

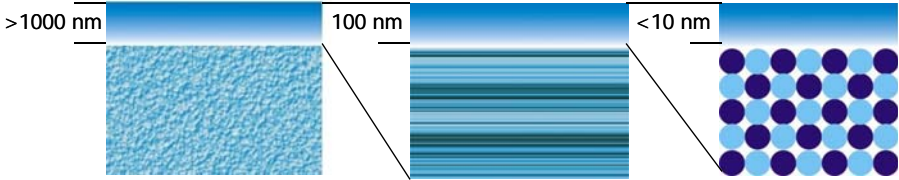
 **Advanced Materials Characterization Workshop** 

X-ray Photoelectron Spectroscopy (XPS) and Auger Electron Spectroscopy (AES)

Rick Haasch, Ph.D.

Supported by the U.S. Department of Energy under grants DEFG02-07-ER46453 and DEFG02-07-ER46471
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What is Surface Analysis?



Bulk Analysis **Thin-film Analysis** **Surface Analysis**

The Study of the Outer-Most Layers of Materials (~10 nm).

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Particle Surface Interactions

Primary beam
(source)

Ions
Electrons
Photons

Secondary beam
(spectrometers, detectors)

Ions
Electrons
Photons

Vacuum

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Spatial resolution versus Detection Limit

Evans
 Analytical Group
 CHARLES EVANS & ASSOCIATES • EVANS PHI • EVANS EAST • EVANS NORTHEAST • EVANS TEXAS • EVANS EUROPA • EVANS TAIWAN
 WWW.EVANSLABS.COM

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Particle Surface Interactions


Photoelectron Spectroscopy

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X-ray Photoelectron Spectroscopy (XPS)


X-ray Photoelectron Spectroscopy (XPS), also known as **Electron Spectroscopy for Chemical Analysis (ESCA)** is a widely used technique to investigate the chemical composition of surfaces.

X-ray¹ Photoelectron spectroscopy, based on the photoelectric effect,^{2,3} was developed in the mid-1960's as a practical technique by Kai Siegbahn and his research group at the University of Uppsala, Sweden.⁴




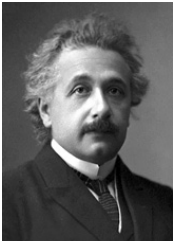
Wilhelm Conrad Röntgen







Heinrich Rudolf Hertz






Albert Einstein





Kai M. Siegbahn



1. W. Röntgen, 1901 Nobel Prize in Physics "in recognition of the extraordinary services he has rendered by the discovery of the remarkable rays subsequently named after him."
2. H. Hertz, *Ann. Physik* 31,983 (1887).
3. A. Einstein, *Ann. Physik* 17,132 (1905). 1921 Nobel Prize in Physics "for his services to Theoretical Physics, and especially for his discovery of the law of the photoelectric effect."
4. K. Siegbahn, Et. Al., *Nova Acta Regiae Soc.Sci., Ser. IV, Vol. 20* (1967). 1981 Nobel Prize in Physics "for his contribution to the development of high resolution electron spectroscopy."

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X-ray Photoelectron Spectroscopy Small Area Detection

X-ray Beam

X-ray penetration depth $\sim 1\mu\text{m}$. Electrons can be excited in this entire volume.

Electrons are extracted only from a narrow solid angle.

10 nm

10 μm - 1 mm dia.

X-ray excitation area $\sim 1\text{ mm}$ -1 cm diameter. Electrons are emitted from this entire area

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Photoelectron and Auger Electron Emission

KE (measured) = $h\nu$ (known) - BE - Φ_{spec} (calibrated)

$E_{\text{incident X-ray}} = h\nu$

Emitted Auger Electron

Free Electron Level

Conduction Band

Fermi Level

Valence Band

BE

2p L2,L3

2s L1

1s K

Calculate: **BE = $h\nu$ - KE - Φ_{spec}**

BE – binding energy depends on Z, i.e. characteristic for the element

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Photoelectron and Auger Electron Emission

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

$E_{\text{incident X-ray}} = h\nu$

Emitted X-ray Photon

The diagram illustrates energy levels for a material. On the left, an incident X-ray photon with energy $h\nu$ excites an electron from a core level (1s) to a free electron level. On the right, an emitted X-ray photon is shown, resulting from an Auger transition where an electron from a higher level (L2, L3) fills the 1s core hole, and another electron from a higher level (L1) is emitted. The diagram also shows the Fermi level, Conduction Band, and Valence Band. The binding energy (BE) is the energy difference between the Fermi level and the core level.

Calculate: $BE = h\nu - KE - \Phi_{\text{spec}}$


BE – binding energy depends on Z, i.e. characteristic for the element

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Photoelectron and Auger Electron Emission

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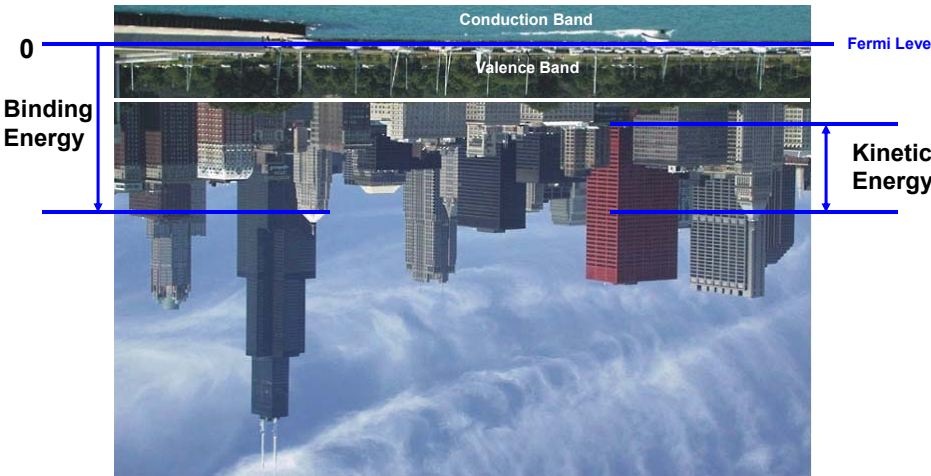
Photoelectron and Auger Electron Emission



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Photoelectron and Auger Electron Emission

Energy Scales



0
Fermi Level
Conduction Band
Valence Band
Binding Energy
Kinetic Energy
Photoelectron Lines
Auger Electron Lines

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Photoelectron and Auger Electron Emission

X-ray Photoelectron Spectroscopy

Conduction Band
Fermi Level
Valence Band

Al $K\alpha$ = 1486.6 eV
Mg $K\alpha$ = 1253.6 eV

XPS can probe all of the orbitals in only the light elements.
e.g. BE C 1s = 285 eV, Mg 1s = 1304 eV, Au 1s \approx 81000 eV

Predominantly, soft X-rays are used.

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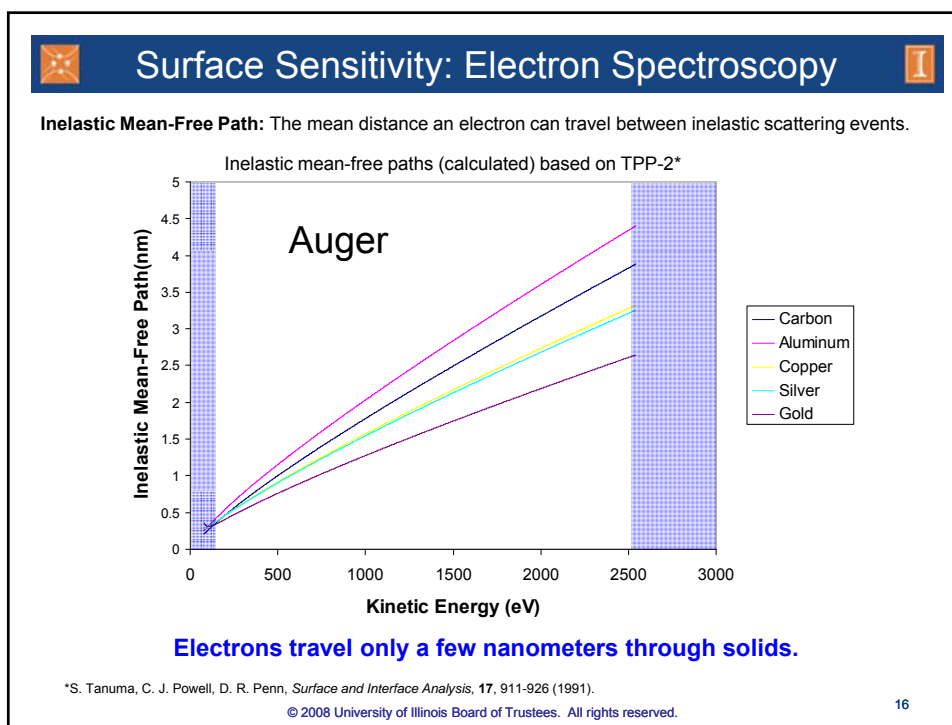
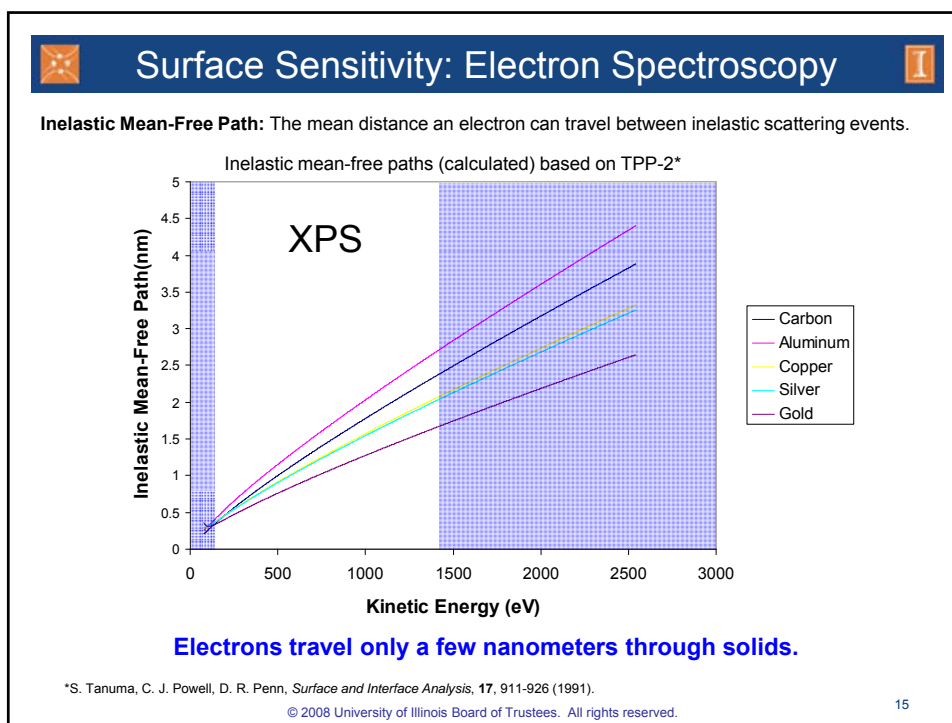
Photoelectron and Auger Electron Emission

