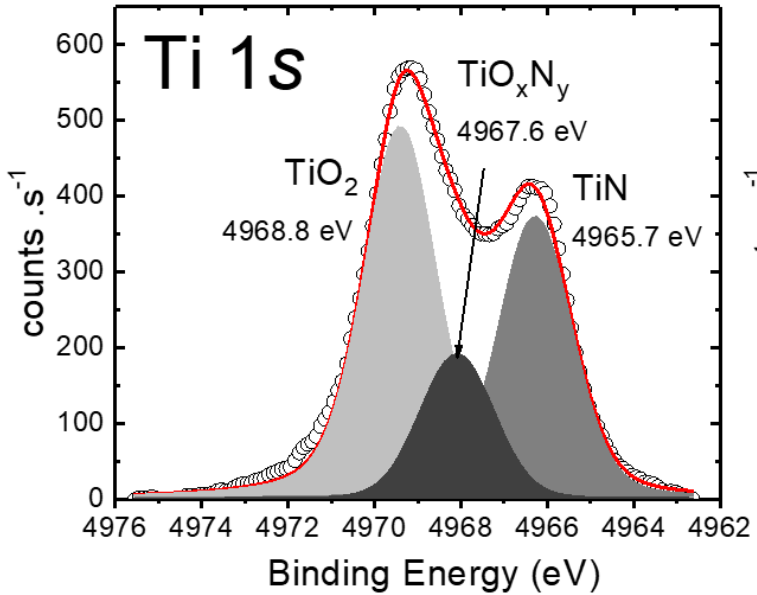


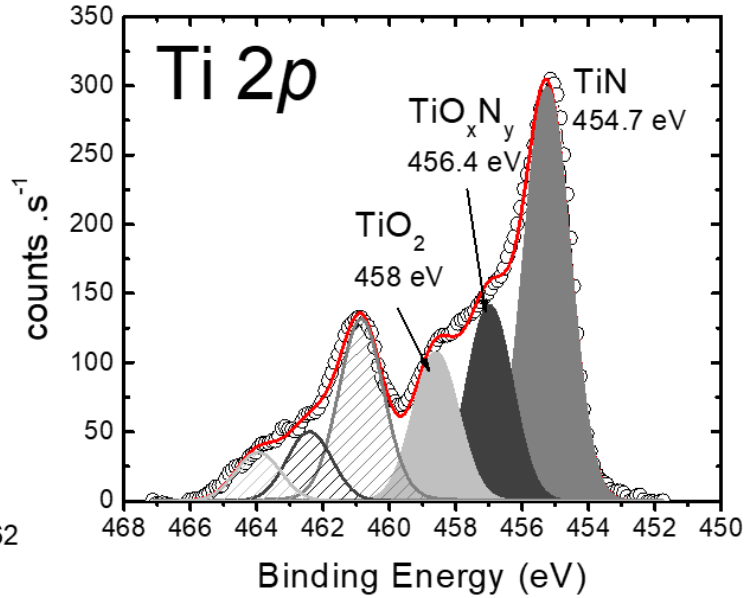


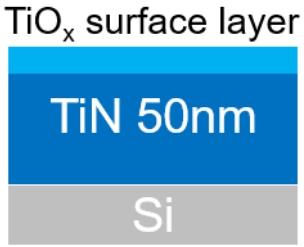
Application case 1: depth profiling

› Different core-level lines provide variable probing depth

Photoelectron	Ti 1s	Ti 2p
Kinetic Energy (KE), eV	449	4962
Material	Pt (TiN)	Pt (TiN)
Inelastic mean-free path, nm	0.77 (1.08)	4.50 (6.93)



