PHI VersaProbe II Scanning XPS Microprobe

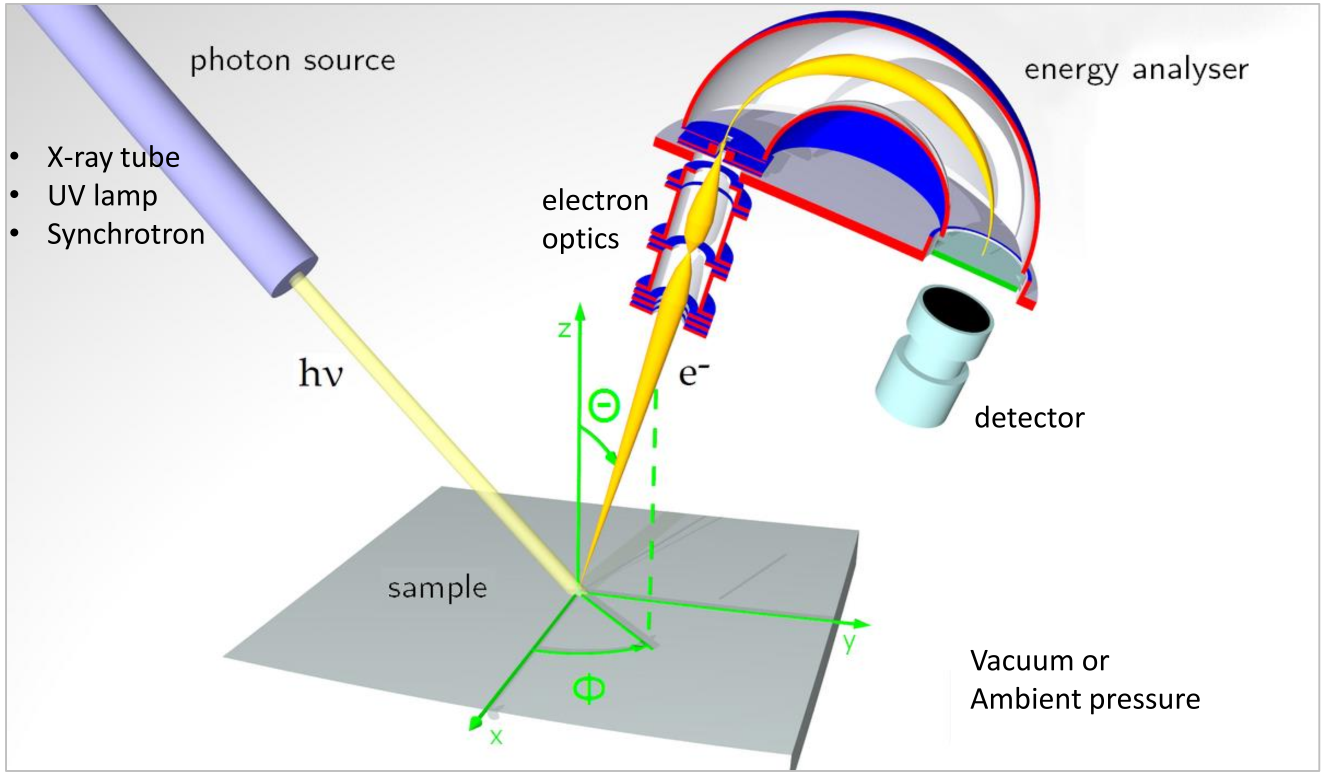
Core Policies

- **DO NOT** let other people use the facility under your account.
- **DO NOT** try to fix parts or software issues by yourself!
- **DO NOT** surf web using instrument computer!
- **Follow** checklist and SOP! **DO NOT** explore program!

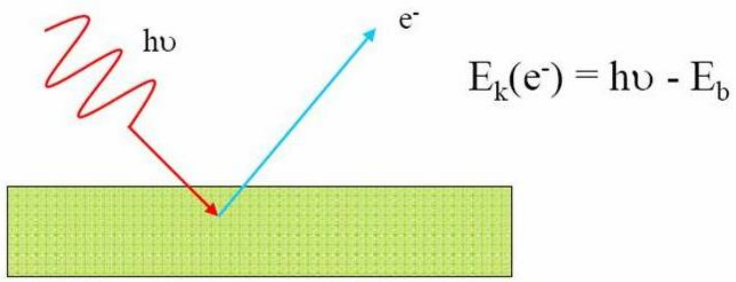
- **Facility usage time** at least twice a month, **OR** receive training again (two practice sessions within one week).
- **No trainings** on monthly users

What is XPS? X-ray Photoelectron Spectroscopy

- Photoelectric effect
- A spectroscopy that records the counts of X-ray induced secondary electrons - photoelectrons as the function of binding energy



- A technique based on photoelectric effect:



