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The Foundation of XPS/XAES

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Outline

- Introduction
  - Terminology, surfaces, types of surfaces.
- The Principles of XPS/XAES
  - Production of photoelectrons, peak labeling, handbooks, books, surface sensitivity, electron escape depth, chemical shift, plasmons, multiplet splitting, shake-up.
- Qualitative Analysis
  - Atomic Electron Levels, identification of elements, energy scale calibration, relaxation effects, Auger parameter, peak widths, lineshapes.
- Quantitative Analysis
  - Sensitivity factors, photoelectron cross-sections, asymmetry, analyser transmission, detection limits, effect of thin overlayers, overlayer thickness on curved surfaces.

Outline

- Instrumentation
  - Vacuum System, X-Ray Sources, electron energy analyzer , ion Gun, Flood Gun, small area analysis, imaging.
- Sample Preparation
  - Bulk, fiber, powder, polymer.
- Data processing
  - Data acquisition, background subtraction, measurement of peak area, curve-fitting.
- Quantitative Compositional Depth Profiling
  - Sputtering, ARXPS.
- Typical Applications of XPS/XAES
  - Carbon materials, silicon materials, transition metal, rare earth element
- Related Technique
  - UPS, REELS, AES, ISS, etc.

Terminology

- XPS-X-ray photoelectron spectroscopy
- ESCA-electron spectroscopy for chemical analysis(Kai Siegbahn)
- PES-photoemission spectroscopy
- HXPS or HAXPES-hard X-ray XPS
- ARXPS-angle-resolved XPS
- TRXPS-total reflection XPS
- NAP-XPS-near ambient pressure XPS
- UPS-ultra-violet photoelectron spectroscopy
- XAES-X-ray induced auger electron spectroscopy

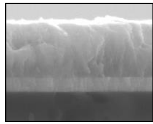
Surface Analysis by XPS

- Identification of elements present at the surfaces-qualitative analysis
- Concentration of elements at the surface-quantitative analysis
- Chemical bonding state of elements-chemistry
- Distribution of elements across the surface-elemental image or map
- Change in composition with depth from the(original) surface-depth profile

Surface

- Processes occurring at the surface are of great practical importance
  - catalysis, coatings, adhesion, corrosion and oxidation, wear and friction, biological implant compatibility, polymers, semiconductors; also at interfaces.
- A surface has ~10^{15}atoms per cm^2.
- If a bulk material is fractured, surfaces are created
  - Initially clean
  - Gases will adsorb on the surfaces if fractured in air.
  - It takes several seconds to adsorb a monolayer in the vacuum Of 10^{-4} Pa.

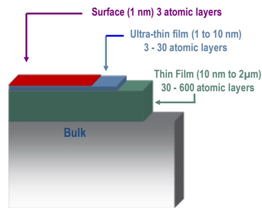
Definition of a Surface



- XPS surface analysis
  - What is a surface?
    - XPS measures
      - Surfaces using XPS and angle resolved XPS
      - Ultra-thin films using XPS and angle resolved XPS (ARXPS)
      - Thin films using XPS in combination with sputter profiling

The modified layer is often far too thin to be characterized with most techniques.

The extreme surface sensitivity of XPS ensures that only the top few nanometers of the sample are analyzed.



Note: Approximate layer thickness only. Actual values depend upon materials

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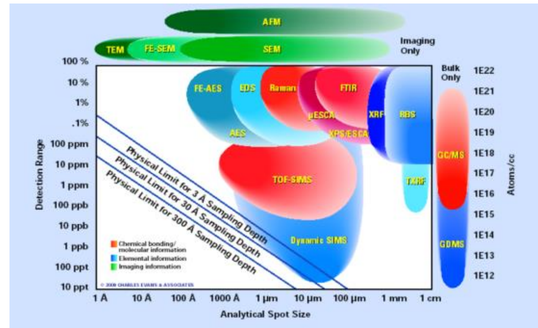
Spot size and Detection Range of XPS



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Historical Background of XPS

1887	1905	1914	1920	1954	1960
Discovery of Photoelectric Effect (Hertz)	Einstein explains the Photoelectric Effect	First Formulation of basic XPS Theory (Rutherford)	Photoelectron spectra of different elements were recorded	Further Development of Method (K. Siegbahn)	First commercial XPS machines available

H. Hertz A. Einstein P. Rutherford K. Siegbahn

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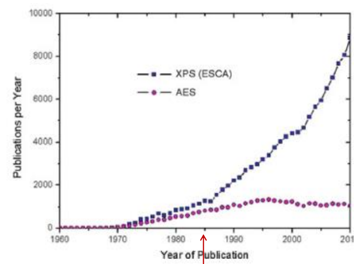
XPS and AES publications per year



multichannel detection and higher analyzer transmission

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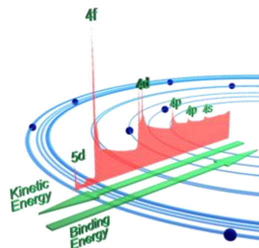
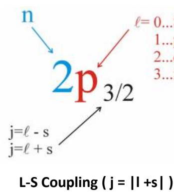
Notation of Atomic Electron Levels





