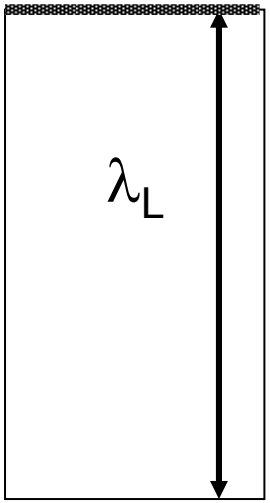
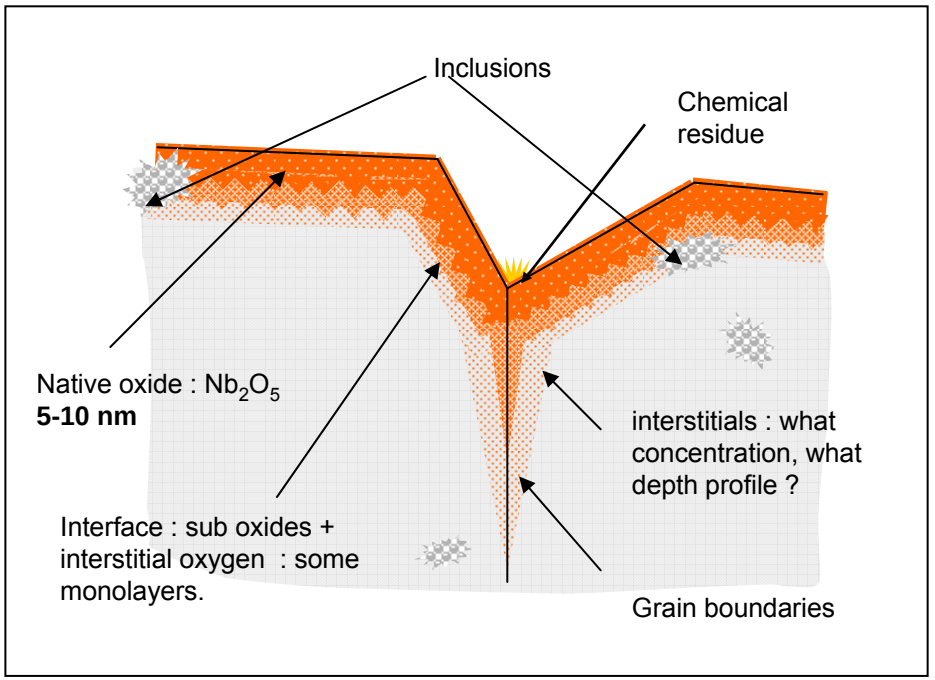
Moderated discussion : Surface Measurements.

λ_L for Nb :
~ 40-50 nm





Limits of RF measurement

- RF measurements @ 2K => ~ 50-100 nm,
- RF measurements @ 10K => ~1µm

But

- (baking) => nm scale modifications ?

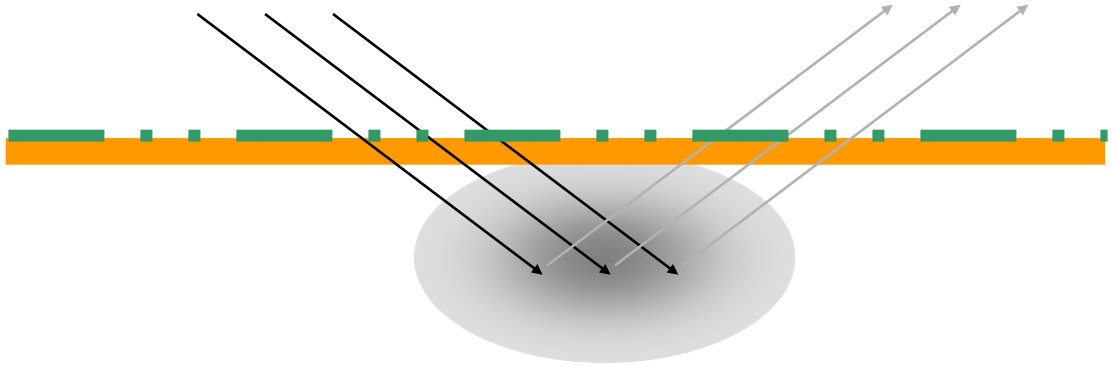
=> We cannot measure very local modification of SC parameters with cavities.

Topics.