$E_k(e^-) = h\upsilon - E_b$

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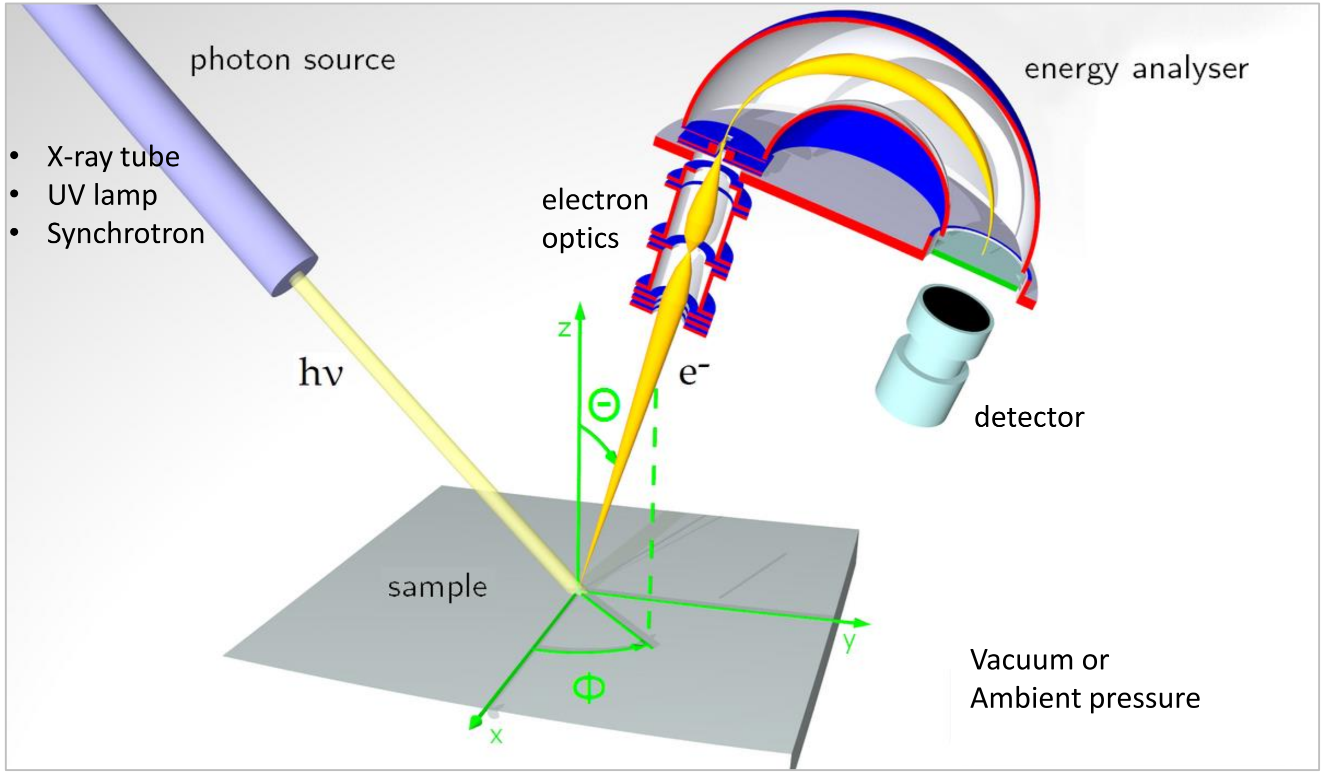

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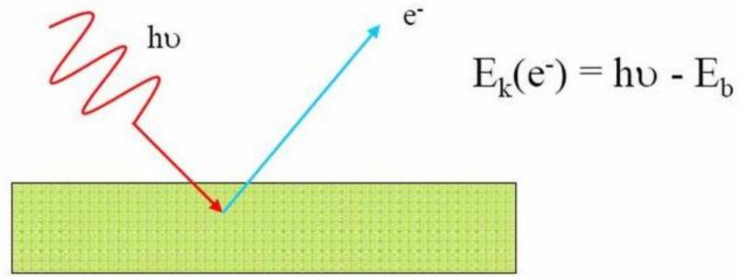