L-S Coupling (j = |l +s|)

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Notation of Atomic Electron Levels

Electron shell levels in spectroscopic (XPS) and X-ray (AES) notation based on principal quantum number, n; orbital angular momentum, l; spin angular momentum, s; and total angular momentum, j

Quantum numbers				Spectroscopic notation (XPS)	X-ray notation (AES)
n	*l*	*s*	*j*		
1	0	+1/2, −1/2	1/2	$1s_{1/2}$	K
2	0	+1/2, −1/2	1/2	$2s_{1/2}$	L_1
2	1	−1/2	1/2	$2p_{1/2}$	L_2
2	1	+1/2	3/2	$2p_{3/2}$	L_3
3	0	+1/2, −1/2	1/2	$3s_{1/2}$	M_1
3	1	−1/2	1/2	$3p_{1/2}$	M_2
3	1	+1/2	3/2	$3p_{3/2}$	M_3
3	2	−1/2	3/2	$3d_{3/2}$	M_4
3	2	+1/2	5/2	$3d_{5/2}$	M_5
					etc.

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Relative Intensities of Doublets

Relative Intensities of Doublets

Subshell	J-values	Ratio
s	1/2	--
p	1/2:3/2	1:2
d	3/2:5/2	2:3
f	5/2:7/2	3:4

The peak area ratios of a core level of an element in different compounds are also nearly the same.

Spin orbital splitting and peak area ratios assist in elemental identification and curve fitting.

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Principle of XPS Analysis

Three-step Model：I) excitation; II) transfer; III) escape.

$$E_B = h\nu - E_{kin} - \Phi_S$$

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Principle of XPS Analysis

$$E_B = h\nu - E_{kin} - \Phi_A$$

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Binding Energies of Elements

Accessible with AlK_{α} Radiation

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XPS Spectrum of Ag

$1s^2\ 2s^2p^6\ 3s^2p^6d^{10}\ 4s^2p^6d^{10}\ 5s^1$

John F M, et al. Handbook of X-ray Photoelectron[M]. Physical Electronics, Inc. 1992.

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XPS Spectrum of Ag

Beamson G, Briggs D. High resolution XPS of organic polymers: the Scienta ESCA300 database. 1992.

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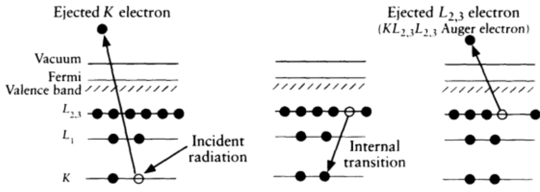
XAES Process

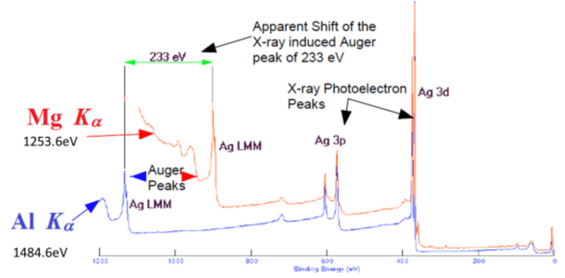
Relaxation of the ionized atom of Figure 1.2 by the emission of a $KL_{2,3}L_{2,3}$ Auger electron



$$E_{KL_1L_{2,3}} \approx E_K - E_{L_{2,3}} - E_{L_{2,3}}$$

XAES Process

$$E_B = h\nu - E_{kin} - \Phi_A$$

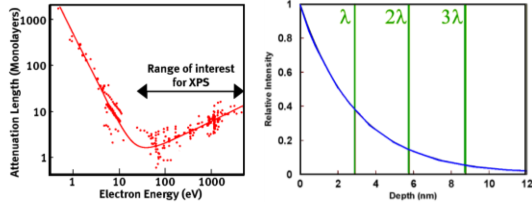


Auger Electron Energies

Accessible with AlK_α Radiation

XPS is a surface sensitive technique

$I = I_0 \exp(-z/\lambda)$



General Curve of λ

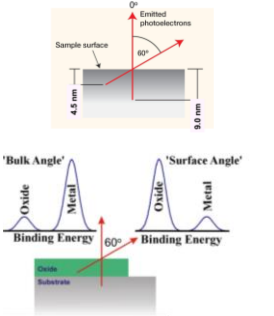
$l \propto \sim E^{0.75}$ (TPP-2M)