- Classic basic tools:
  - e.g. : SIMS, XPS, AUGER, X-Ray techniques… Other profiling techniques
- Extension of these techniques to high level precision:
  - e.g. use of synchrotron sources, time of flight analysis ect…
- Surface morphology:
  - @ atomic resolution STM, AFM…
  - @ larger scale: profilometry, replicas…
- Dedicated techniques for RF Superconductivity :
  - magnetic measurements, RRR, I, field emission.
- Emerging techniques :
  - 3D Atom Probe Tomography
  - Δ measurement by photoemission

What surface technique ?

Need for sensitivity and depth resolution



- too superficial => STM, LEED, REED…
  - need to cross ~ 5 nm Nb_2O_5 , only indirect info on the SC matrix; STM/AFM = OK for morphology
- too “deep” => EDX, electron probe…
  - Explores ~1 µm depth
  - Only relative information
- roughness sensitive => X-Rays, reflectometry…
  - Work on monoXstal, special sample preparation

Limits of classic techniques(SIMS, XPS)

- 1rst golden rule : what you see is not (always) what's in your sample
  - e.g. XPS :
    - very sensitive to the detection angle => to compare spectra the sample holder must be @ same location!
    - Very sensitive to ion sputtering : creates suboxides
- 2nd golden rule (Murphy's law variant)

  (sensitivity x accuracy) goes like (time x money)$^{-1}$
  - e.g. SIMS
    - Standard : explores ~some µm, with ~100 nm depth resolution
    - Same set up, more accurate settings + UHV => some nm resol^{n}
    - TOF-SIMS : atomic ML resolution + indirect access to chemical info You can observe significant changes upon surface treatments BUT if you change location on the sample … changes too ! (≠ grain orientations ?)

  => Very useful for qualitative exploration, not trivial for quantitative

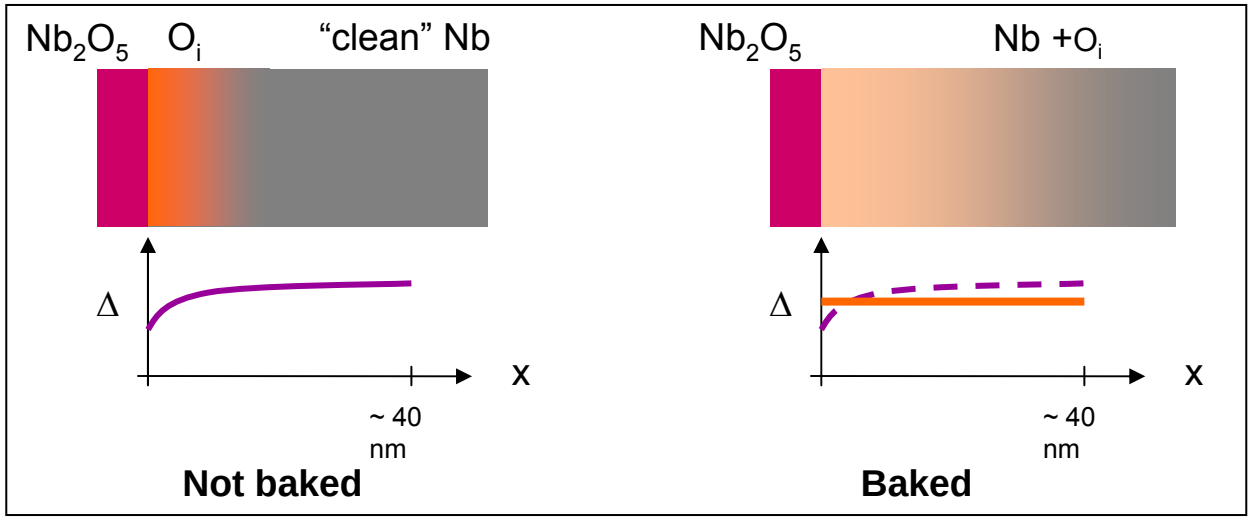
  (cf J. Kauffman, A. Wu/G. Rao)

Gain in sensitivity/resolution: «exotic» techniques

- 3D Atomic probe tomography (D.N Seidman)
- grazing X-Ray depth resolved techniques (M. Delheusy)
- Δ measurement by photoemission (myself)

How can Oi influence superconductivity ?

