Surface and Thin Film Analysis for Compound Semiconductors

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Analysis: What and How?

Probe (primary) → **Sample** → Modified Probe

→ Response (secondary)

***Probe*:**	photo	***Response*:**	photo
	electron		electron
	ion		ion
	phonon		phonon

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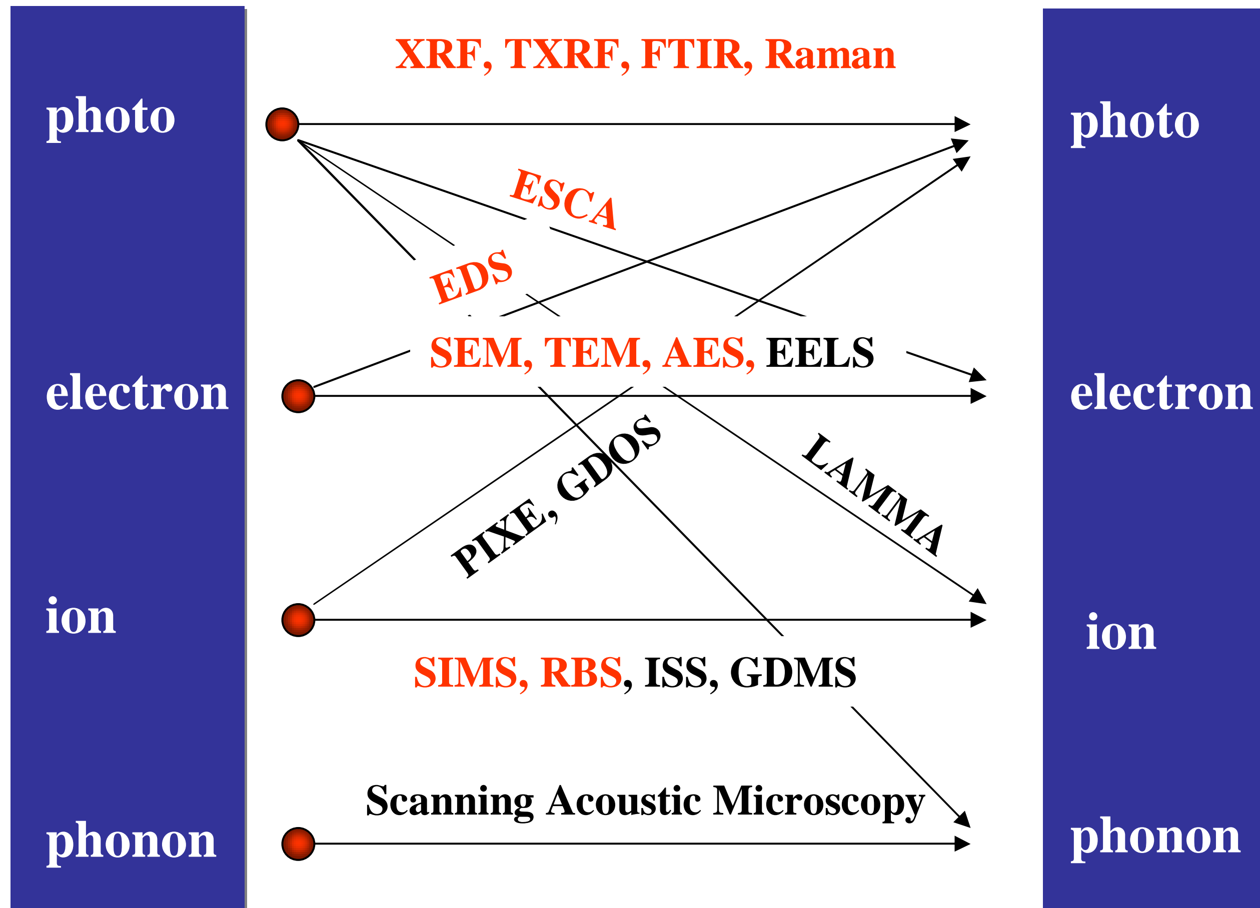
Common Surface and Microanalytical Tools