95% of the signal < 3 λcosθ

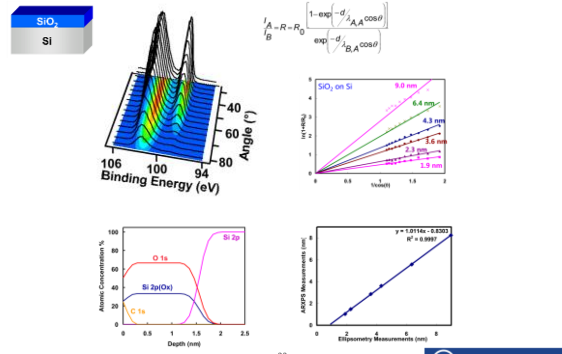
Seah M P, Dench W A. Quantitative electron spectroscopy of surfaces: a standard data base for electron inelastic mean free paths in solids[J]. Surface and interface analysis, 1979, 1(1): 2-11.

ARXPS

- By changing the electron collection angle, the XPS information depth varies.
- Electrons acquired at a grazing ("surface") angle come exclusively from a shallow region of the sample.
- Electrons acquired at a near-normal ("bulk") angle may come from deeper into the sample.
- Non-destructive compositional depth profile and thickness measurement of ultra-thin film.



ARXPS

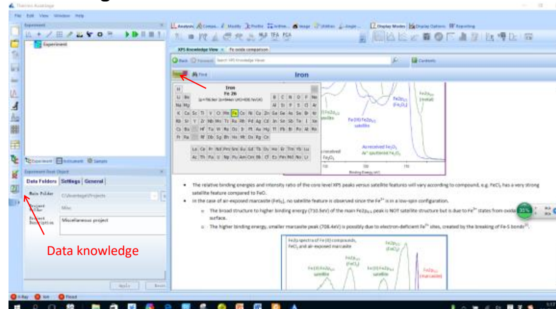


Identification of elements

- Start with the most intense peak
- Compare position with table/handbook
- Looking for confirming peaks
- Continue until all peaks are identified
- Check relative intensities
- Quantification uses the area of one peak from each element detected

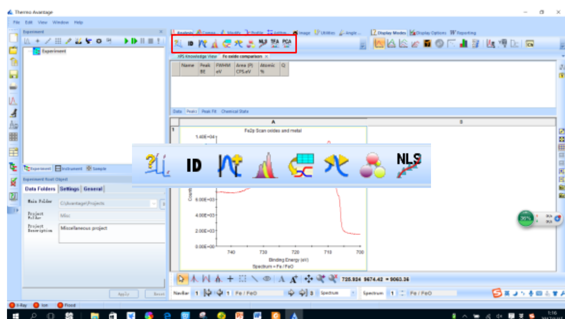
Avantage

- Avantage-Thermo Fisher



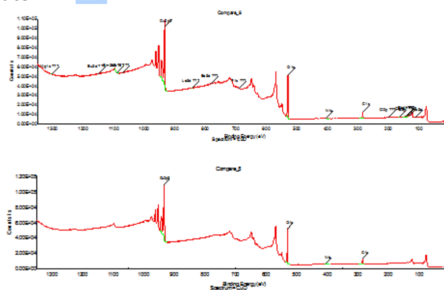
http://xpssimplified.com/periodictable.php

Avantage



Identification of elements——Avantage

- Auto ID



Identification of elements——Avantage

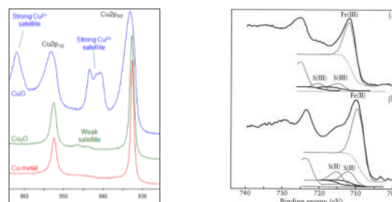
- Manual ID

- Quantification

Name	Peak BE	FWHM eV	Area (P) CPS.eV	Atomic %	Q
O1s	529.75	1.21	86346.56	49.06	1
Cu2p3	933.17	2.8	220046.7	29.66	1
C1s	284.72	1.54	14507.32	19.96	1
N1s	404.93	0.77	1483.77	1.32	1

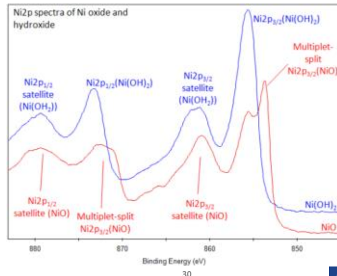
Shake-up Satellites

- Shake-up satellites may occur when the outgoing photoelectron simultaneously interacts with a valence electron and excites it (shakes it up) to a higher-energy level.
- The most notable examples being the 2p spectra of the d-band metals and the π—> π * transition brought about by C Is electrons in aromatic organics.