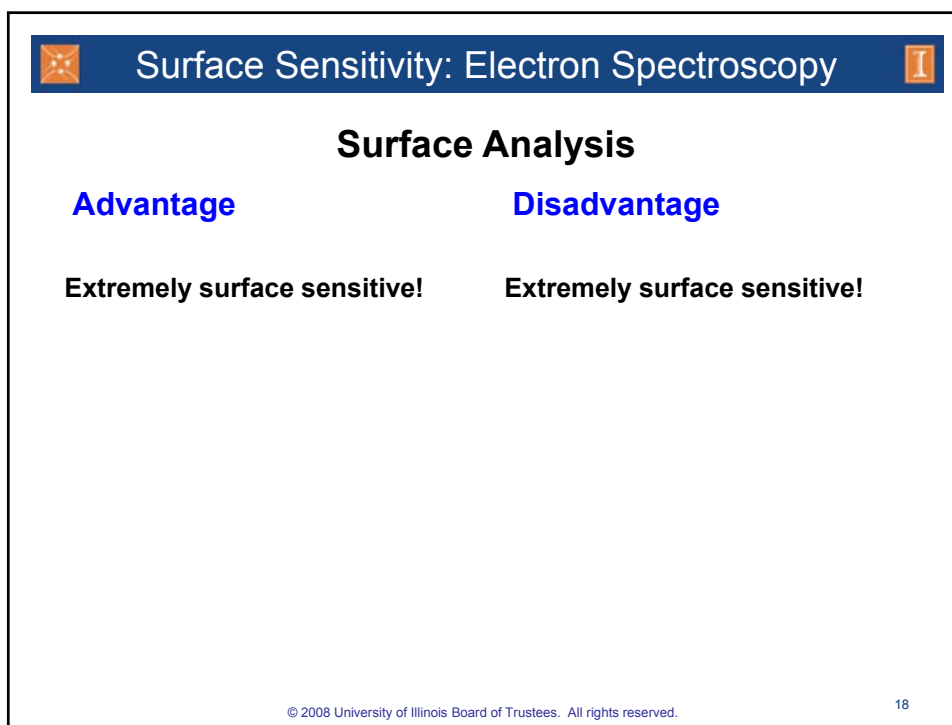
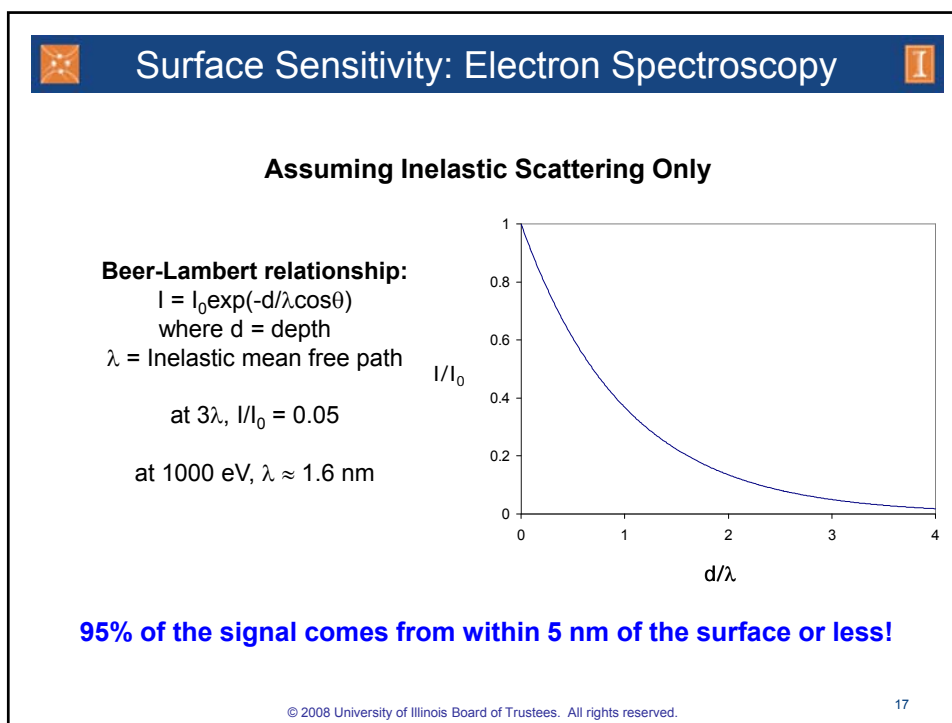
Ultraviolet Photoelectron Spectroscopy

Conduction Band
Fermi Level
Valence Band

He II = 40.8 eV
He I = 22.4 eV

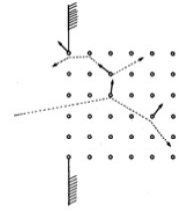
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Ion Sputtering

0.5 – 5 keV Ar⁺



Linear cascade
a series of binary collisions

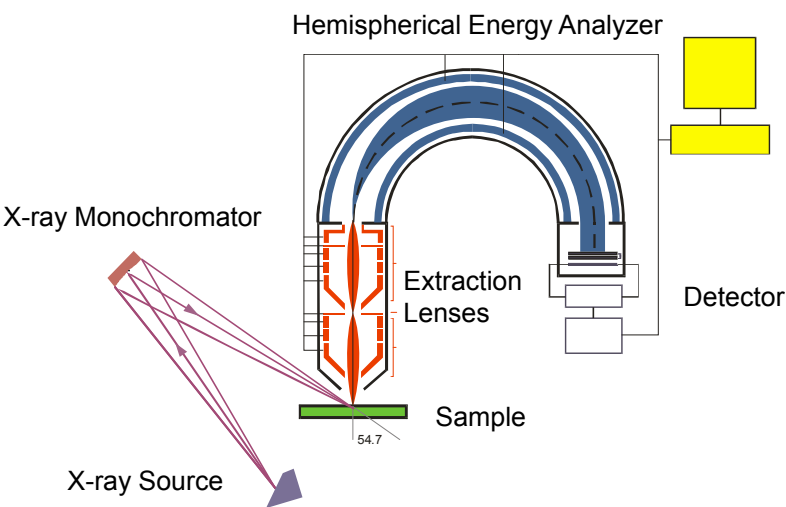
- Ions striking a surface interact with a number of atoms in a series of collisions.
- Recoiled target atoms in turn collide with atoms at rest, generating a collision cascade.
- The initial ion energy and momentum are distributed among the target recoil atoms.
- When $E_i > 1$ keV, the cascade is "linear", i.e., approximated by a series of binary collisions in a stationary matrix.

Causes physical and chemical damage

P. Sigmund, "Sputtering by ion bombardment: theoretical concepts," in Sputtering by particle bombardment I, edited by R. Behrisch, Springer-Verlag, 1981
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X-ray Photoelectron Spectrometer

X-ray Source



X-ray Monochromator

Hemispherical Energy Analyzer

Extraction Lenses

Detector

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X-ray Photoelectron Spectrometer

Physical Electronics PHI 5400

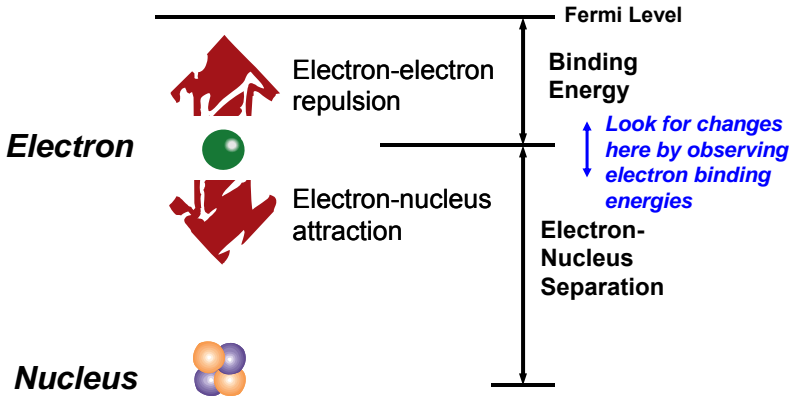


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Elemental Shifts

Pure Element



Fermi Level
Binding Energy
Look for changes here by observing electron binding energies
Electron-Nucleus Separation

Electron
 Electron-electron repulsion
 Electron-nucleus attraction
Nucleus

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Elemental Shifts

Periodic Table of the Elements

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Elemental Shifts

Core Level Binding Energies

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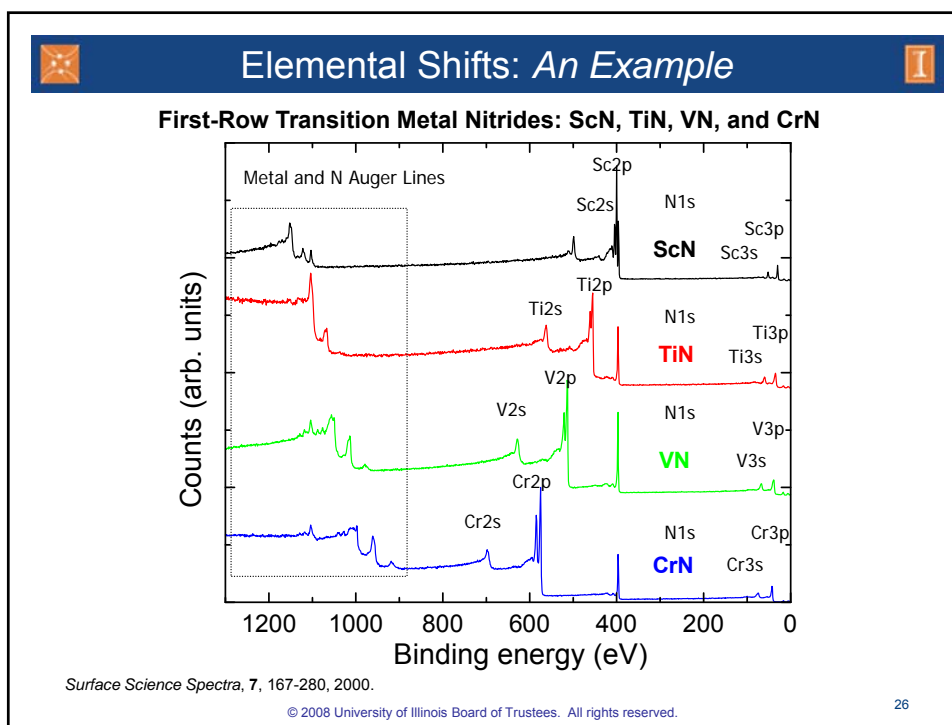
Elemental Shifts

First-Row Transition Metals

3	4	5	6	7	8	9	10	11	12
21	22	23	24	25	26	27	28	29	30
Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
44.96	47.88	50.94	52.00	54.94	55.85	58.93	58.69	63.55	65.38

Binding Energy (eV)			
Element	2p _{3/2}	3p	Δ
Sc	399	29	370
Ti	454	33	421
V	512	37	475
Cr	574	43	531
Mn	639	48	591
Fe	707	53	654
Co	778	60	718
Ni	853	67	786
Cu	933	75	858
Zn	1022	89	933