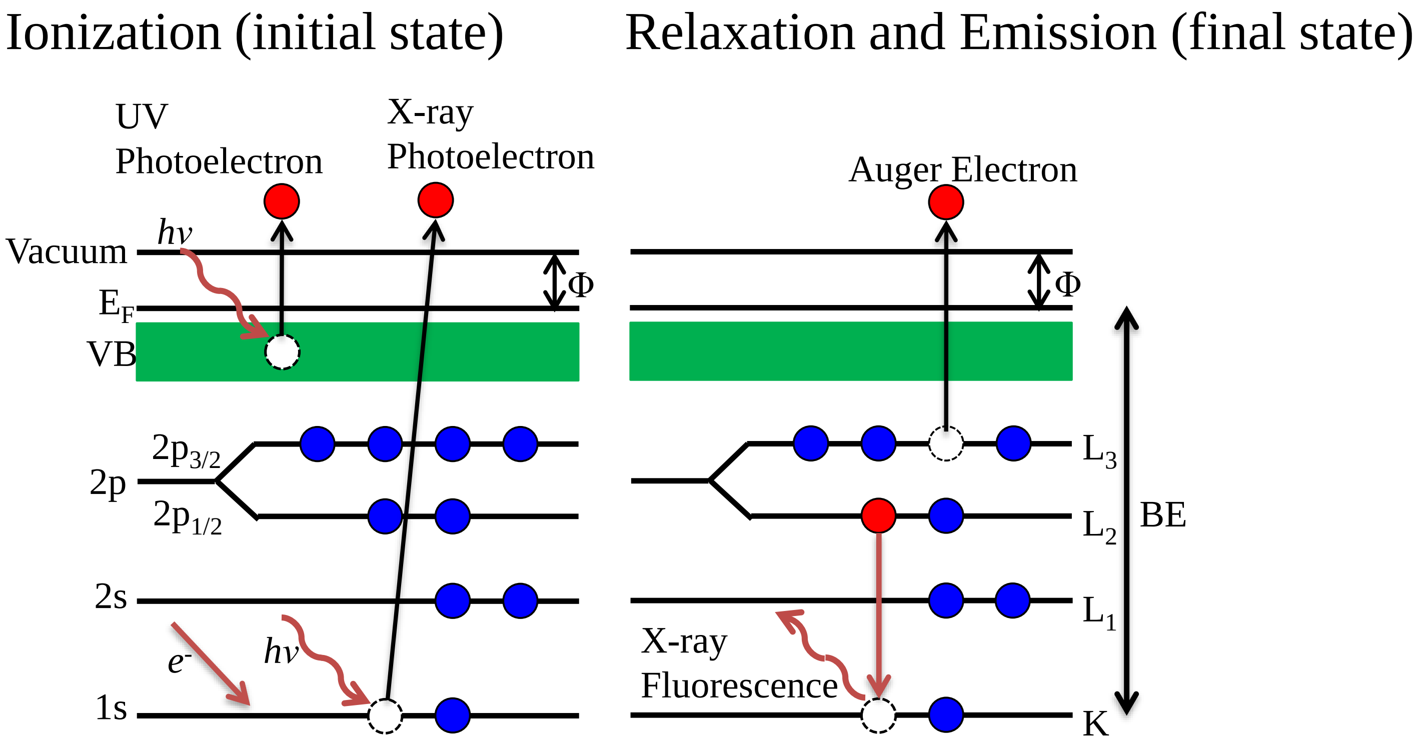
$$E_k(e^-) = h\upsilon - E_b$$

What kinds of samples for XPS?

- Vacuum compatible: low vapor pressure under 10^{-8} Pascal
- Conductive or insulating

How XPS works?

- XPS detects the number of photoelectrons at different kinetic energies (KE)
- The photoelectron binding energy can then be calculated, characteristic of elements within the sample volume



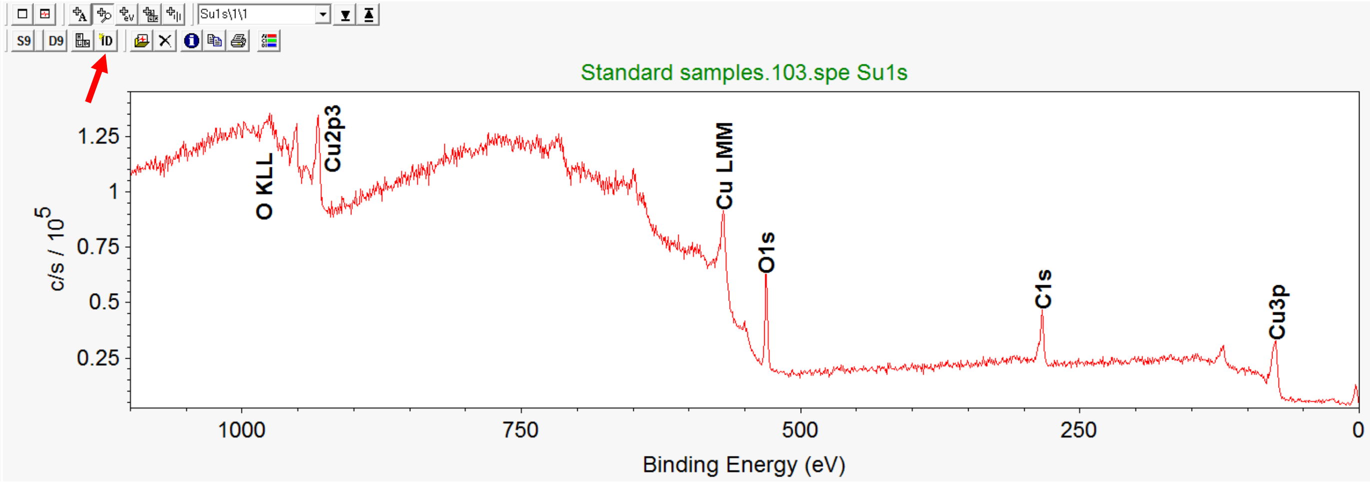
KE (measured) $= h\nu - BE - \Phi_{spec}$

$BE = h\nu$ - KE - Φ_{spec}

$KE\ (KLL) = BE(K) - BE(L_2) - BE(L_3)$

XPS Main Features

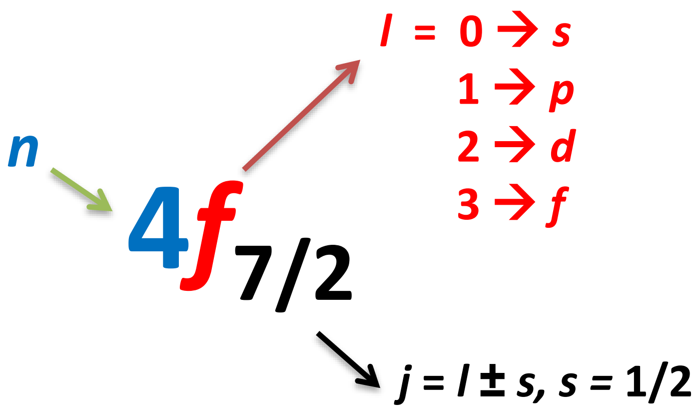
- Core level splitting
- Auger peaks
- Stepped background ← inelastic secondary electrons



