X-Photoelectron Spectroscopy XPS

SURFACE ANALYSIS

MSE-351

Anna Igual Munoz

1.- Introduction XPS vs other techniques

2.-Instrumentation

3.-Spectral interpretation

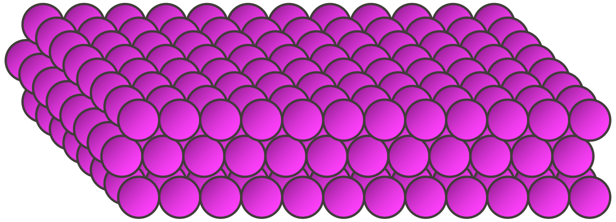
4.-Quantification

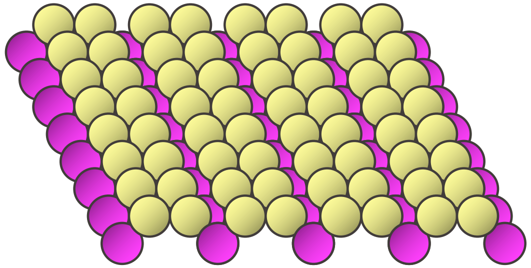
5.-Depth profiling

6.-Applications

Surface properties to be measured

- **Chemical composition**, chemical bonds?
- Surface morphology, symmetries, patterns, .. Smooth, rough, periodic, crystalline, amorphous…?
- Thermodynamic, electrical, magnetic properties?





Surface analysis techniques

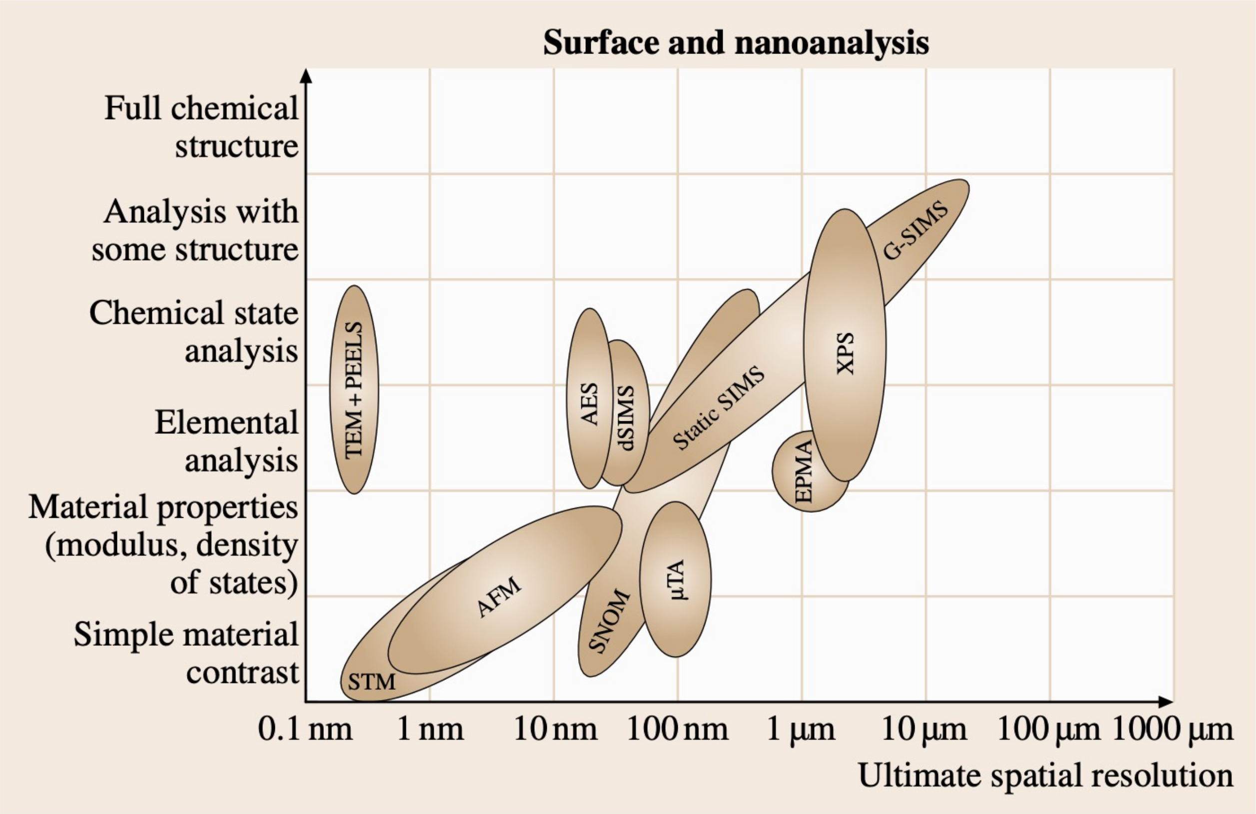
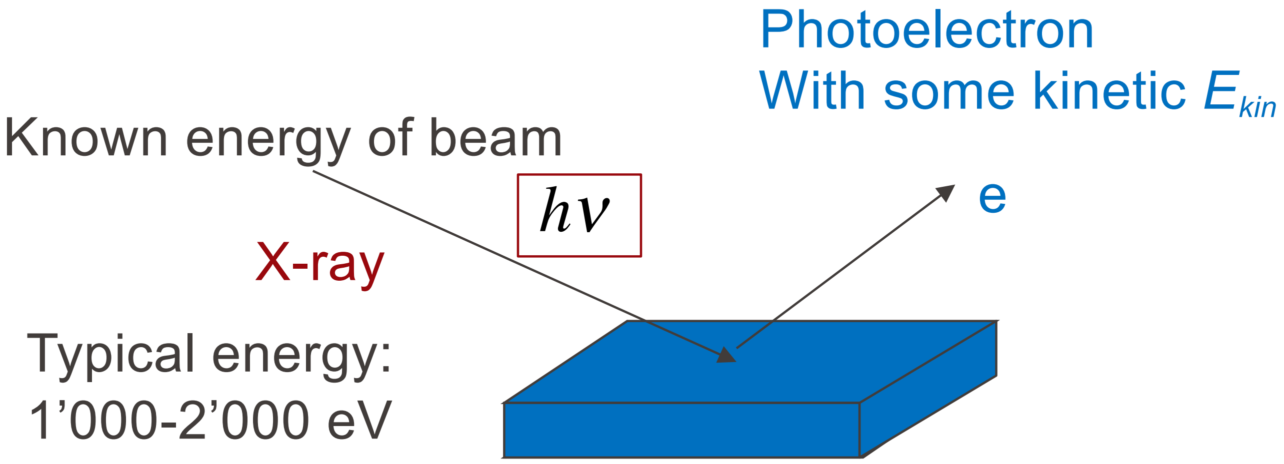


Fig. 6.1 The resolution and information content of a range of analytical methods (STM = scanning tunneling microscopy, AFM = atomic force microscopy, TEM = transmission electron microscopy, PEELS = parallel electron energy loss spectroscopy, SNOM = scanning near-field optical microscopy, µTA = microthermal analysis, EPMA = electron probe microscopy) that can be used at surfaces (after *Gilmore* et al. [6.1])

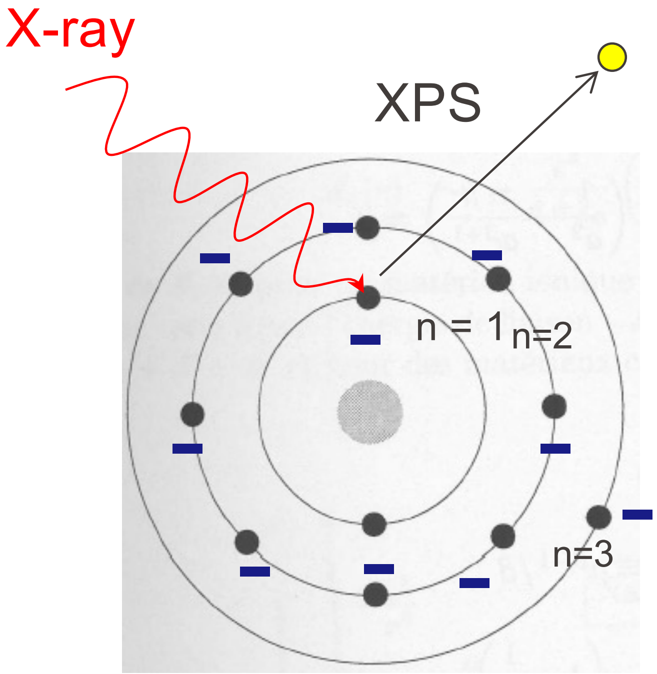
Which method should one use?

- What can be measured?
  - Presence of an element, qualitative or quantitative analysis, binding state, isotopes...
- Sensitivity
  - Detection limit
- Lateral and depth resolution
  - Spatial distribution, layer thickness
- Cost/means
- Sample preparation
- Time needed for a measurement

Principle of XPS



Typical energy:
1’000-2’000 eV



Analysis consists first in measuring the intensity of photoelectron emission as a function of their kinetic energy E_{kin} that can be converted into electron binding energy E_b by known X-Ray beam energy *hv:* $\mathbf{E_b = h\nu - E_{kin} - \Phi}$

Φ is the work function of the spectrometer

Binding energy

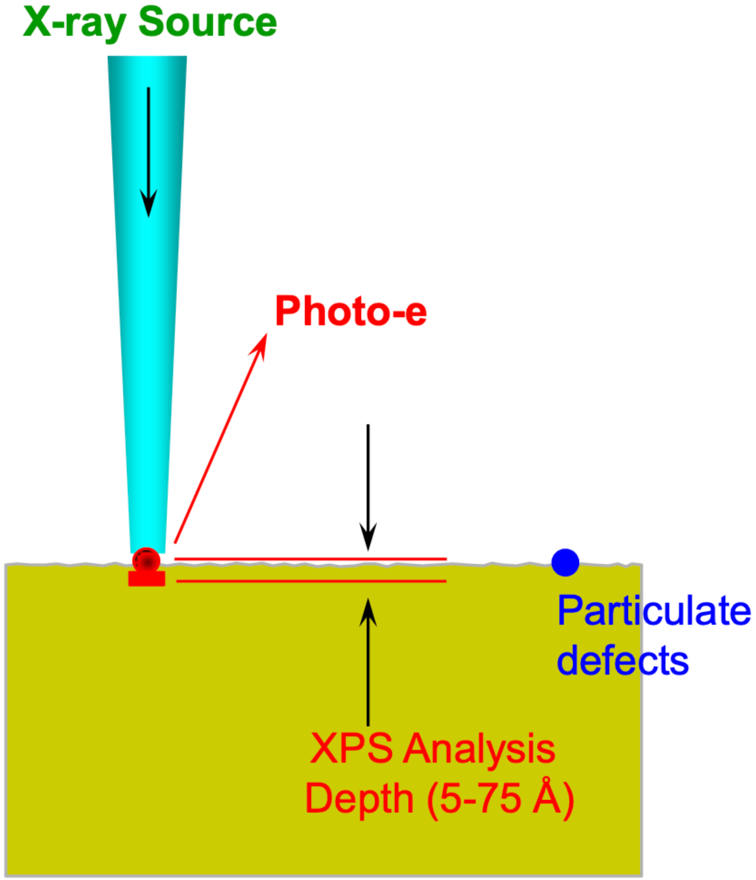
$$E_b = h\nu - E_{kin} - \Phi$$

- Electron binding energy is the parameter of fundamental interest, the spectrometer can be set up to record the spectrum on this energy scale.

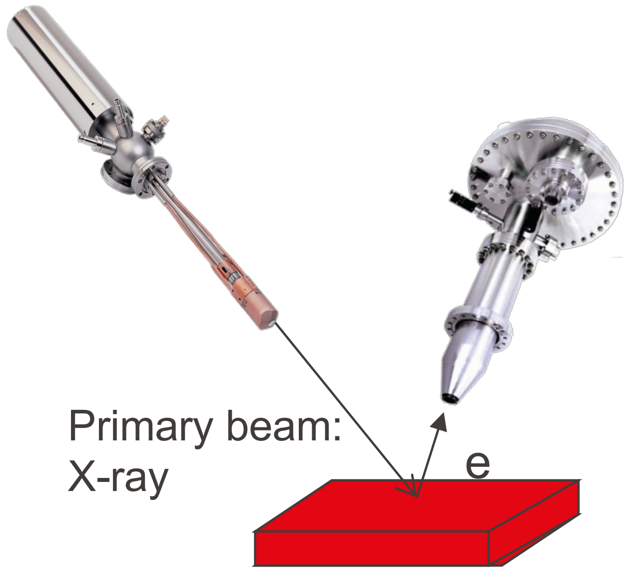
- Φ value can capture the uncertainties for the insulating/nonconductive samples (which does not have fixed fermi levels, can be affected by charging)

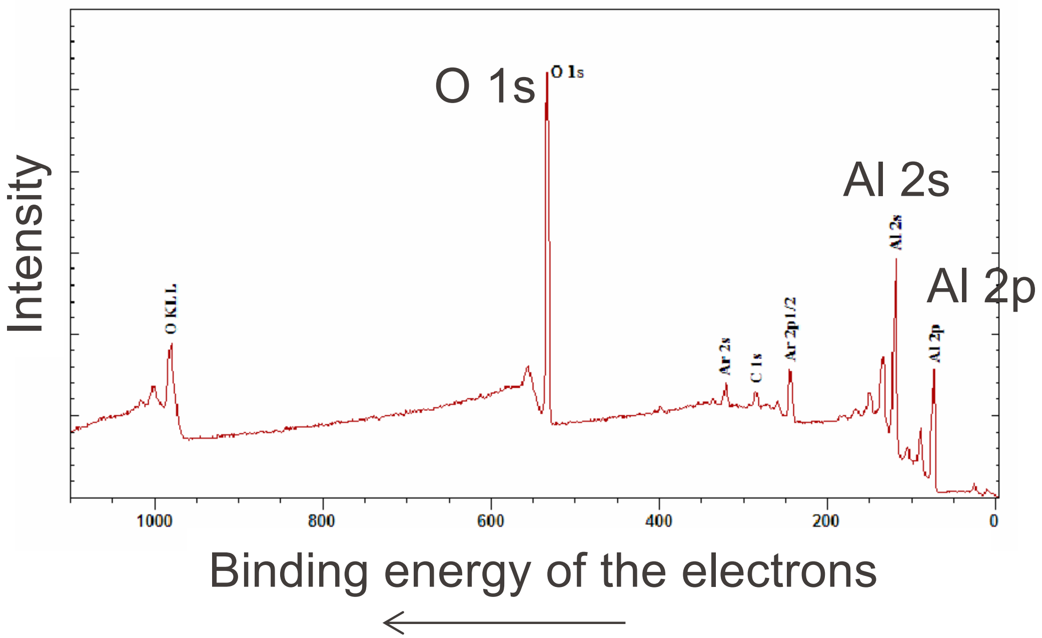
XPS a surface analysis technique

- Only the generated photoelectrons from the top surface about 5 to 75A can have enough energy to pop out of the sample surface.



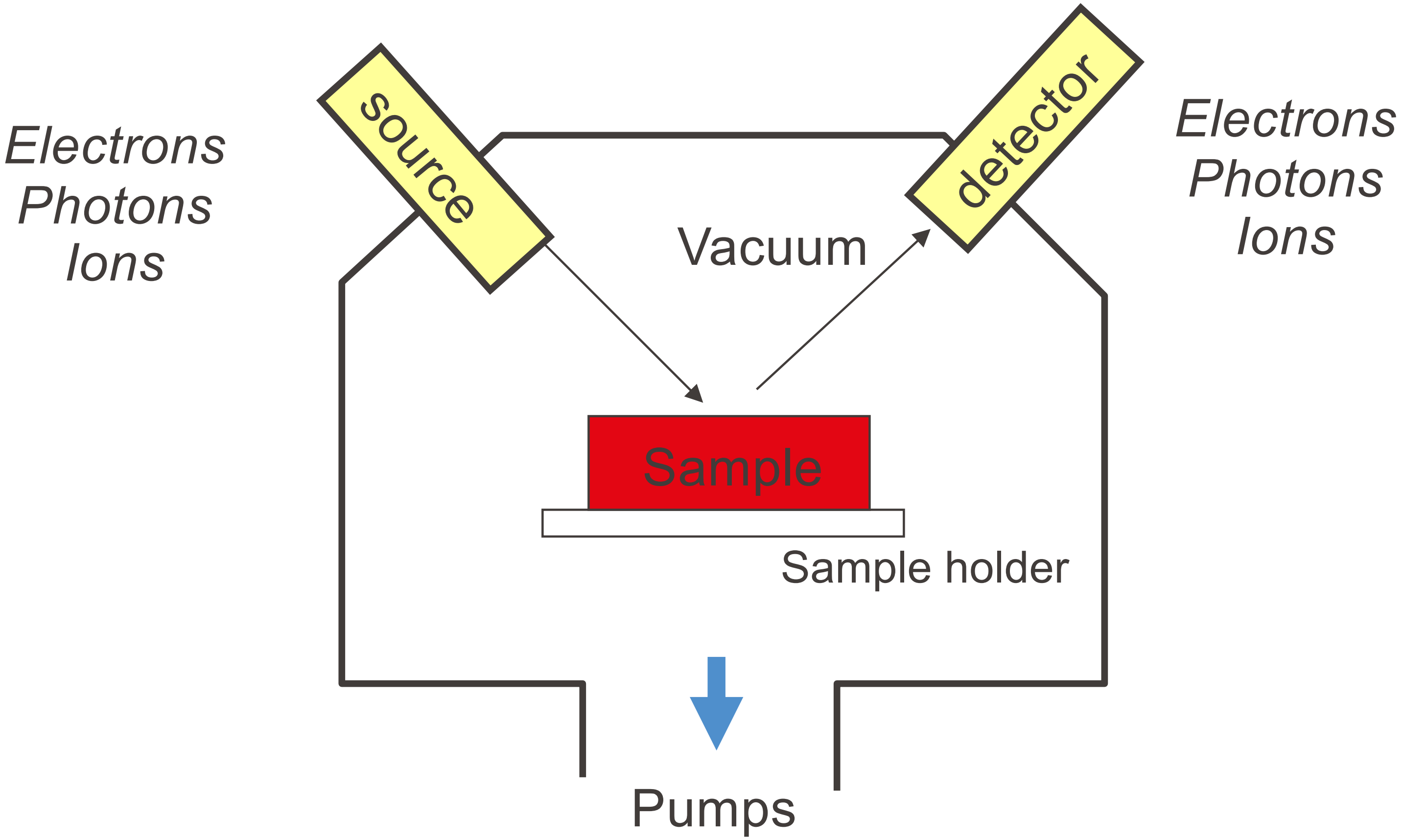
XPS: X-ray Photo Electron Spectroscopy





The XPS spectrum shows distinct peaks characteristic of specific surface atoms. Their intensities are proportional to their concentration.

Principle of different surface analysis methods



	AES Auger Electron Spectroscopy	**XPS** X-Ray Photoelectron Spectroscopy	**SIMS** Secondary Ion Mass Spectroscopy	**ISS** Ion Scattering Spectroscopy
Incident radiation (KeV)	Electrons (1 - 10)	Photons (1 - 2)	Ions (0.5 - 2)	Ions (0.5 – 2)
Analysed radiation	Auger electrons	Photoelectrons	Secondary ions	Backscattered ions
Analysed depth (atomic layers)	2 - 10	3 - 15	2 - 10	1 - 2
Detection limit, % at	0.1 – 1	0.1 - 2	10^{-4} - 1	0.1 - 1
Quantitative analysis	yes	yes	limited	partial
Particular characteristics	High spatial resolution (10-100 nm), conductive samples	Detection of oxidations state	High sensitivity, isotope detection	High depth resolution

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Vacuum: why

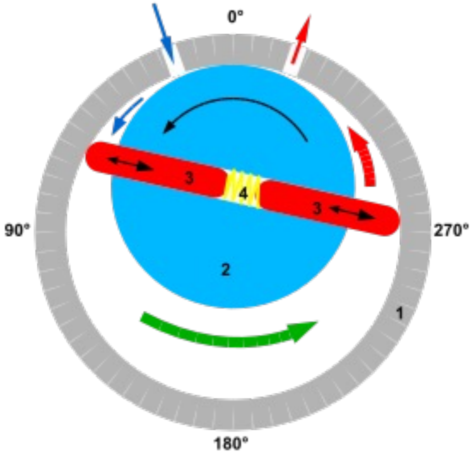
- In surface analysis vacuum is required for:

1. **Minimize collisions** of secondary particles emitted form the sample with gas molecules leading to loss of signal intensity. For this a pressure below 10^{-6} torr (1.33 10^{-4} Pa) is required.
2. **Minimize surface contamination** by residual gases present in the analysis chamber that could adsorb on the surface. From gas kinetic theory a gas monolayer forms by adsorption in 1 s at a pressure of 10^{-6} torr (1.33 10^{-4} Pa). In practice, a **pressure in the range 10^{-7} – 10^{-8} Pa** is sufficient to limit adsorption for surface analysis.