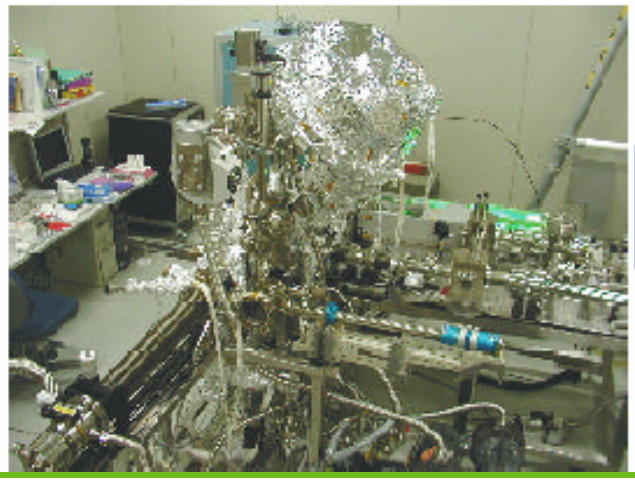
- [O_i] might affect very locally the superconducting gap Δ (d_{char} ~ 1nm)

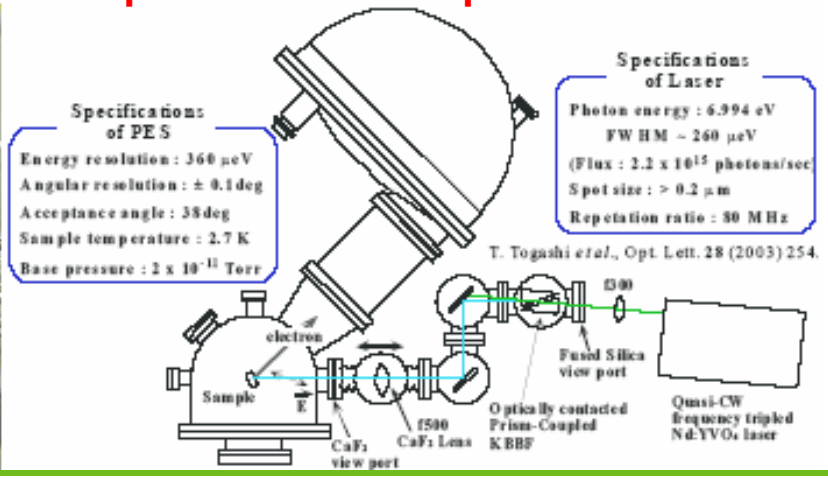


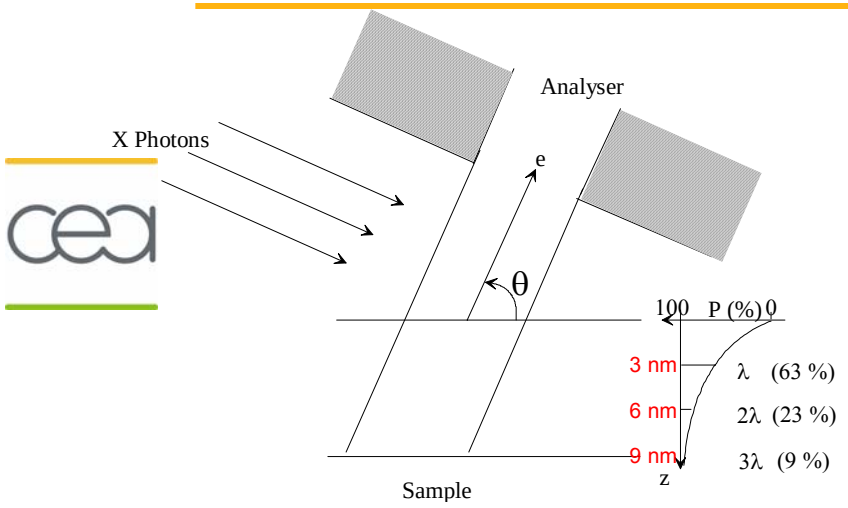
$Rs \sim e^{-\Delta/T}$

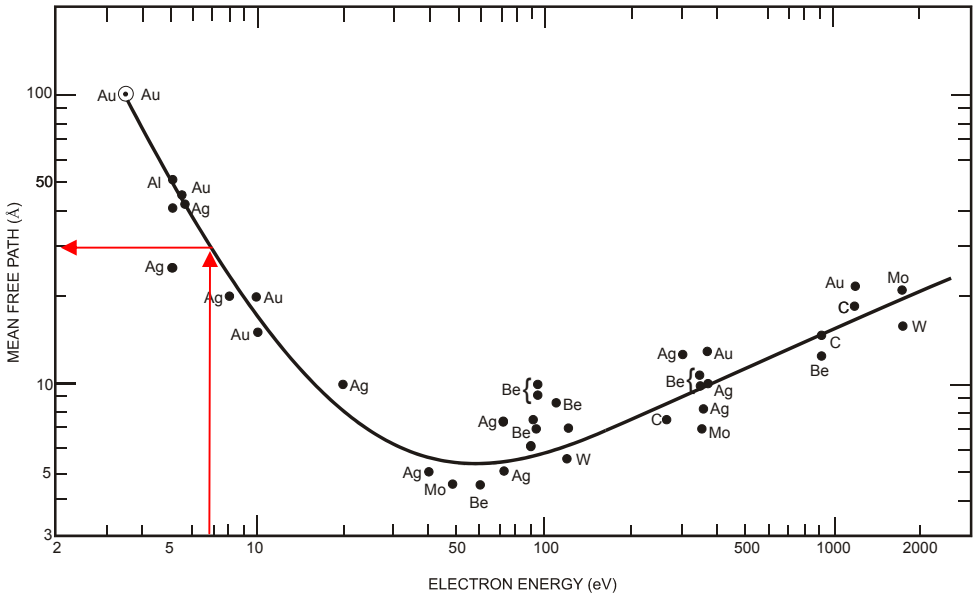
- Need for a nm sensitive probe !

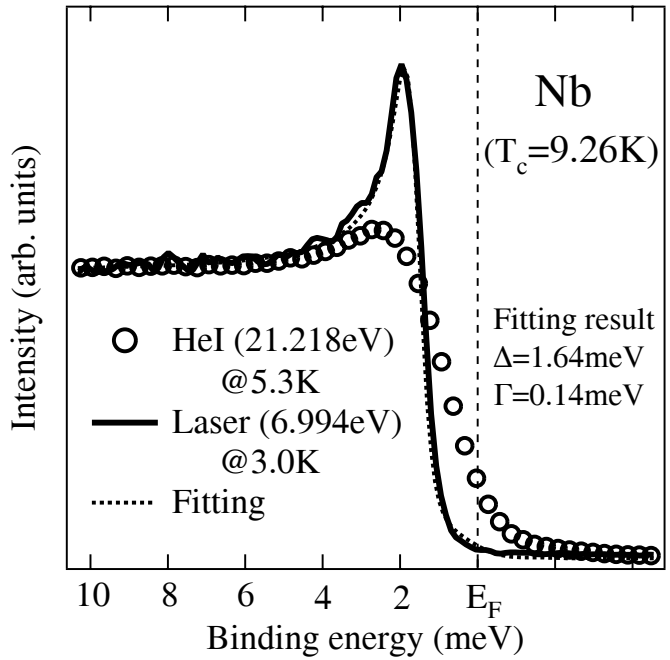
=> Ultrahigh resolution laser photoemission spectrometer





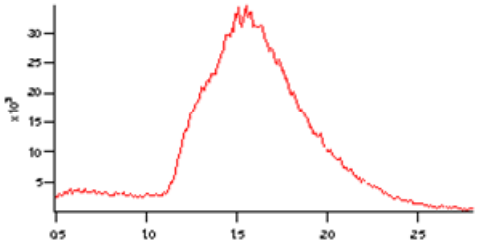