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Chemical Shifts

Electronegativity Effects

Carbon-Oxygen Bond

Oxygen Atom

Valence Level
C 2p

Core Level
C 1s

C 1s Binding Energy

↓ Shift to higher binding energy

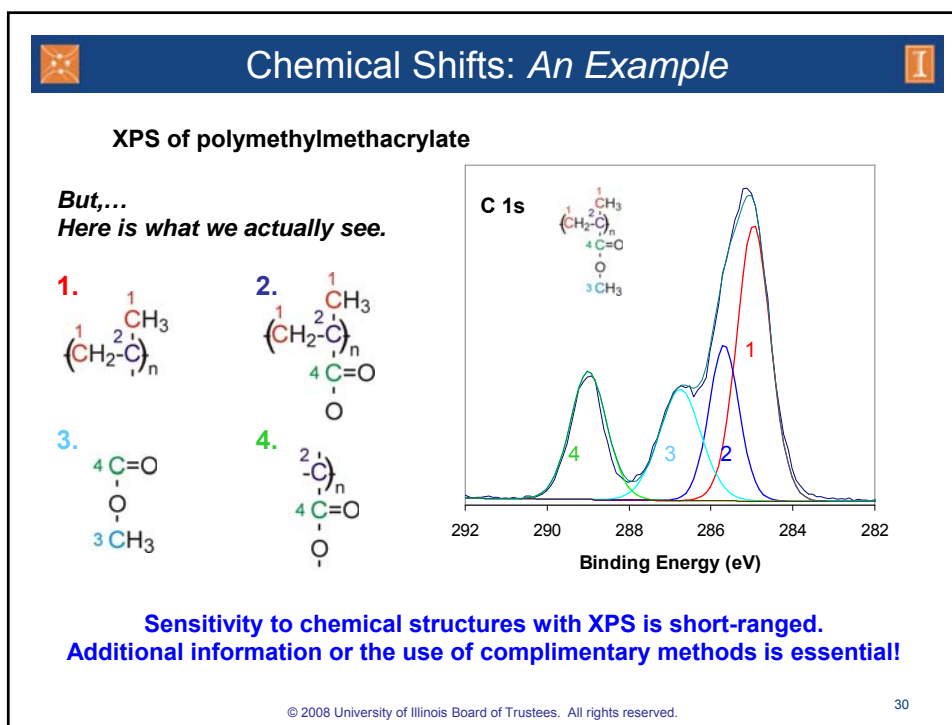
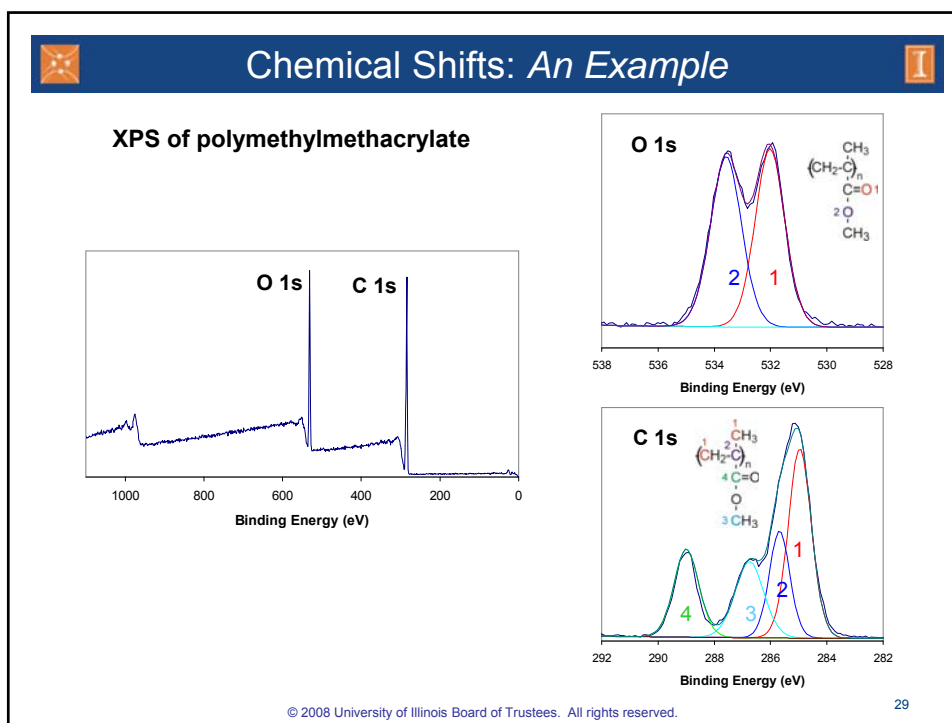
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Chemical Shifts

13	14	15	16	17
5	6	7	8	9
B	C	N	O	F
10.81	12.01	14.01	16.00	19.00

Functional Group	C-H, C-C	C 1s Binding Energy (eV)
hydrocarbon	C-H, C-C	285.0
amine	C-N	286.0
alcohol, ether	C-O-H, C-O-C	286.5
Cl bound to C	C-Cl	286.5
F bound to C	C-F	287.8
carbonyl	C=O	288.0

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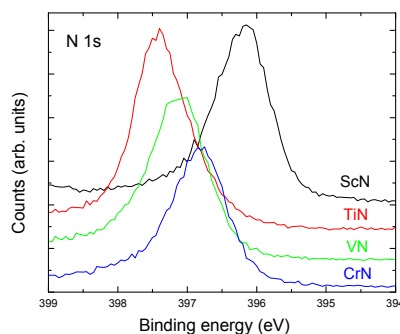
Chemical Shifts: An Example



N 1s spectra of First-Row Transition Metal Nitrides: ScN, TiN, VN, and CrN

p-d hybridization 8 MO's

	Anti-bonding e-/Formula Unit (nominal)	Binding Energy, eV
ScN	-0.17 (0)	396.1
TiN	1 (1)	397.3
VN	1.9 (2)	397.0
CrN	2.9 (3)	396.7



Surface Science Spectra, 7, 167-280, 2000.

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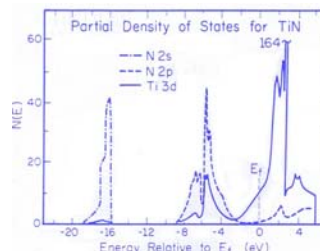
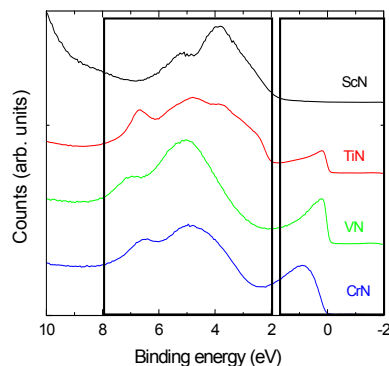
Ultraviolet Photoelectron Spectroscopy: An Example



First-Row Transition Metal Nitrides: ScN, TiN, VN, and CrN

Partially hybridized N 2p M 3d anti-bonding states and M 3d bonding states

- As the number of electrons per unit cell increases, from ScN to CrN, the metal 3d bands begin to fill pushing the Fermi level above the minimum in the density-of-states (DOS). The Fermi edge then becomes more dominant and the N 2p bands move to higher binding energy.



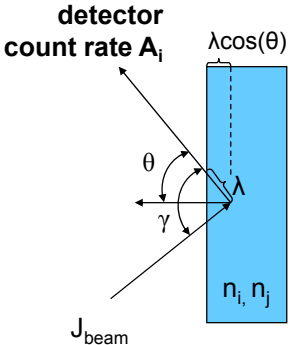
Surface Science Spectra, 7, 167-280, 2000.

J.-E. Sundgren, B.O. Johansson, A. Rockett, S.A. Barnett, J.E. Greene, *American Institute of Physics Conference Proceedings*, **149(1)**, 95-115 (1986).

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Quantitative Surface Analysis: XPS



Assuming a Homogeneous sample:
 $A_i = \text{detector count rate}$

$A_i = (\text{electrons/volume})(\text{volume})$
 $A_i = (N_i \sigma_i(\gamma) J T(E_i)) (a \lambda_i(E_i) \cos \theta)$

Sample Dependent Terms
 where: $N = \text{atoms/cm}^3$
 $\sigma(\gamma) = \text{photoelectric (scattering) cross-section, cm}^2$
 $\lambda(E_i) = \text{inelastic electron mean-free path, cm}$

Instrument Dependent Terms
 $J = \text{X-ray flux, photon/cm}^2\text{-sec}$
 $T(E_i) = \text{analyzer transmission function}$
 $a = \text{analysis area, cm}^2$
 $\theta = \text{photoelectron emission angle}$

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Quantitative surface analysis: XPS

By assuming the concentration to be a relative ratio of atoms, we can neglect the terms that depend only on the instrument:

$$N_i = A_i / \sigma_i T(E_i) \lambda_i(E_i)$$

It is difficult to accurately determine λ_i so it is usually neglected. Modern acquisition and analysis software can account for the transmission function.

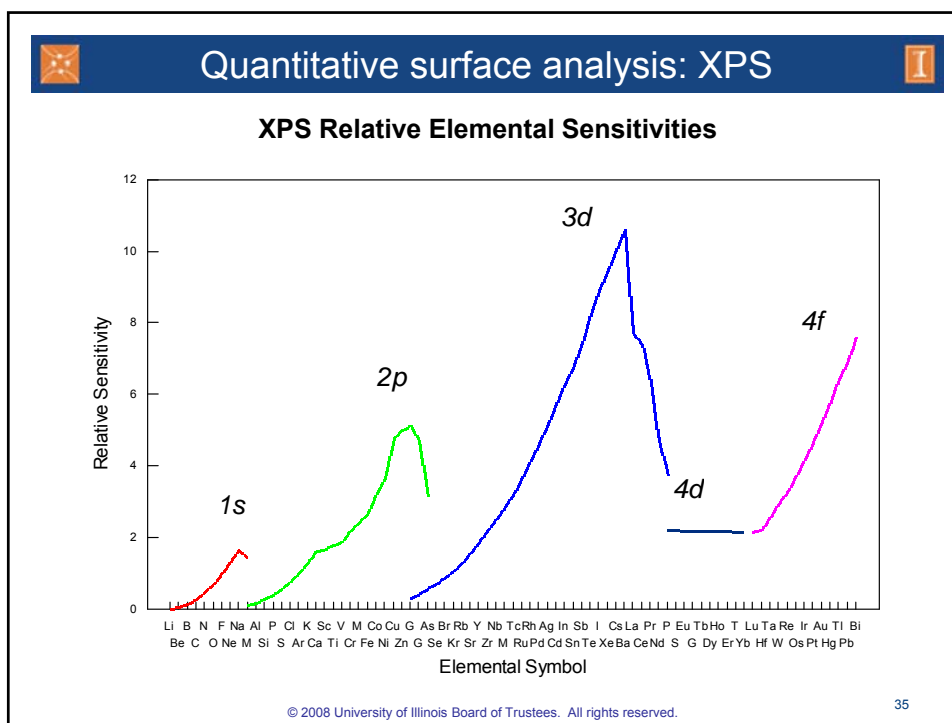
$$N_i = A_i / S_i$$

$$C_i = A_i / S_i / \sum A_{i,j} / S_{i,j}$$

The values of S are determined theoretically or empirically with standards.

XPS is considered to be a semi-quantitative technique.

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Quantitative surface analysis: *An Example*