Vacuum: how (pumps)

Rotary vane pumps

Range 10^5 to 10^{-1} Pa



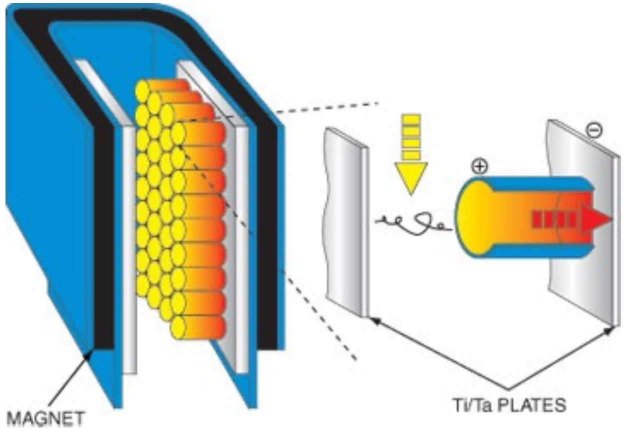
1. pump housing
2. rotor
3. vanes
4. spring

Turbopumps

Range 10^{-1} to 10^{-7} Pa

Ion pumps

Range 10^{-6} to 10^{-8} Pa

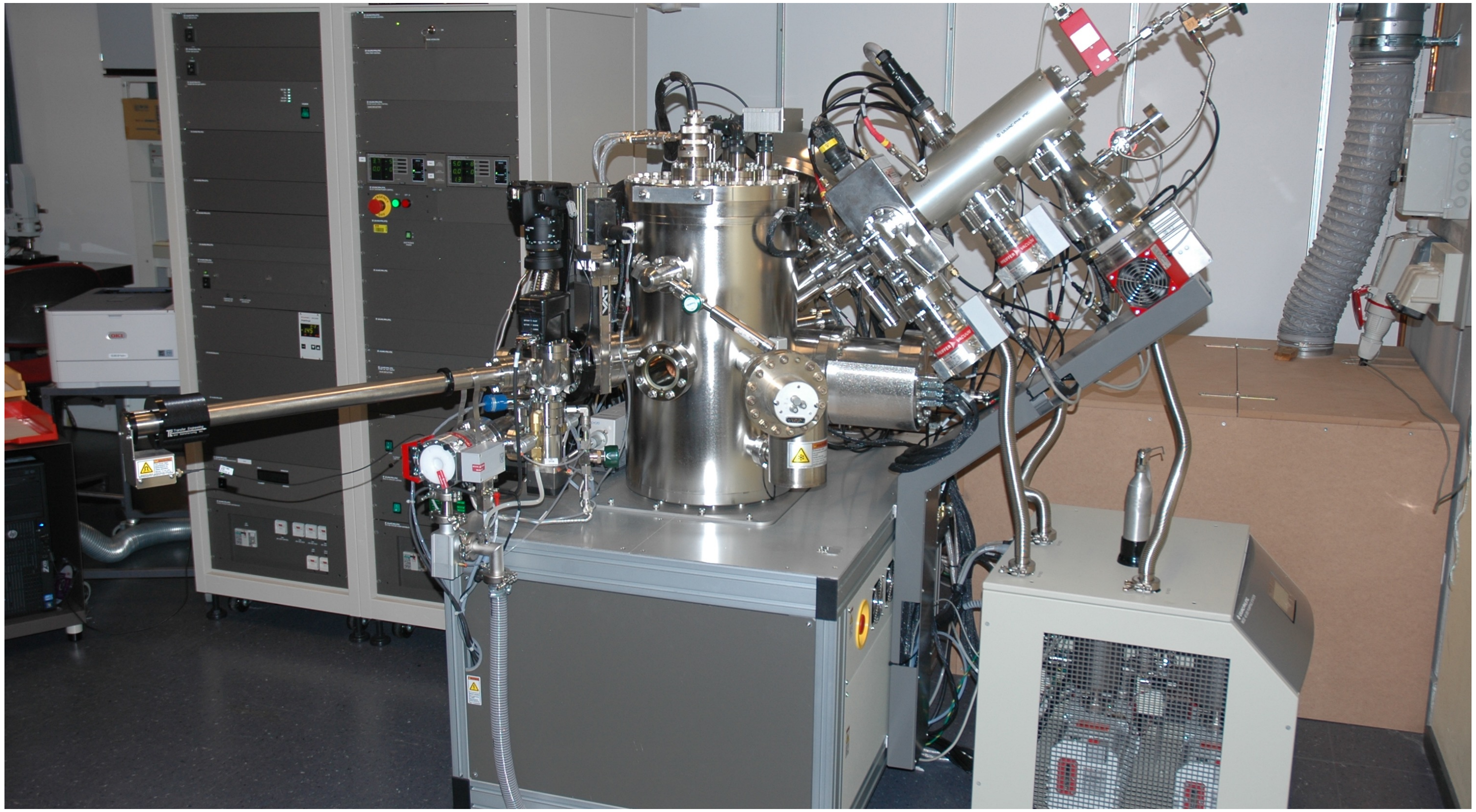


Source: wikipaedia

Vacuum: how (design)

1. Appropriate design of the vacuum chamber to ensure appropriate pumping speed in all points (avoid recessed geometries or bottlenecks)
2. Use of bake out (heating) of the vacuum chamber to stimulate desorption of gases from the walls.
3. Use appropriate materials for the construction (stainless steel, copper seals, viton seals for valves).
4. Provide sample introduction systems with entry locks

XPS VersaProbe II instrument of EPFL-MHMC



Vacuum: how (samples)

1. Samples must be clean and free of surface contaminations (manipulating samples with hands makes surface analysis impossible, storage in open air has to be avoided).

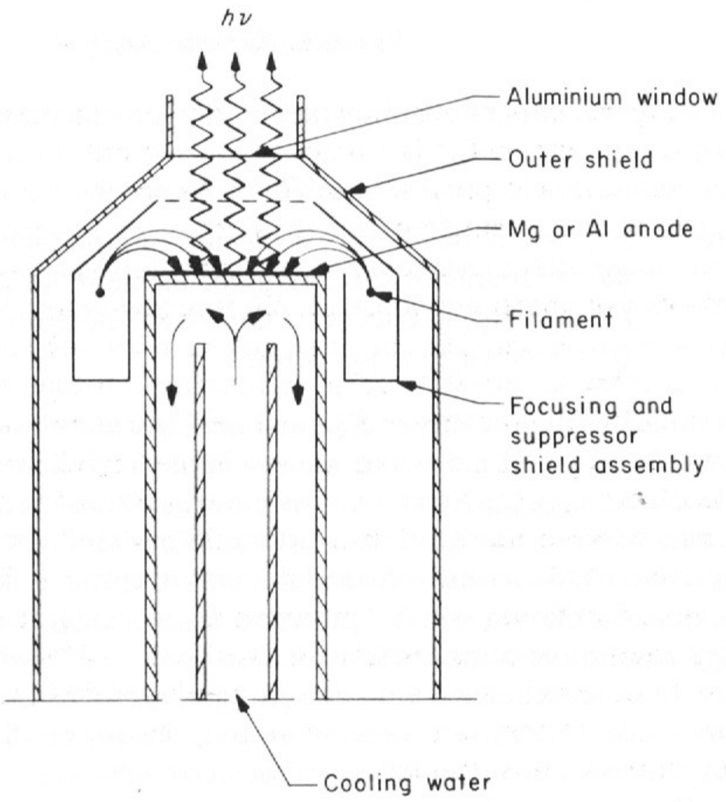
2. Samples should not contain solvents or volatile compounds (no oil, no water, no …).

Contamination layers mask the sample surface and worsen vacuum.

Clean surfaces and samples are a prerequisite for successful surface analysis !!!

X-Ray sources

- A hot filament emits electrons that are accelerated towards the positively charged anode.
- The impinging electrons generated fluorescence radiation hv.
- The shield assembly and the aluminium window blocks unwanted electron flows and radiation.
- The anode requires strong cooling through water flow.



Choice of anode material

The choice of the anode material is a compromise between:

- Depth of information that increases with X-Ray line energy hv.
- Energy resolution of the signal that worsen with increasing X-Ray line width.

$$E_b = hv - E_{kin} - const$$

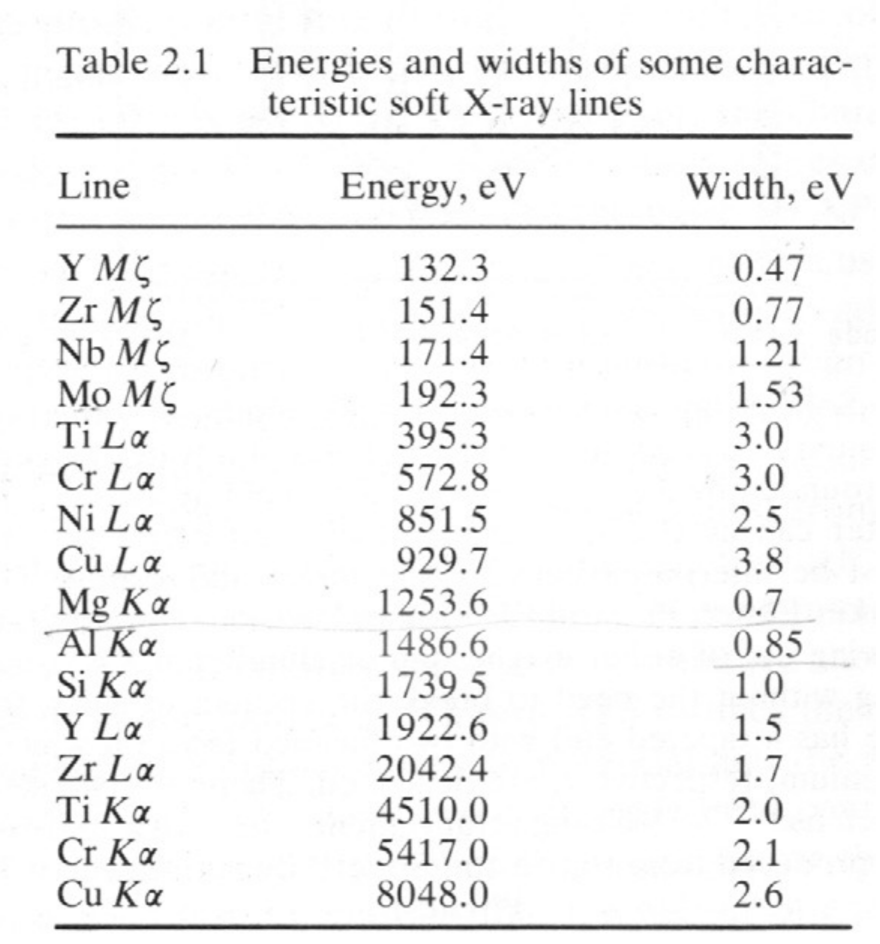


Table 2.1 Energies and widths of some characteristic soft X-ray lines

Line	Energy, eV	Width, eV
Y $M\zeta$	132.3	0.47
Zr $M\zeta$	151.4	0.77
Nb $M\zeta$	171.4	1.21
Mo $M\zeta$	192.3	1.53
Ti $L\alpha$	395.3	3.0
Cr $L\alpha$	572.8	3.0
Ni $L\alpha$	851.5	2.5
Cu $L\alpha$	929.7	3.8
Mg $K\alpha$	1253.6	0.7
Al $K\alpha$	1486.6	0.85
Si $K\alpha$	1739.5	1.0
Y $L\alpha$	1922.6	1.5
Zr $L\alpha$	2042.4	1.7
Ti $K\alpha$	4510.0	2.0
Cr $K\alpha$	5417.0	2.1
Cu $K\alpha$	8048.0	2.6

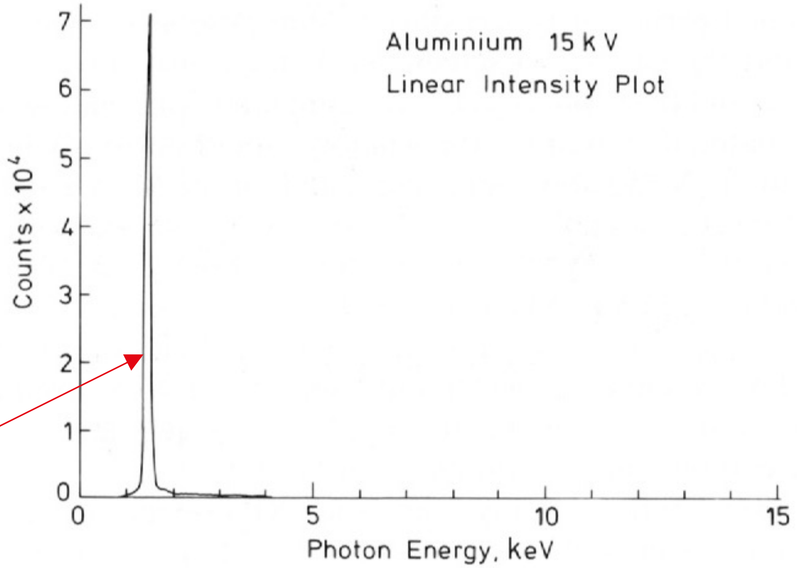
X-ray and Bremstrahlung

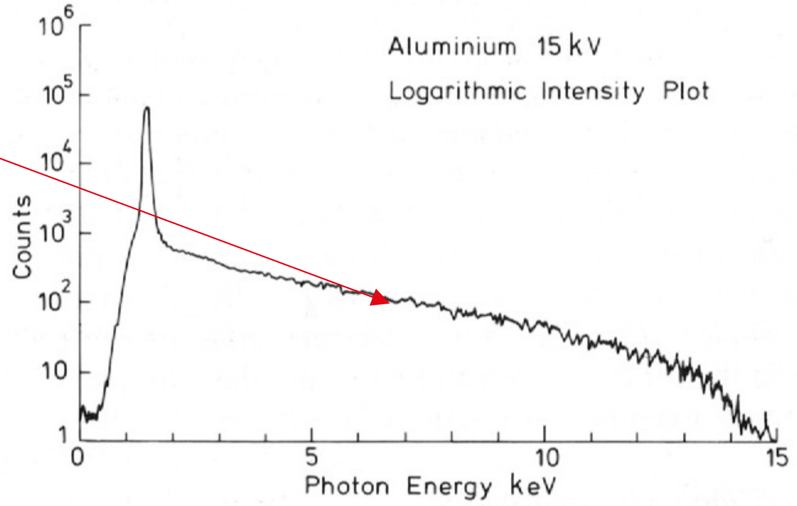
X-Ray emission spectrum of an Al anode bombarded by 15 kV electrons.

Two features:

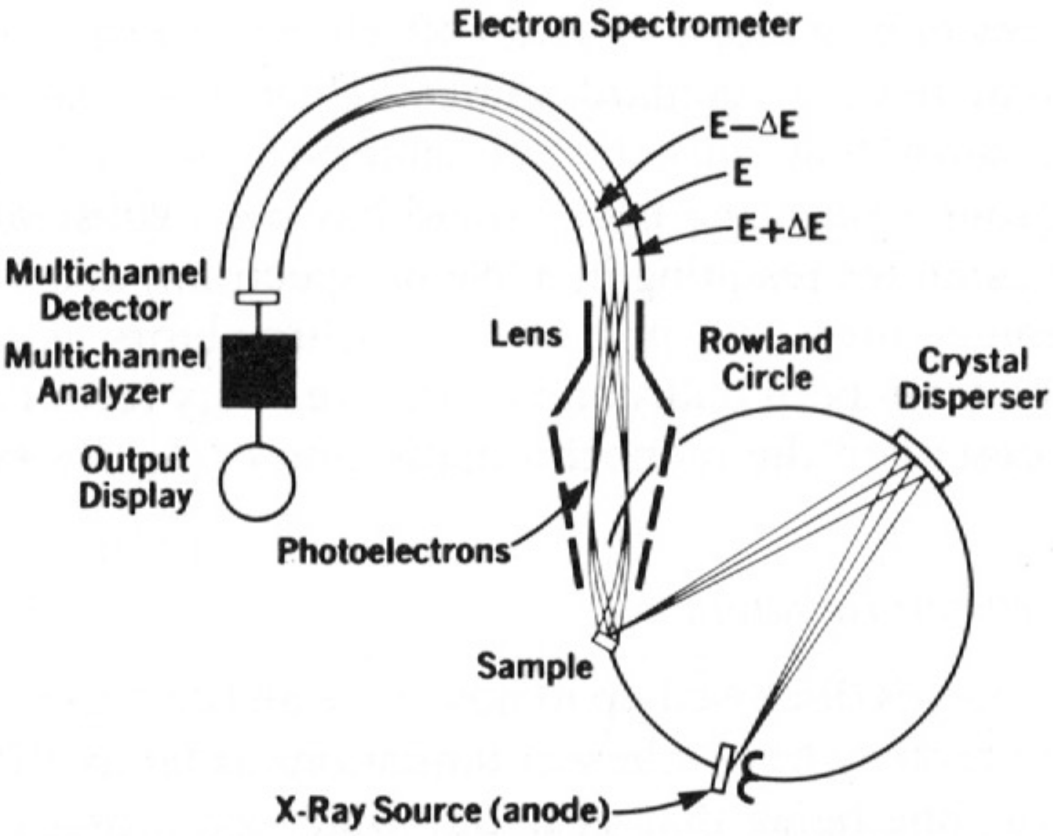
- Characteristic strong K_α line
- Broad Bremstrahlung background

Bremstrahlung: The X-ray output consists of a continuous energy distribution extending up to incident electron energy, with higher intensity at the characteristic K_α energy.





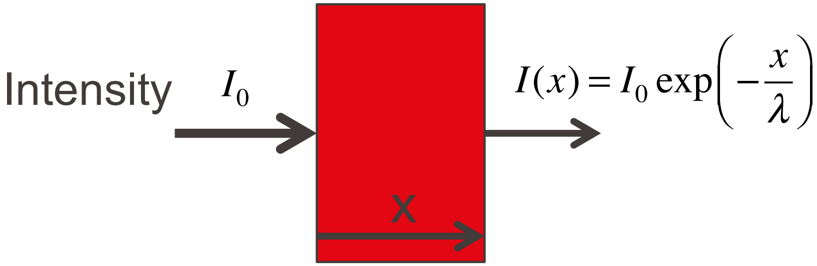
Elimination of Bremstrahlung through crystal monochromators



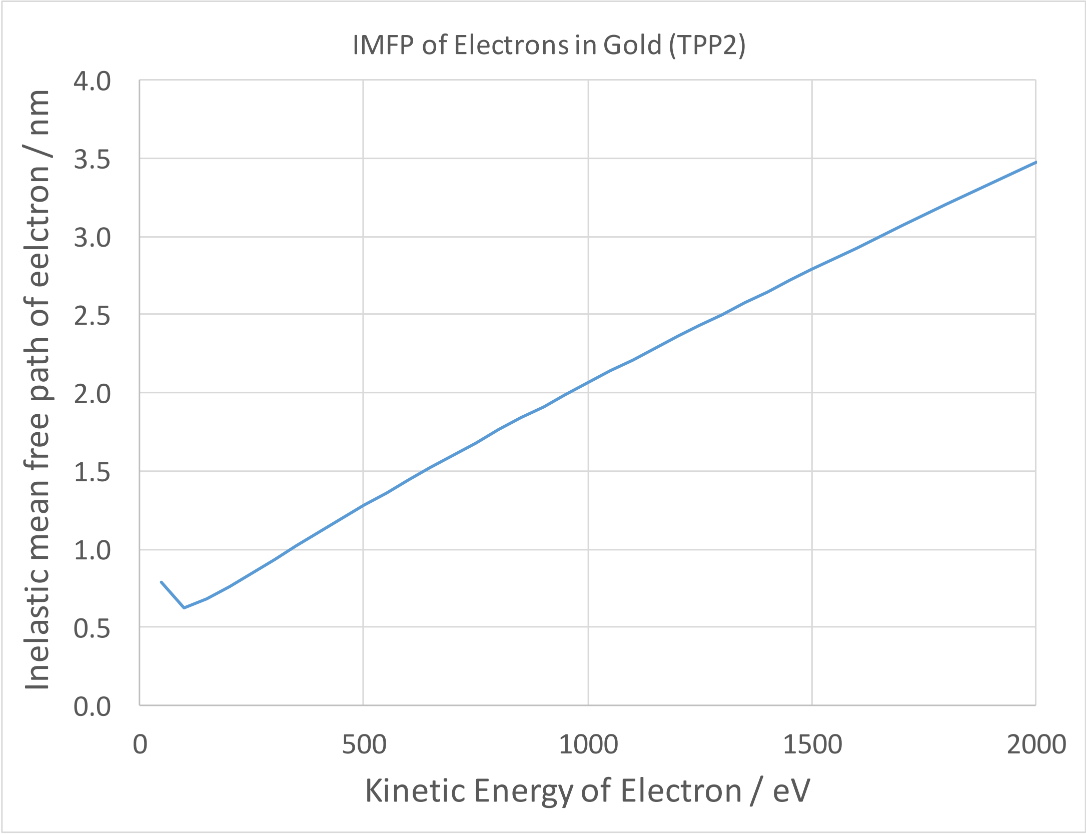
The crystal filters the characteristic line through Bragg’s diffraction.

For Al anodes quartz crystal are used as monochromators.

Electron mean free path λ in solids



$$I(x) = I_0 \exp\left(-\frac{x}{\lambda}\right)$$



The electrons exhibit a very small mean free path of a few nm’s.
The escape only if emitted by the first few monolayers.

The x-rays penetrate depending on the energy much deeper, typically 100 µm.

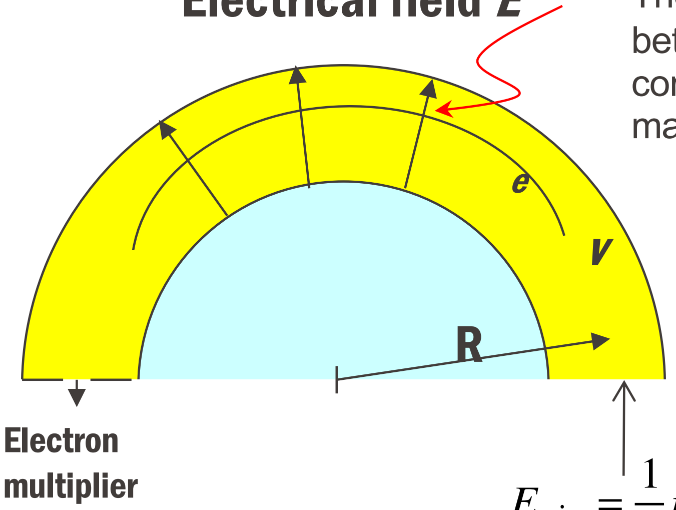
The Concentric Hemispherical Analyser CHA

To measure the number of photo-ejeted electrons as a function of their energy

Hemispherical Condenser

CHA: Concentric hemispherical analyser

Electrical field *E*



The electric field is swept between zero and the value corresponding to the maximum E_{cin}.