Multiplet Splitting

- Multiplet splitting of a photoelectron peak may occur in a compound that has unpaired electrons in the valence band, and arises from different spin distributions of the electrons of the band structure.
- Mn, Cr(3s levels), Co, Ni (2p3/2 levels), and the 4s levels of the rare earths



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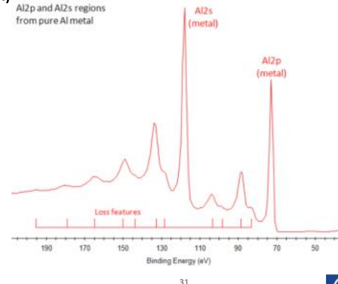
Plasmons

- They arise when the outgoing electron excites collective oscillations in the conduction band electrons and thus suffers a discrete energy loss (or several losses in multiples of the characteristic plasmon frequency, about 15 eV for aluminium).



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Chemical Shift

- The binding energy of core electrons can change when the chemical environment changes.
- Generally, the binding energy of core levels increase when valence electrons are removed from when valence electrons are removed from an atom.
- Generally, the biding energy of core levels decrease when valence electrons are added to an atom.
- The chemical shift of Some d-band metals and rare earths is not so significant or abnormal(Ce→CeO2,-2eV).

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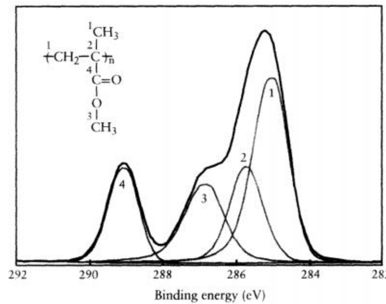
Chemical Shift of C1s



C 1s spectrum of poly(methyl methacrylate)

Beamson G, Briggs D. High resolution XPS of organic polymers: the Scienta ESCA300 database. 1992.

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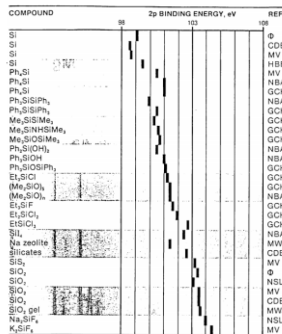
Chemical Shift of Si2p



Wagner C D et al. Handbook of X-ray photoelectron spectroscopy[M]. Perkin-Elmer, 1979.

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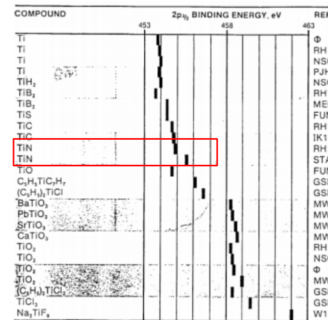
Chemical Shift of Ti2p3



Wagner C D et al. Handbook of X-ray photoelectron spectroscopy[M]. Perkin-Elmer, 1979.

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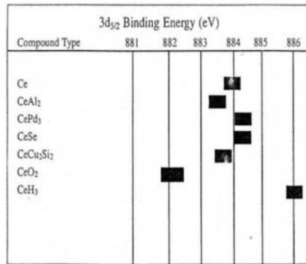
Chemical Shift of Ce3d5



John F M, et al. Handbook of X-ray Photoelectron[M]. Physical Electronics, Inc. 1992.

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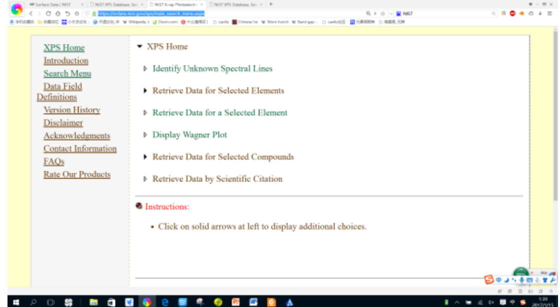
NIST

https://srdata.nist.gov/xps/main_search_menu.aspx



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NIST

- Ti 2P3

Ti	2p3/2	TiO2	458.50	Click
Ti	2p3/2	TiO2	458.70	Click
Ti	2p3/2	TiO2	458.50	Click
Ti	2p3/2	TiO2	458.80	Click
Ti	2p3/2	TiO2	459.00	Click
Ti	2p3/2	TiO2	458.33	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	459.20	Click
Ti	2p3/2	TiO2	459.60	Click
Ti	2p3/2	TiO2	458.00	Click
Ti	2p3/2	TiO2	458.75	Click
Ti	2p3/2	TiO2	458.70	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	458.60	Click
Ti	2p3/2	TiO2	458.70	Click
Ti	2p3/2	TiO2	459.20	Click
Ti	2p3/2	TiO2	459.00	Click
Ti	2p3/2	TiO2	458.60	Click

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NIST

- Ti 2P3

Lorentzian Width (eV):	
Measurement Information:	
Use of X-ray Monochromator:	No
Excitation Energy:	Al
X-ray Energy:	
Overal Energy Resolution (eV):	
Calibration:	
Charge Reference:	Adventitious carbon
Energy Scale Evalution:	Reliable, with one-point correction of energy scale
Specimen Information:	
Specimen:	crystal
Method of Determining Specimen Composition:	
Method of Determining Specimen Crystallinity:	
Specimen Temperature (K):	300
Sample Quality:	Adequate
Comment:	
Notes:	Anatase.