First-Row Transition Metal Nitrides: ScN, TiN, VN, and CrN

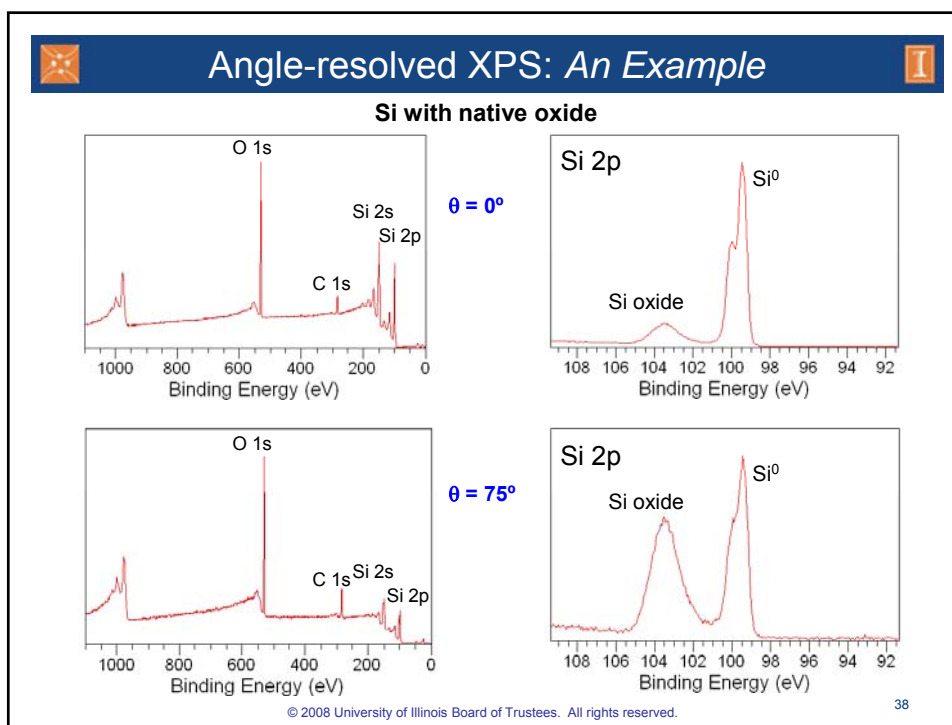
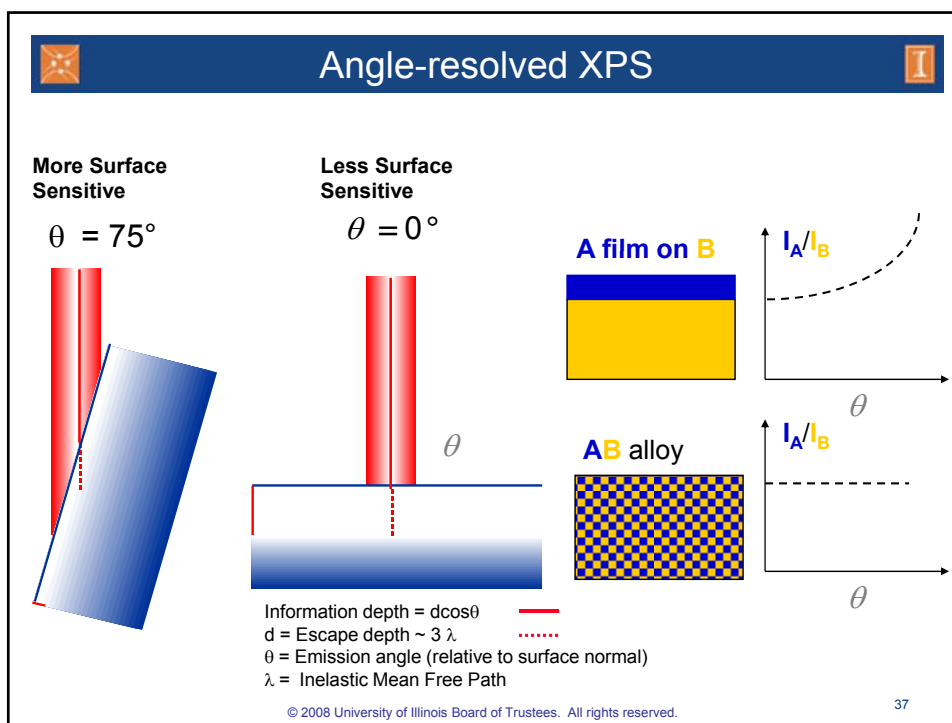
XPS Analysis			ScN	TiN	VN	CrN
Binding energy (eV)	Metal 2p _{3/2}	Major peak	400.4	455.1	513.2	574.4
		Satellite ^a		457.9	515.5	575.5
	Metal 2p _{1/2}	Major peak	404.9	461.0	520.7	584.0
		Satellite ^a		463.8	523.0	585.1
	N 1s		396.1	397.3	397.0	396.7
Composition (N/metal)	As Deposited		1.13	1.00	1.02	0.73 ^b
	After ion bombardment		0.99	0.73	0.46	0.55 ^b
	Bulk value from RBS		1.11±0.03	1.02±0.02	1.06±0.02	1.04±0.02

a. The satellite is due to a transition into a relaxed final state
 b. The composition determination of the CrN layers by peak fitting is less reliable because the commonly used Shirley method for background subtraction does not accurately describe the experimental data.

Nitrogen/Metal peak ratio decreases after sputtering

Surface Science Spectra, 7, 167-280, 2000.

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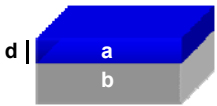


Angle-resolved XPS: An Example

SiO₂ Layer thickness calculation: Two-Layer Model- thin overlayer

Assuming only inelastic scattering *and* photoelectrons from layer a will not undergo an inelastic scattering event

Beer-Lambert relationship:
 $I = I_0 \exp(-d/\lambda \cos\theta)$



$\frac{I_a}{I_b}$

→

$d/\lambda = \ln(c_a/c_b + 1) \cos\theta$

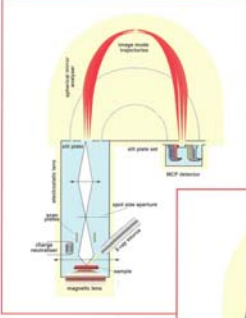
Step 1:
a=Carbon, b=SiO₂+Si
with $\lambda=3$ nm, d=0.3 nm

Step 2:
a=Carbon+SiO₂, b=Si
with $\lambda=3$ nm, d=1.5 nm

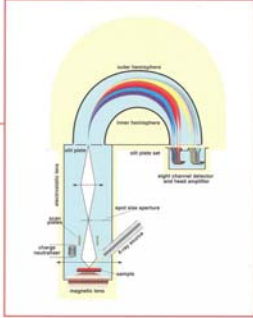
0.3 nm Carbon
1.2 nm SiO ₂
Si

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Imaging X-ray Photoelectron Spectrometer




Imaging Mode:
Spherical mirror analyzer With the entrance aperture open, a spatially dispersed image (real image) is projected onto the detection plane.

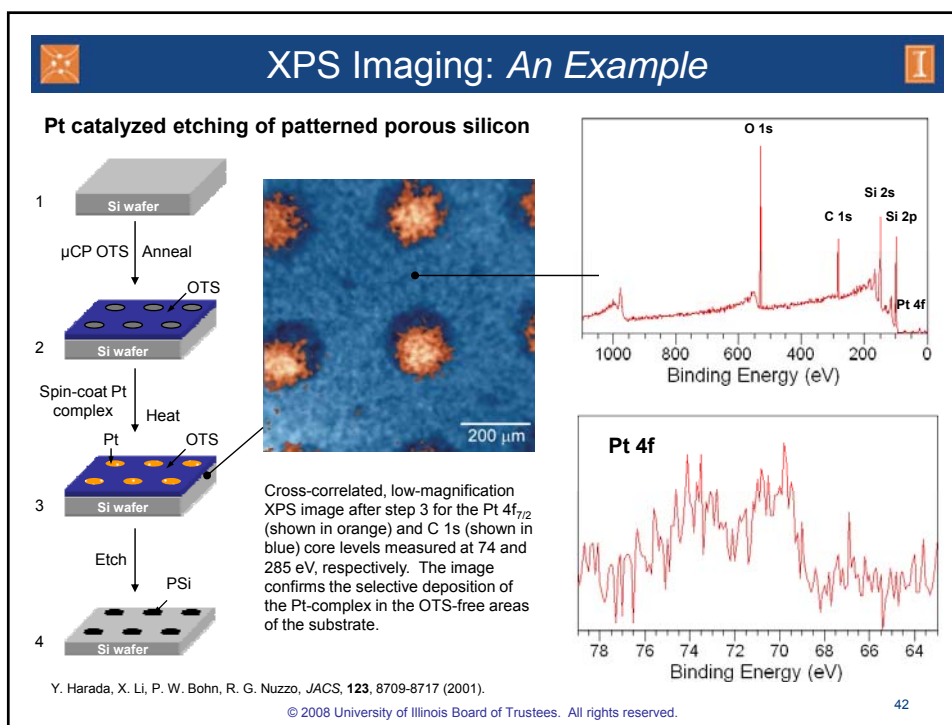
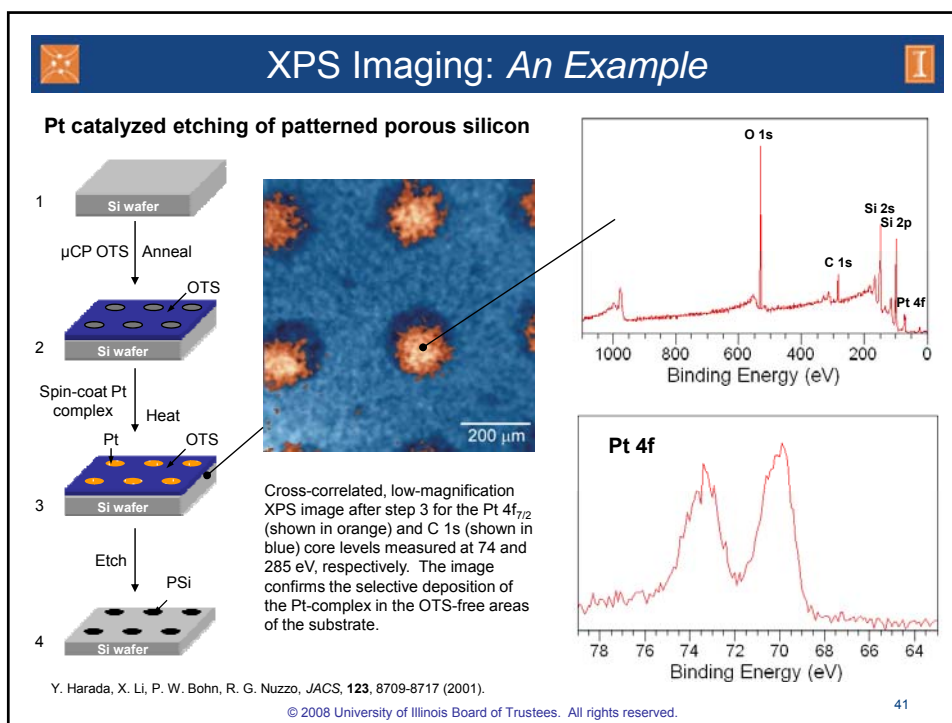


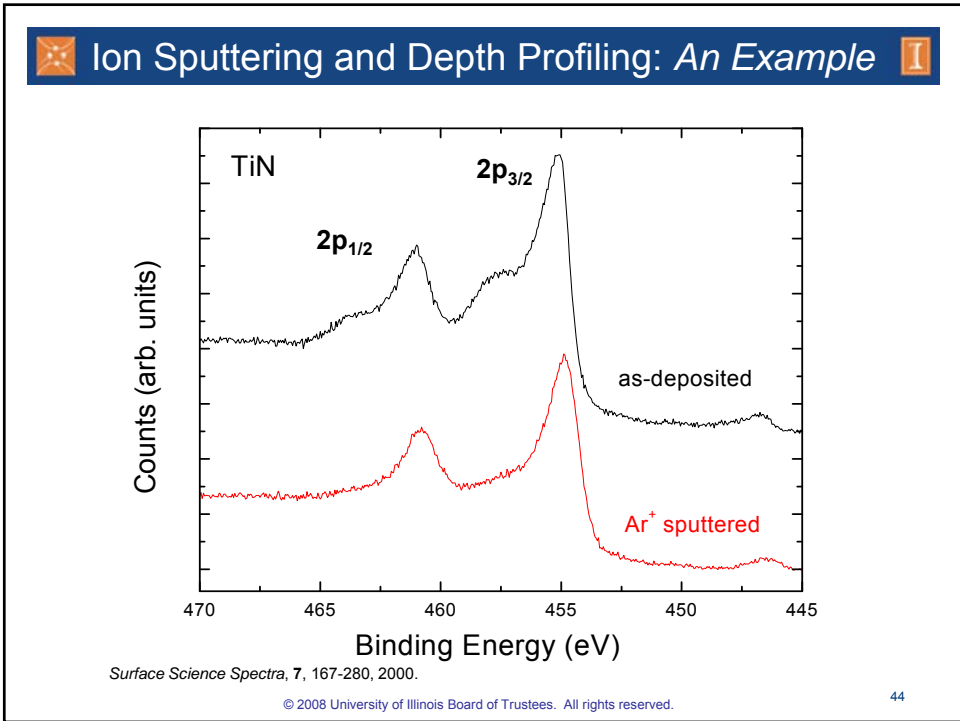
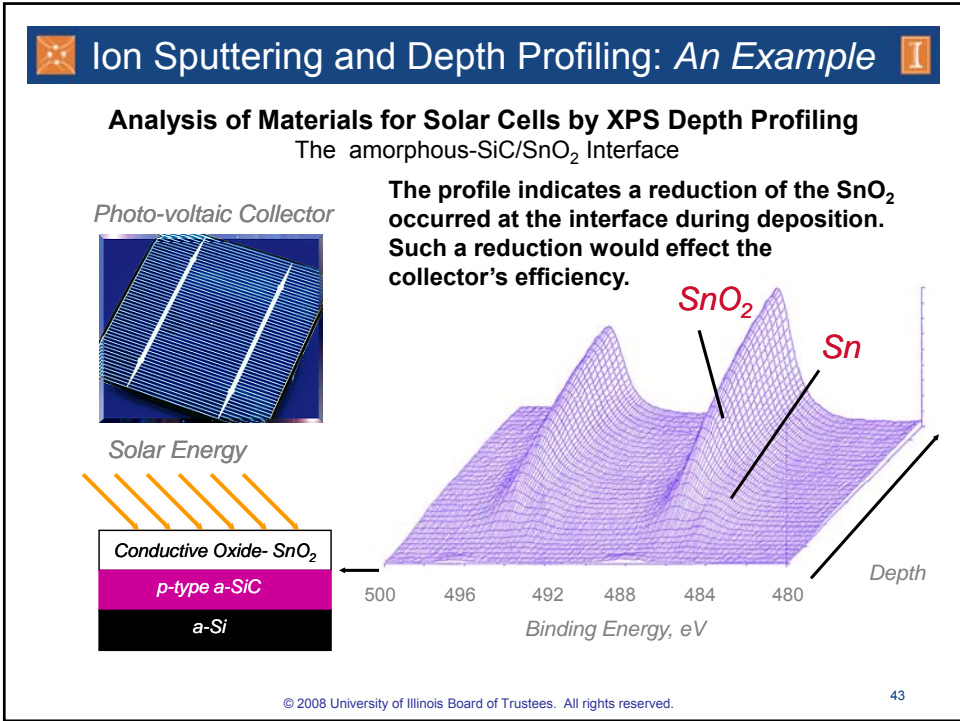
Spectroscopy Mode:
Hemispherical energy analyzer With the entrance aperture closed, an energy dispersed image (reciprocal image *i.e.* spectrum) is projected onto the detection plane.