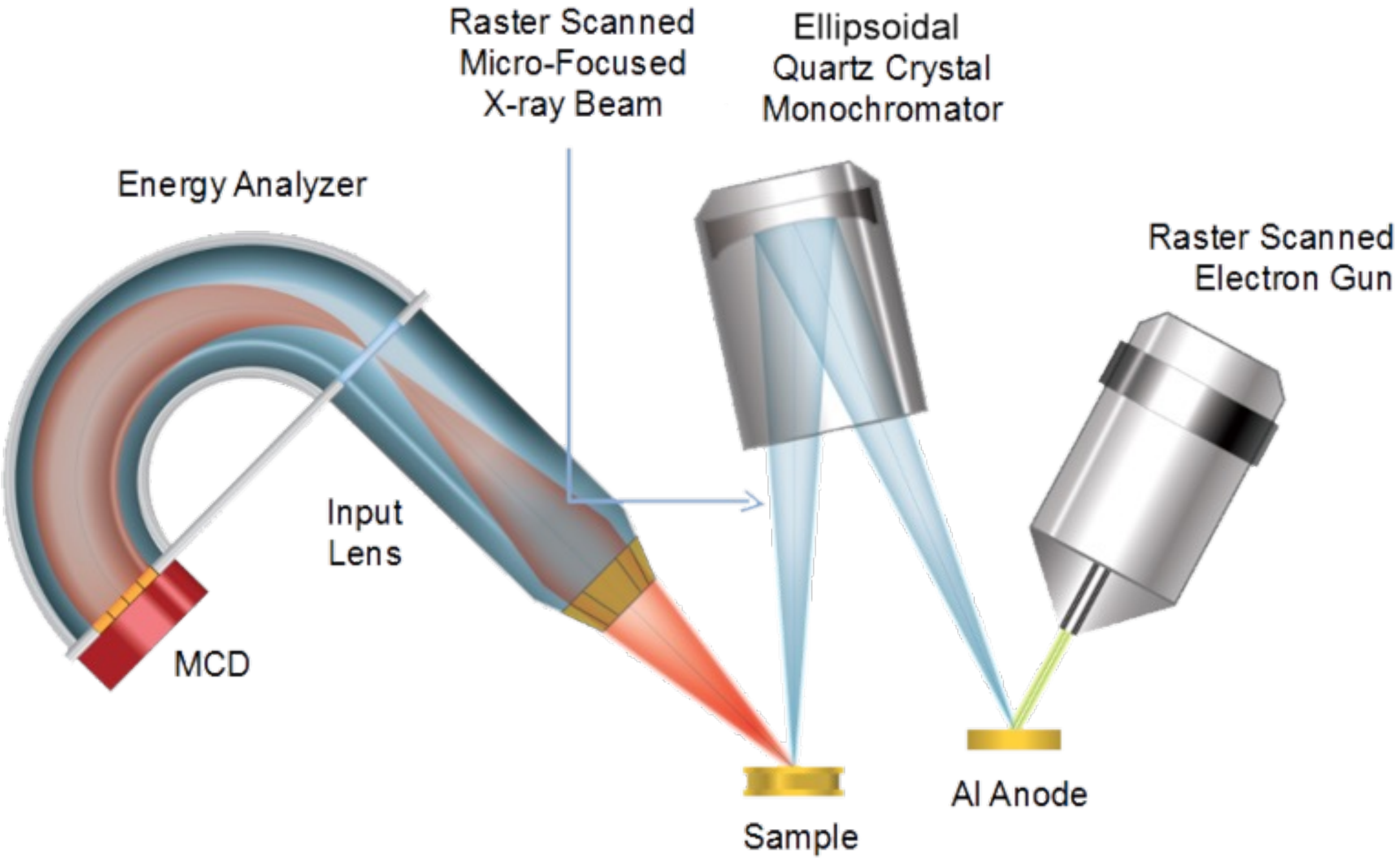
$$E_{cin} = \frac{1}{2}mv^2$$

$$-eE = m_e\frac{v^2}{R}$$

The electrons arriving at the detector (electron multiplier) have a kinetic energy of:

$$E_{cin} = \frac{eER}{2}$$

Summary of main instrumentation in XPS



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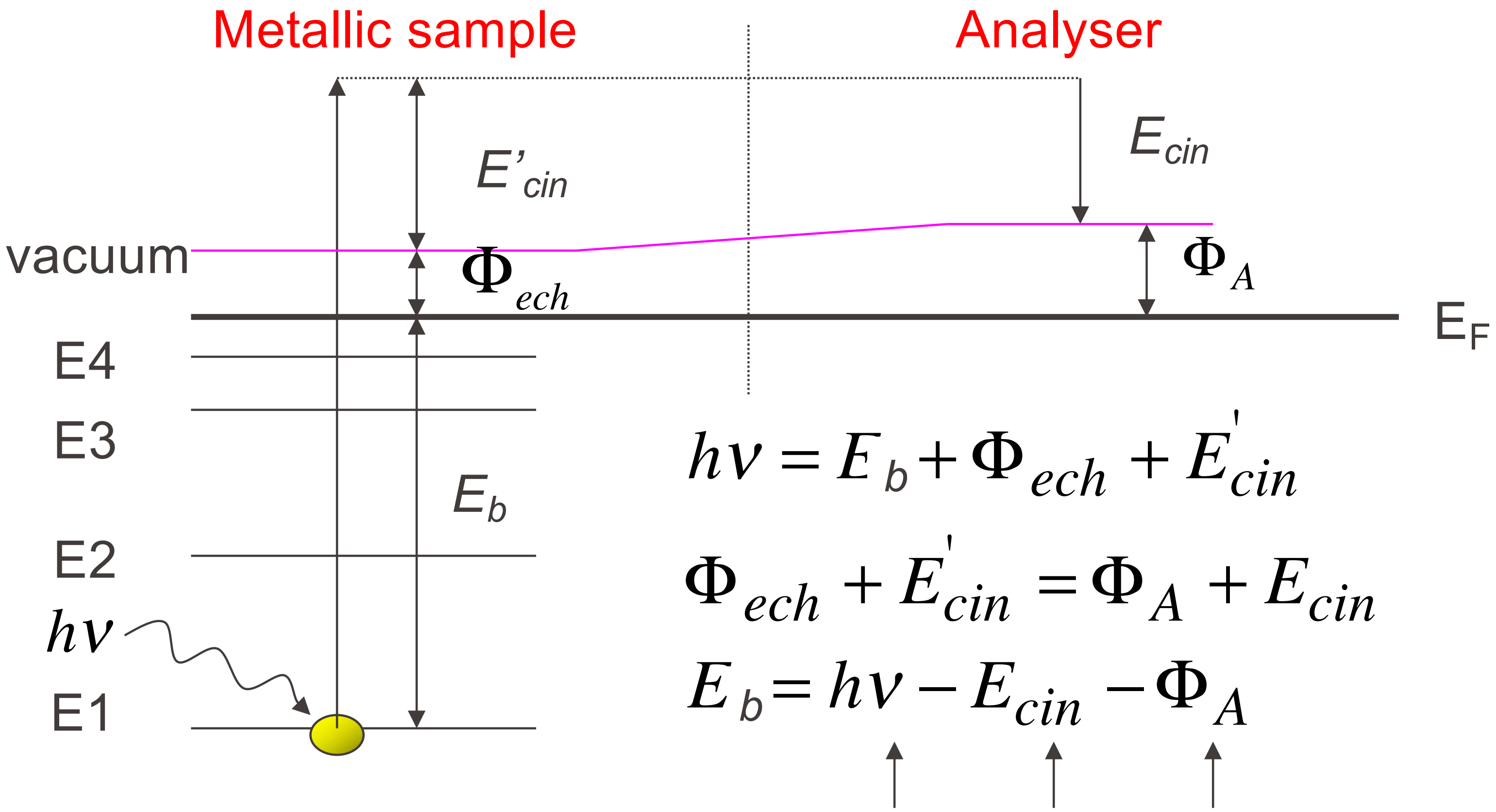
Extraction of binding energy



$$h\nu = E_b + \Phi_{ech} + E'_{cin}$$

$$\Phi_{ech} + E'_{cin} = \Phi_A + E_{cin}$$

$$E_b = h\nu - E_{cin} - \Phi_A$$

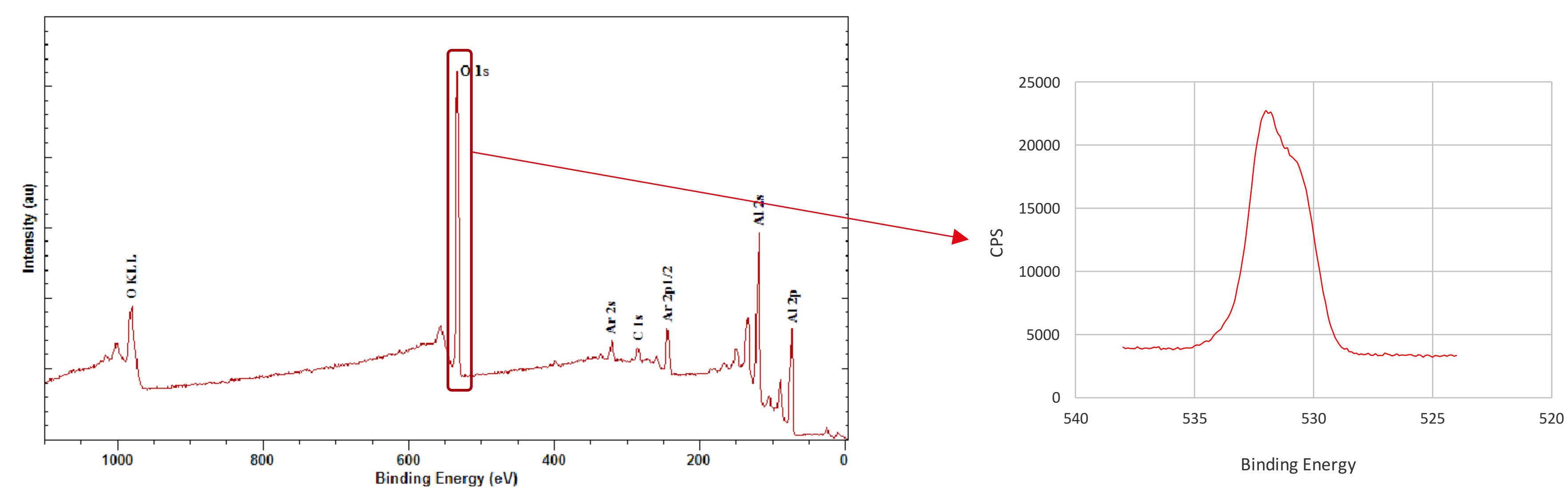
Known Measured Known by calibration

Binding energy E_b

Calibration of the energy scale

- Usually the binding energy cannot be extracted directly form the equation $E_b = h\nu - E_{kin} - \Phi_A$ because:
  - The Fermi levels of the analyzer and of the sample do not necessarily coincide.
  - Loss of electrons from the sample can cause electrical charging.
- Most of the samples exhibit an adventitious hydrocarbon contamination which energy is 284.8 eV. The energy scale can then be calibrated by shifting the spectrum to lower or higher energy until the adventitious carbon peak corresponds to 284.8 eV.

Survey and multiplex spectra



Survey spectra are measured over a large range of energy with the scope to identify the elements present on the surface. To keep acquisition time reasonably short the energy resolution is weak (1 recorded point every 1-2 eV)

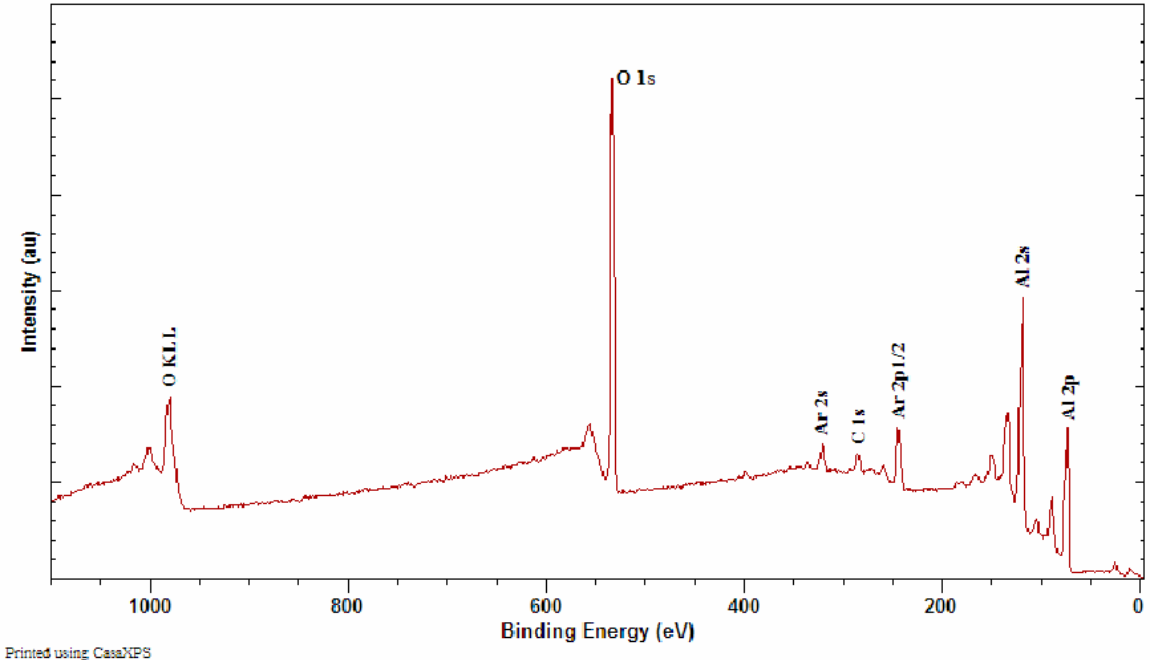
In **multiplex measurements** characteristic peaks of each identified element are acquired with increased resolution (1 recorded point every 0.1 – 0.2 eV) in order to identify the fine structure of the peak and its precise energy position.

Spectral features

- Primary structure of spectra
    - Background
    - XPS Peaks
    - Doublets
    - Auger peaks

- Secondary structure
    - Shake-up peaks in insulators
    - Plasmon peaks in metals

Background



The background is not constant under the peaks, as it is formed by photoelectrons that have lost a little bit of energy in inelastic and elastic interactions with other electrons (e.g. from valence band). (BG is larger to the left).

The procedure of Shirley calculates at a given energy a background that is proportional to the integral of signal intensity at higher kinetic energy (lower binding energy).

XPS peaks: nomenclature (I)

Elements can exhibit several XPS peaks (depending on involved electrons) that have to be precisely defined.

This is done by considering the quantum numbers n, l and j = l + s, where s is the spin number (either +1/2 or -1/2).

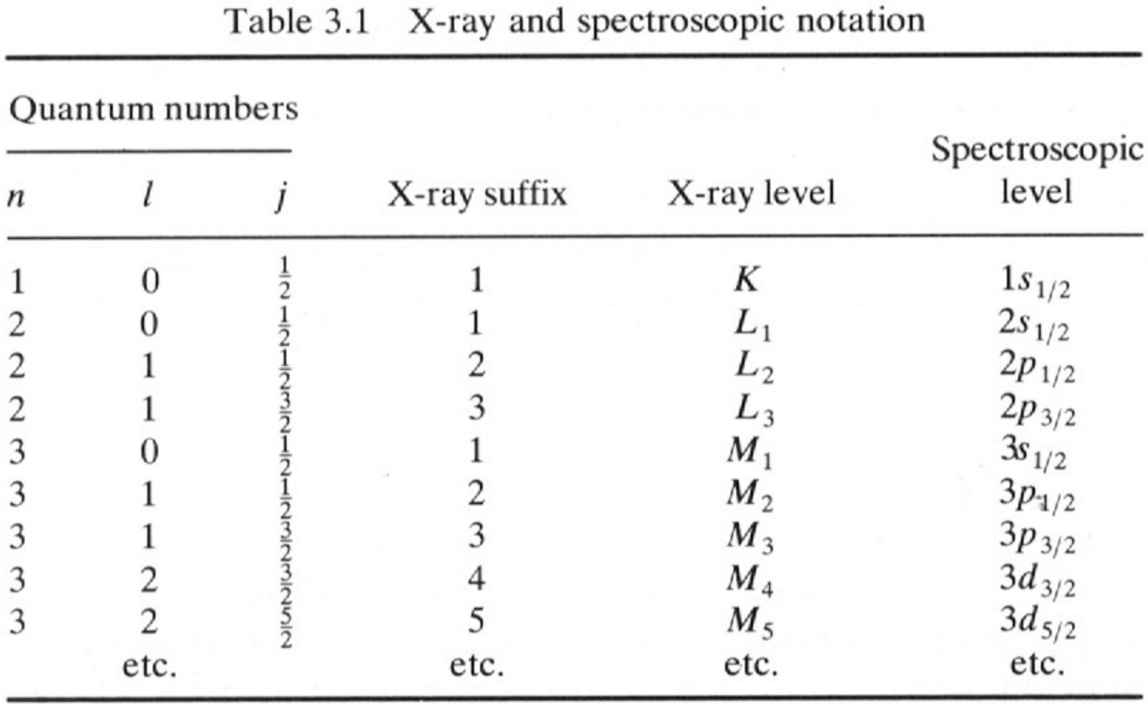
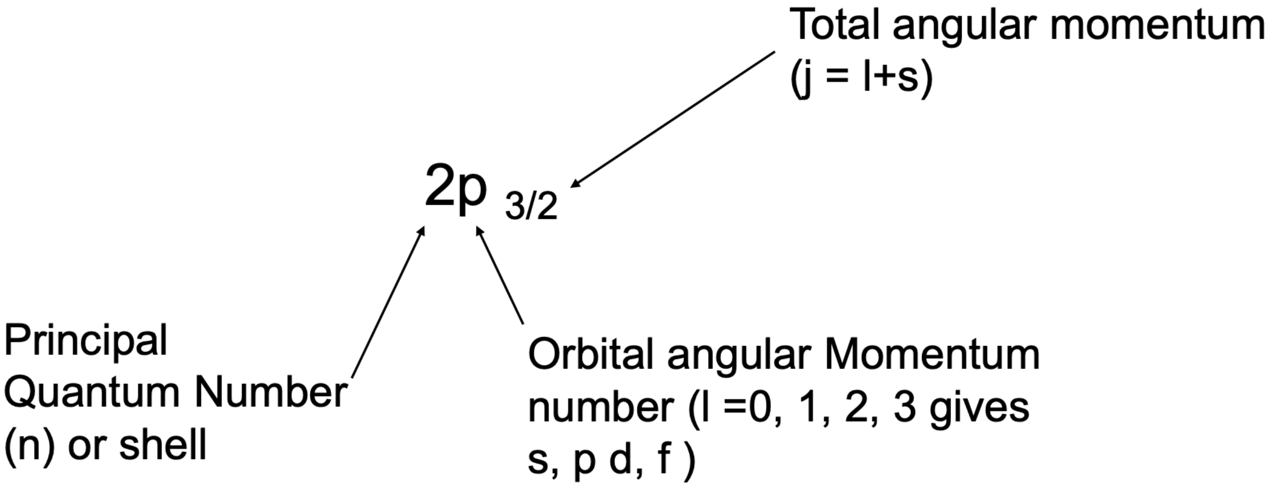


Table 3.1 X-ray and spectroscopic notation

Quantum numbers					
n	l	j	X-ray suffix	X-ray level	Spectroscopic level
1	0	$\frac{1}{2}$	1	K	$1s_{1/2}$
2	0	$\frac{1}{2}$	1	L_1	$2s_{1/2}$
2	1	$\frac{1}{2}$	2	L_2	$2p_{1/2}$
2	1	$\frac{3}{2}$	3	L_3	$2p_{3/2}$
3	0	$\frac{1}{2}$	1	M_1	$3s_{1/2}$
3	1	$\frac{1}{2}$	2	M_2	$3p_{1/2}$
3	1	$\frac{3}{2}$	3	M_3	$3p_{3/2}$
3	2	$\frac{3}{2}$	4	M_4	$3d_{3/2}$
3	2	$\frac{5}{2}$	5	M_5	$3d_{5/2}$
	etc.		etc.	etc.	etc.

XPS peaks: nomenclature (II)

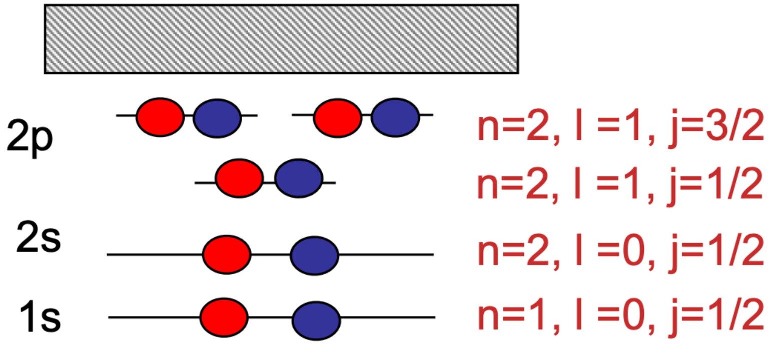


$2p_{3/2}$

Total angular momentum (j = l+s)

Principal Quantum Number (n) or shell

Orbital angular Momentum number (l =0, 1, 2, 3 gives s, p d, f)



n=2, l =1, j=3/2

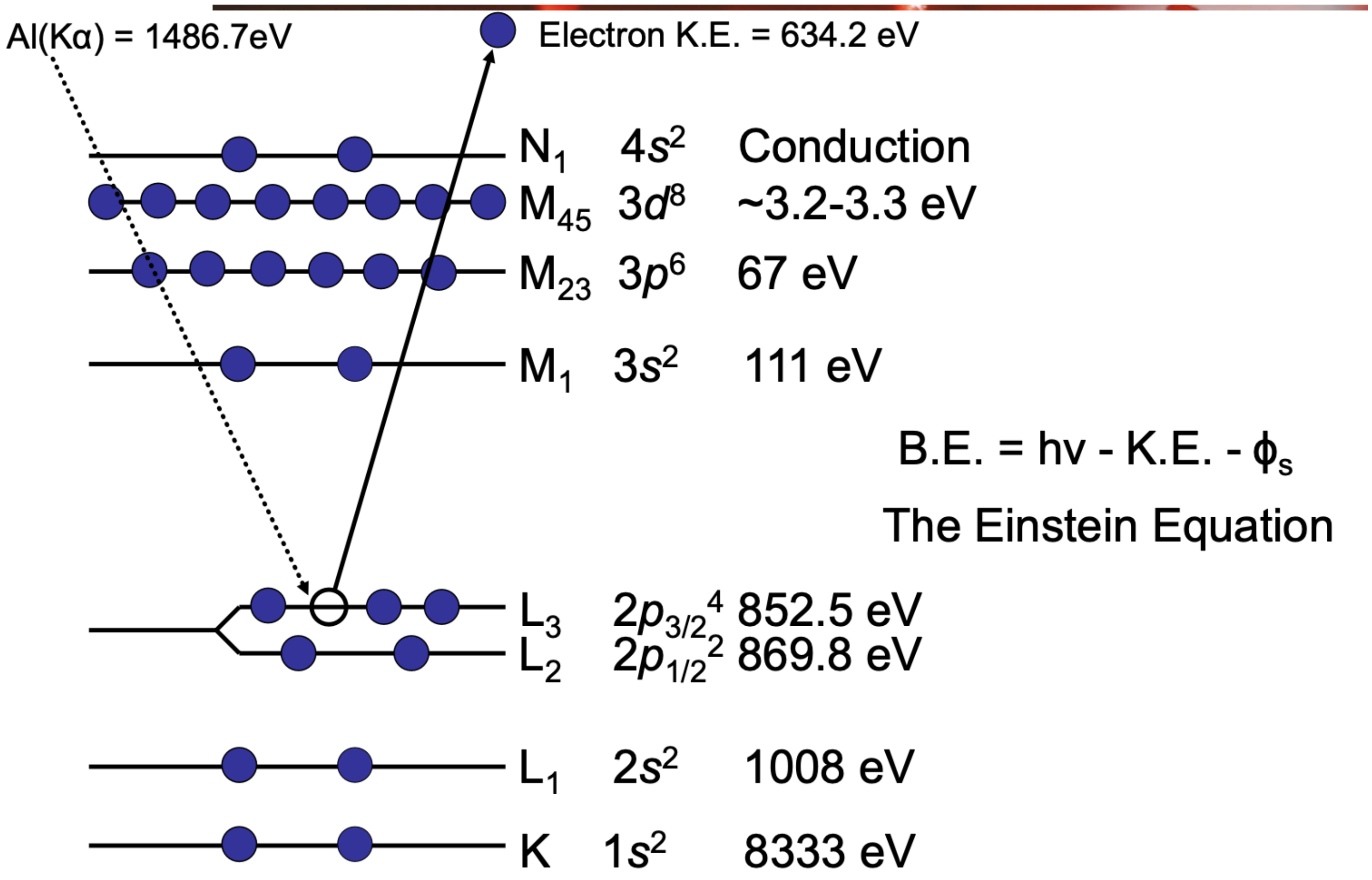
n=2, l =1, j=1/2

n=2, l =0, j=1/2

n=1, l =0, j=1/2

Degeneracy = 2j+1

Photoelectron ejection: example Ni $2p_{3/2}$



Al(Kα) = 1486.7eV

Electron K.E. = 634.2 eV

N_1 $4s^2$ Conduction

M_{45} $3d^8$ ~3.2-3.3 eV

M_{23} $3p^6$ 67 eV

M_1 $3s^2$ 111 eV

$$B.E. = h\nu - K.E. - \phi_s$$

The Einstein Equation

L_3 $2p_{3/2}{}^4$ 852.5 eV

L_2 $2p_{1/2}{}^2$ 869.8 eV

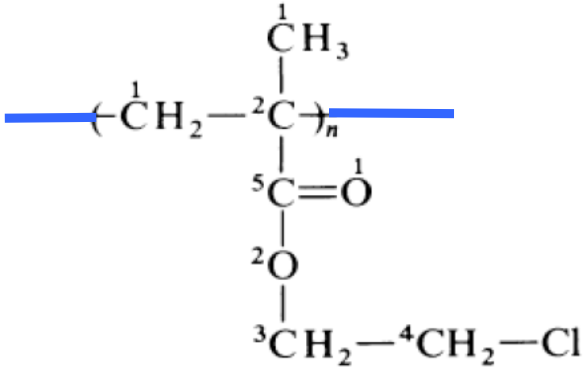
L_1 $2s^2$ 1008 eV

K $1s^2$ 8333 eV

XPS peaks: example of a polymer (poly (2-chloroethyl methacrylate) spectrum evaluation