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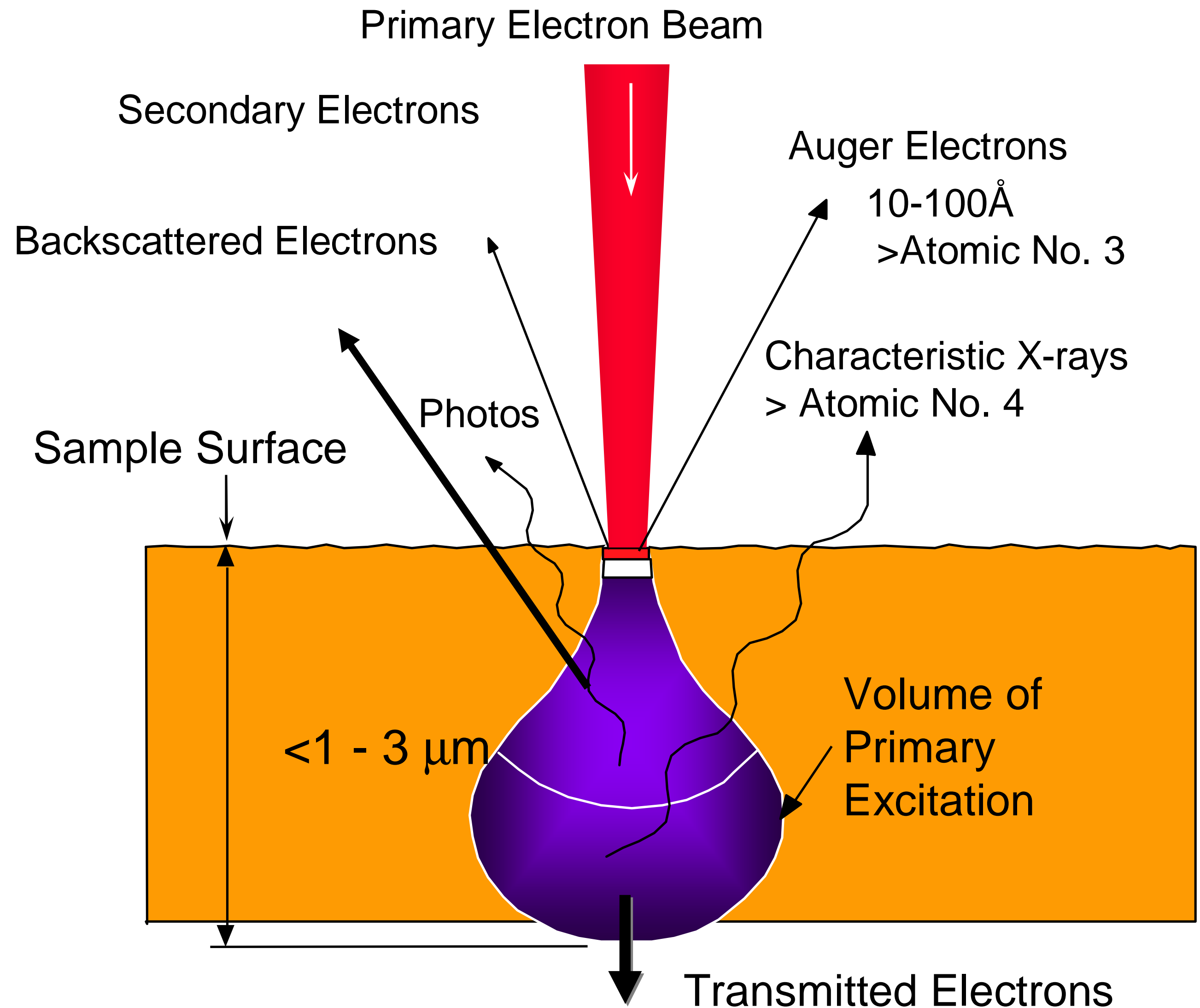
e⁻ BeamTechniques