KE →

← ***BE***

XPS Peak Notation



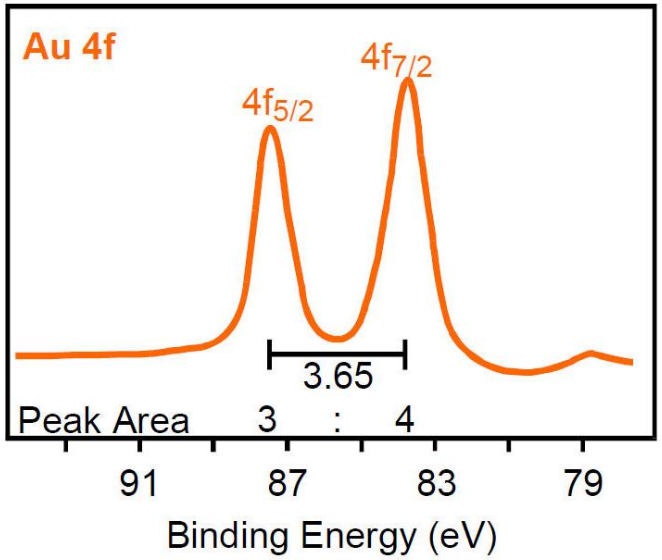
n

$4f_{7/2}$

$l = 0 \rightarrow s$
$1 \rightarrow p$
$2 \rightarrow d$
$3 \rightarrow f$

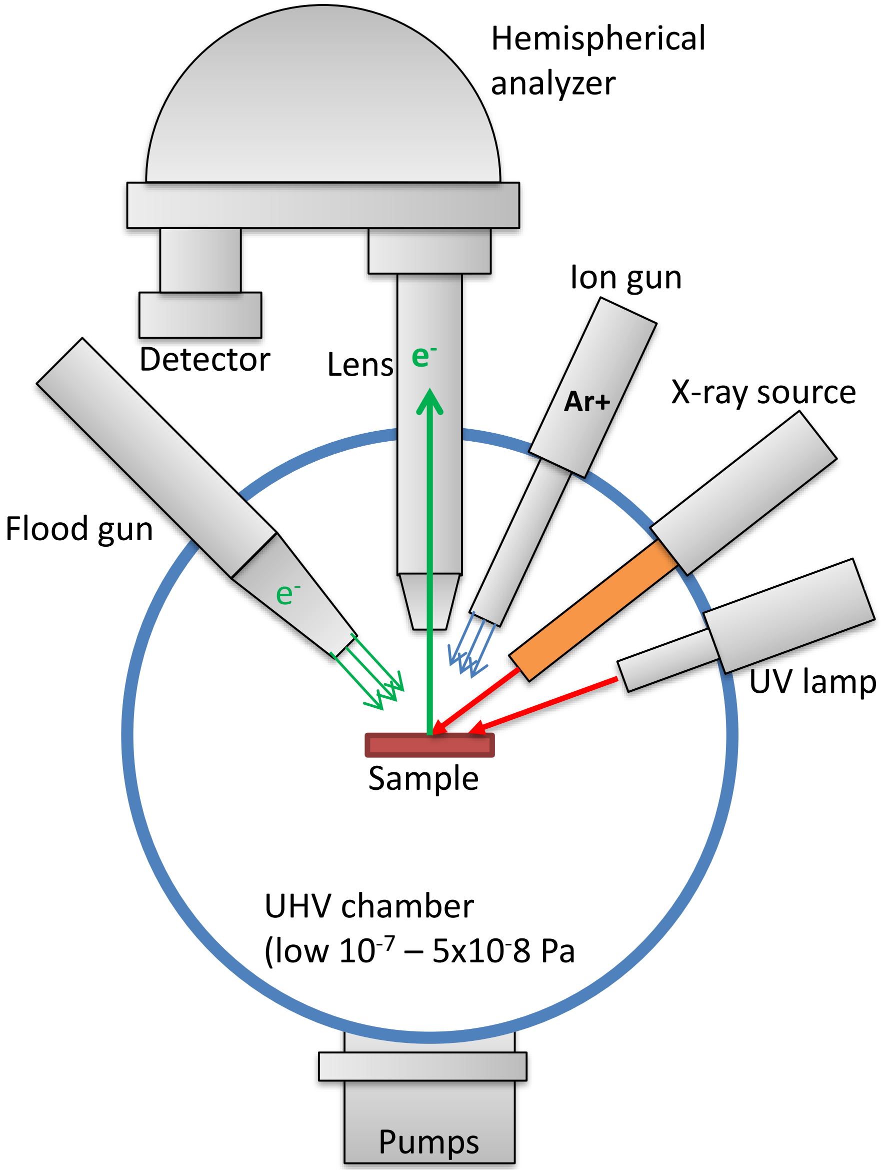
$j = l \pm s,\ s = 1/2$

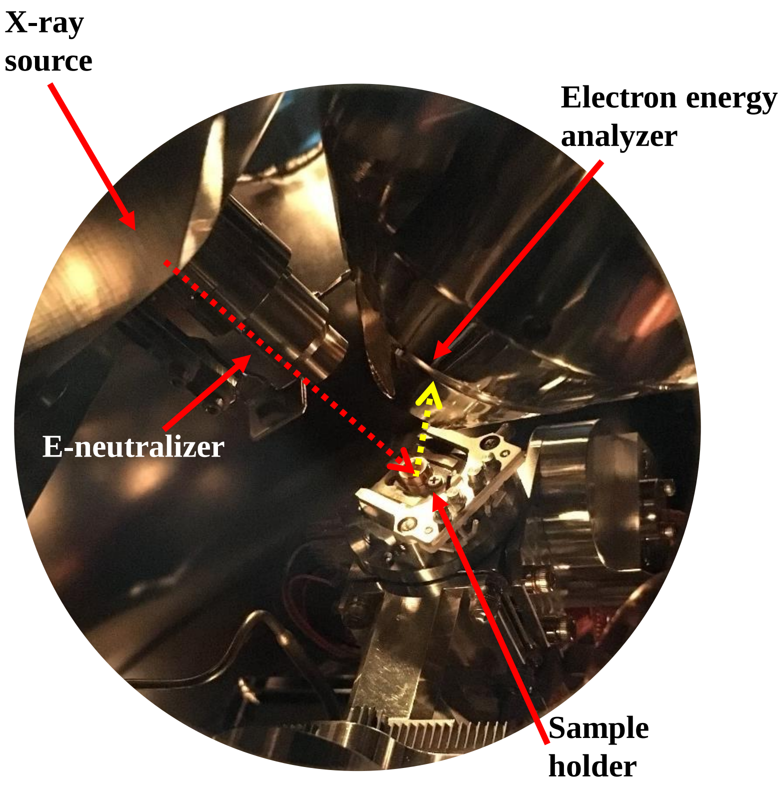
Spin-orbital splitting with *l* > 0



Orbital	*l*	*j*	Degeneracy (2*j* + 1)	Peak area ratio	Electron level
s	0	1/2	1	-	1*s*
p	1	1/2, 3/2	2, 4	1 : 2	$2p_{1/2}$, $2p_{3/2}$
d	2	3/2, 5/2	4, 6	2 : 3	$3d_{3/2}$, $3d_{5/2}$
f	3	5/2, 7/2	6, 8	3 : 4	$4f_{5/2}$, $4f_{7/2}$