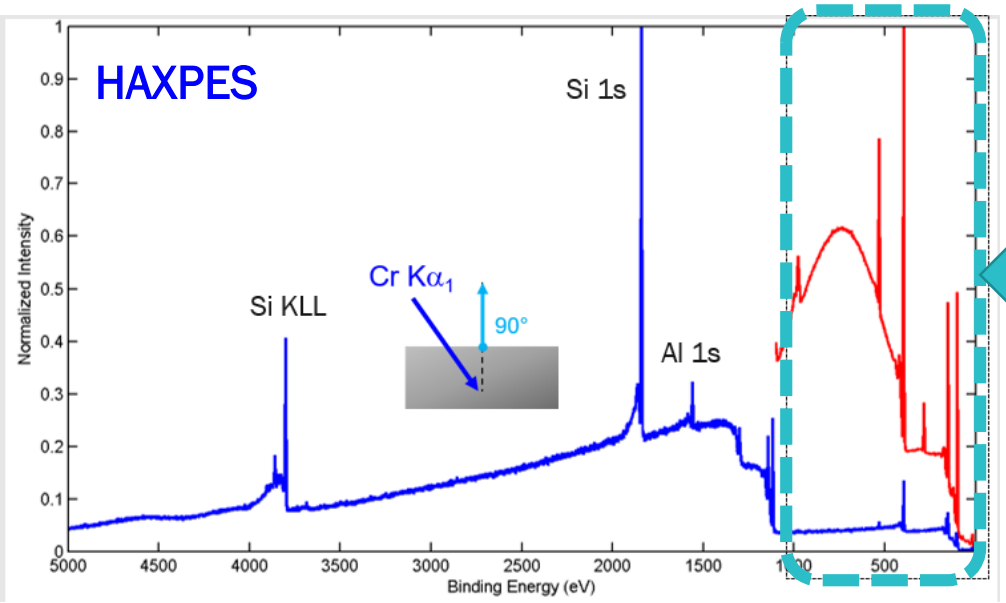


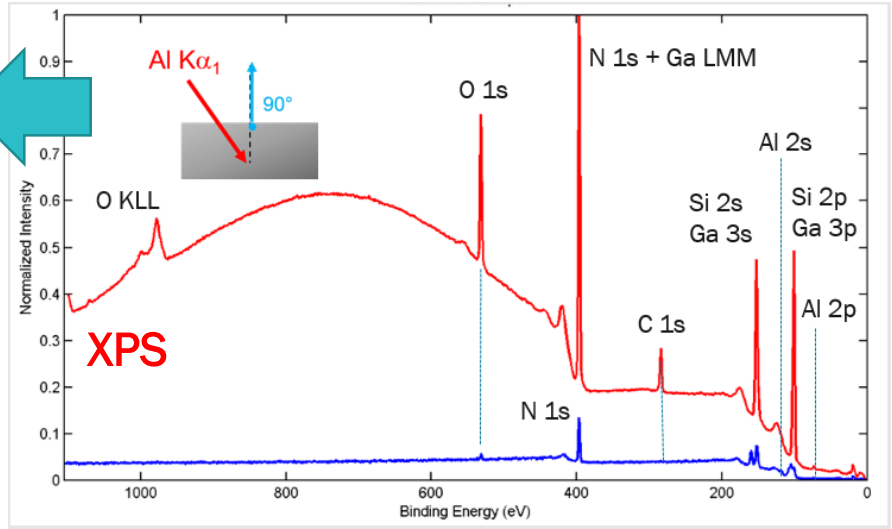


O. Renault, E. Martinez *et al.*, *Surf. Interface Anal.* **50**, 1158 (2018).

Application case 2: more accessible lines

- Core-line overlapping in XPS: case of stacks of III/ IV-nitrides
- HAXPES enables analyzing isolated, high-energy levels