- SEM
- AES
- TEM
- EDS
- CL
- LEED



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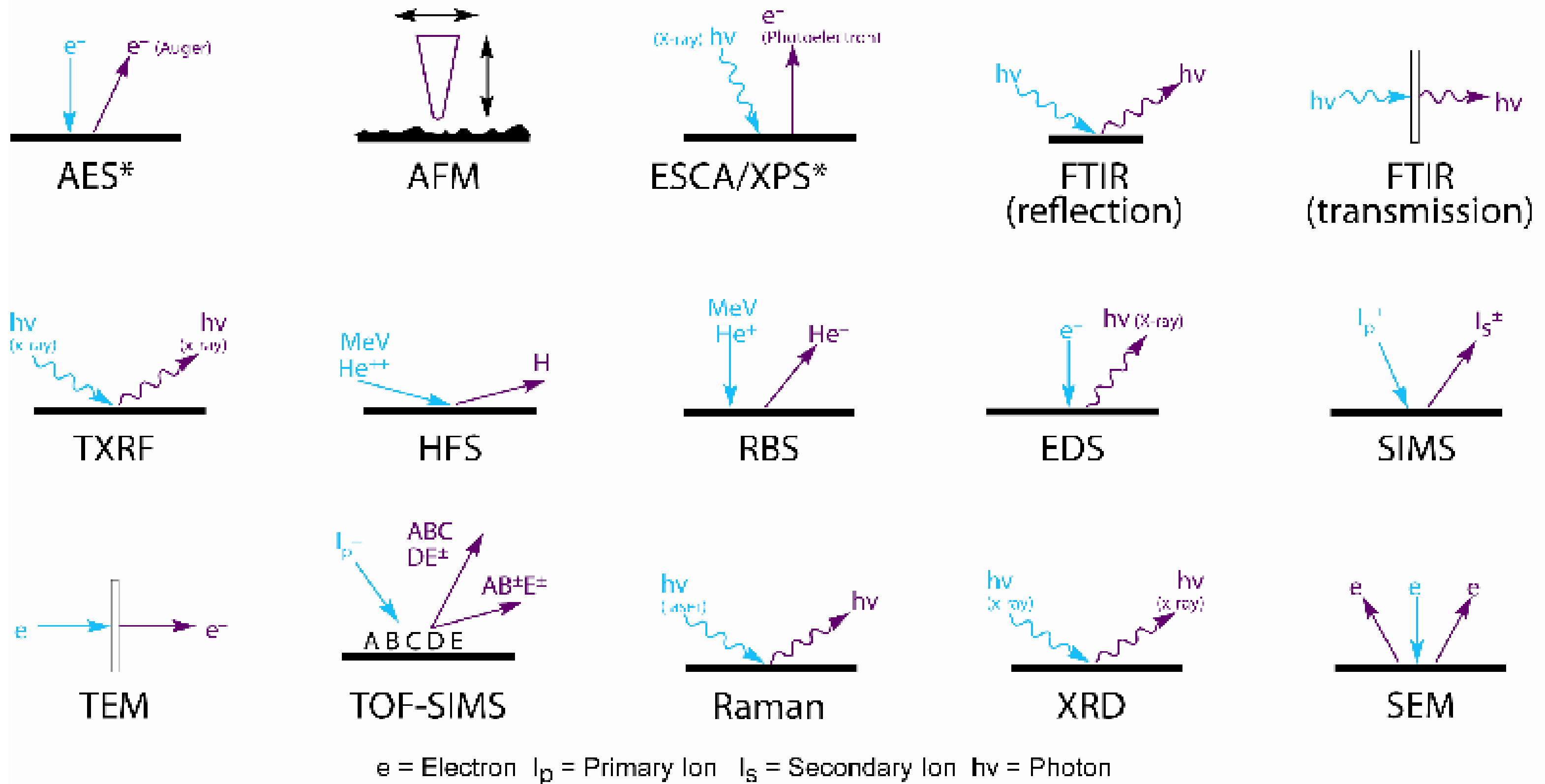
General Principles of Analysis Techniques