XPS Instrumentation





PHI *VersaProbe* II XPS

UHV system (< 10^{-8} Torr)
- Surface clean
- Longer photoelectron path length

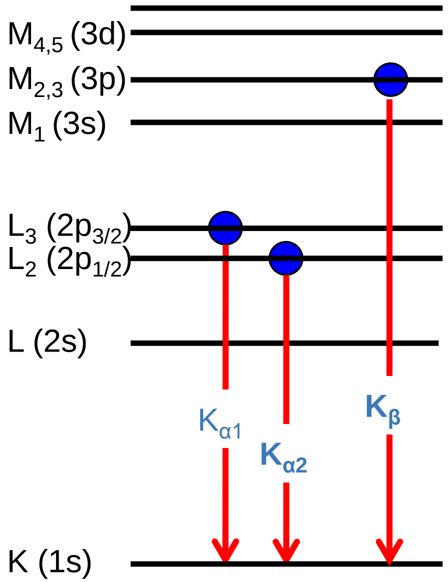
Electron analyzer
- Lens to collect photoelectrons
- Analyzer to filter electron energies
- Detector to count electrons

X-ray source
- Al Kα 1486.6 eV; Mg Kα 1256.6 eV
- Monochromated using quartz crystal

Low-energy electron flood gun
- Insulating samples

Ion gun
- Sample cleaning
- Depth profiling
- For polymers, cluster ion sources may be required

X-ray Dual Anode Source



$M_{4,5}$ (3d)

$M_{2,3}$ (3p)

M_1 (3s)

L_3 ($2p_{3/2}$)

L_2 ($2p_{1/2}$)

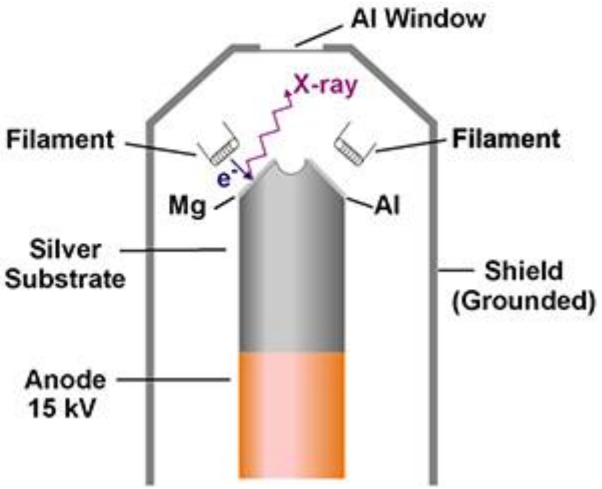
L (2s)

K (1s)

$K_{\alpha1}$

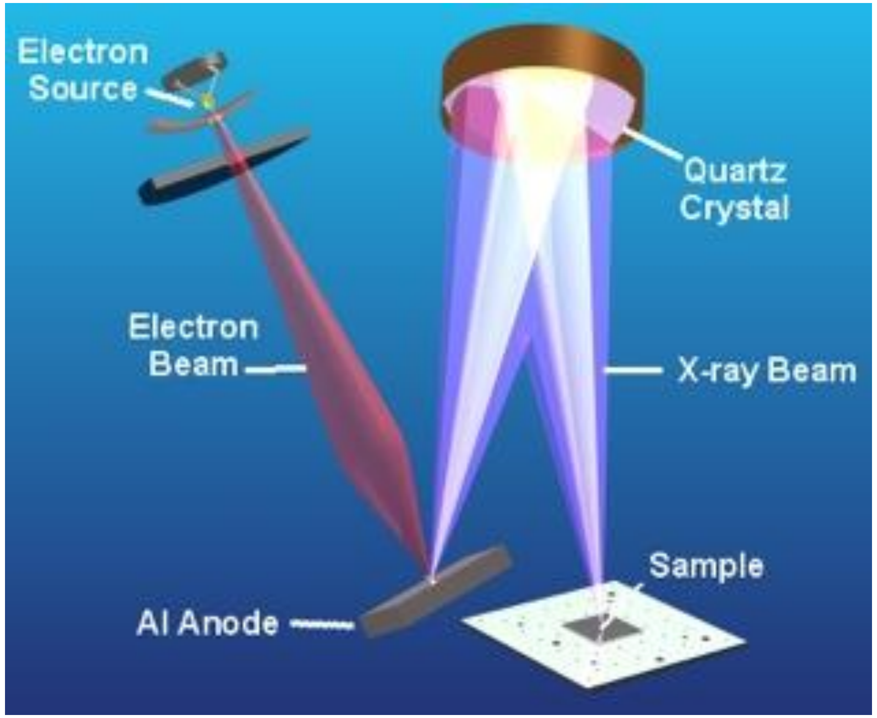
$K_{\alpha2}$

K_{β}



X-ray lines	Line Energy (eV)	Width (eV)
Mg $K_{\alpha1,2}$	1253.6	0.70
Al $K_{\alpha1,2}$	1486.6	0.85