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XAES and Auger Parameter

- Some elements has more larger XAES chemical shift than XPS.

Chemical Shift	Cu→ Cu2O	Zn→ ZnO	Mg→ MgO	Ag→ Ag2SO4	In→ In2O
XPS	0.1	0.8	0.4	0.2	0.5
XAES	2.3	4.6	6.4	4.0	4.6

- Auger Parameter

$$\alpha=E_B+E_k$$

E_B is the binding energy of the most intense photoelectron emission peak and E_k is the kinetic energy of the Auger transition

Independent of any electrostatic charging of the specimen.

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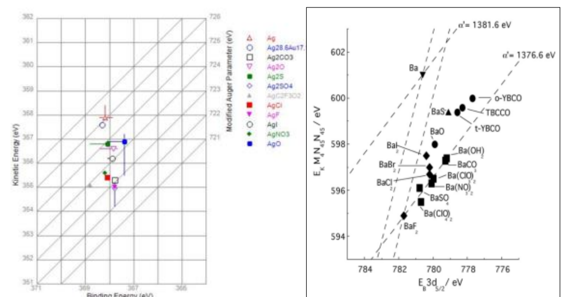
Wagner plot



https://srdata.nist.gov/xps.

Satta M, Moretti G. Auger parameters and Wagner plots[J]. Journal of Electron Spectroscopy and Related Phenomena, 2010, 178: 123-127.

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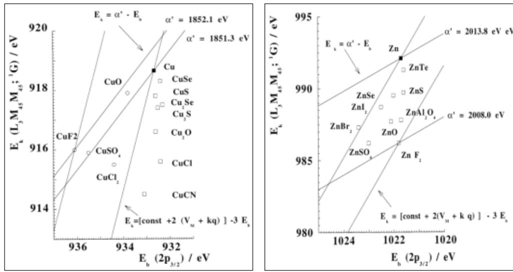
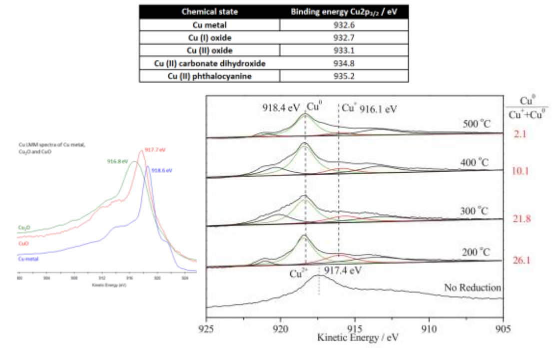
Wagner plot



- Moretti G. Auger parameter and Wagner plot in the characterization of chemical states by X-ray photoelectron spectroscopy: a review[J]. Journal of electron spectroscopy and related phenomena, 1998, 95(2): 95-144.

XAES



Chemical state	Binding energy $Cu2p_{3/2}$ / eV
Cu metal	932.6
Cu (I) oxide	932.7
Cu (II) oxide	933.1
Cu (II) carbonate dihydroxide	934.8
Cu (II) phthalocyanine	935.2

Quantitative Analysis

First-principles method:

$$I = JN\sigma\lambda\cos(\theta)T$$

J -- x-ray photon flux

N -- number of atom per cm^3

σ -- photo-ionized cross section

λ -- mean free path

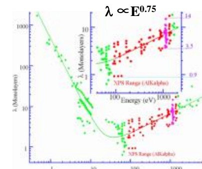
θ -- accept solid angle of the analyzer

T -- transmission of analyzer ($T \propto E^{-1}$)

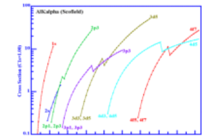
$$S = \sigma\lambda T \propto \sigma E^{-0.25}$$

Hofmann S. Springer Science & Business Media, 2012.

$$n_x = \frac{I_x / S_x}{\sum_i I_i / S_i}$$



Seah M P, Dench W A. Surface and interface analysis, 1979.



Scofield J H. Journal of Electron Spectroscopy and Related Phenomena, 1976

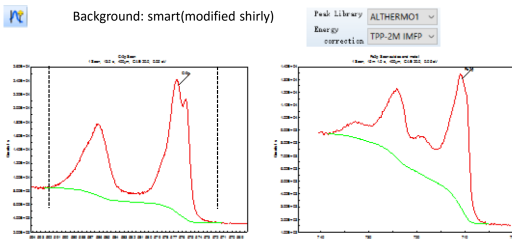
Wagner RSF

$S \propto \sigma E^{-0.34}$, $S_{F1s}=1$.

Table 5. Atomic Sensitivity Factors (ASF)

Wagner C D et al. Handbook of X-ray photoelectron spectroscopy[M]. Perkin-Elmer, 1979.