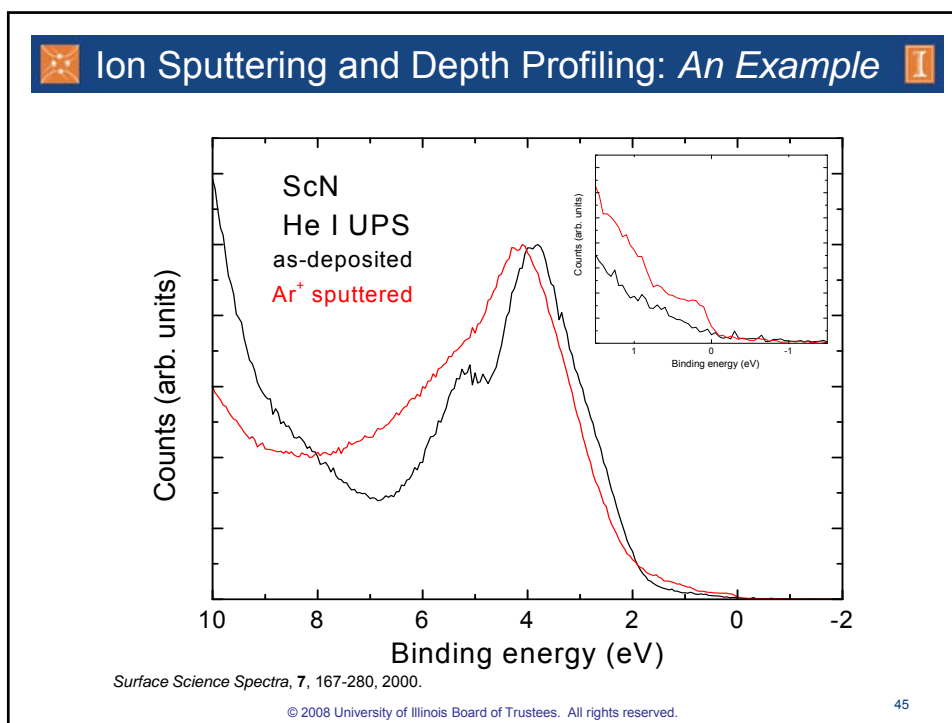
Kratos AXIS Ultra



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XPS and UPS- A Summary

<p>XPS</p> <p>Elements: Li and above.</p> <p>Sensitivity: 0.1 – 1 atomic %</p> <p>Destructive: No, some beam damage to sensitive materials.</p> <p>Elemental Analysis: Yes, semi-quantitative without standards, quantitative with standards, not a trace analysis technique.</p> <p>Chemical State Information: Yes, for most elements.</p> <p>Depth Resolution: 0.5 – 5 nm.</p> <p>Lateral Resolution: Spectroscopy- 1 mm to 40 mm, Imaging- 5 mm.</p> <p>Sample Types: Solid UHV-compatible, conducting, semiconducting and insulating.</p>	<p>UPS</p> <p>Elemental Analysis: Not usually, sometimes from low BE core levels.</p> <p>Destructive: No, some beam damage to sensitive materials.</p> <p>Chemical State Information: Yes, but complicated from valence levels, for core levels same as XPS.</p> <p>Depth Resolution: 0.5 – 5 nm.</p> <p>Lateral Resolution: Several mm.</p> <p>Sample Types: Solid UHV-compatible, conducting or semiconducting are best.</p>
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✦

Auger Electron Spectroscopy


ℐ

Auger Electron Spectroscopy (AES), is a widely used technique to investigate the composition of surfaces.

First discovered in 1923 by **Lise Meitner** and later independently discovered once again in 1925 by **Pierre Auger**¹.



Lise Meitner



Pierre Victor Auger

1. P. Auger, *J. Phys. Radium*, 6, 205 (1925).

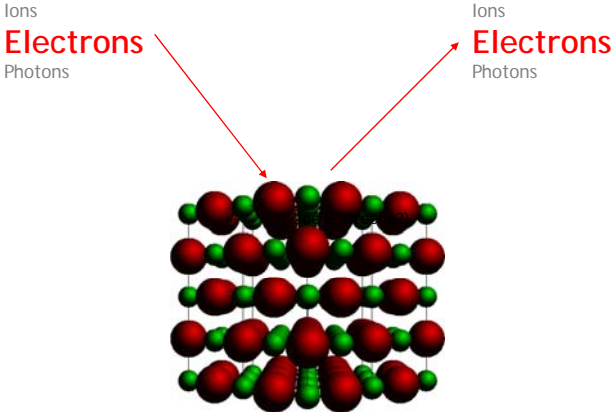
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✦

Particle-Surface Interactions

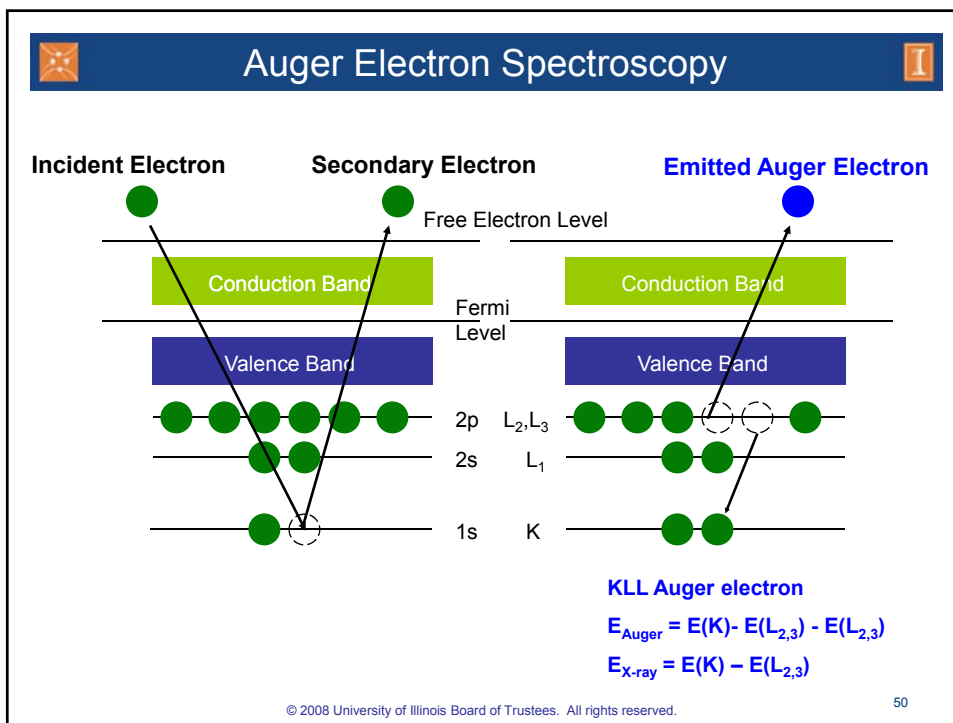
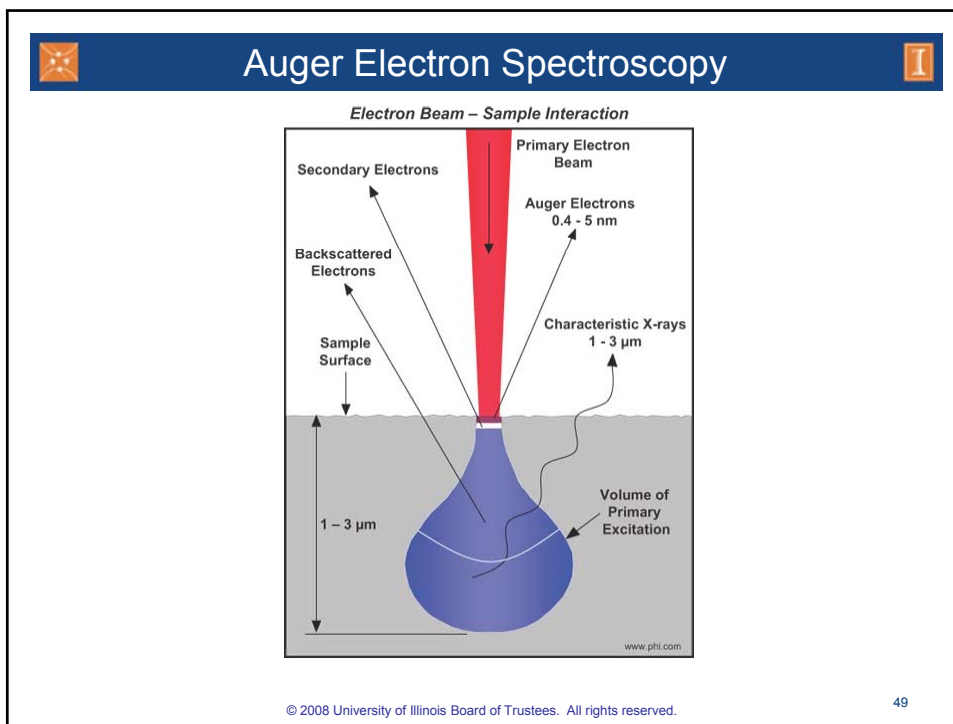
ℐ