X-ray monochromator



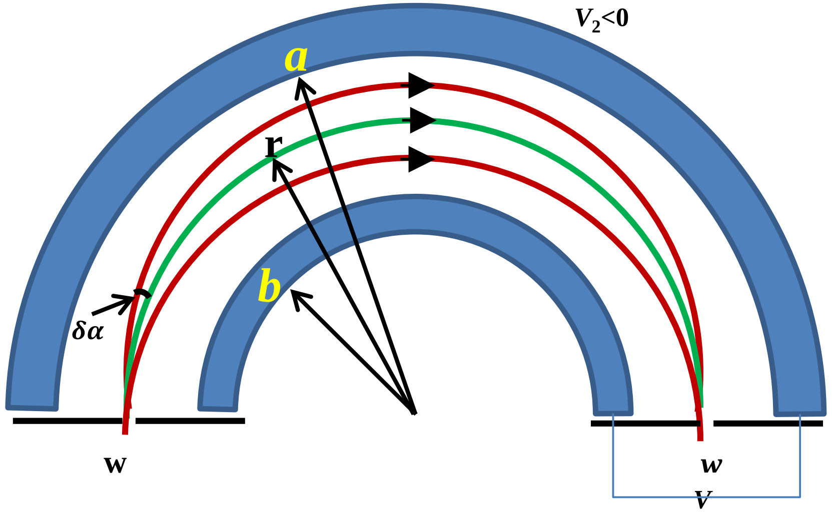
- Narrow peak width
- Reduced background
- No satellite & Ghost peaks

$$\boxed{n\lambda = 2d\sin\theta}$$

For quartz $(10\bar{1}0)$ surface:

n = diffraction order
d = 0.42 nm (lattice constant)
$\theta = 78.5^{o}$
λ = 0.83 nm for Al Kα

Spherical Capacitor Analyzer (SCA)



Pass energy:

$$E_0 = eV_0 = \frac{V}{\left(\frac{b}{a} - \frac{a}{b}\right)}$$

Analyzer Resolution:

$$\Delta E = E_0\left(\frac{w}{a+b} + \frac{\delta\alpha^2}{4}\right)$$

Where the mean radius $r = \frac{a+b}{2}$

V_0: the median equipotential surface of radius r

V: the potential applied between inner (radius b) and outer (radios a) shells

w: entrance and exit slit widths

$\delta\alpha$**:** angular deviation of the electron trajectories at the entrance with respect to the center line

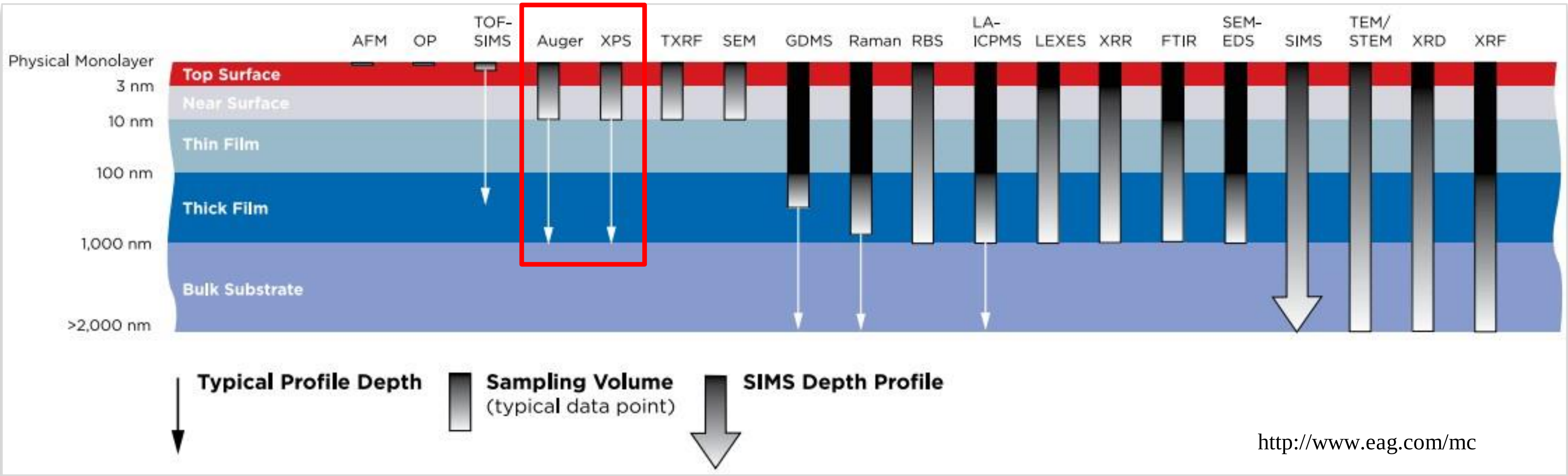
For the PHI SCA : $E_0 = 0.56V$ and $\Delta E = 0.015E_0$

Typical $E_0 = 100$ eV → $\Delta E = 1.5$ eV

Why are we interested in XPS?