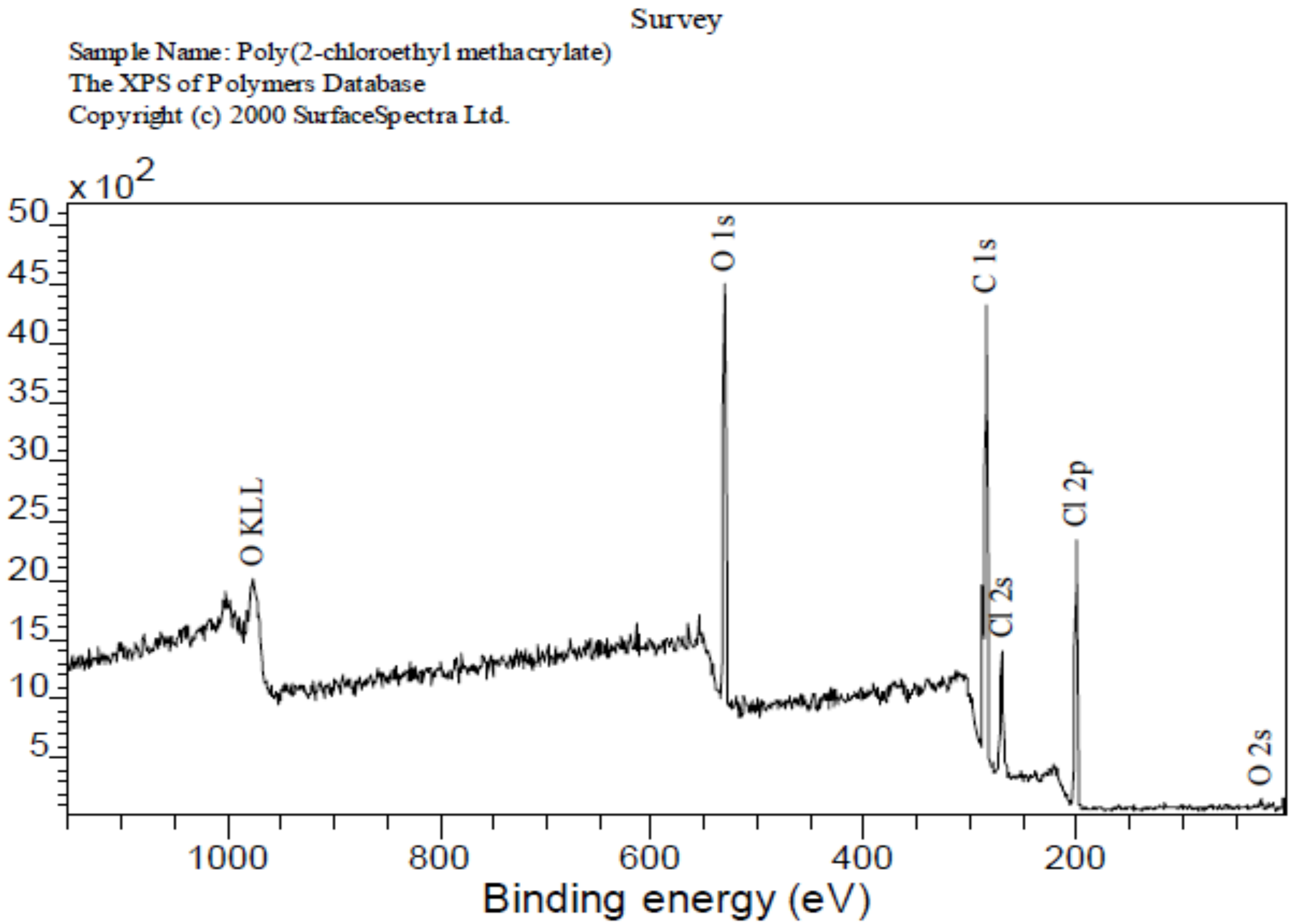
Formula: $C_6O_2Cl_1$

(hydrogen is not considered as not detectable in XPS)

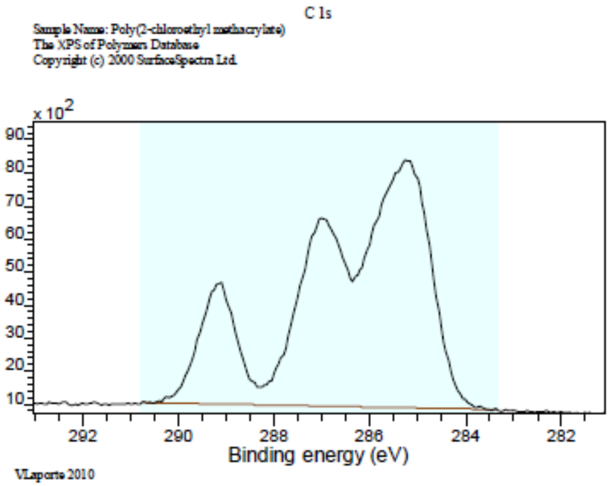


Backbone:
-C-C-

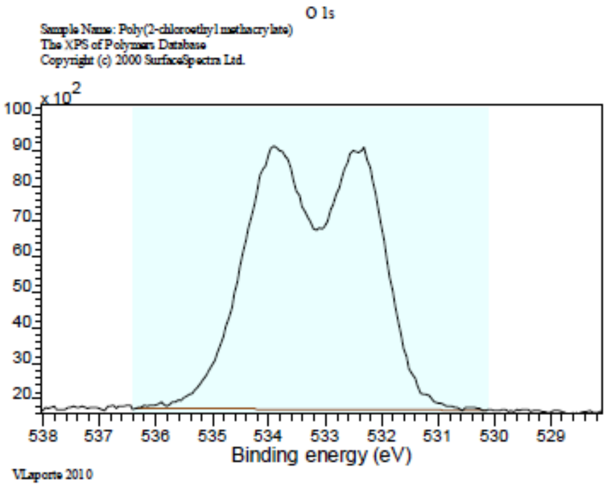
Survey spectra



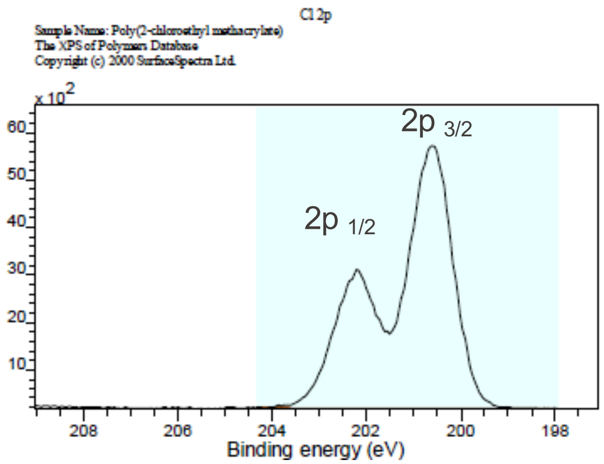
Multiplex spectra



Carbon



Oxygen

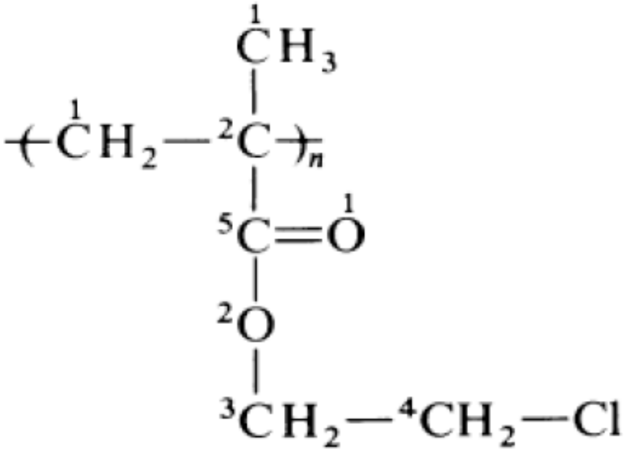


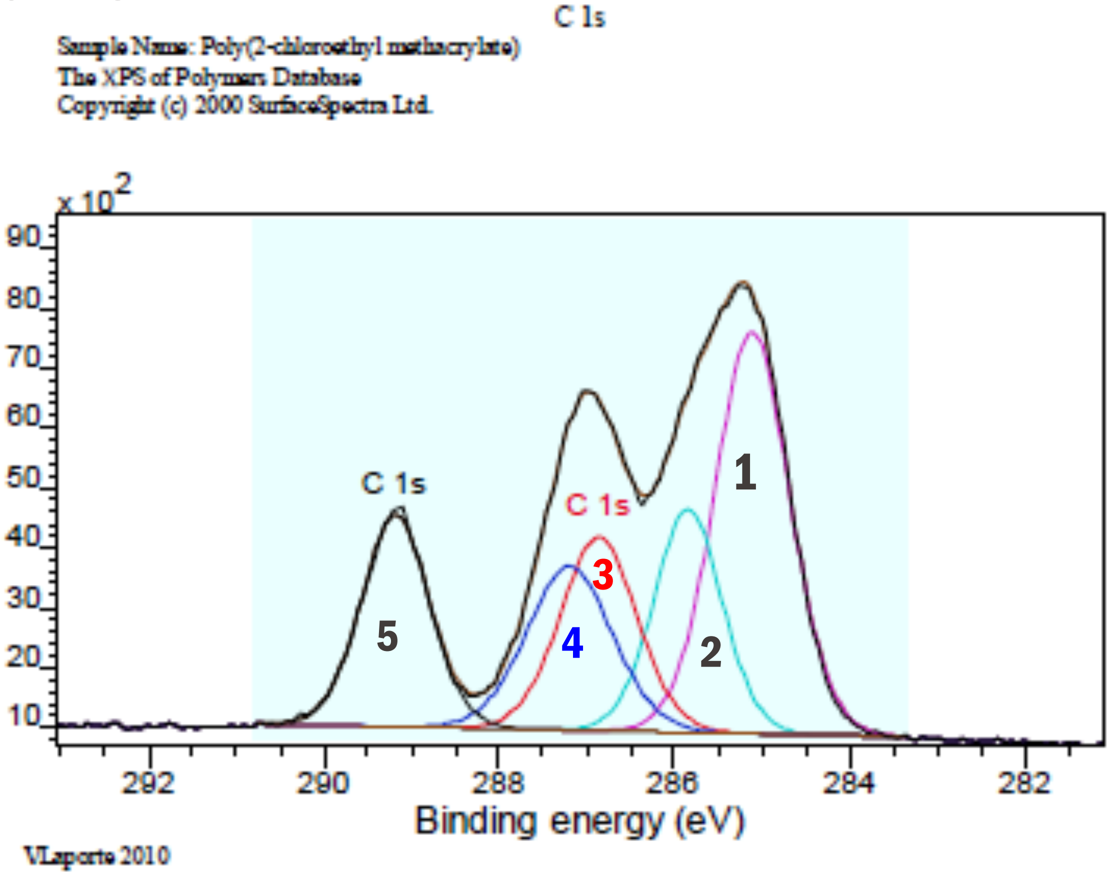
Chlorine

The multiplex spectra reveal a fine structure of each peak involving several sub-peaks.

- For carbon and oxygen 1s peaks this indicates different oxidation states.
- In case of a 2p peak we expect a doublet splitting due to the quantum number j (the area ratio between 2p $_{3/2}$ and 2p $_{1/2}$ is expected to be 2:1. The peak energy at 200.5 eV corresponds to Cl in organic molecules.

Chemical shift of C 1s



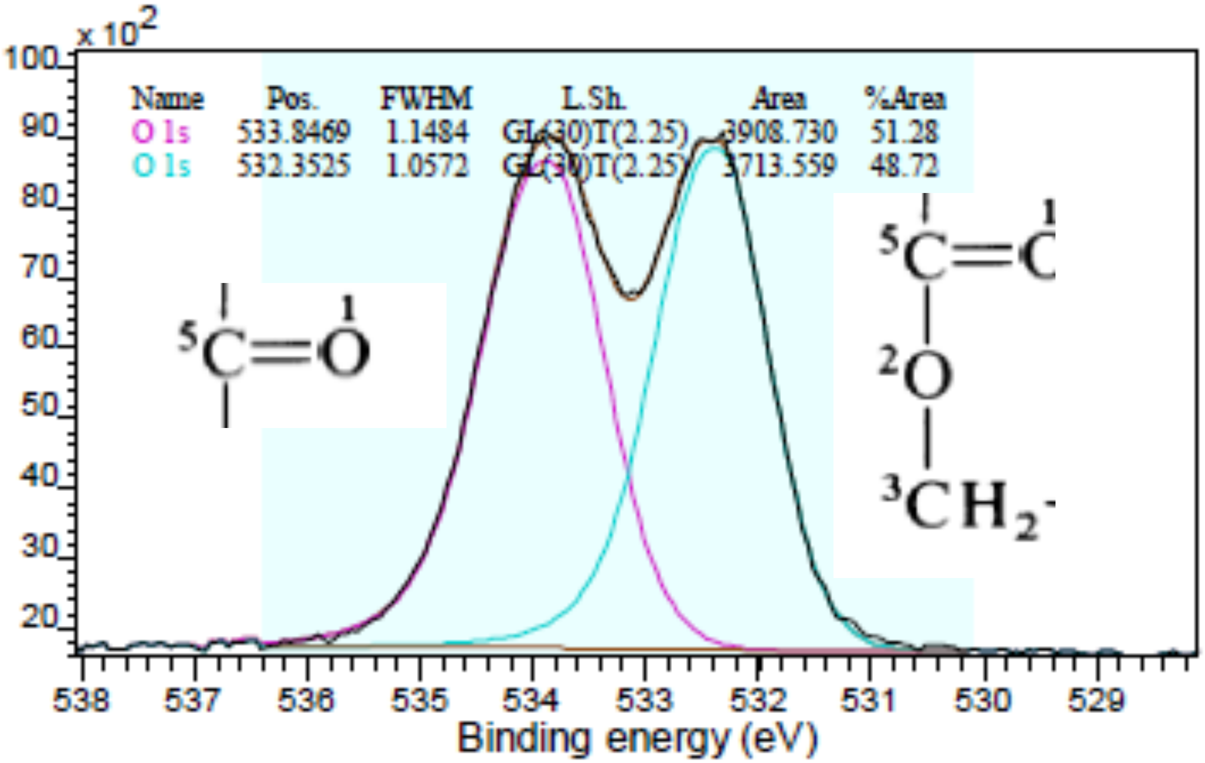

C 1s

Sample Name: Poly(2-chloroethyl methacrylate)
The XPS of Polymers Database
Copyright (c) 2000 SurfaceSpectra Ltd.

VLaporte 2010

The different chemical bonds of carbon atoms change the binding energy of the electrons and can be identified in the XPS spectrum.

Chemical shift of O 1s

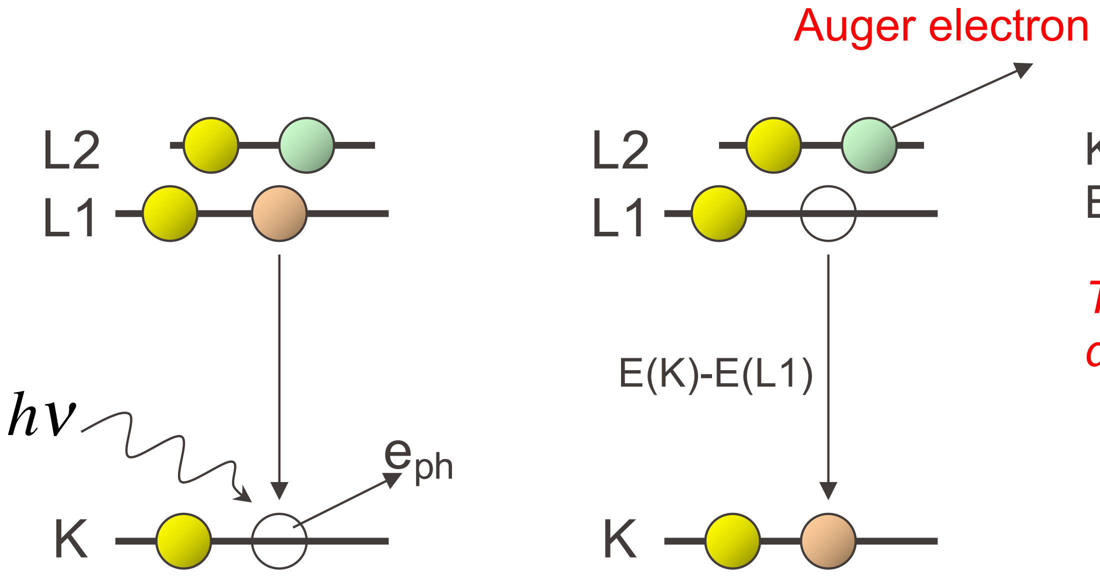


Name	Pos.	FWHM	L.Sh.	Area	%Area
O 1s	533.8469	1.1484	GL(30)T(2.25)	3908.730	51.28
O 1s	532.3525	1.0572	GL(30)T(2.25)	3713.559	48.72

O attracts electrons: less binding energy for 1s state

Auger electrons

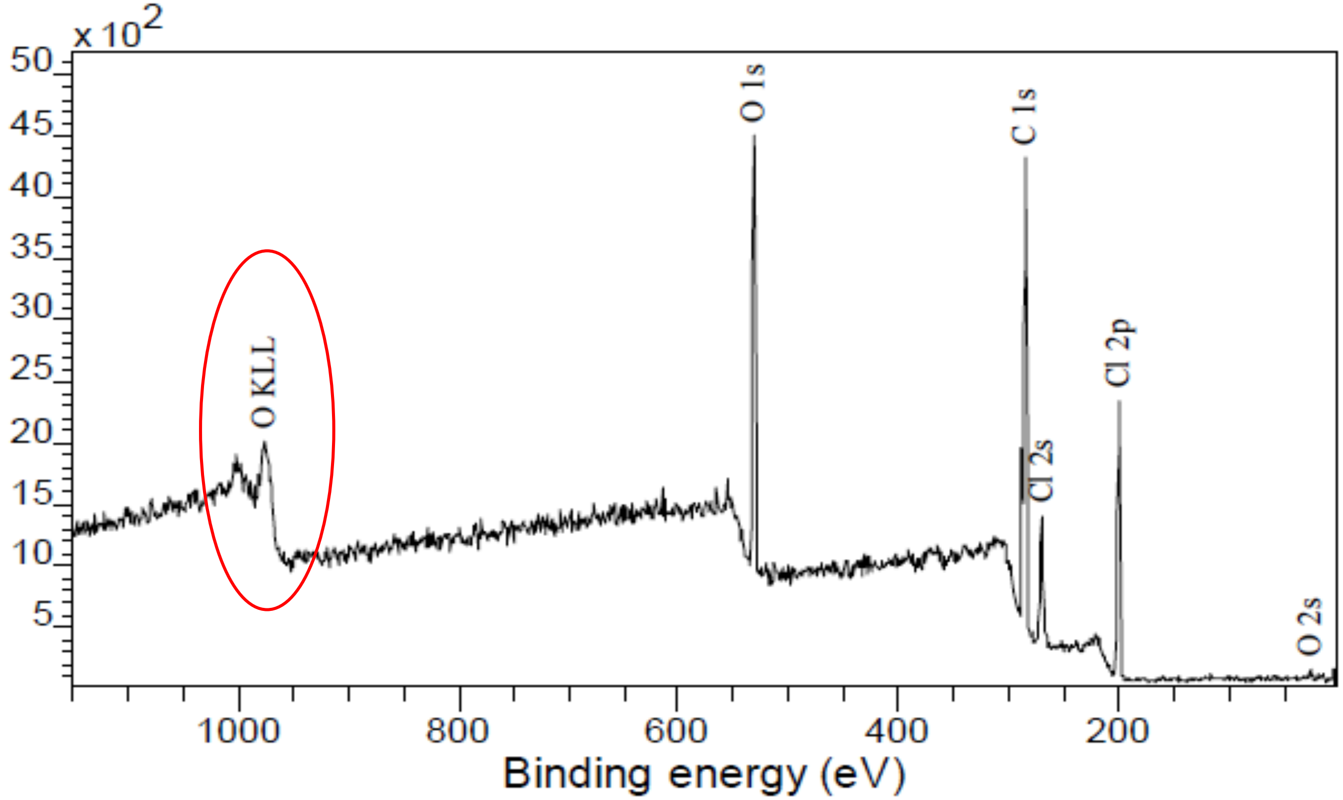
- After the photoelectron emission, the atom makes a relaxation, i.e., an electron from a higher shell occupies the empty position left by the photoelectron. The energy gain is carried away by an AUGER electron.