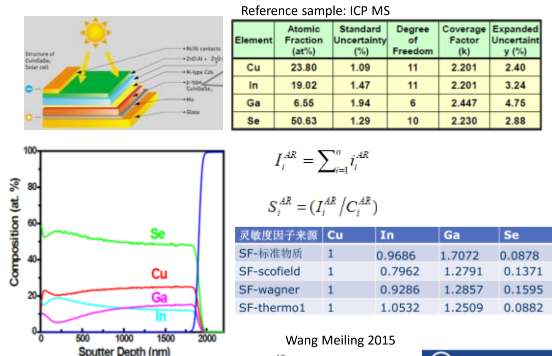
Quantitative Analysis——Avantage



An error of 15% is generally quoted

Name	Peak BE	FWHM eV	Area (P) CPS.eV	Atomic %	Q
Cr2p	576.41	2.76	100275.5	52.29	1
Fe2p	710.64	4.34	99084.06	47.71	1

Experimental RSF--CIGS



Reference sample: ICP MS

Element	Atomic Fraction (at%)	Standard Uncertainty (%)	Degree of Freedom	Coverage Factor (k)	Expanded Uncertainty (%)
Cu	23.80	1.09	11	2.201	2.40
In	19.02	1.47	11	2.201	3.24
Ga	6.55	1.94	6	2.447	4.75
Se	50.63	1.29	10	2.230	2.88

$$I_i^{AR} = \sum_{i=1}^{n} i_i^{AR}$$

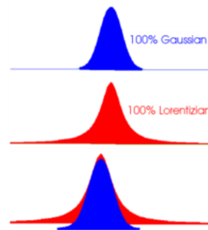
$$S_i^{AR} = (I_i^{AR} / C_i^{AR})$$

灵敏度因子来源	Cu	In	Ga	Se
SF-标准物质	1	0.9686	1.7072	0.0878
SF-scofield	1	0.7962	1.2791	0.1371
SF-wagner	1	0.9286	1.2857	0.1595
SF-thermo1	1	1.0532	1.2509	0.0882

Wang Meiling 2015

Curve fitting

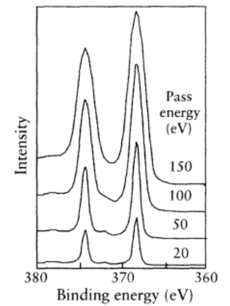
- Combinations of Gaussian and Lorentzian functions have been generally used.
- Augmented by asymmetric tail functions.
- Also by defining a lineshape using data acquired from a reference sample.
- Reasonable Peak is the most important, followed by FWHM, L/G ratio, and then the asymmetric tail functions.



FWHM

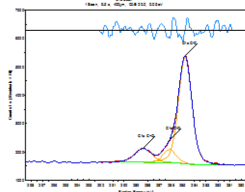
- It is mathematical combination of
  - the natural width of core level, E_L
  - Phonon broadening, E_p
  - the width of the X-ray line, E_x
  - The energy resolution of the analyzer, E_A
- If they are all Gaussian functions or shapes, the measured energy resolution, E_M would be given by:

$$E_M^2 = E_L^2 + E_p^2 + E_x^2 + E_A^2$$



Carbon contamination

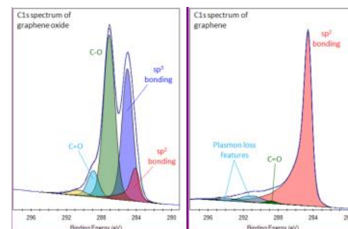
- Adventitious carbon contamination is commonly used as a charge reference for XPS spectra.



Ref	Name	Peak BE	Height CPS	Height Ratio	Area CPS.eV	Area Ratio	FWHM fit param (eV)	L/G Mix (%) Convolve	Tail Mix (%)	Tail Height (%)	Tail Exponent
D	C1s C-C	284.80	3760.93	1.00	5566.10	1.00	1.31	22.88	100.00	0.00	0.0000
							0.5 : 3.5		fixed	fixed	fixed
E	C1s C-O	286.14	460.75	0.12	681.89	0.12	1.31	22.88	100.00	0.00	0.0000
							D*1	D*1	fixed	fixed	fixed
F	C1s C=O	288.40	474.32	0.13	910.23	0.16	1.70	22.88	100.00	0.00	0.0000
							0.5 : 3.5	D*1	fixed	fixed	fixed

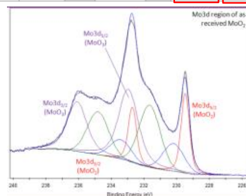
Graphene

- Functionalized graphene will have a complex C1s spectrum, containing sp2 and sp3 components.
- The sp3 carbon peak should be 1eV to the higher binding energy side of the sp2 component.