- Surface sensitive technique
- Chemical shift detection → XPS is also named as Electron Spectroscopy for Chemical Analysis (ESCA)

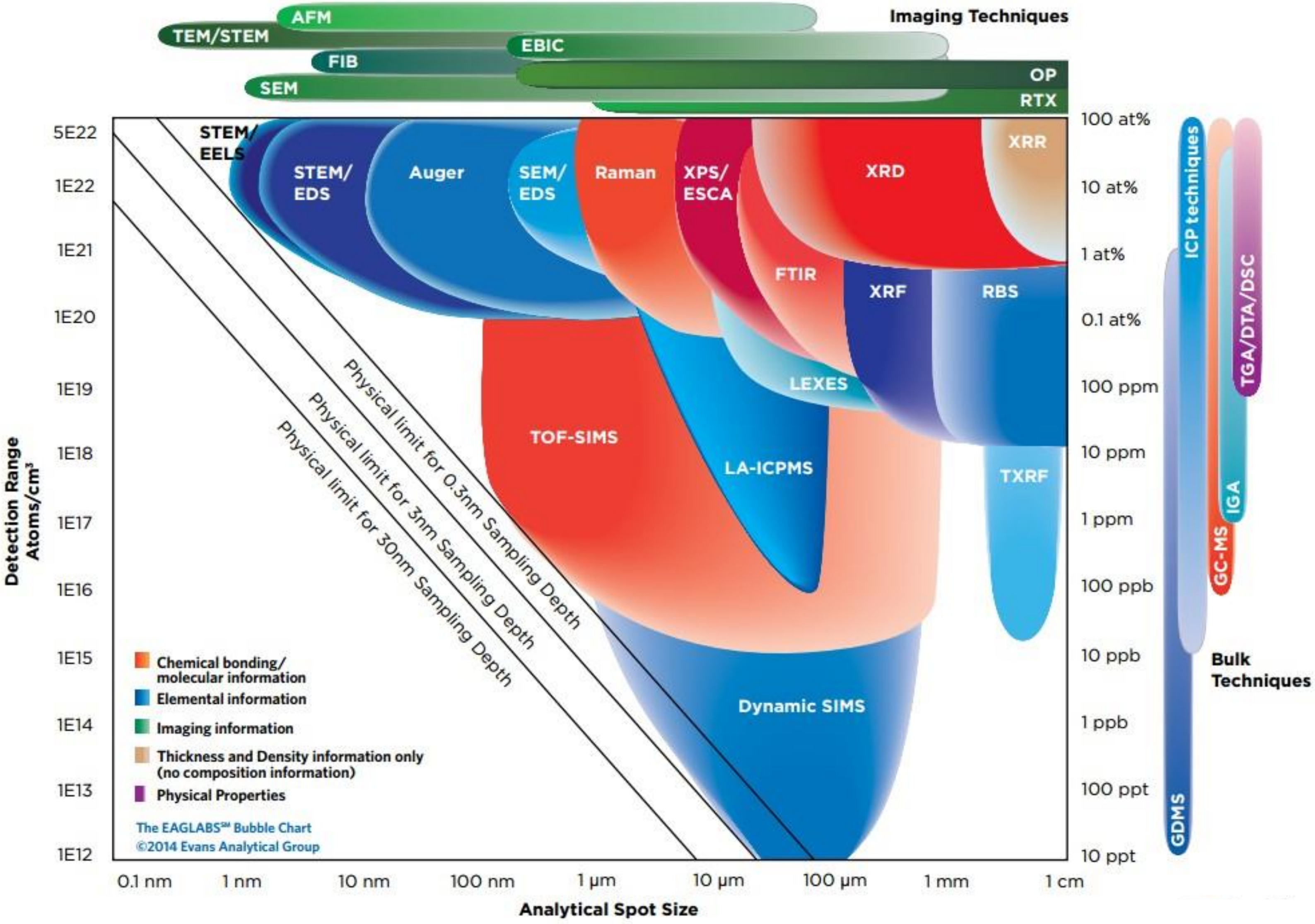
Typical Analysis Depths for Techniques



http://www.eag.com/mc

XPS detects electron signals in the near surface region (0 ~ 10 nm)

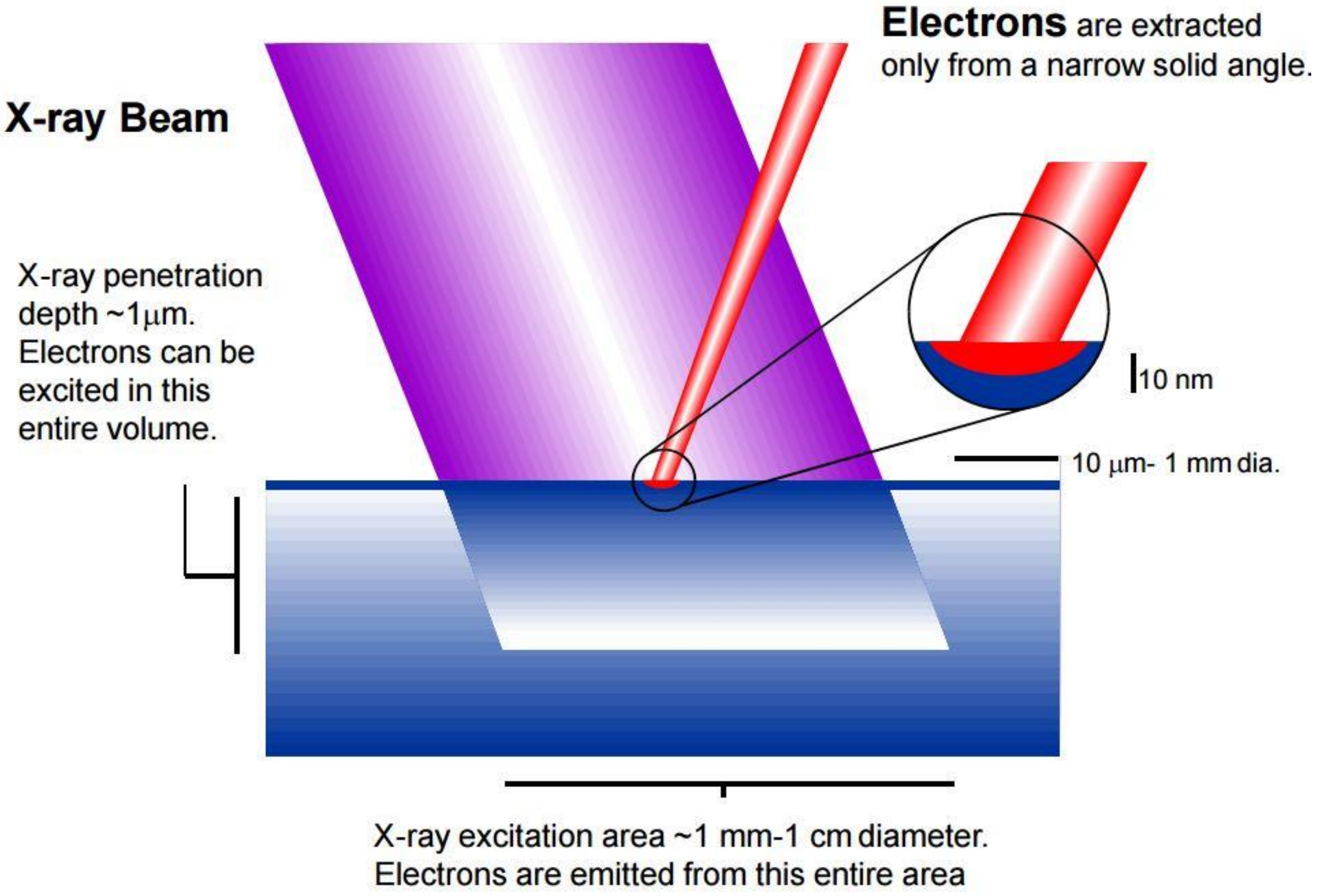
Analytical Resolution vs. Detection Limit