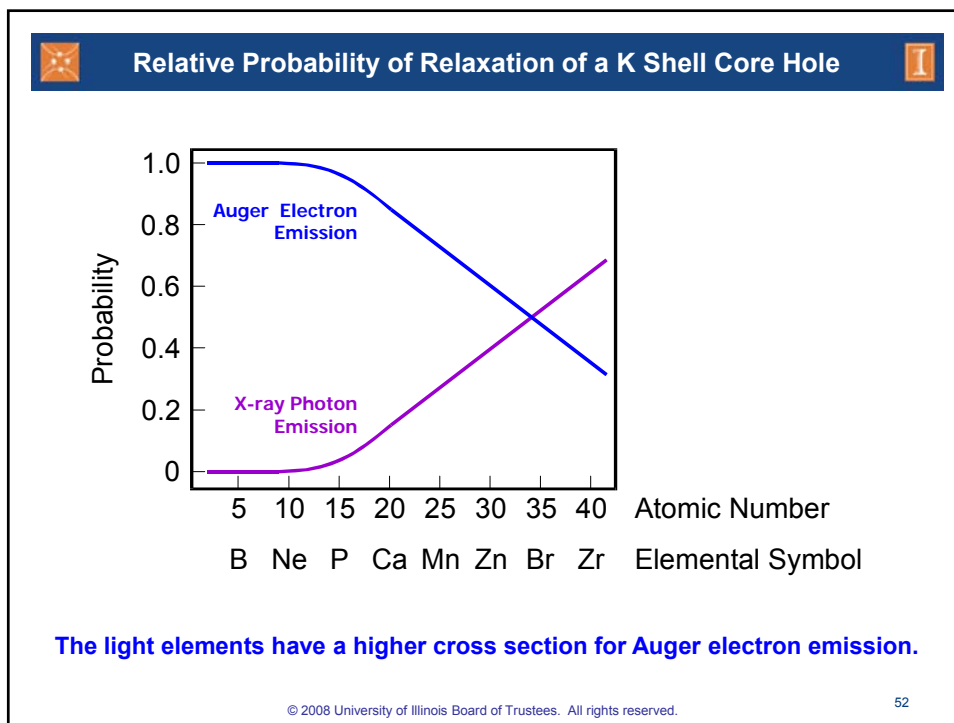
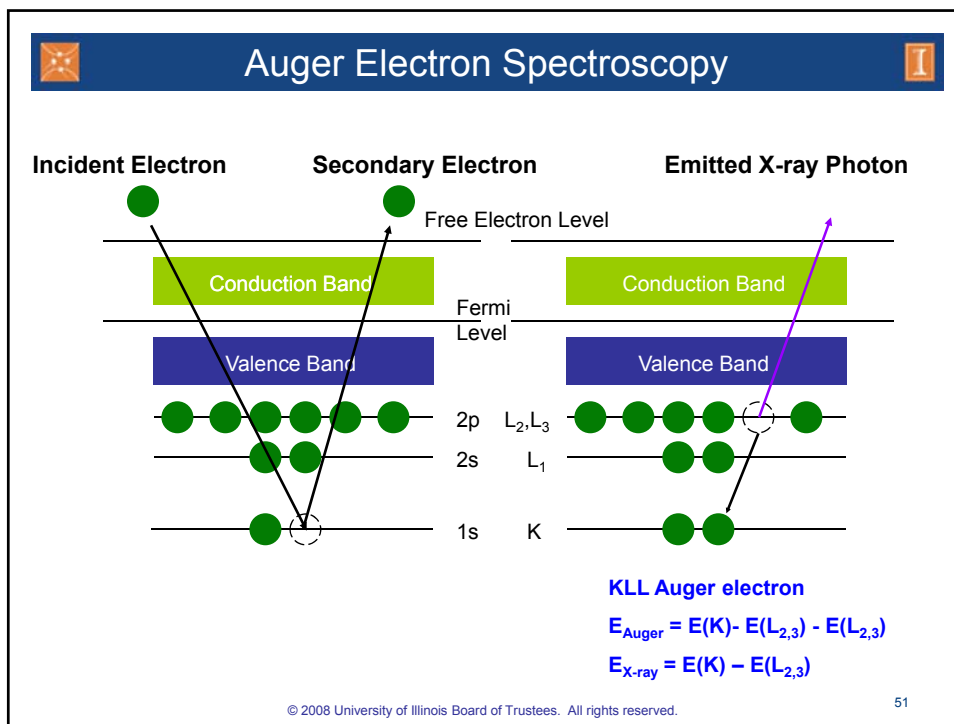
Auger Electron Spectroscopy



Vacuum


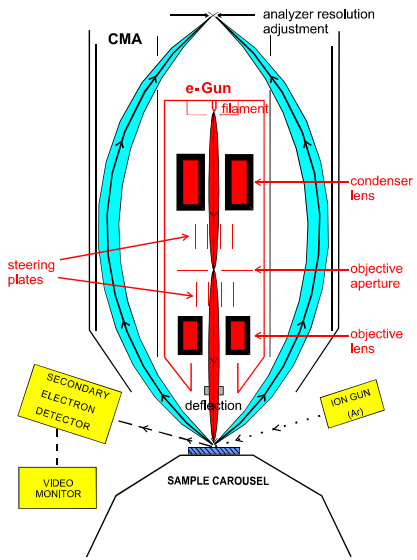
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Scanning Auger Electron Spectrometer

SCANNING AUGER SYSTEM

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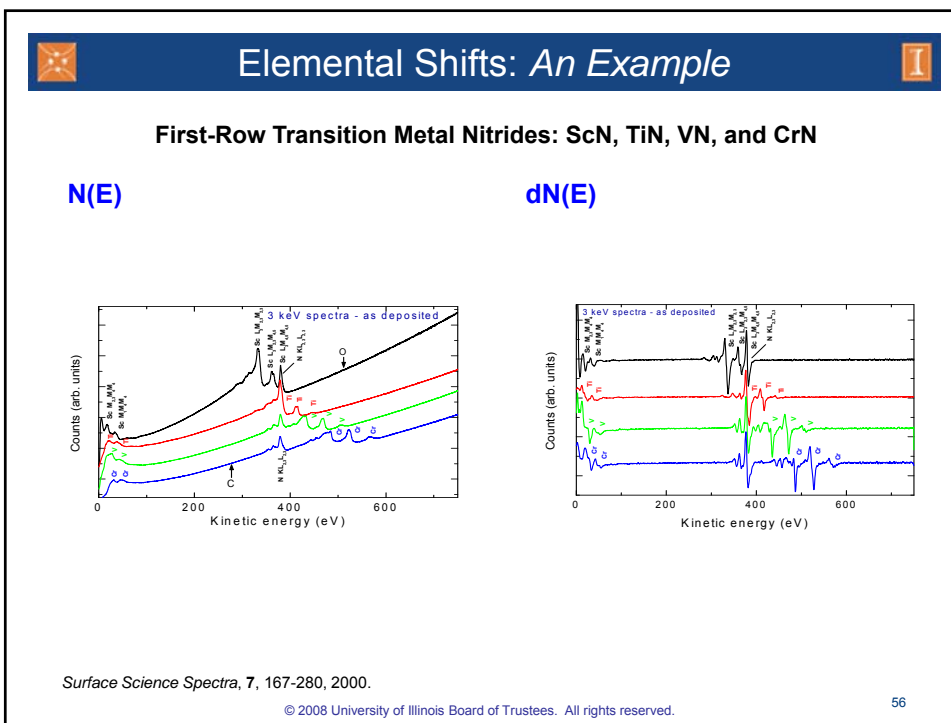
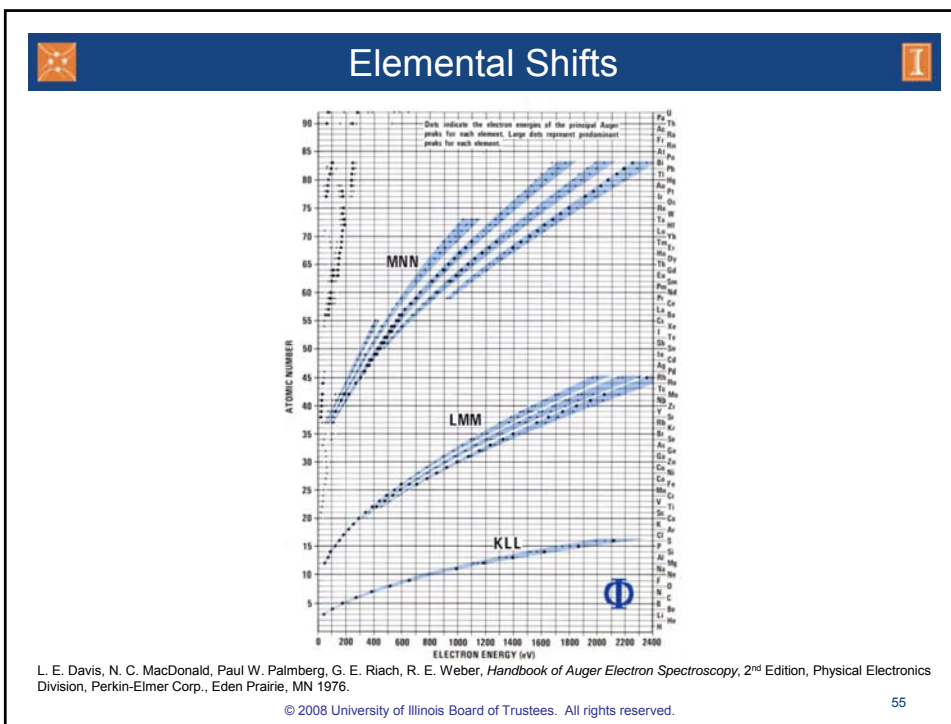
Elemental Shifts

First-Row Transition Metals

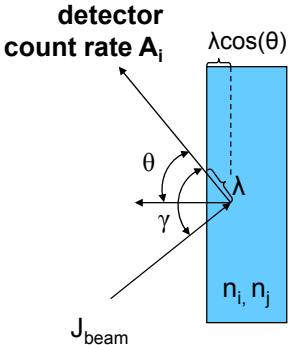
3	4	5	6	7	8	9	10	11	12
21	22	23	24	25	26	27	28	29	30
Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
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Co	778	60	718
Ni	853	67	786
Cu	933	75	858
Zn	1022	89	933

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Quantitative surface analysis: AES



Assuming a Homogeneous sample:
 A_i = detector count rate

$A_i = (\text{electrons/volume})(\text{volume})$
 $A_i = (N_i \sigma_i(\gamma) \chi_i (1+r) J T(E_i)) (a \lambda_i(E_i) \cos \theta)$

Sample Dependent Terms
 where: N = atoms/cm³
 $\sigma(\gamma)$ = ionization (scattering) cross-section, cm²
 χ_i = Auger transition probability
 r = secondary ionization coefficient
 $\lambda(E_i)$ = inelastic electron mean-free path, cm

Instrument Dependent Terms
 J = Electron flux, electron/cm²-sec
 $T(E_i)$ = analyzer transmission function
 a = analysis area, cm²
 θ = Auger electron emission angle

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Quantitative surface analysis: AES

By assuming the concentration to be a relative ratio of atoms, we can neglect the terms that depend only on the instrument:

$$N_i = A_i / \sigma_i \chi_i (1+r) T(E_i) \lambda_i(E_i)$$

It is difficult to accurately determine λ_i and r , so they are usually neglected. Modern acquisition and analysis software can account for the transmission function.