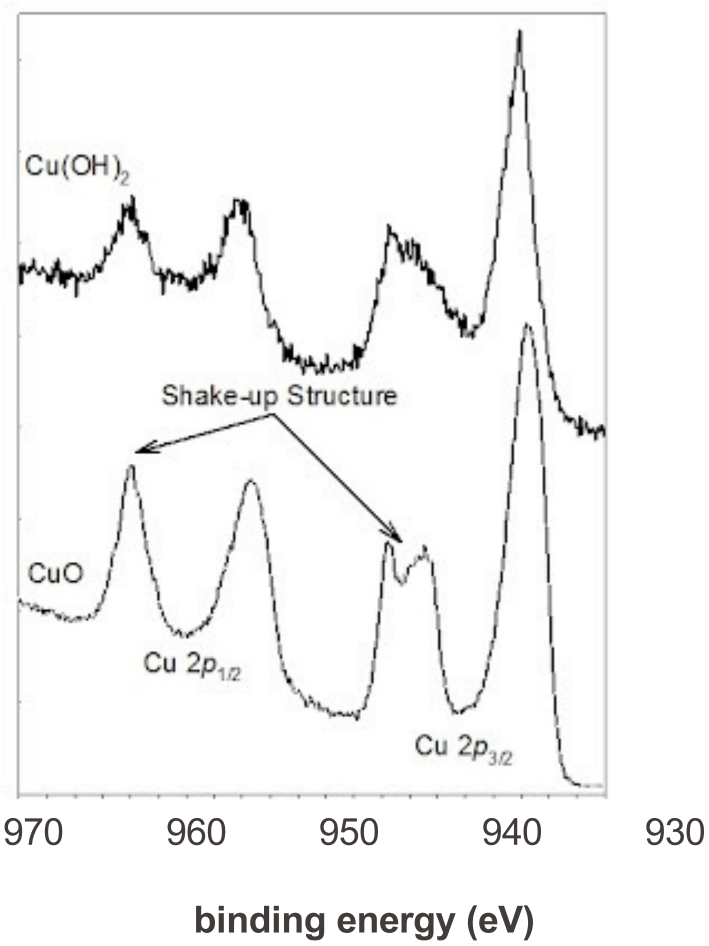
Kinetic Energy:
E(K L1 L2)=E(K)-E(L1*)-E(L2*)

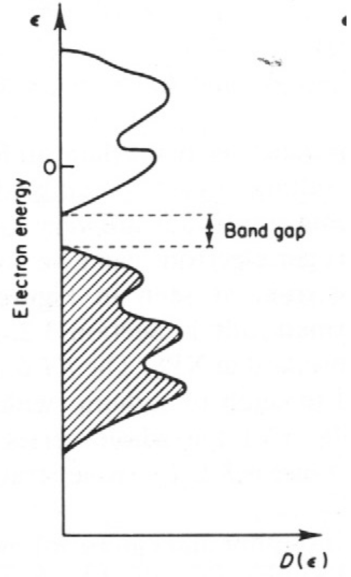
This energy does not depend on the source!!

Auger peaks in XPS



Shake up peaks





During their ejection process core photoelectrons can excite electrons from the valence band to the conduction band and thus loose energy (band gap energy). This generate satellite peaks called “shake up” peaks.

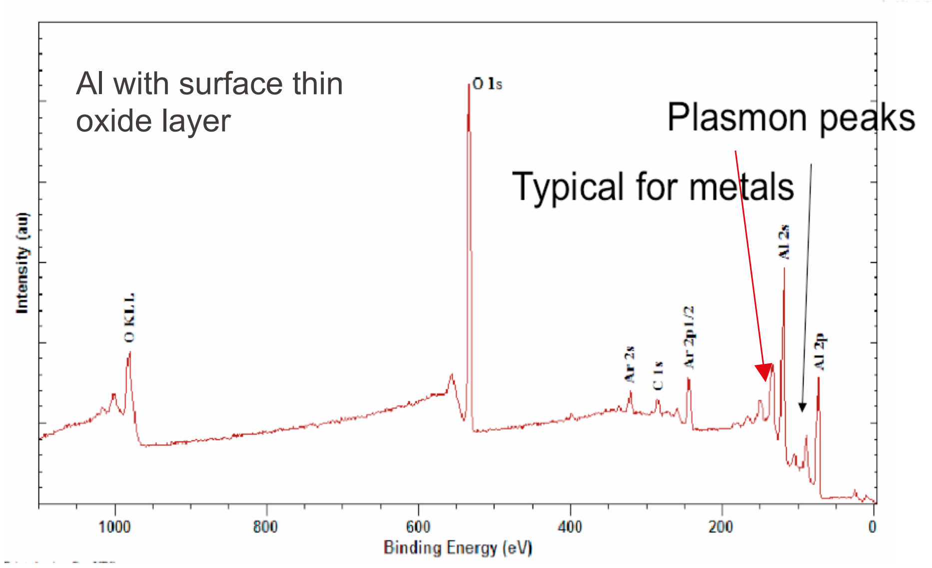
In metals the bands overlap and no discrete satellite peak generates. Instead, peaks in metals are asymmetric.

High density of states at the Fermi level results in the high degree of core level asymmetry.

Plasmon peaks

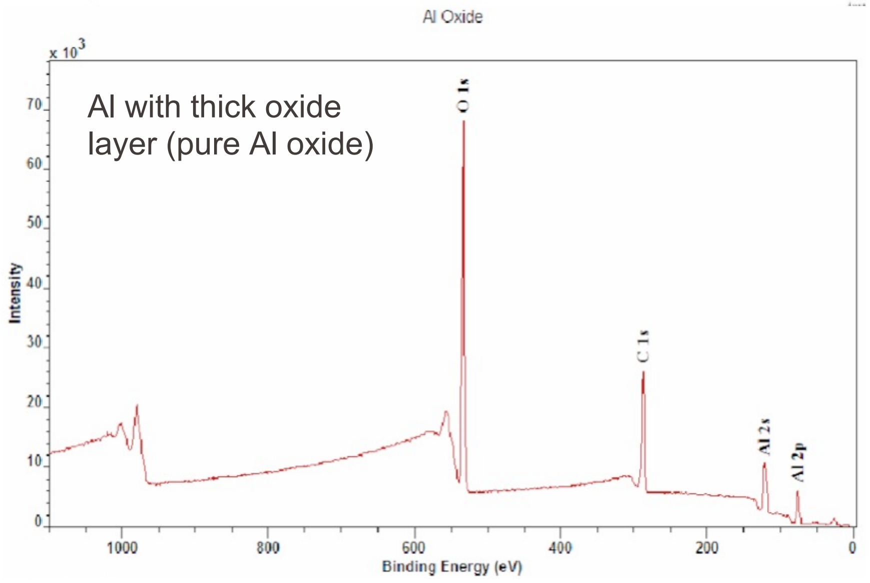
Plasmons are collective vibration modes of ”free” electrons against the positive ion lattice. Photoelectrons excite plasmon vibrations with energies of one to several times the basic plasmon peak.

After long oxidation: There are no plasmon peaks anymore, proving that these peaks are due to metallic electron states.


Al with surface thin oxide layer

Plasmon peaks

Typical for metals


Al with thick oxide layer (pure Al oxide)

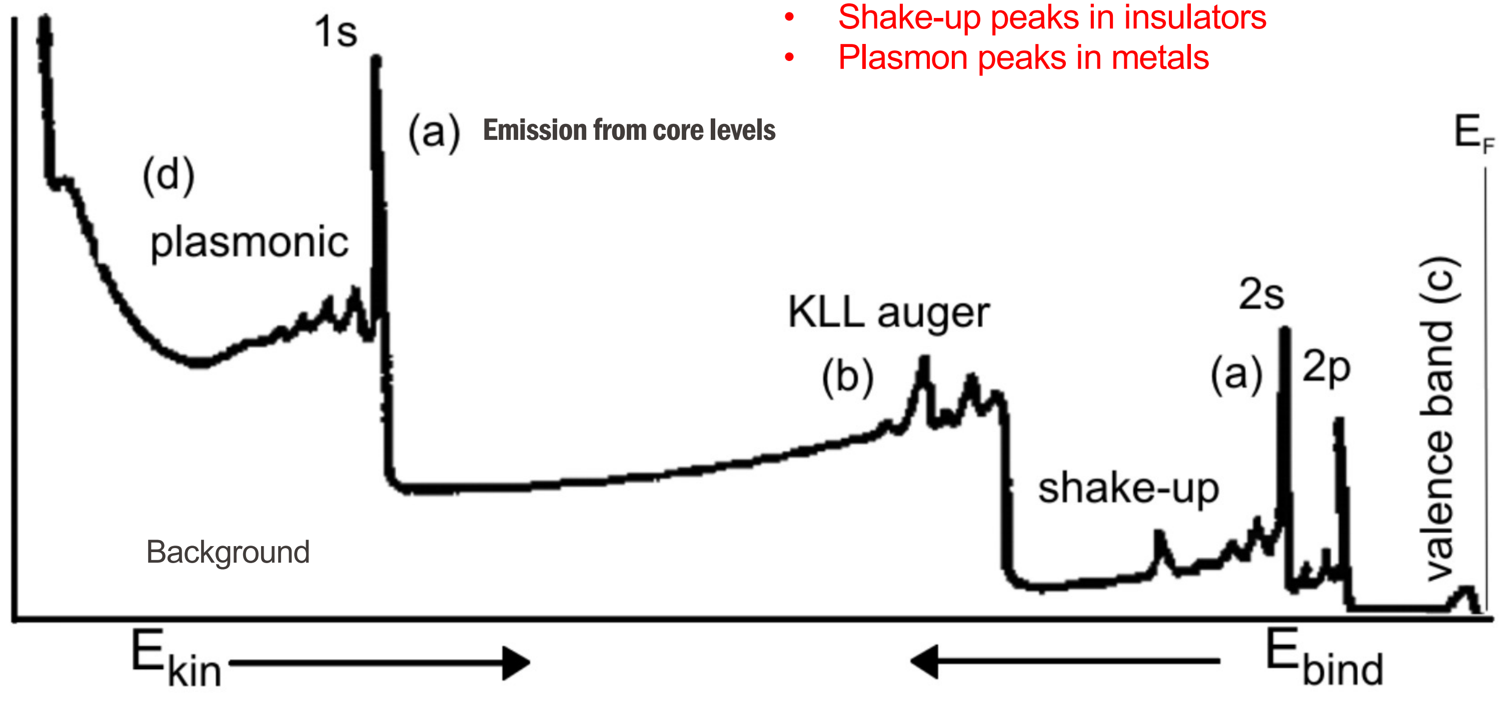
Typical XPS overview spectrum of a solid

Primary structure of spectra

- Background
- XPS Peaks
- Doublets
- Auger peaks

Secondary structure

- Shake-up peaks in insulators
- Plasmon peaks in metals



1.- Introduction XPS vs other techniques
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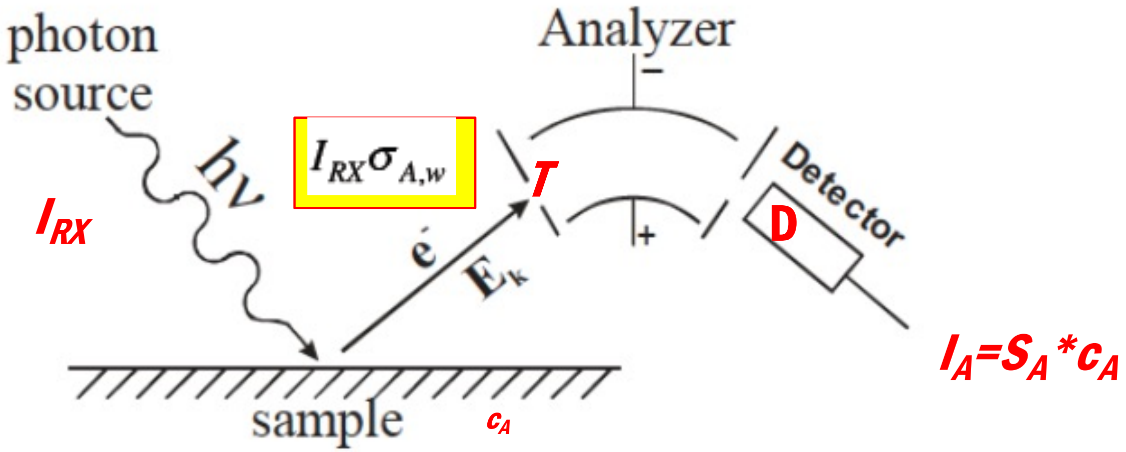
The intensity I_A of a XPS peak



$$I_A = I_{RX}\sigma_{A,w}\cdot T(E_{cin})\cdot D(E_{cin})\cdot c_A \equiv S_A \cdot c_A$$

Overall Sensitivity

Concentration of atom type A

Detectivity of electron multiplier (charge per electron detected)

Transmission factor of apparatus (likelyhood that p.e. arrives at entry of analyzer)

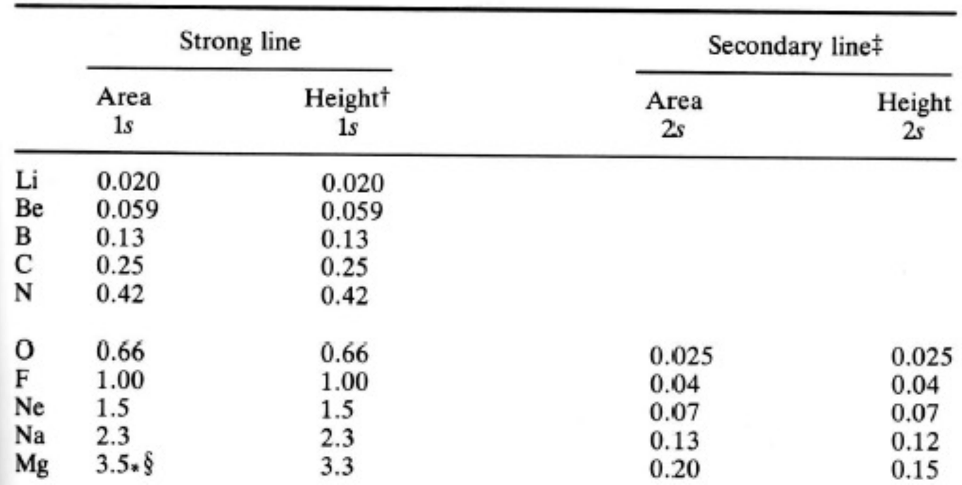
Cross section for photoelectron creation

X-ray photon flux

Quantitative analysis (homogeneous solids)

$$c_A = \frac{I_A / S_A}{\sum_{k=1}^{n} I_k / S_k} = \frac{I_A / RSF_A}{\sum_{k=1}^{n} I_k / RSF_k}$$

Relative atomic **S**ensitivity **F**actors (RSF)



	Strong line		Secondary line‡	
	Area 1*s*	Height† 1*s*	Area 2*s*	Height 2*s*
Li	0.020	0.020		
Be	0.059	0.059		
B	0.13	0.13		
C	0.25	0.25		
N	0.42	0.42		
O	0.66	0.66	0.025	0.025
F	1.00	1.00	0.04	0.04
Ne	1.5	1.5	0.07	0.07
Na	2.3	2.3	0.13	0.12
Mg	3.5*§	3.3	0.20	0.15

- The RSF are instrument specific, essentially because of different T and D factors.
- For the doublets one has to clarify if the RSF concerns both peaks together, or separately (both exists).
- Cross sections are affected by the composition of the solid, so that reliability of the RSF method is approx. 5%.

Cross-section as critical parameter

- The cross-section is the most important parameters influencing relative sensitivity factors.

- Detection limits are typically 0.1 atomic % for elements with low cross-section core levels.

Chemical State Analysis

- Although core level BEs are sufficiently unique to allow the clear-cut identification in most of the cases, they are not fixed values.
- Different chemical environments give rise to different core levels. These core level shifts give rise to “Chemical Shifts”.
- The chemical shifts range for any element is quite small, typically less than 10eV and peak widths are the order of 1eV, the **dynamic range is rather poor.**

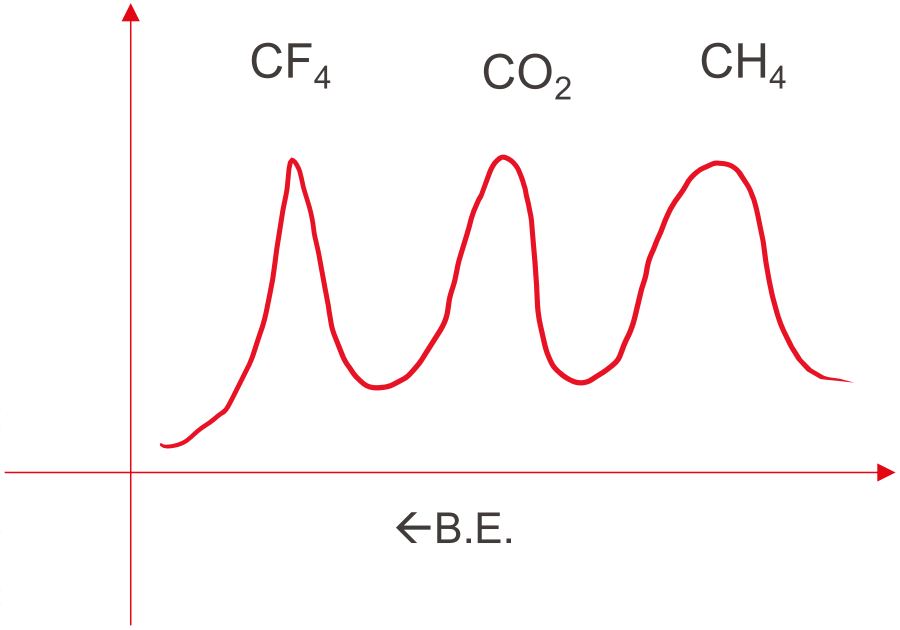
For data analysis this has some repercussions

1. The binding energies can overlap and need to be resolved (Curve fitting needs understanding of different line shapes
2. Background selection is important

Chemical State Analysis

CH_4 will have low BE

CF_4 will have high BE

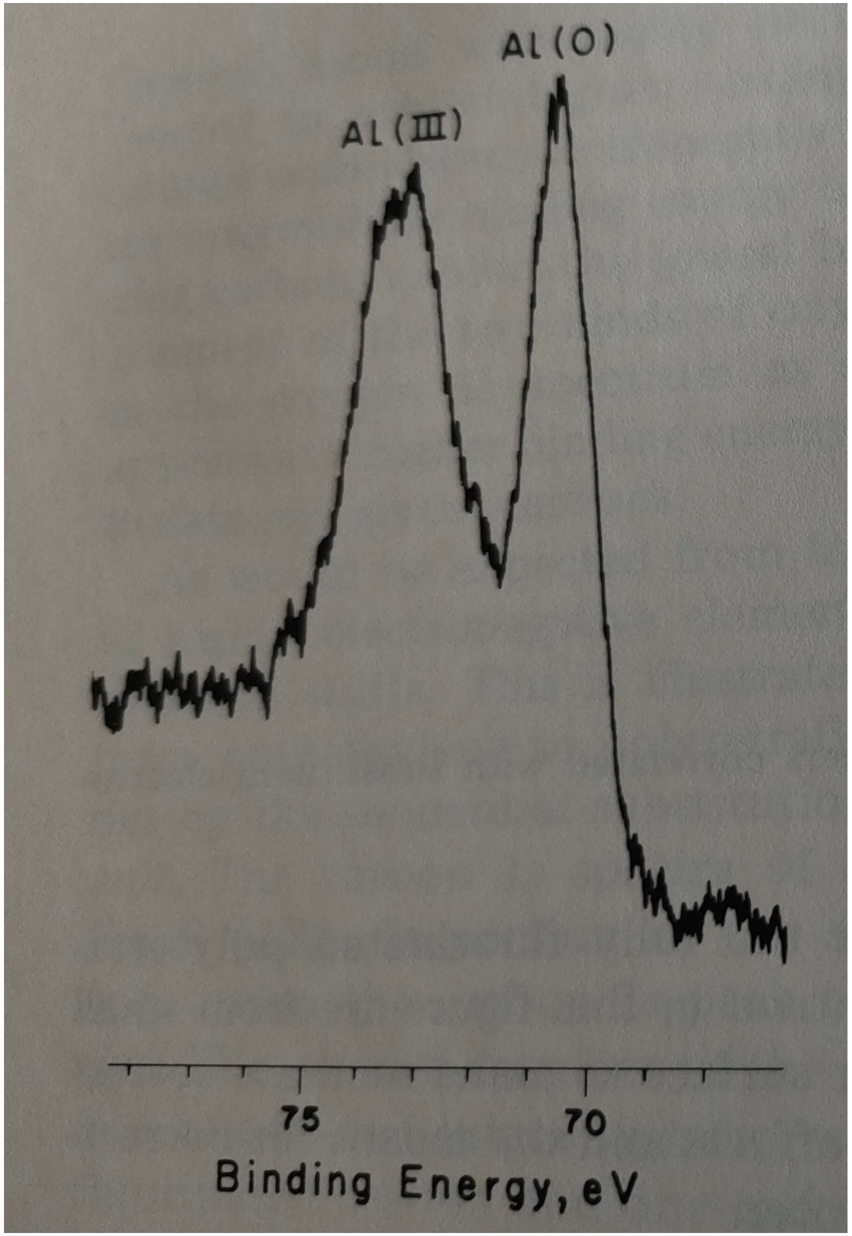


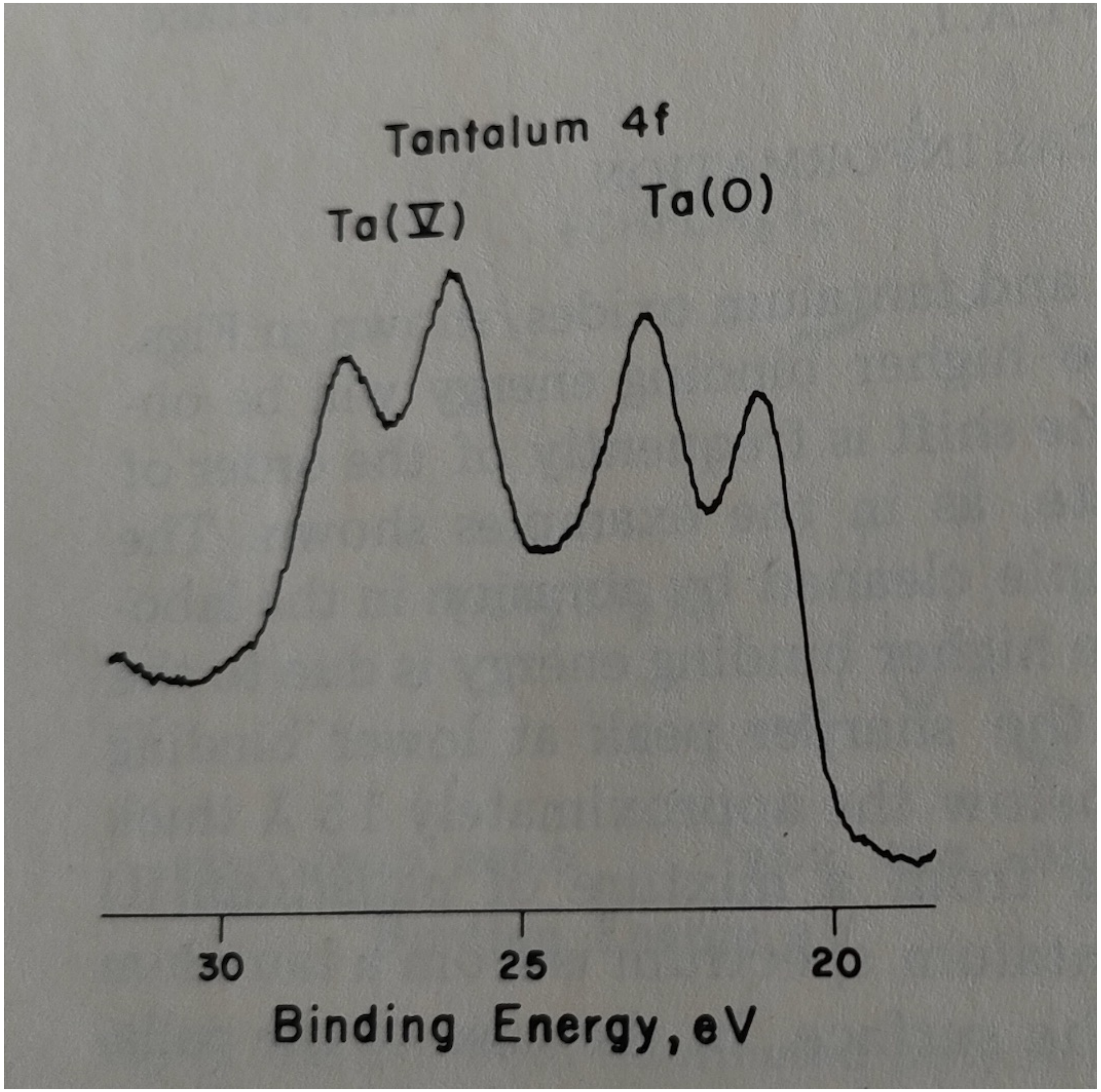
Chemical shift is also related to overall charge of the atom.