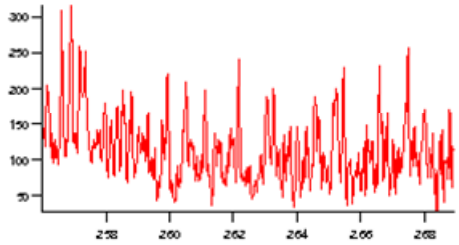
Angle resolved method + in situ baking => profiling should be possible with nm resolution !

As received



EPV.eps

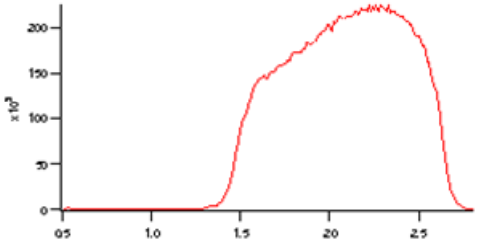
EPV.eps Valence structure of electron polished sample without preperation at 5K.



EPEF.eps

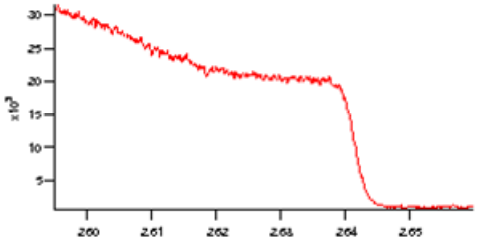
EPEF.eps Spectrum near EF of electron polished sample without preperation at 5K.