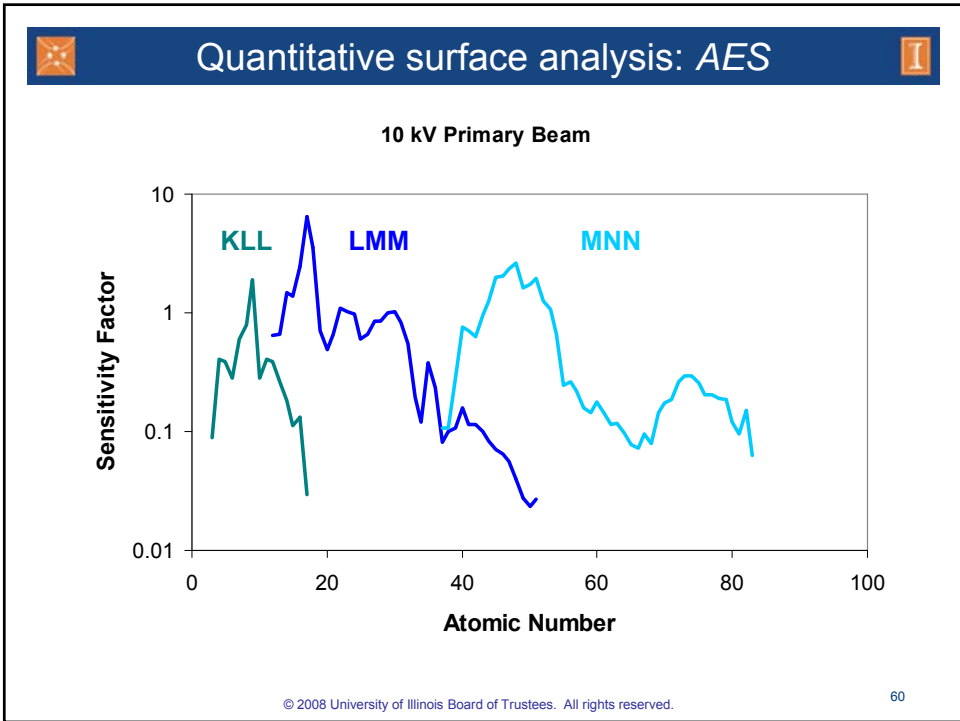
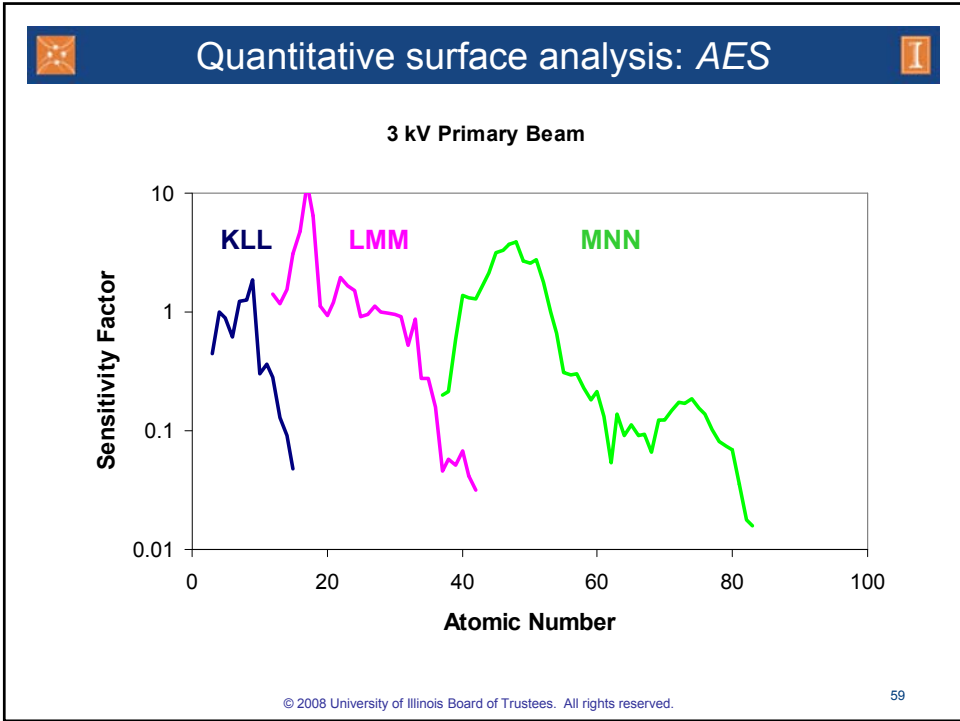
$$N_i = A_i / S_i$$

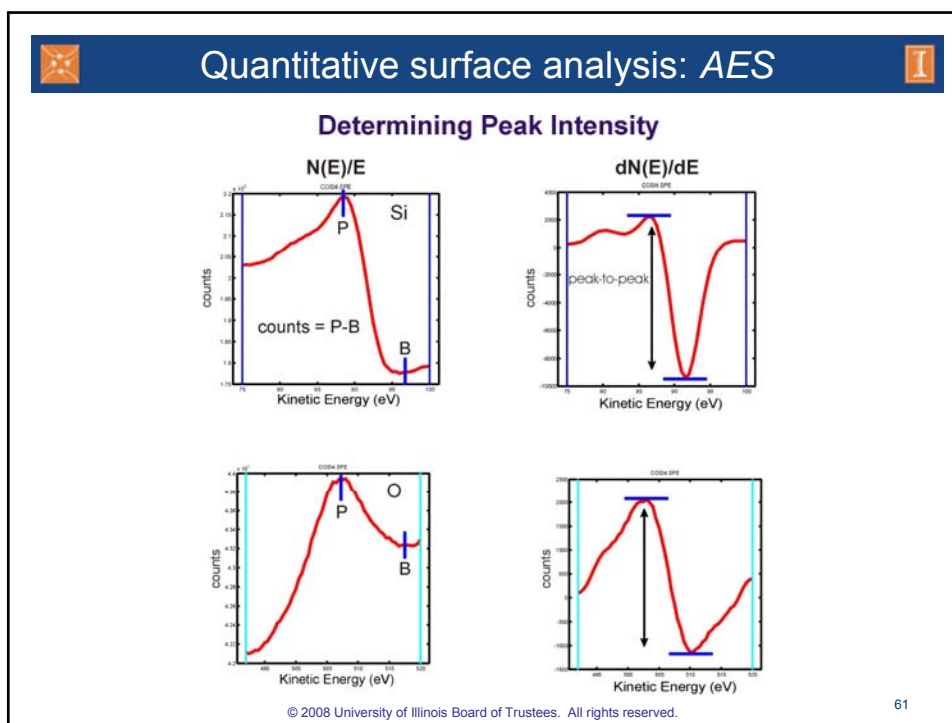
$$C_i = A_i / S_i / \sum A_{i,j} / S_{i,j}$$

The values of S are determined theoretically or empirically with standards.

AES is considered to be a semi-quantitative technique.

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Quantitative surface analysis: *An Example*

First-Row Transition Metal Nitrides: ScN, TiN, VN, and CrN

AES Analysis		ScN	TiN	VN	CrN
Peak energy	Metal $L_3M_{2,3}M_{2,3}$ (α)	337.0	384.2	435.4	486.8
	Metal $L_3M_{2,3}M_{4,5}$ (β)	367.2	417.4	472.0	527.8
	N $KL_{2,3}L_{2,3}$ (γ)	382.2 ^a	... ^b	382.4	381.6
Intensity	As-deposited	I_γ/I_α 1.00	... ^b	1.95	1.69
		I_γ/I_β 2.00	2.52 ^b	1.43	1.30
	After ion bombardment	I_γ/I_α 1.01	... ^b	1.54	1.14
		I_γ/I_β 1.82	2.10	1.01	0.94
Bulk composition from RBS		1.06±0.03	1.02±0.02	1.04±0.02	1.02±0.02

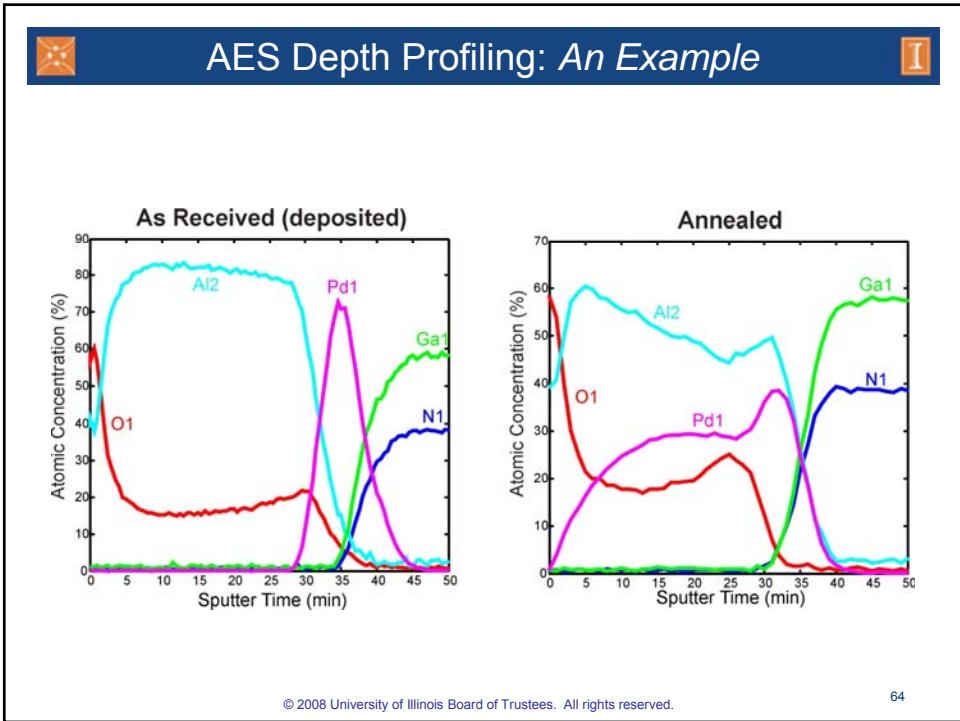
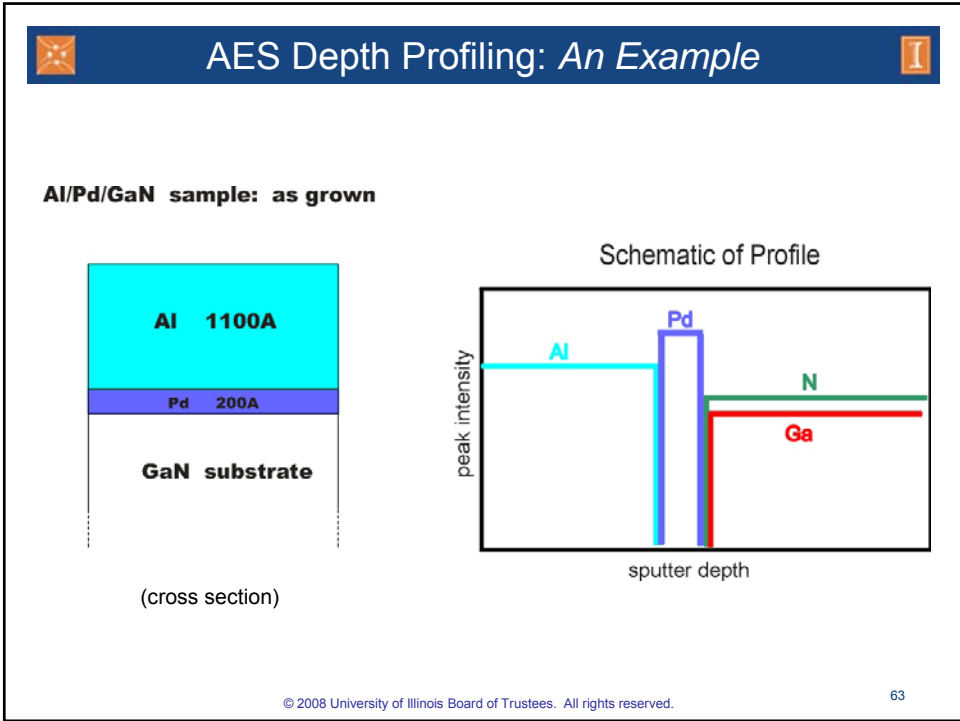
a. The N $KL_{2,3}L_{2,3}$ peak overlaps with the weak Sc $L_3M_{4,5}M_{4,5}$ peak (see spectra). The latter peak is ~6% of the Sc $L_3M_{2,3}M_{2,3}$ in the pure metal spectrum.

b. For the TiN AES spectrum, the N $KL_{2,3}L_{2,3}$ and the Ti $L_3M_{2,3}M_{2,3}$ exhibit severe overlap (see spectra). Therefore, the peak position of N $KL_{2,3}L_{2,3}$ is omitted in the table and the listed peak intensity ratio corresponds to the sum of N $KL_{2,3}L_{2,3}$ and Ti $L_3M_{2,3}M_{2,3}$ divided by Ti $L_3M_{2,3}M_{4,5}$ (i.e., $I_{\alpha+\gamma}/I_\beta$).