Reduced Charge → Increased Binding energy

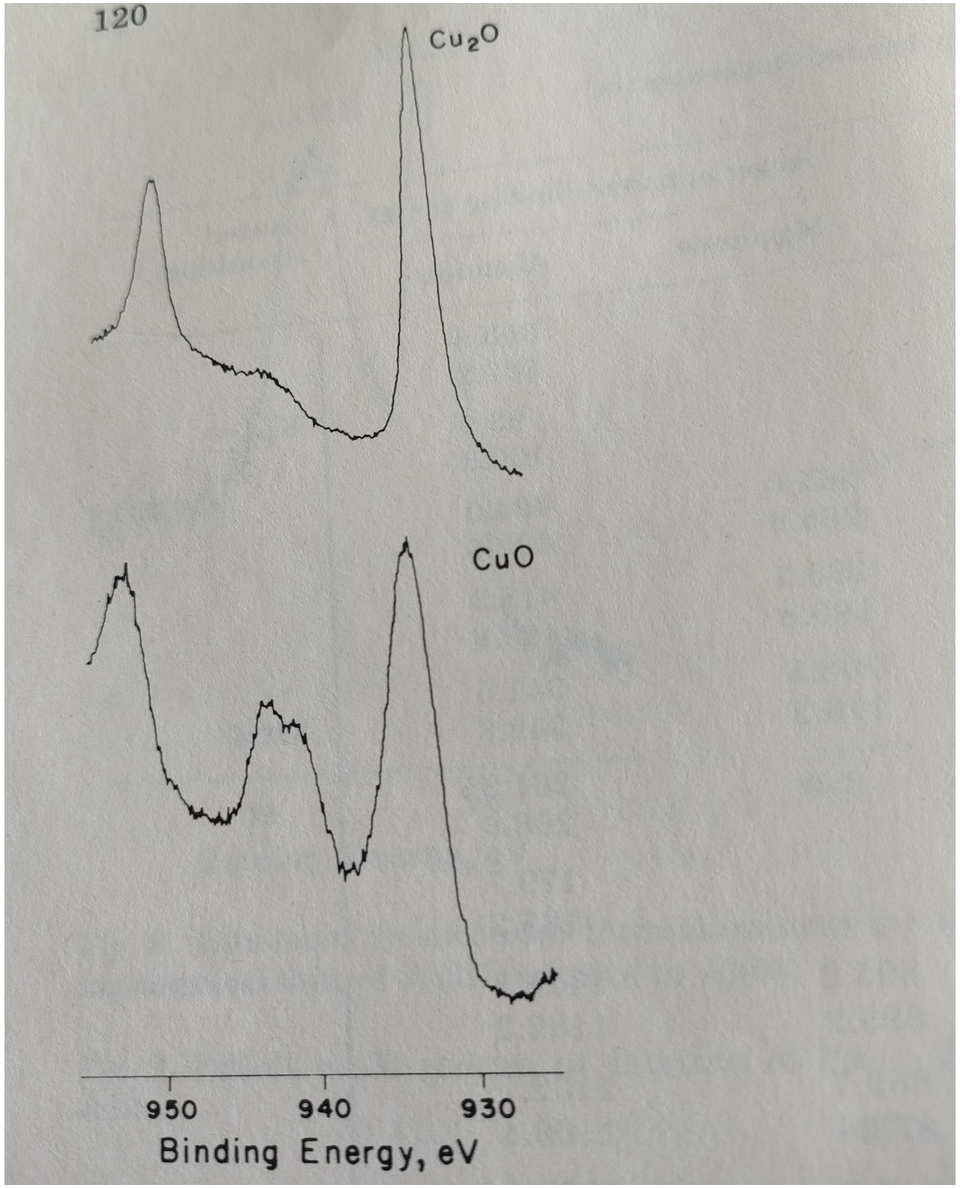
- No. of substituents
- Substituent electronegativity
- Formal oxidation state (depends on ionicity, covalency of bonding)

E


AL(III)
AL(O)
75
70
Binding Energy, eV


Tantalum 4f
Ta(V)
Ta(O)
30
25
20
Binding Energy, eV

Copper 2p spetra



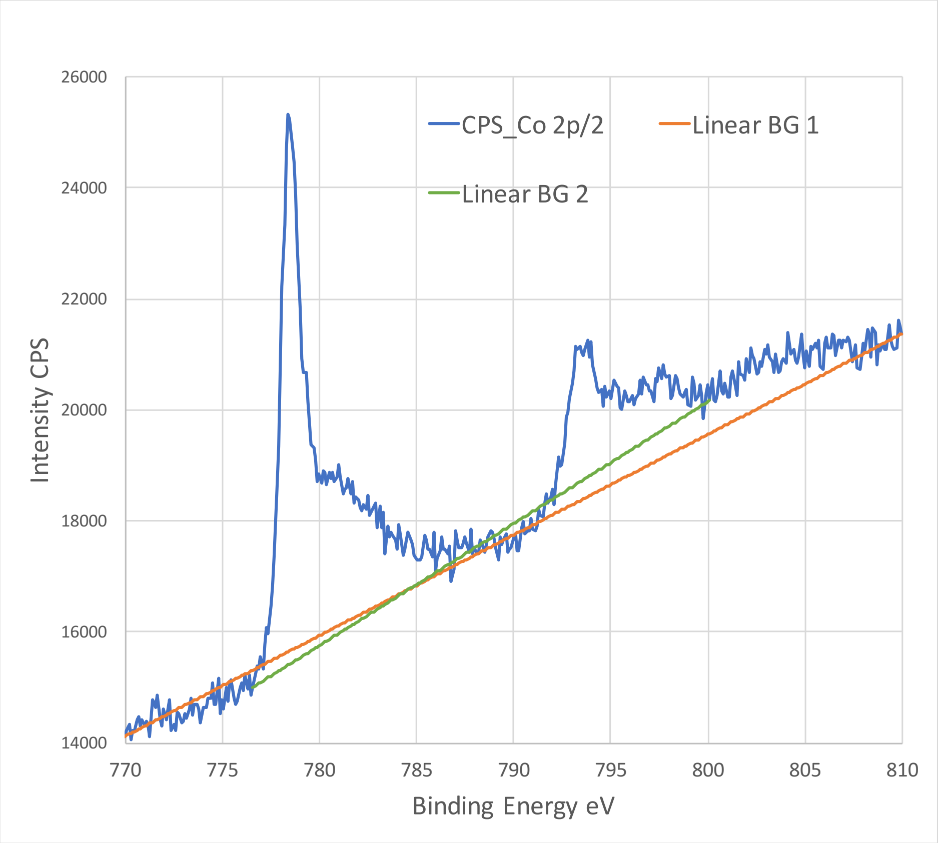
The intensity I_A of a XPS peak

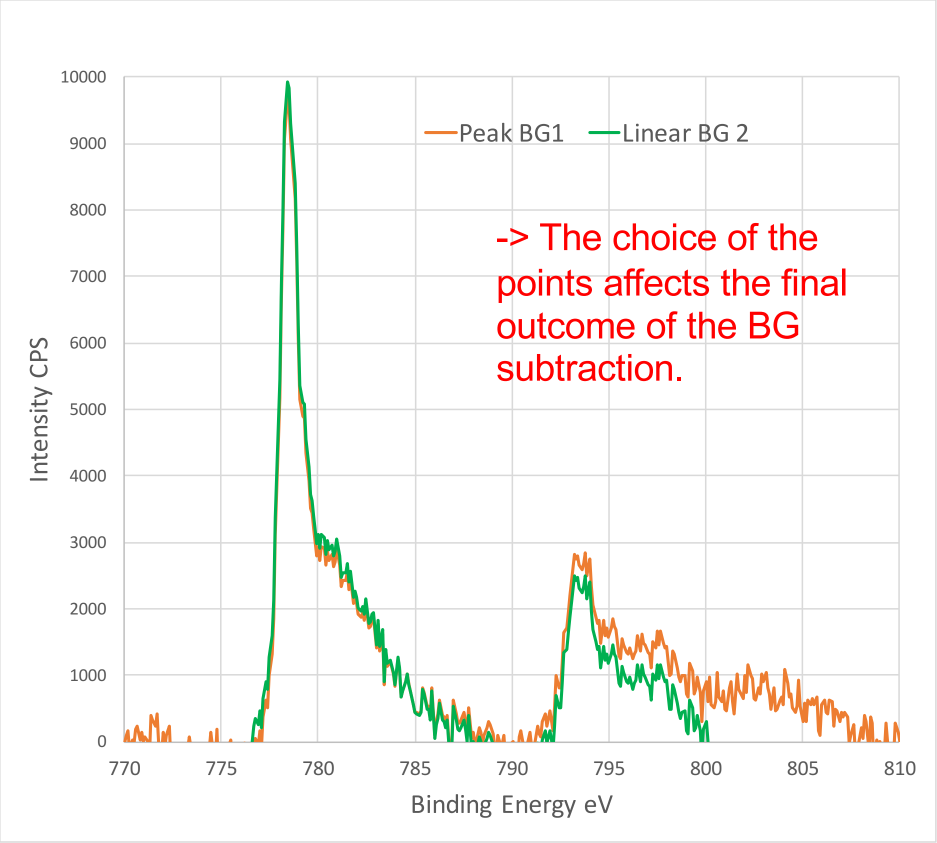
- Background subtraction
  - Linear
  - Shirley

- Peak intensity determination
  - Height
  - Area

Linear background subtraction

BG as a straight line between two suitably chosen points



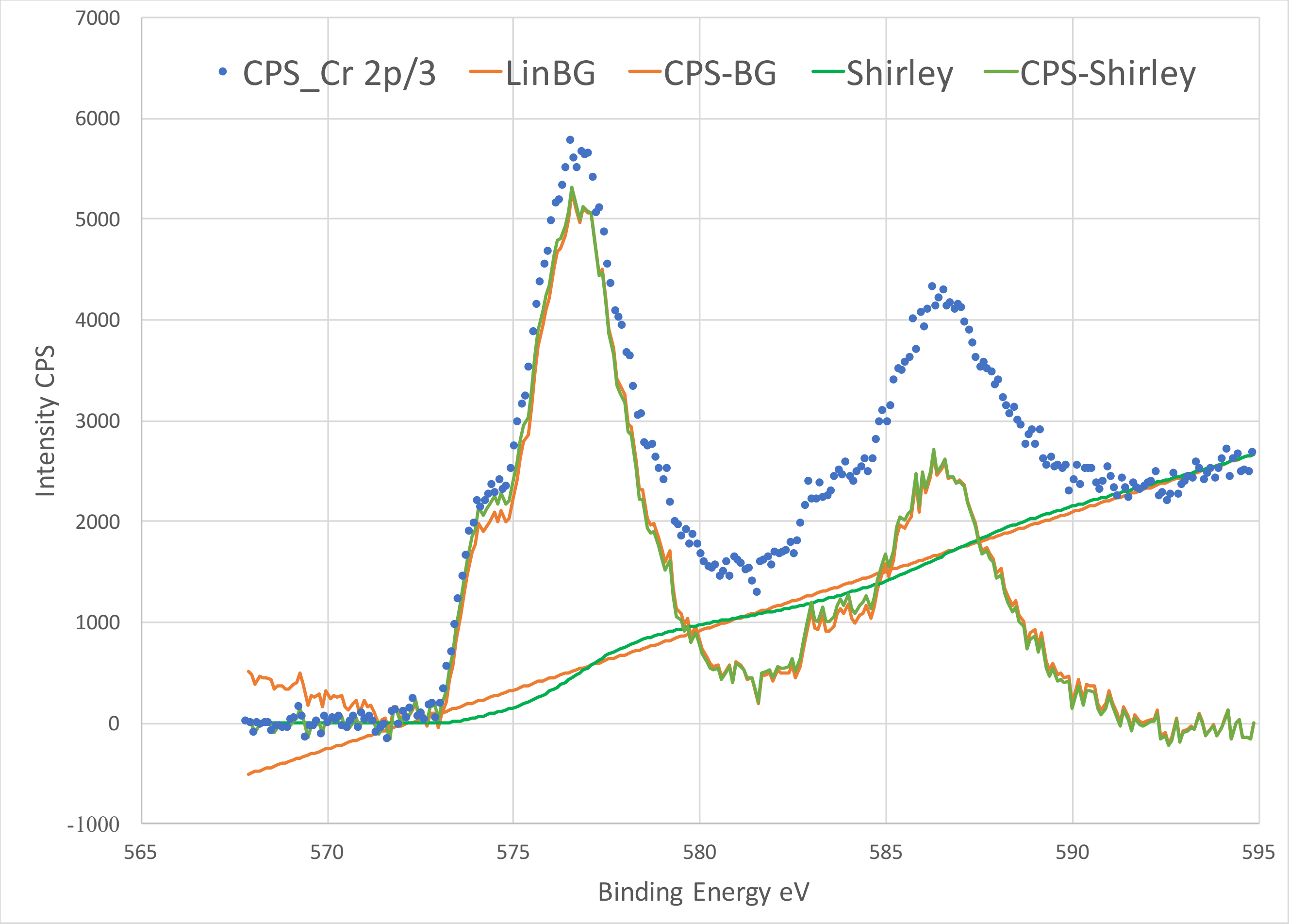


-> The choice of the points affects the final outcome of the BG subtraction.

Shirley Background Subtraction
(comparison with linear BG)

The BG at a point is proportional to the intensity of the total peak area above the background and to higher kinetic energy.

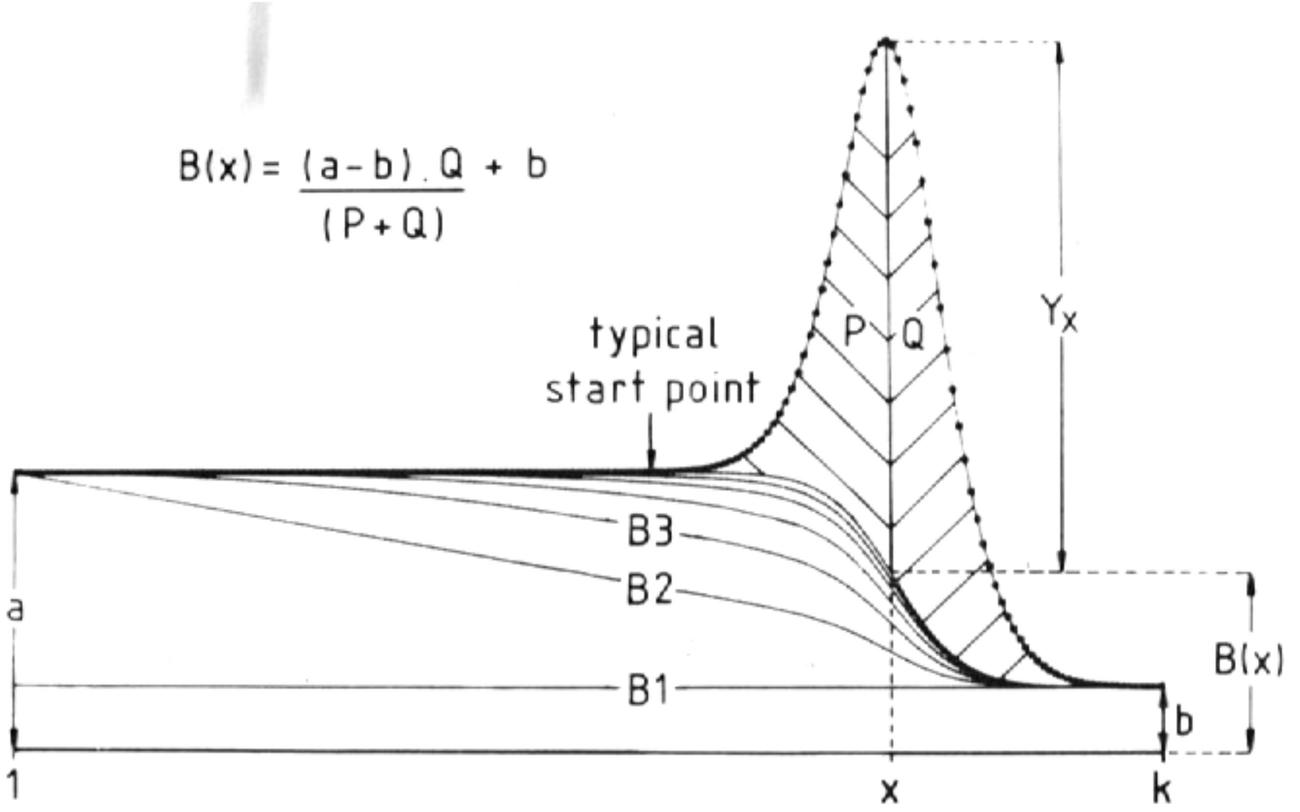
-> The Shirley BG removal has more physical meaning than the linear BG.
It is also highly dependent of the chosen end points (not shown).



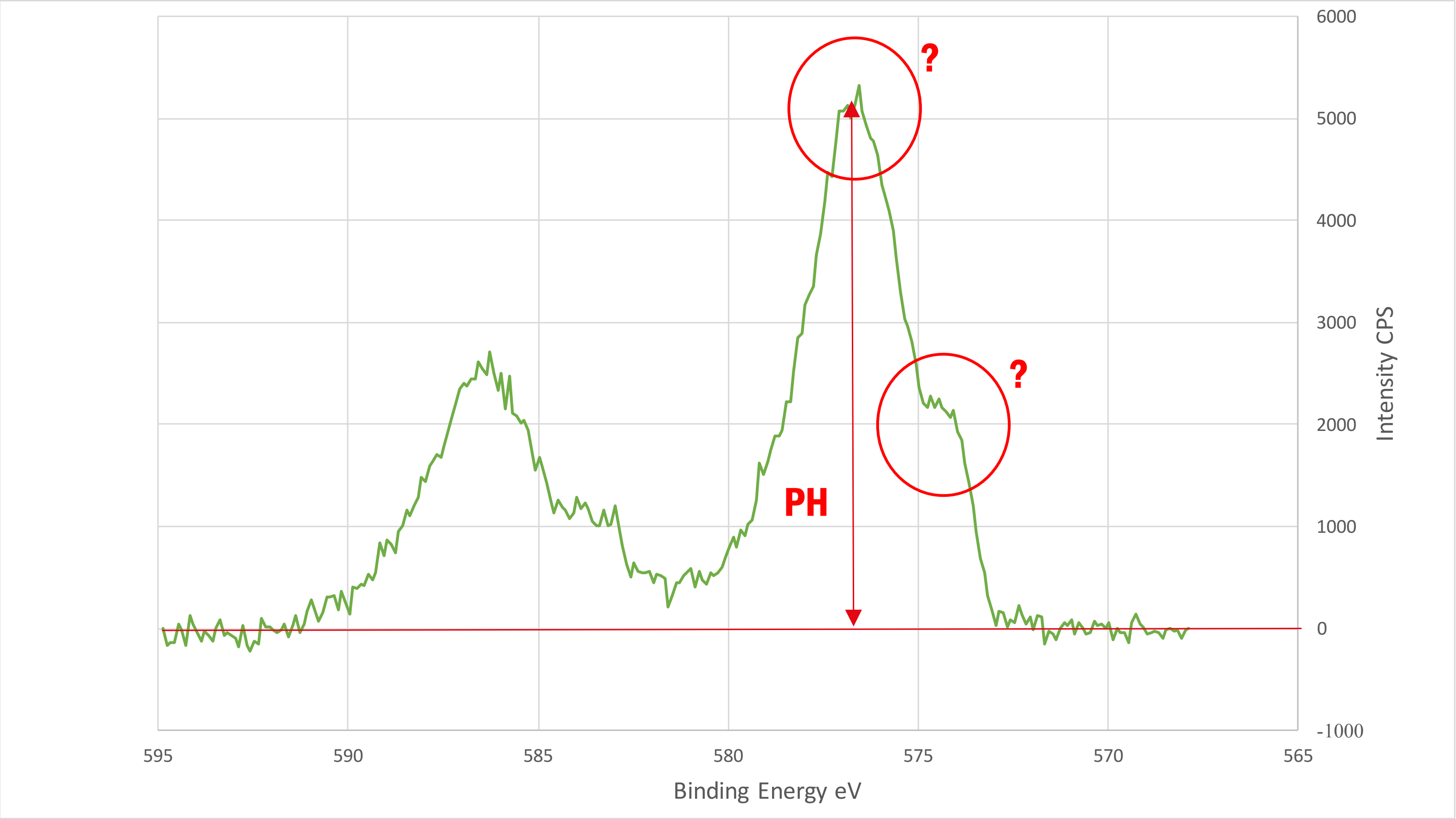
Iterative Shirley Background Calculation

The Shirley BG is an iterative process to be repeated x-times till no significant change occurs (convergence).