After Ar Sputtering



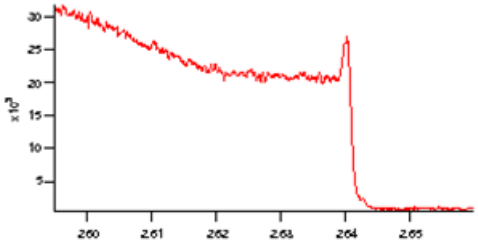
EPVsp.eps

EPVsp.eps Valence structure of electron polished sample after Ar sputtering at 5K.



EPEFsp10K.eps

EPEFsp10K.eps Spectrum near EF of electron polished sample after Ar sputtering at 10K.



EPEFsp5K.eps

EPEFsp5K.eps Spectrum near EF of electron polished sample after Ar sputtering at 5K.

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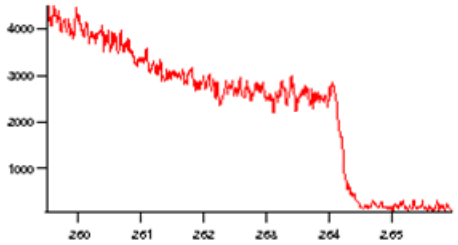
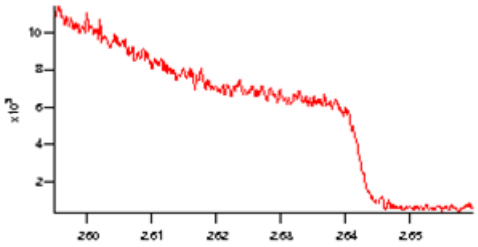
After Ar Sputtering+ annealing



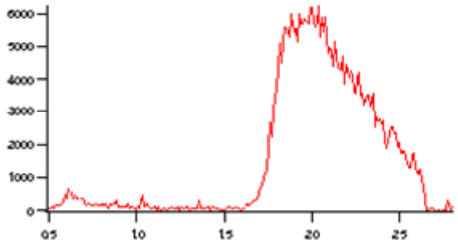
EPspan5K.eps

EPEFspan5K.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 5K.



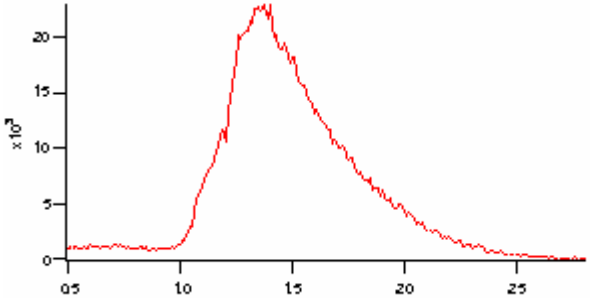
EPspan10K.eps

EPEFspan10K.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 5K.

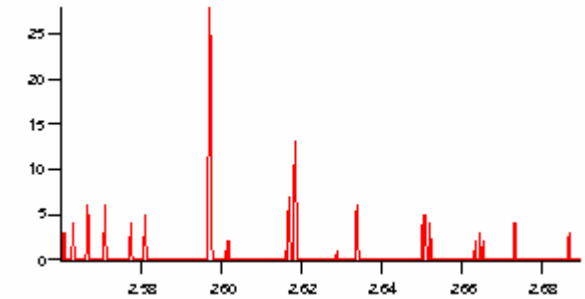


EPVspan.eps

EPVspan.eps Valence structure of electron polished sample after Ar sputtering and 2h anneal at 5K.



CPV.eps Valence structure of chemical polished sample without preperation at 5K.