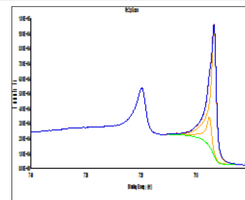
Molybdenum oxides

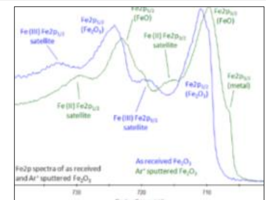
Ref	Name	Peak BE	Height CPS	Height Ratio	Area CPS.eV	Area Ratio	FWHM fit param (eV)	L/G Mix (%) Convolve	Tail Mix (%)	Tail Height (%)	Tail Exponent
A	Mo3d5 sub	231.65	7599.31	0.81	19310.00	1.00	2.12	33.72	100.00	0.00	0.0000
		231.61 : 231.81						fixed	fixed	fixed	fixed
B	Mo3d3 sub	234.78	5250.85	0.56	13279.15	0.69	2.12	33.72	100.00	0.00	0.0000
		A+3.15 (+0.2 -0.1)	A*0.691				A*1	fixed	A*1	A*1	A*1
C	Mo3d5 MoO3	233.09	6642.52	0.71	8276.16	0.43	1.04	33.72	100.00	0.00	0.0000
		232.96 : 233.16					fixed	fixed	fixed	fixed	fixed
D	Mo3d3 MoO3	236.14	4589.74	0.49	5718.51	0.30	1.04	33.72	100.00	0.00	0.0000
		C+3.15 (+0.2 -0.1)	C*0.691				fixed	fixed	C*1	C*1	C*1
E	Mo3d5 MoO2	229.52	9342.14	1.00	7247.27	0.38	0.65	33.72	100.00	0.00	0.0000
							0.5 : 3.5	A*1	fixed	fixed	fixed
F	Mo3d3 MoO2	232.66	6455.08	0.69	5007.60	0.26	0.65	33.72	100.00	0.00	0.0000
		E+3.15 (+0.2 -0.1)	E*0.691				E*1	E*1	E*1	E*1	E*1



Unsymmetrical Peak

Ref	Name	Peak BE	Height CPS	Height Ratio	Area CPS.eV	Area Ratio	FWHM fit param (eV)	L/G Mix (%) Convolve	Tail Mix (%)	Tail Height (%)	Tail Exponent
A	Fe2p3 metal	706.67	84714.94	1.00	134633.45	1.00	0.83	49.49	56.03	0.00	0.0320
							0.5 : 3.5				
B	Fe2p3 metal multiplet	707.52	19489.56	0.23	30741.88	0.23	0.83	47.25	56.03	0.00	0.0320
		A+0.85					A*1		A*1	A*1	A*1





Biesinger M C, Payne B P, Grosvenor A P, et al. Resolving surface chemical states in XPS analysis of first row transition metals, oxides and hydroxides: Cr, Mn, Fe, Co and Ni[J]. Applied Surface Science, 2011, 257(7): 2717-2730.
Biesinger M C, Lau L W M, Gerson A R, et al. Resolving surface chemical states in XPS analysis of first row transition metals, oxides and hydroxides: Sc, Ti, V, Cu and Zn[J]. Applied Surface Science, 2010, 257(3): 887-898.

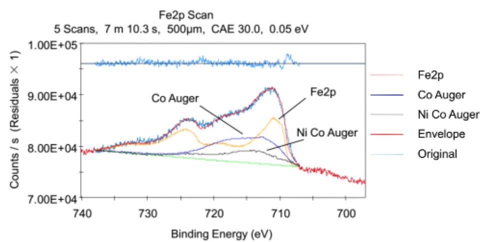
NLLSF

- Non-Linear Least Squares Fitting (NLLSF) assumes that the data is composed of a sum of component spectra but that the peak positions may due to charging, especially in Auger analysis where charge compensation may not be easily controlled.
- In NLLSF, the reference spectra are determined as before, but at every level in the profile, the peak positions are adjusted to give the mathematical best fit.
- The advantage that NLLSF has over normal peak fitting is that real peak shapes compared with G/L mixtures can be applied to sets of data.