Note that the algorithm is essentially divergent so that after a first convergence the obtained results may start diverging.



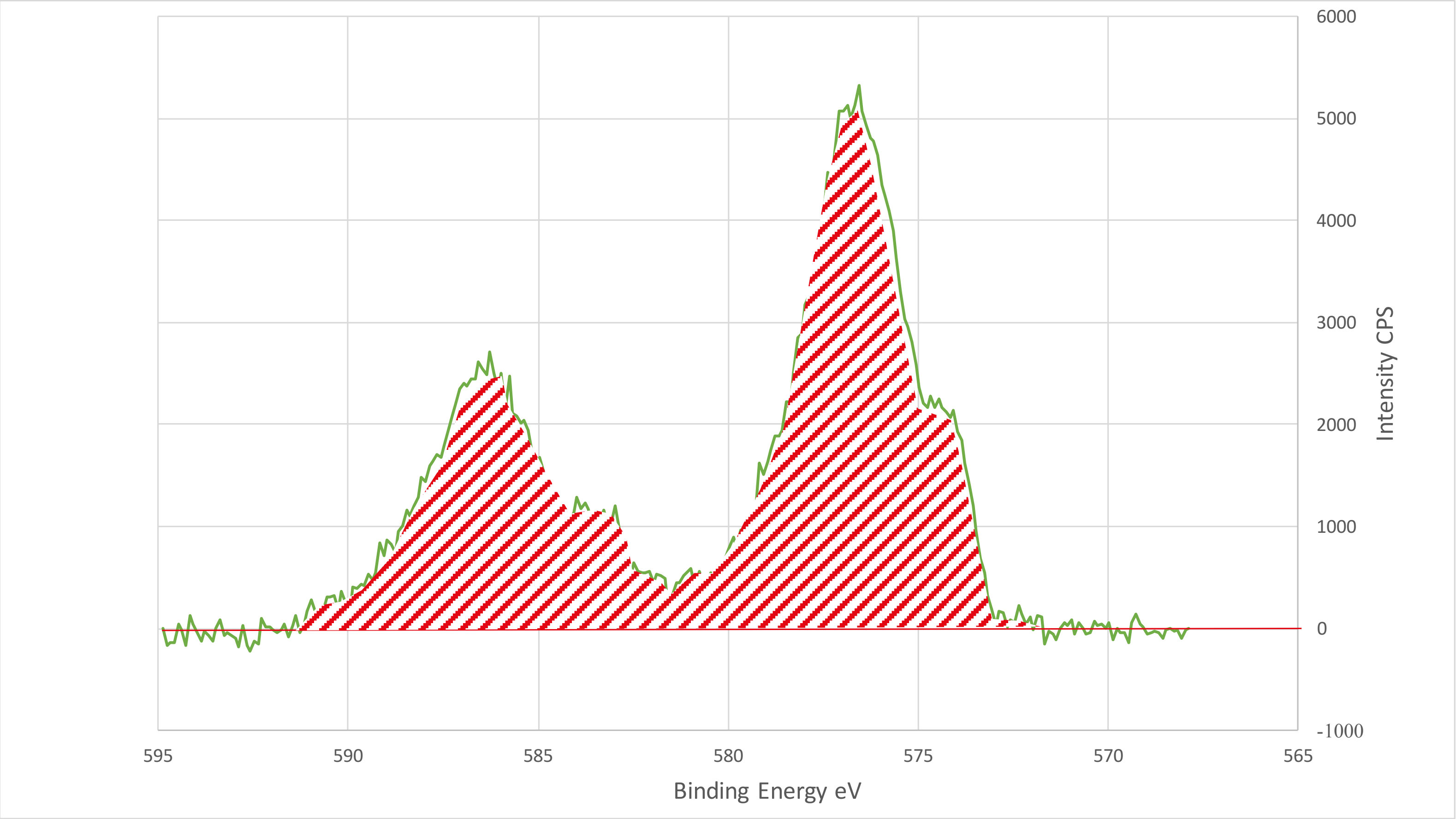
Peak intensity determination



Peak height PH is a simple method but not really reliable especially when an element is present in different chemical states or if secondary features appears (shake up and plasmon satellites).

PH is also affected by noise on the measurement.

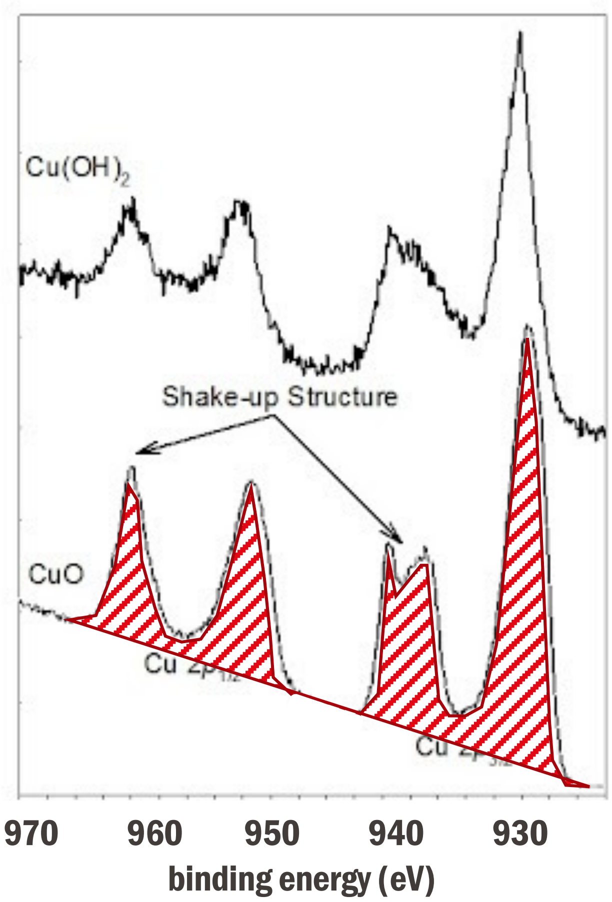
Peak area determination



Peak area determination after BG removal can be efficiently carried out by using computers.

The energy range for peak area calculation must be tuned to the one used to determine RSF (doublet or singlet)!

Peak area determination



When peak areas are determined, secondary structures such a shake up satellites must be considered.

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Different methods for depth profiling

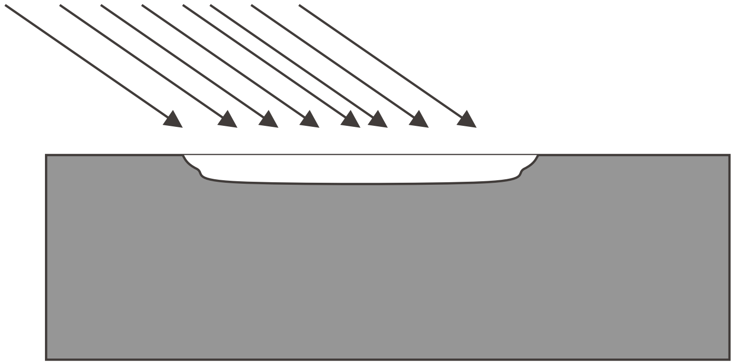
1. Depth profiling by ion etching (ion milling).

2. The finite escape depth of XPS electrons allows for a specific analysis of ultrathin surface layers by angle variation

Escape depth is determined as the depth measured normal to the surface at which intensity drops to 1/e i.e 36.8%, of the original value.

Depth analysis by ion etching

Erosion of the sample surface by an ion beam (ex Ar+ ions)



- 3 nm/min

The etching rate is about constant in an homogeneous solid. With simultaneous XPS spectra -> depth profile.

Sometimes the ionic bombardment changes the surface composition!

Sputter rates

Sputter rates are calibrated using standard thin film of known thickness such as Ta_2O_5 or SiO_2 films
They are usually expressed an thickness of removed material per unit time sputtering, i.e. nm/min

The sputter rate depends on type of ions, their energy and they current

Sputter rated depend on materials:

Material	**Relative sputter rate at 4keV Ar+**
Ta_2O_5	1
Si	0.9
SiO_2	0.9
Pt	2.2
Cr	1.4
Al	1
Au	4.1

A simple ion source

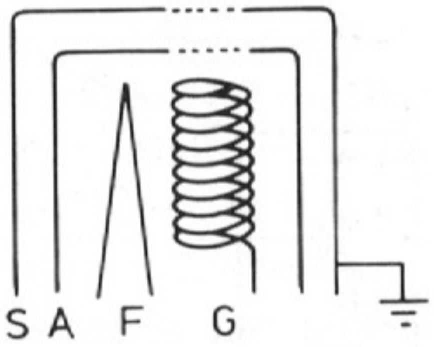


Figure 2.17 Design of an ion gun based on an ionization gauge geometry. The hot filament F emits electrons to a positively biased grid G, within which the electrons make several oscillations before being collected. With argon admitted to the gun at pressures between 10^{-5} and 10^{-4} torr, argon ions are produced by ionization in the grid space. The positive ions are then accelerated from the grid space towards a cylindrical electrode A surrounding the filament and grid, through a potential gradient of about 200 V. The ions pass out into the system through meshes in A and S, an outer screening can at earth potential. The mesh apertures are offset to avoid any contamination from the filament striking the specimen. By floating the filament–grid–accelerator with respect to earth, the ion energy can be varied from 200 to 500 eV. At an argon pressure of 10^{-4} torr, the ion current density on the specimen is about 1 μA/cm^2

Modern ion gun

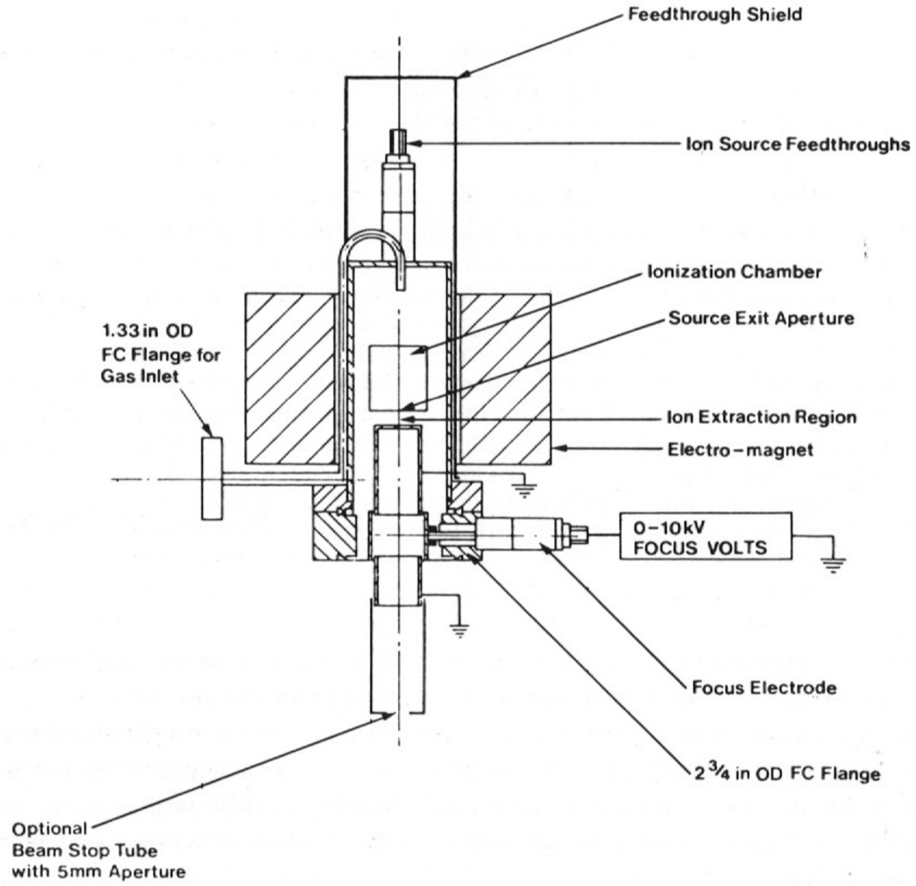
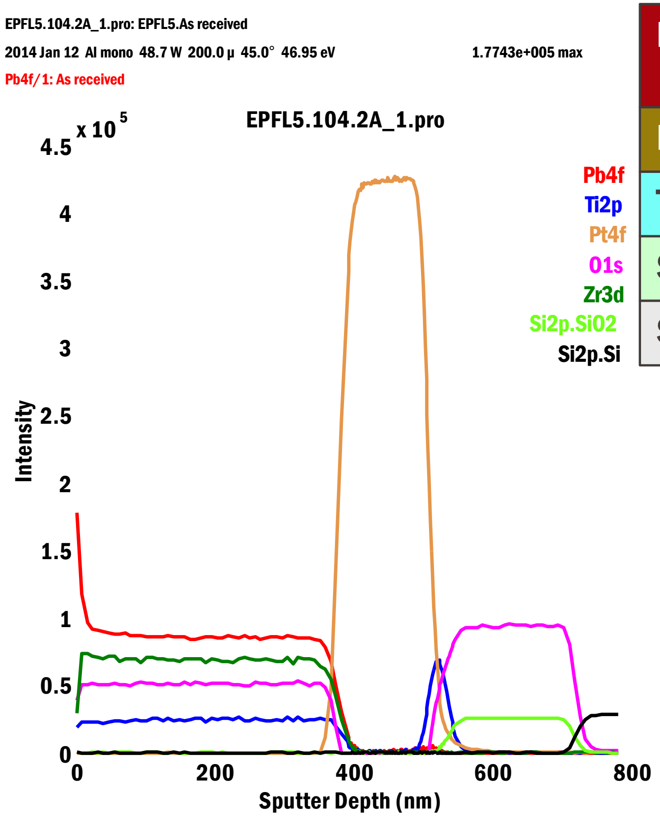


Figure 2.18 Design of an ion gun using a Penning discharge for production of positive ions. Argon gas is fed directly into the back of the gun in this case, rather than being admitted to the whole system, and since its exit speed is restricted the pressure in the interior of the gun is much higher than the pressure of argon in the system. During operation the pressure in the system is between 3×10^{-7} and 3×10^{-6} torr. A high voltage and an axial magnetic field are applied to an ionization chamber in which a discharge is induced and positive ions are extracted from the discharge into a focusing region. Ion energy can be varied betwen 500 eV and 10 keV, and the gun can deliver between 10 and 100 μA into an area of diameter about 5 mm, according to the pressure of argon in the source. (Reproduced by permission of Vacuum Generators Ltd, East Grinstead)

Depth profiling on a multilayer sample



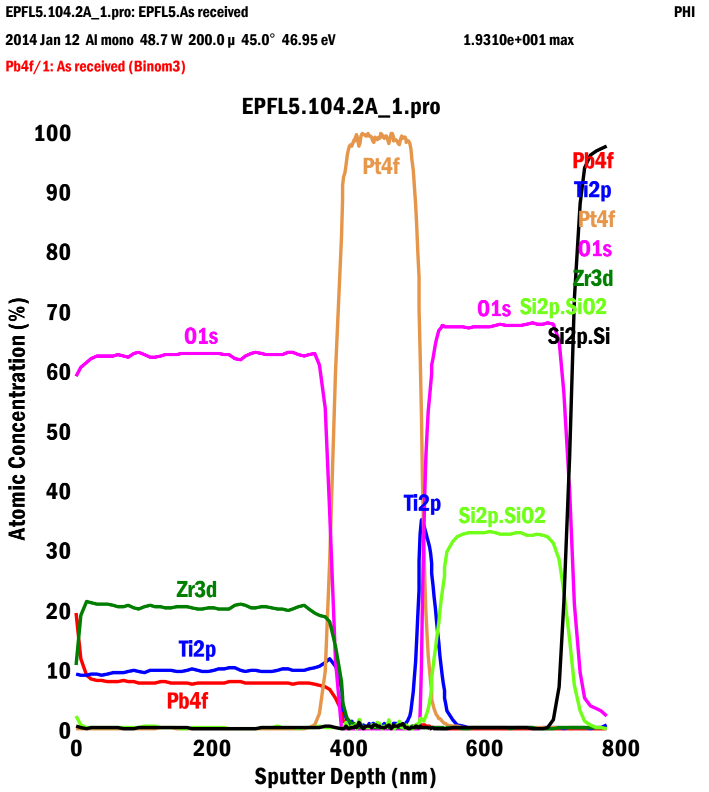
$PbZr_{0.5}Ti_{0.5}O_3$ 1 µm

Pt 0.1µm

TiO_2 30 nm

SiO_2 500 nm

Si

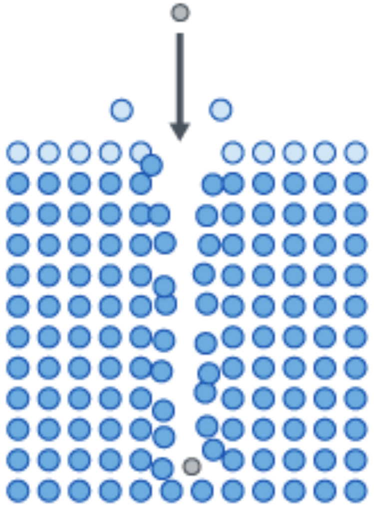


O: 60 %
Pb:20 %
Zr: 10 %
Ti: 10%

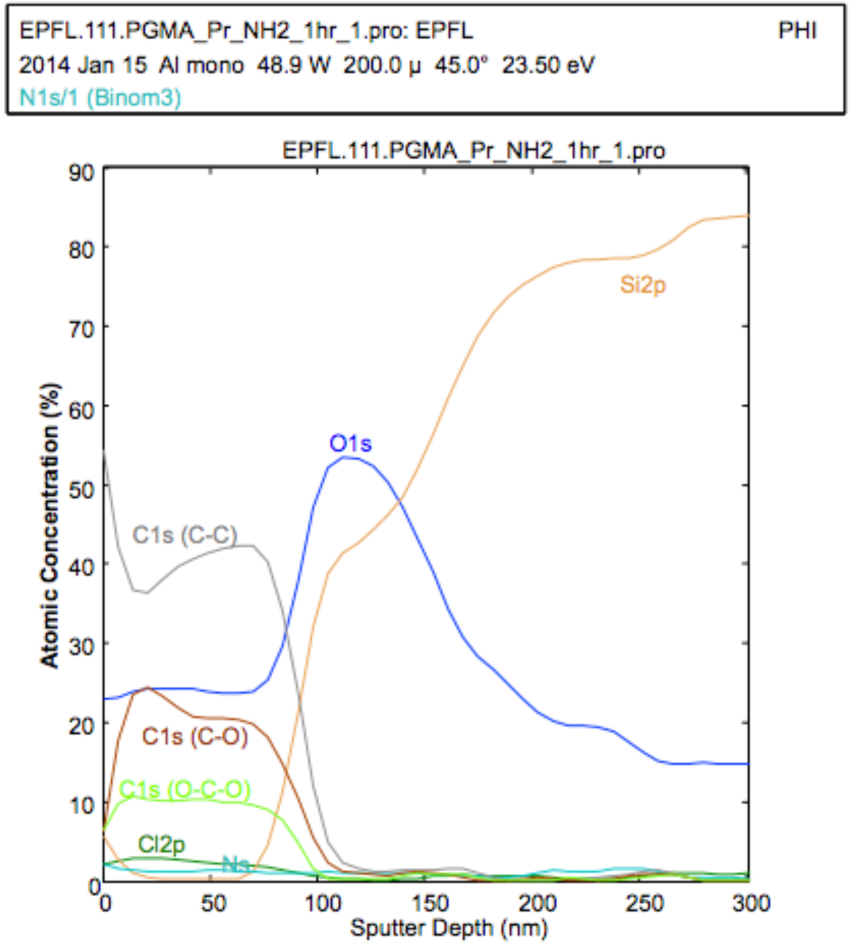
The concentration of Pb is decreased, the one of Zr increased by ion etching

Ion induced artefacts: example PGMA coating on silicon substrate

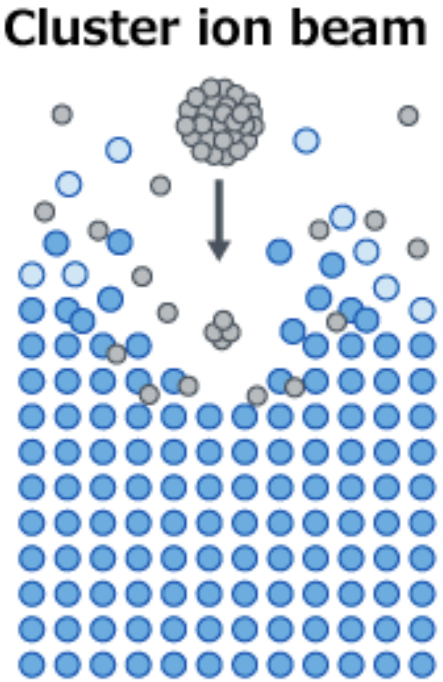
Single atomic ion beam



Ions penetrate deep into the substrate and cause damage → analysis of inorganic materials