- XPS resolution can be reached below 10 µm
- XPS detection limits: ppt range

Why XPS is Surface Sensitive?

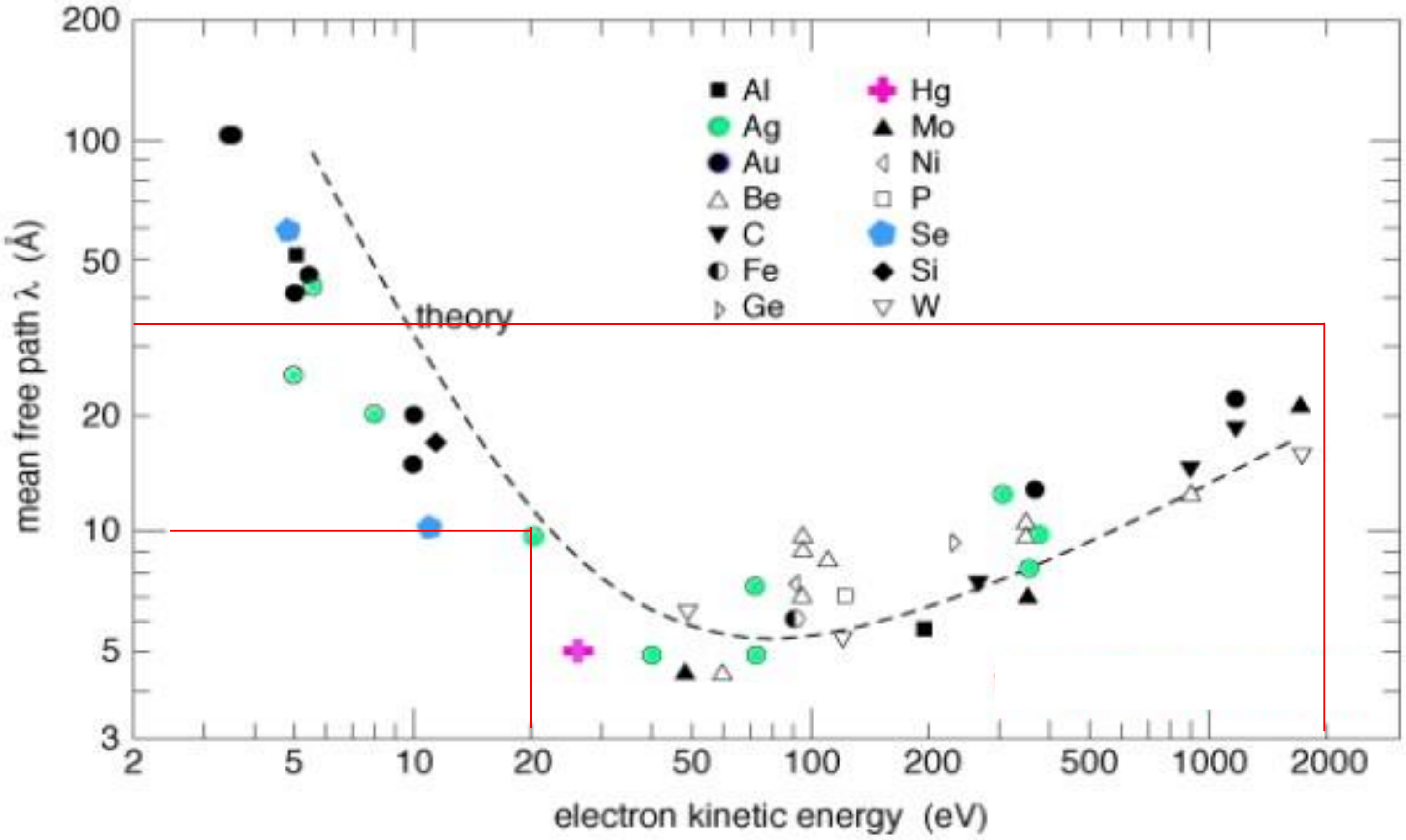
- Inelastic scattering of photoelectrons



Electron Inelastic Mean Free Path (IMFP)

- The average distance an electron travels through a solid before losing energy through inelastic collisions.

“**Universal Curve**” - λ (IMFP) *vs* kinetic



λ = 1 ~ 3.5 nm for X-ray photoelectrons