Nitrogen/Metal peak ratio decreases after sputtering

Surface Science Spectra, 7, 167-280, 2000.

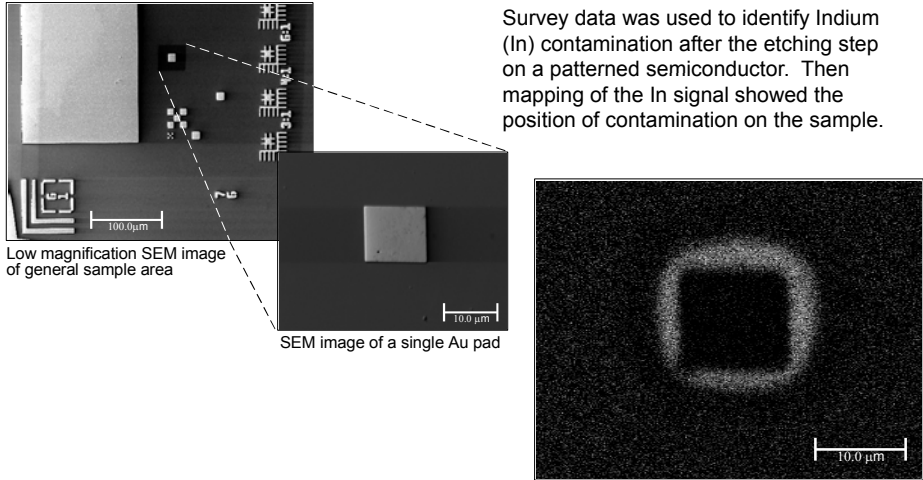
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AES Imaging and Mapping: *An Example*

Contamination on patterned semiconductor

Survey data was used to identify Indium (In) contamination after the etching step on a patterned semiconductor. Then mapping of the In signal showed the position of contamination on the sample.



Low magnification SEM image of general sample area

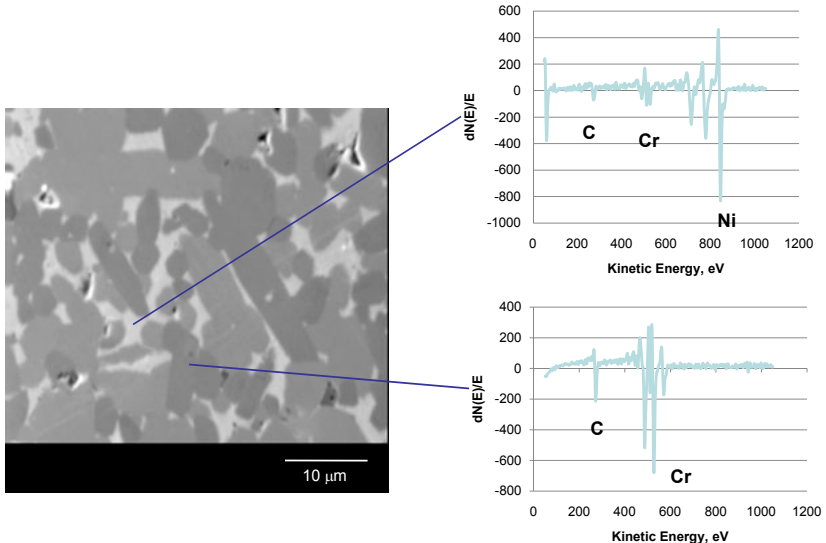
SEM image of a single Au pad

In Map of single Au pad

From research by D. Ahmari/G. Stillman, Electrical and Computer Engineering Dept.
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AES Imaging and Spectroscopy: *An Example*



10 μm

$dN(E)/E$

C Cr Ni

Kinetic Energy, eV

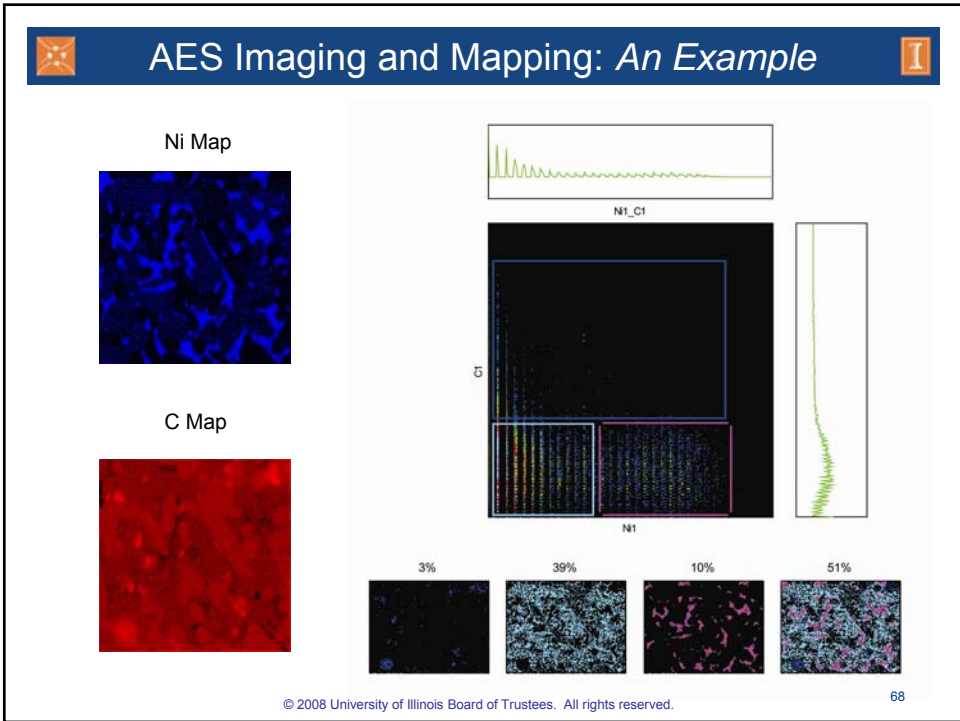
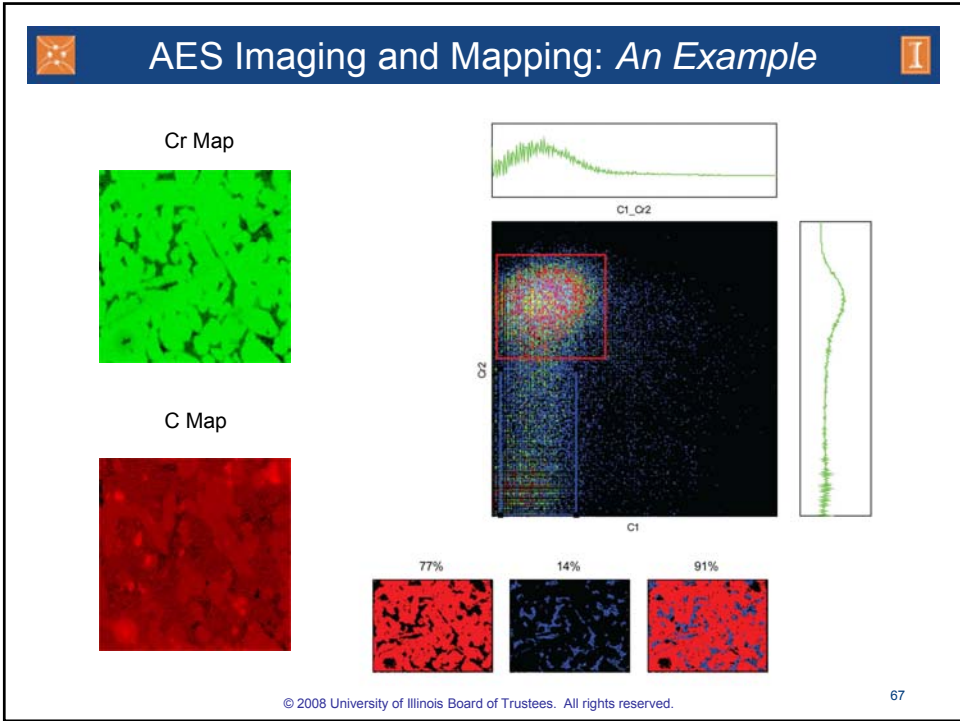
$dN(E)/E$

C Cr

Kinetic Energy, eV

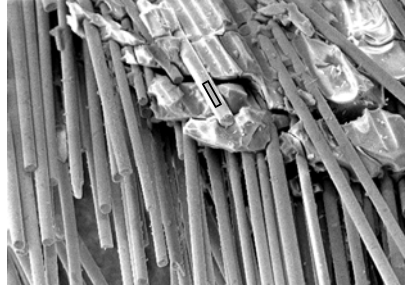
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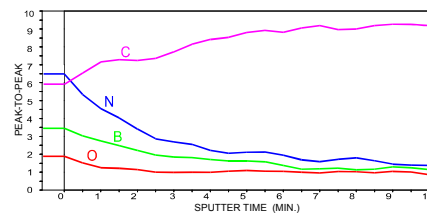
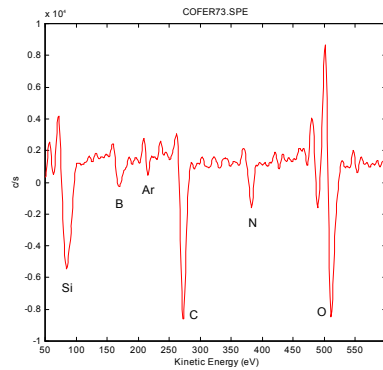


AES Imaging and Depth Profiling: An Example

Electron Beam in combination with an SED detector allows for imaging of the sample to select the area for analysis.



Fracture surface of Carbon fibers in BN matrix - analysis area outlined in black



From research by C. Cofer/J. Economy, Materials Science Dept.
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AES: A Summary

AES

Elements: Li and above.

Sensitivity: 0.1 – 1 atomic %

Destructive: No, some beam damage to sensitive materials.

Elemental Analysis: Yes, semi-quantitative without standards, quantitative with standards, not a trace analysis technique.

Chemical State Information: Yes, for some elements, sometimes requires high-resolution analyzer.

Depth Resolution: 0.5 – 5 nm.

Lateral Resolution: 500 nm.

Sample Types: Solid UHV-compatible, conducting, semiconducting.

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Surface Analysis

Technique Comparison

	AES	XPS	D-SIMS	TOF-SIMS
Probe Beam	Electrons	Photons	Ions	Ions
Analysis Beam	Electrons	Electrons	Ions	Ions
Spatial Resolution	8 nm	9 μm	2 μm	0.1 μm
Sampling Depth	0.5 – 7.5 nm	0.5 – 7.5 nm	0.1 – 1 nm	0.1 – 1 nm
Detection Limits	0.1 – 5 atom %	0.01 – 0.1 atom %	1 ppm*	1 ppm*
Quantification	Good Semi-quantitative	Excellent Semi-quantitative	Challenging Large matrix effects	Challenging Large matrix effects
Information Content	Elemental	Elemental Chemical bonding	Elemental	Elemental Molecular
Insulator Analysis	Challenging	Excellent**	Good**	Excellent**
Organic Analysis	Electron beam damages organics	Excellent	DC ion beam damages organics	Excellent in static mode
Depth Profiling	Excellent for small areas	Excellent for insulating materials	Excellent for speed and sensitivity	Excellent for sensitivity

* 1 ppm sensitivity is achieved by consuming the sample surface
** requires effective charge neutralization apparatus

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