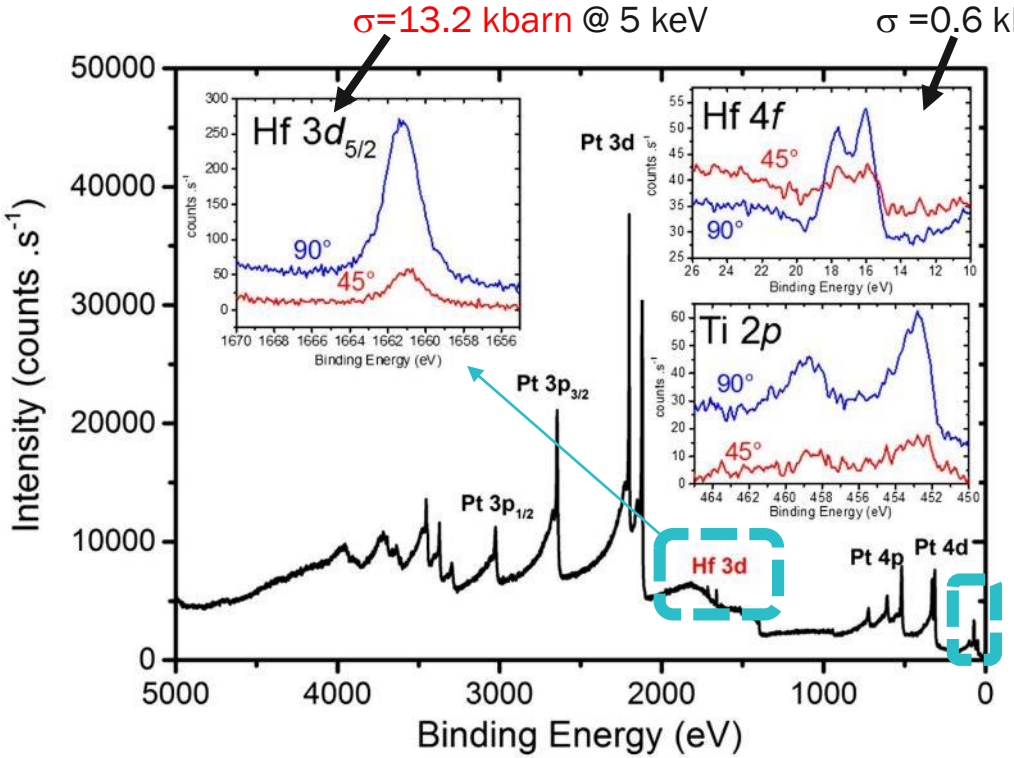


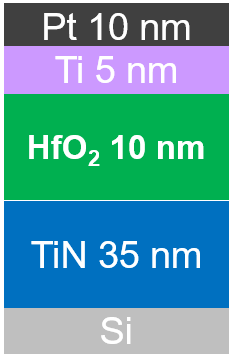


- O. Renault - Lab-scale HAXPES @PFNC

Application case 3: buried interfaces

- **Resistive memory devices:** access to critical oxide-electrode interface
- Available high energy core-line provides **enhanced sensitivity (ionization cross-section)**

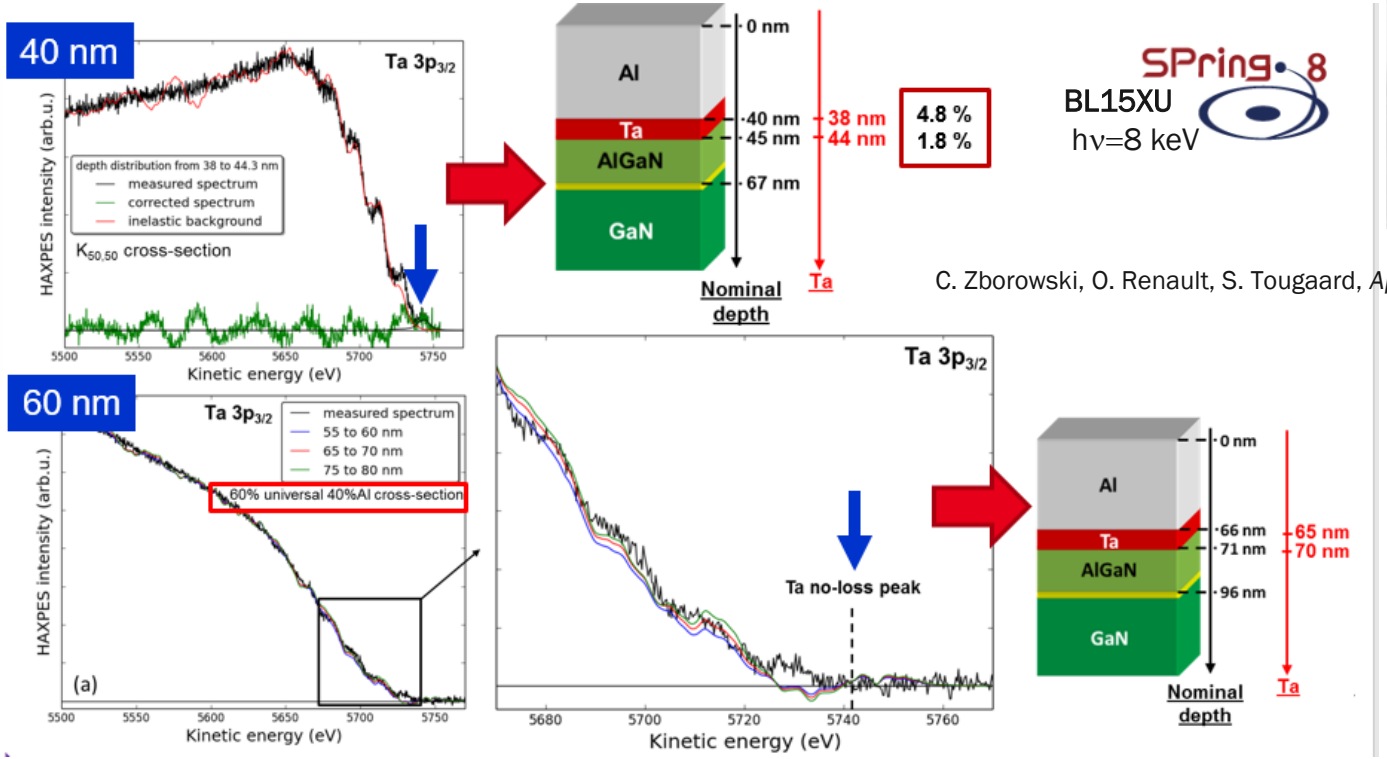




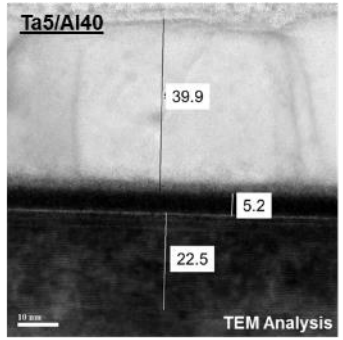
O. Renault, E. Martinez *et al.*, *Surf. Interface Anal.* **50**, 1158 (2018).