NLLSF

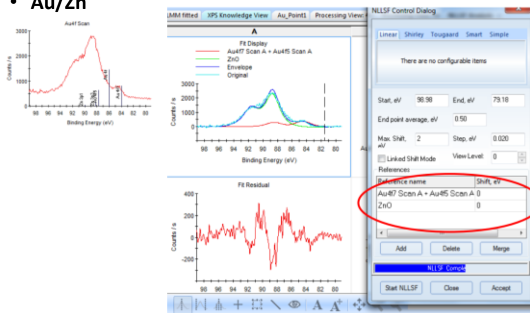
NLLSF

- Fe2p, Co Auger, Ni Auger



NLLSF

- Au/Zn



What XPS looks like？

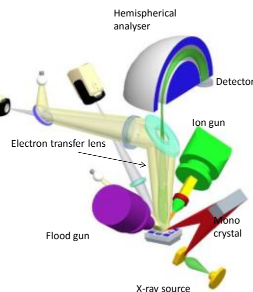
KRATOS AXIS Supra

PHI 5000 VersaProbe III

Thermo Fisher ESCALAB 250Xi

XPS instrumentation

- UHV System
  - Ultra-high vacuum keeps surfaces clean
  - Allows longer photoelectron path length
- X-ray source
  - Typically Al Kα radiation
  - Monochromated using quartz crystal
- Electron analyser
  - Lens system to collect photoelectrons
  - Analyser to filter electron energies
  - Detector to count electrons
- Low-energy electron flood gun
  - Analysis of insulating samples
- Ion gun
  - Sample cleaning
  - Depth profiling
  - For polymers, cluster ion sources may be required



Monochromated X-ray sources

Crystal Adjustment
Crystal
500mm Rowland Circle
Angle Adjustment
Electron Gun
Anode
Sample

0.16 ev
Al Kα1,2
after monochromatization
1ev

Principle:
Bragg diffraction

$$2d\sin\theta_B = \lambda$$

Al K_a: $\lambda \sim 8.339$Å
Quartz: $d \sim 4.25$Å $\Rightarrow \theta_B = 78^0$

Interface.Miner.

Electron energy analyzer

H–
H+
Photoelectrons
Detector
KE < E_p
KE = E_p
KE > E_p

Pass Energy(E_0):

$$E_0 = e\Delta V\left(\frac{R_2}{R_1} - \frac{R_1}{R_2}\right)^{-1}$$

$$\Delta V = V_2 - V_1$$

Energy resolution:

$$\frac{\Delta E}{E_0} \approx \frac{W_2}{2R_0} + (\delta\alpha)^2$$

Intensity
Pass energy (eV)
150
100
50
20
380 370 360
Binding energy (eV)

Since W_2 and $\delta\alpha$ is small in CHA, so the energy resolution is high for XPS.

Interface.Miner.

Electron Flood Gun

X-ray
e⁻
~2eV-20eV
Electrons optics

Differential (non-uniform) surface charging
K.E.= hν - B.E. - ϕ_{spec} - 5V
K.E.= hν - B.E. - ϕ_{spec} - 1V
+5V +2V +3V +4V +1V +2V
Sample
Broadening of peak

$$E_B = h\nu - E_{kin} - \Phi_A$$

Charging effect and compensation

Interface.Miner.

Electron Flood Gun

polymer
Flood gun off
Flood gun on
Binding Energy (eV)

(a) Quartz
Flood gun off
(b)
Flood gun on
1200 1000 800 600 400 200 0
Binding Energy (eV)

Interface.Miner.

Ar Iron Gun

Clean the contaminated surface

Before sputtering
After 200eV Ar^+ sputtering
B 1s
oxide
BN
N 1s
BNO?
BN

Depth profile with ion sputtering

XPS analysis
Ar^+ sputtering
XPS analysis

Interface.Miner.

CIGS solar cell

CIGS
Mo
Cr
Steel
Atomic Percent (%)
Etch Depth (nm)

Interface.Miner.

Sample Preparation

- Volatile？
- Magnetic？
- Conductive？
- Size？
- Powder？
  - Tablet press
  - Conducting resin
- Fiber？
  - Tablet press
  - Conducting resin
  - Hang cross a gap

Spectroscopy Acquiring

Experiment
Mono Al 200um
Point
Survey
C1s Scan
O1s Scan
Cr2p Scan
Fe2p Scan
Ni2p Scan
Valence

XAES？

Summary of XPS Capabilities

- Elemental analysis(H,He)
- Chemical state information
- Quantification (sensitivity about 0.1 atomic %)
- Small area analysis (5 μm spatial resolution)
- Chemical mapping
- Depth profiling
- Ultrathin layer thickness
- Suitable for insulating samples

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  - http://xpsdata.com/
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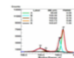
- Software
  - Avantage http://info1.thermoscientific.com/forms/CAD-MOL-AvantageSoftwareGateway-EN-0000-RE-KR
  - Casaxps http://www.casaxps.com/
  - MultiQuant http://aki.ttk.mta.hu/XMQpages/XMQhome.htm

AD

- 测试谷店铺
  - 名称：Interface.Miner.
  - 主营业务
    - XPS/UPS咨询、培训及图谱分析
    - 扫描电镜截面样品制备



XPS/UPS咨询及图谱分析

XPS/UPS咨询及图谱分析

服务介绍

XPS或UPS图谱数据处理及相关咨询

服务商介绍

本人毕业于国内一流高校，导师在国内较早开展AES/XPS研究及应用，目前在一作，近两年专职从事XPS/UPS研究及应用，曾在国际权威会议（IVC 20th）做层分析。

Thanks for Your Attention！