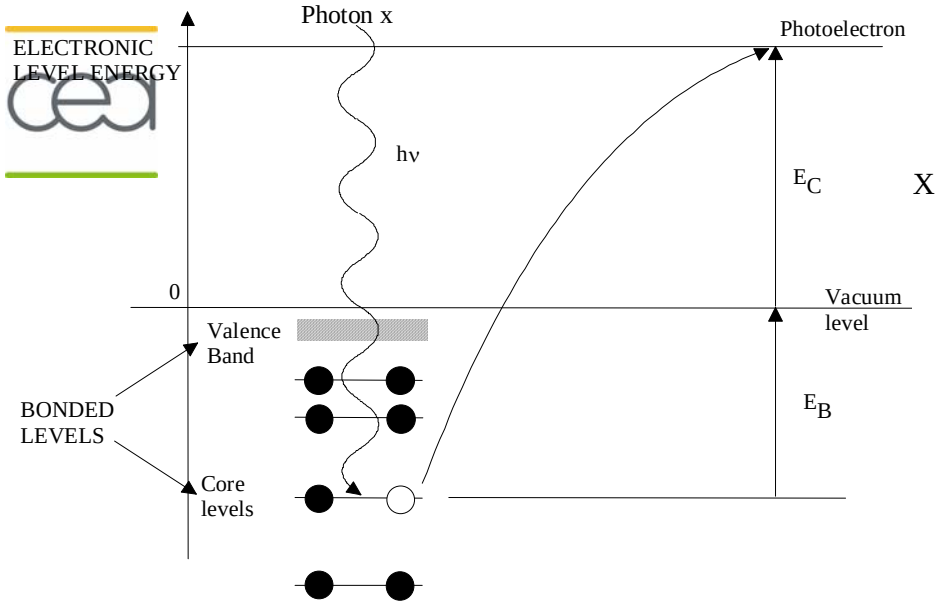
CPEF.eps Spectrum near EF of chemical polished sample without preperation at 5K.

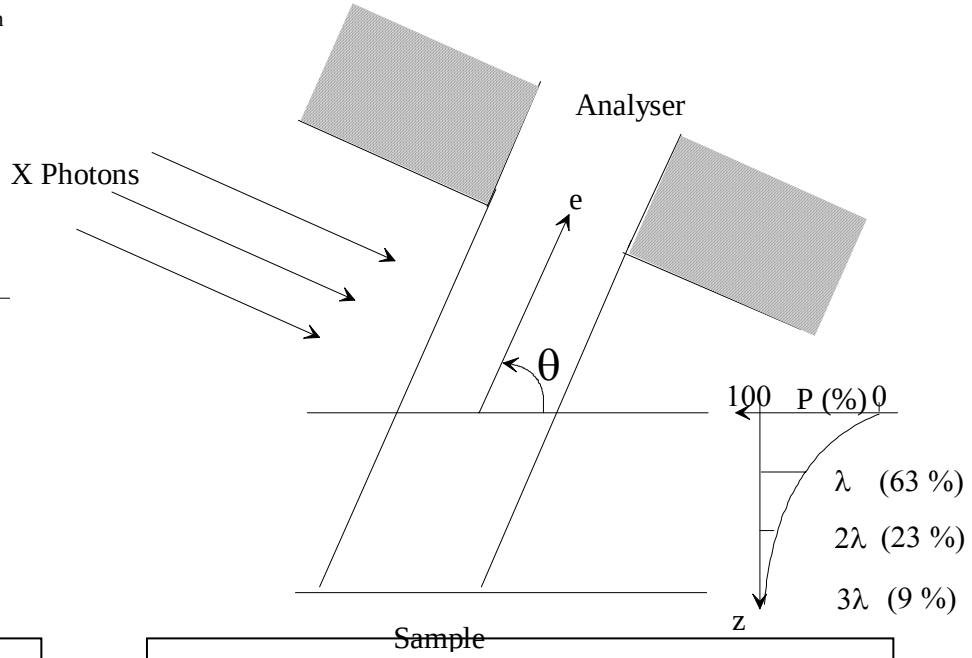
Profiling techniques

- Ion sputtering : troubles with preferential O sputtering…
  - Reduces depth sensitivity to ~ >10 nm
  - Only relative information, no way to tell oxide/Oi
- Thus : be careful with profiling (XPS, Auger…. and SIMS in standard conditions)
- Profiling with depth sensitivity :
  - TOF-SIMS (but tricky to tell oxides / O_i)
  - Angle-resolved techniques
- Angle resolved photoemission (XPS/ESCA)
  - Chemical sensitivity
  - Profiling
  - But ….

Photoemission (≡ ESCA ≡ XPS) / Auger

A interesting way to get info from depth under oxide





$E_B = h\nu - E_C$

E_B(bonding energy) is characteristic from one element ; and is influenced by electronegativity of bonded neighbors ⇒ chemical environment information.

- λ~ 0.5-2nm (Auger)
- λ~ 5nm (XPS)

But :

- Not very sensitive (0.5 At%)
- Deconvolution = very « tricky »

For $[x_i] < 10\%$, ∃ deconvolution signal ≠ ∃ physical cpd !!!