Application case 4: inelastic background analysis

- Probing depth: 3× mean free path for elastic peaks (core-levels)
  < 8× mean free path for energy losses (inelastic background)



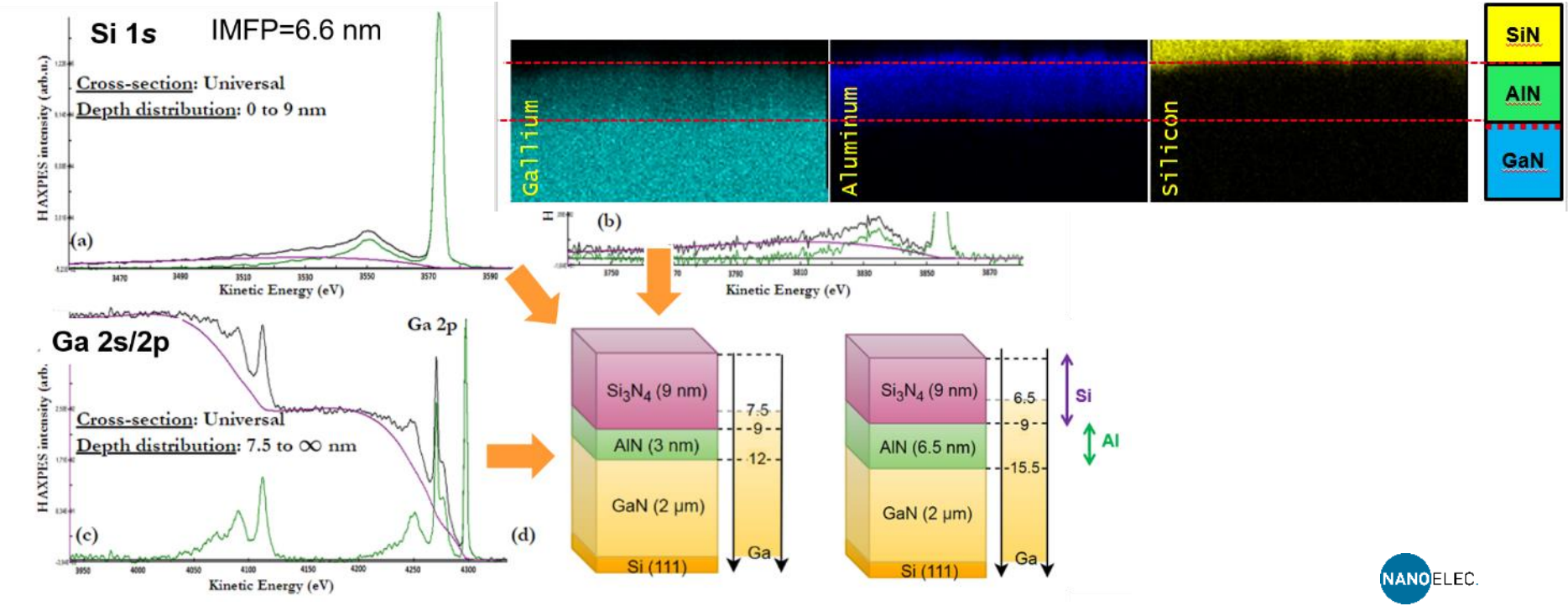
SPring-8
BL15XU
hν=8 keV



C. Zborowski, O. Renault, S. Tougaard, *Appl. Surf. Sci.* **432** 60 (2018)

Application case 4: inelastic background analysis

- Probing depth: < 8× mean free path for energy losses (inelastic background)
- Sub-surface diffusion phenomena accessible quantitatively



Conclusion

- Hard X-ray Photoelectron Spectroscopy is now getting in the lab
- It offers x3 probing depth (30 nm), up to 80 nm thanks to inelastic background analysis

- It is expected to become a standard tool for characterizing sub-surface phenomena in materials
- In device technology, buried interfaces are being analyzed non-destructively
- Synchrotron excitation will still remain useful for high-energy resolution needs

The results presented here were obtained at the Platform For NanoCharaterization, with the support of the « Recherche Technologique de Base » (RTB)program from french ministery of research.

Thank you for your attention

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