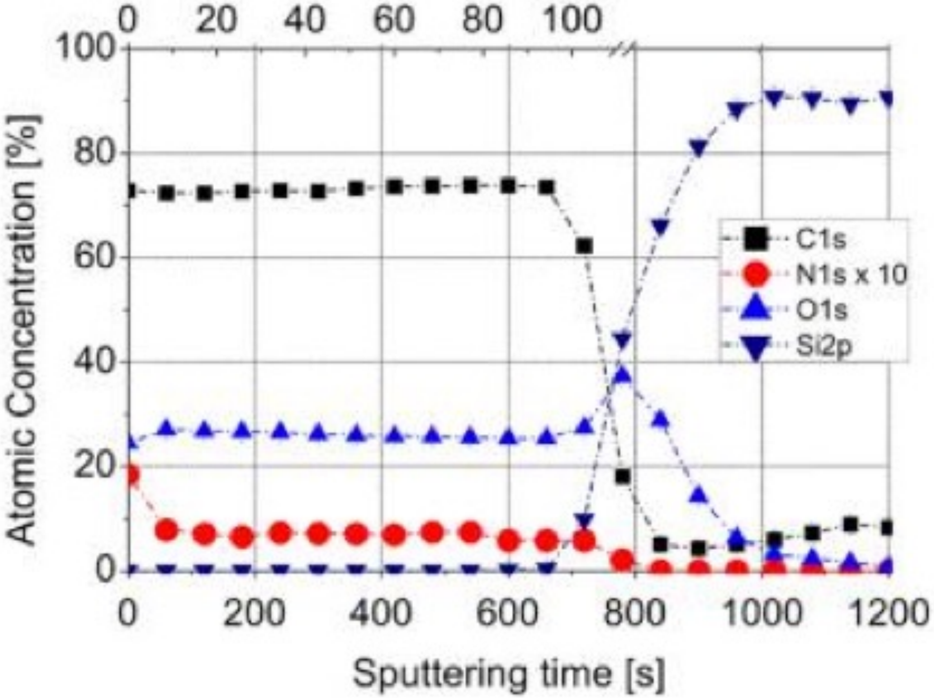


Bombardment by cluster ions limits damage

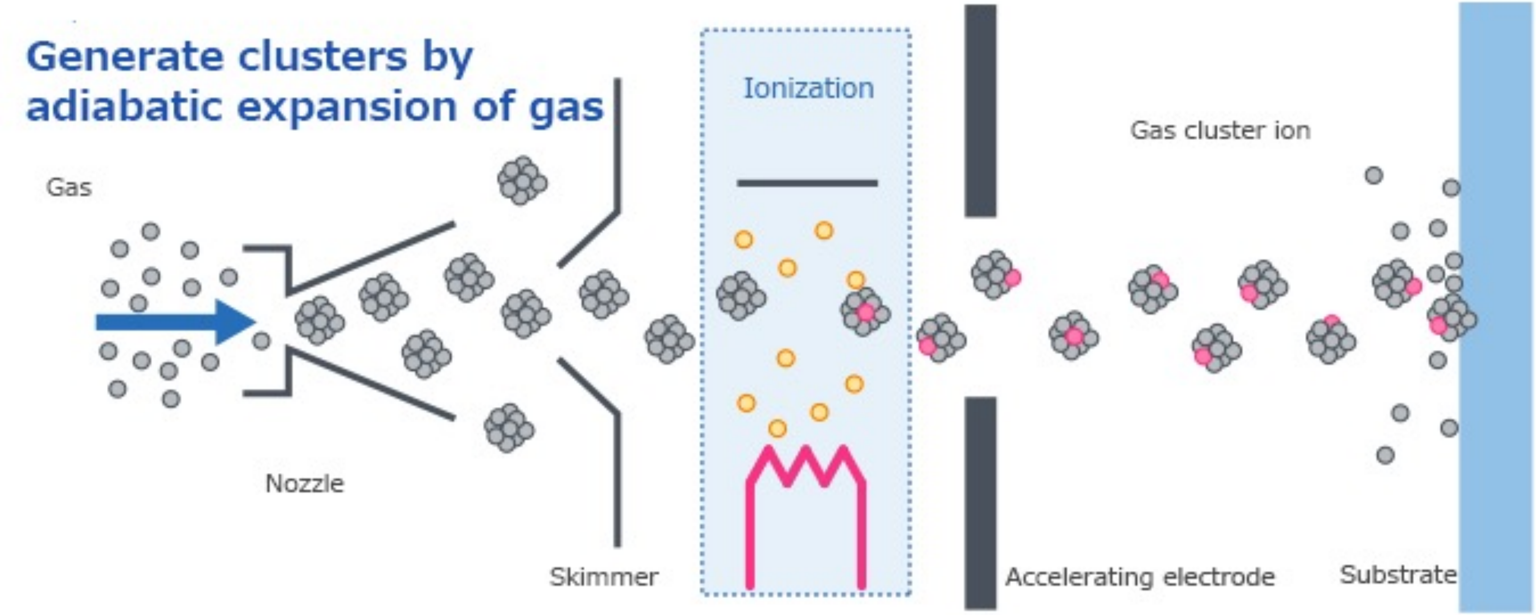


The energy per element is small, limiting damage to the vicinity of the surface → organic

Depth profile of PGMA measured using C60 cluster ion sputtering



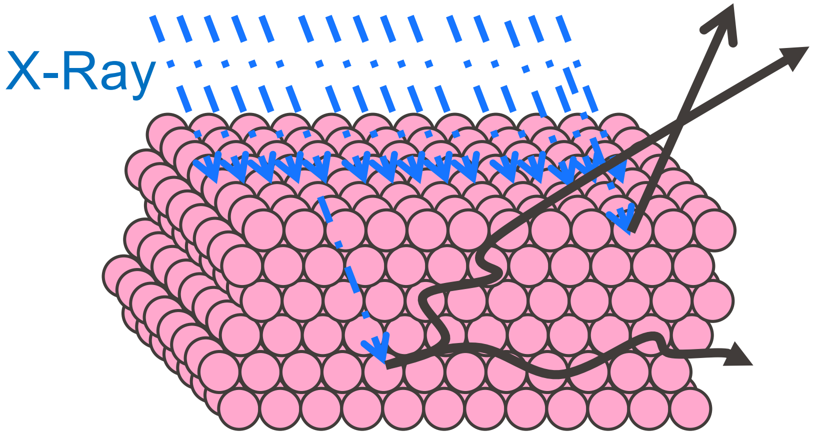
Gas cluster ion gun



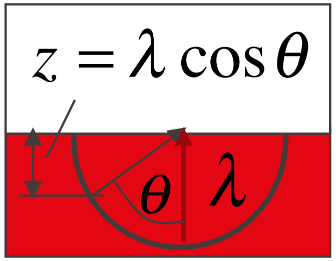
Clusters: C60 molecules,
Ar5000+, Ar10000+

Depth of photoelectrons: concept of the mean free path

The penetration depth of the X-ray beam is much deeper than the escape depth of photoelectrons. The X-ray dose within the volume sending out photoelectrons is thus homogeneous.



The depth (z) and the direction (angle θ) play both a role



$$z = \lambda \cos\theta$$

For contributing to the XPS peak, a photoelectron must not have an inelastic collision on his path to the surface. A mean free path λ is defined for the path free of inelastic collisions. A photoelectron escaping from the surface has its origin (in average) in an hemisphere of radius λ .

Escape depth *z* of electrons

$$Z = \lambda\,(E_{kin})\cos\Theta$$

$$\lambda = K\,a^{1.5}\,(E_{kin})^{0.5} \qquad \text{(empirical)}$$

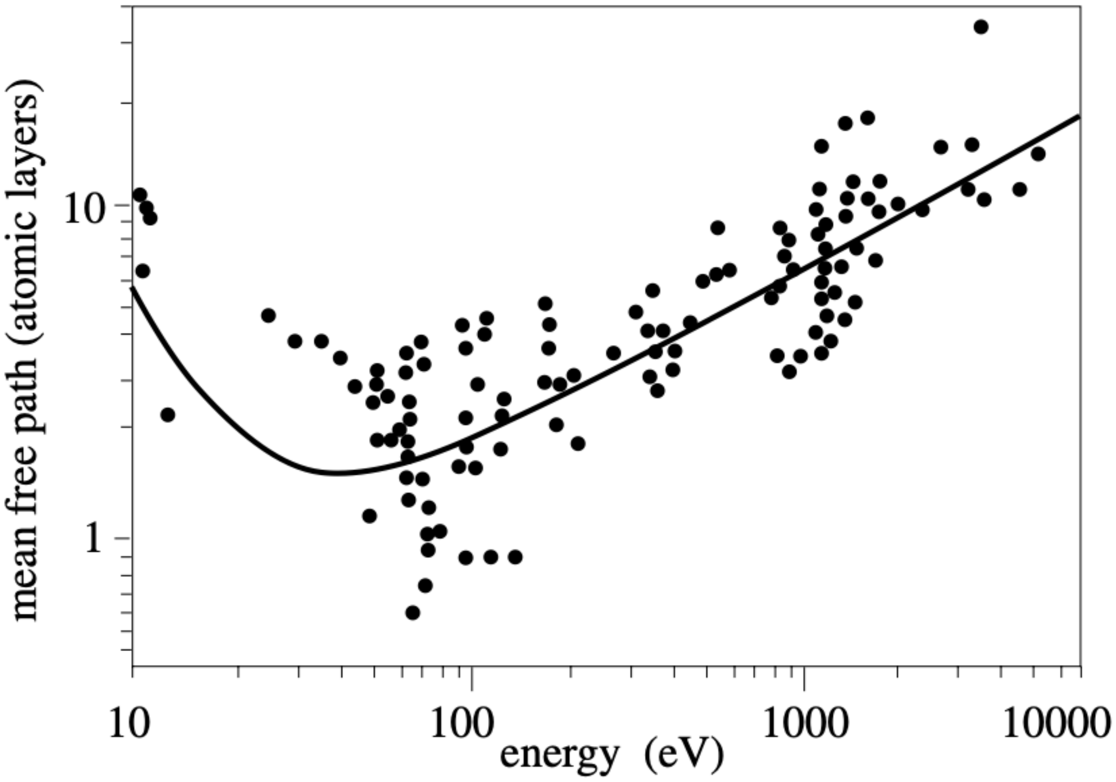
λ = electron mean free path
K = 0.41 (pure elements), 0.72 (inorganic compounds), 0.11 (polymers)
a = thickness of a monolayer (interatomic distance) ***in nm***

$$a = \sqrt[3]{\frac{A}{\rho N_A n}}$$

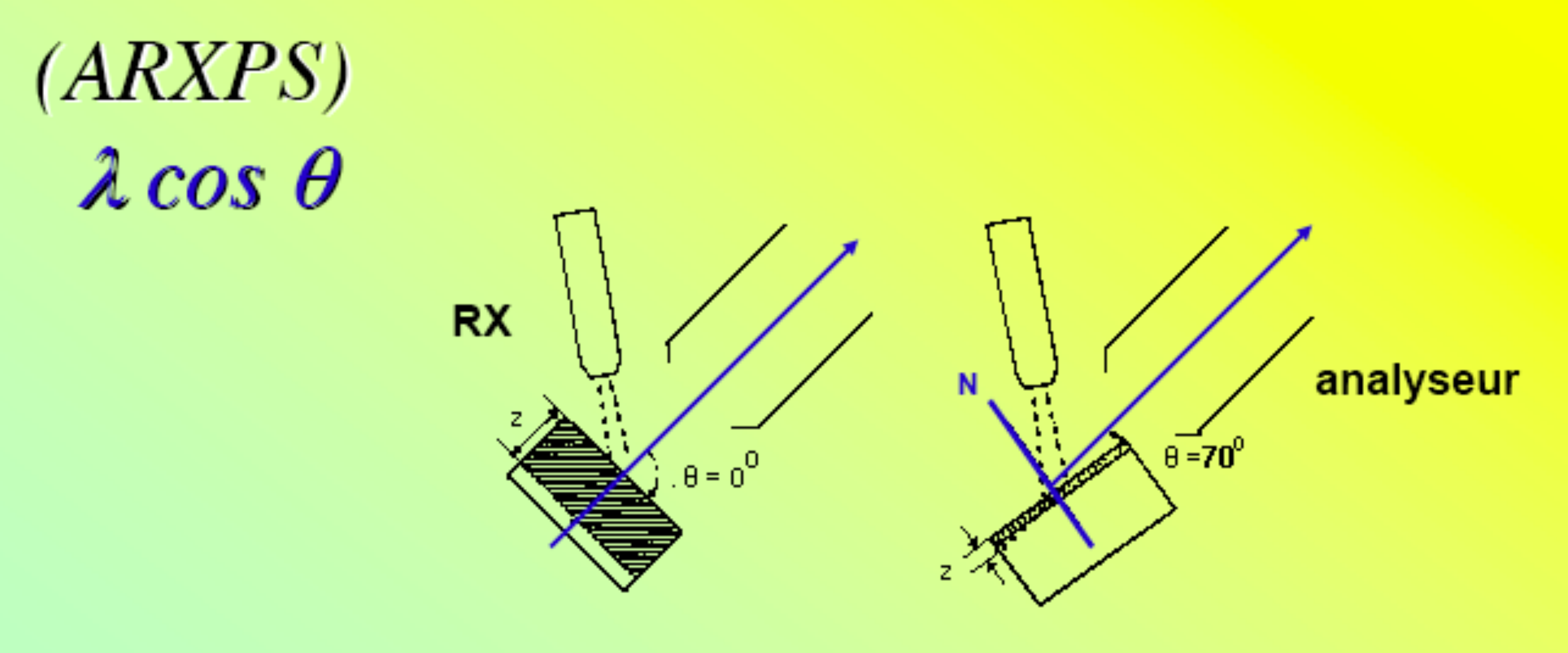
A: molecular weight
ρ: density
N_a: Avogadro number
n: number of atoms in the molecule

More modern and accurate formalisms exist (see TPP formulas)

Variation of the mean free path of electrons in metals as a function of the kinetic energy

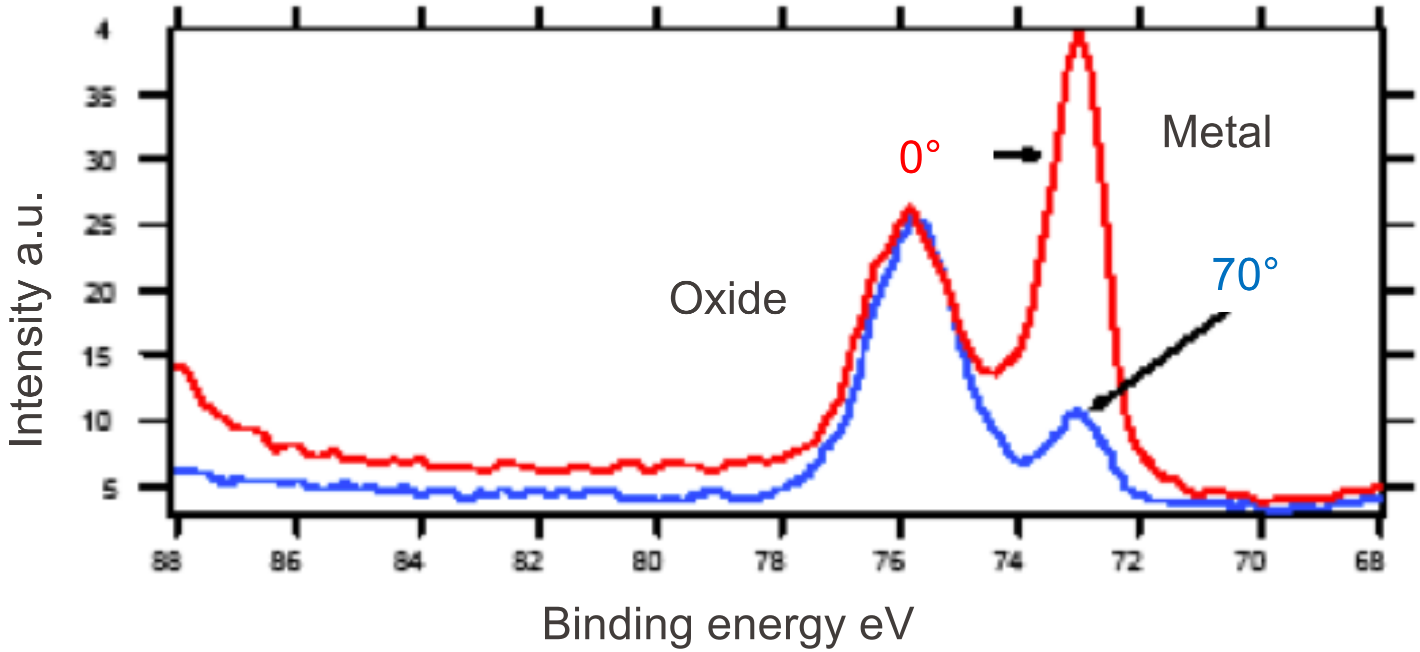


Variation of emission angle

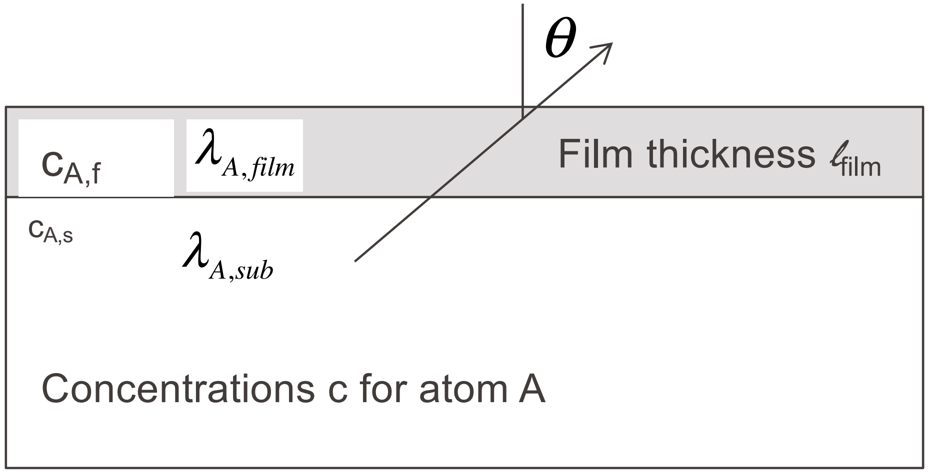


-AR- XPS makes possible electron detection from different depths.
-Provides information about the thickness and composition of ultra-thin films.
-measurements are non-destructive, unlike sputter profiling.

Angle resolved XPS analysis of Al_2O_3 film on Al substrate.



Determination of film thickness from XPS measurements



The intensity emitted from the substrate is attenuated by the $\exp(-\ell_{film} / \lambda_{A,film} \cos\theta)$ factor

This allows for the determination of the film thickness if $\ell_{film} < \lambda_{A,film}$

$$\ell_{\text{film}} = \lambda_{A,film} \cos\Theta \ln\left(1 + \frac{I_{Af}\, c_{A,s}\, \lambda_{A,sub}}{I_{As}\, c_{A,f}\, \lambda_{A,film}}\right)$$

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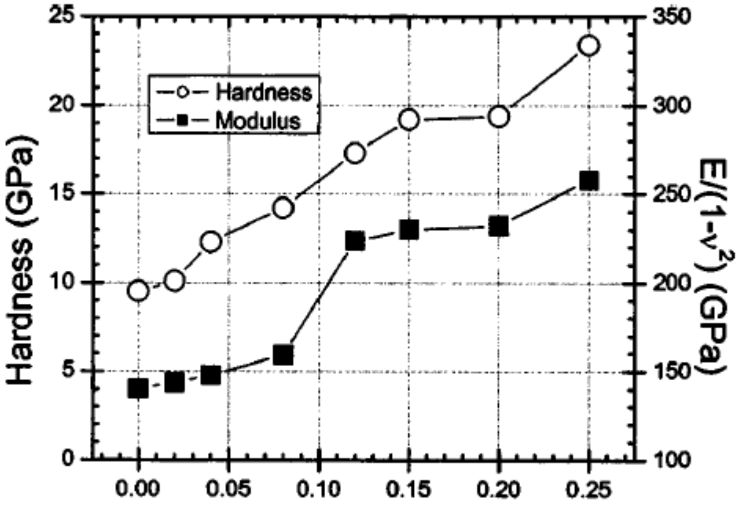
Applications for XPS

- Thin film research
- Corrosion / Tribology
- Organic layers (self assembled monolayers, polymers)
- Alloys
- Aging
- ….

Effect of nitrogen of hardness and chemical state of TiAlCrN hard coatings

Hardness – degree of nitridation



Partial pressure of N2 in reaction gas (Ar+N_2)

Effect of nitrogen pressure on the hardness and chemical states of TiAlCrN coatings

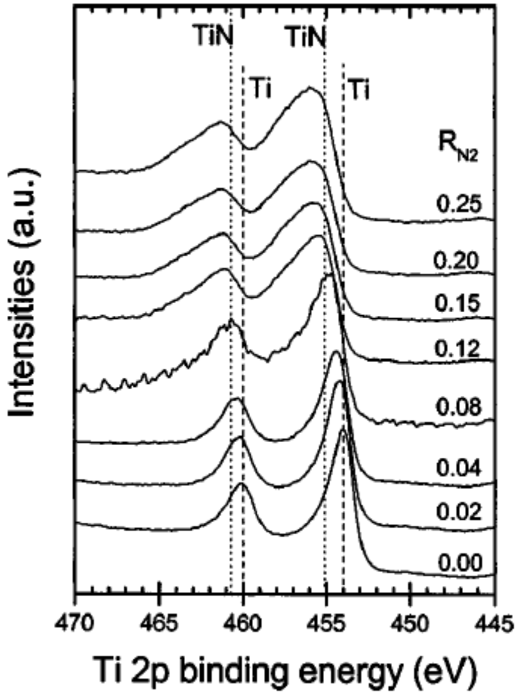


FIG. 6. Evolution of Ti 2*p* XPS spectra as R_{N_2} increases. The binding energies corresponding to TiN and pure Ti were shown for comparison.

Sullivan et al., 2005

J. Vac. Sci. Technol. A 23(1), Jan/Feb 2005 0734-2101/2005/23(1)/78/7/$19.00 ©2005 American Vacuum Society 78

XPS: Summary 1

- The importance of XPS is based on the fact that the energy levels of the electrons in deeper shells are very close to the ones of the free atom, and thus it allows for atom identification.

- The slight displacement of the binding energies due to chemical interactions (chemical shift) allows for an additional information on present chemical bonds.

- One can combine XPS with ion etching in order to obtain a depth profile. Attention: The concentrations can be falsified by sputter yields that differ!

XPS: Summary 2

- Chemical analysis of surfaces from Li to U in ultra high vacuum
- Determination of bonding state
- Identify solid materials
- Depth profiling to some micrometers
- Analyzed surface = 5-10 monolayers
- Sensitivity limit: 0.1-1 at % of a ML.
- Energy resolution: 0.3 eV
- There are various X-ray sources, with and without monochromators
- Charge compensation on insulating samples
- Lateral resolution: 5-100 µm.
- Accuracy in concentration quantification >= 5%