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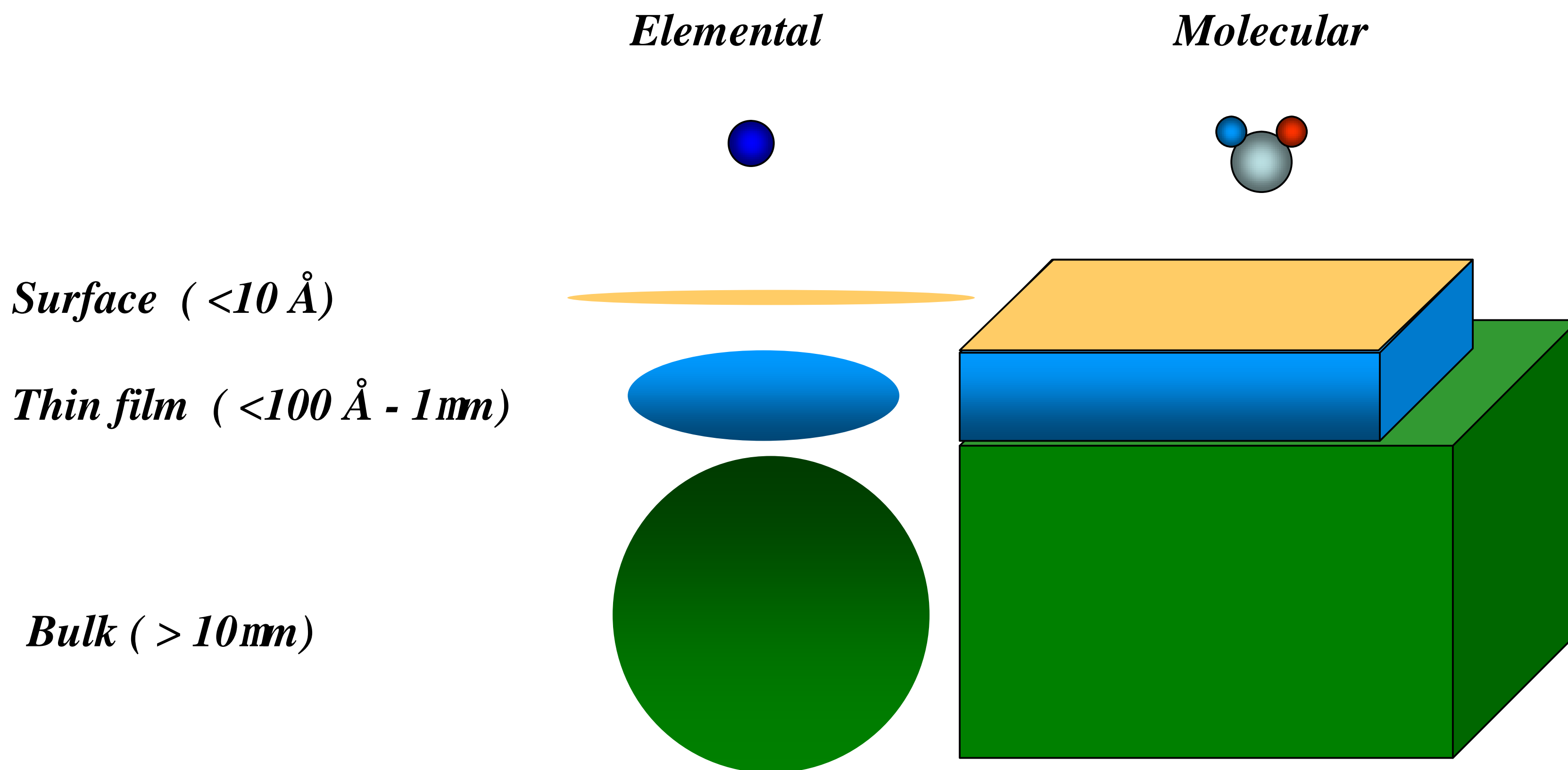
Types of information provided by chemical analysis



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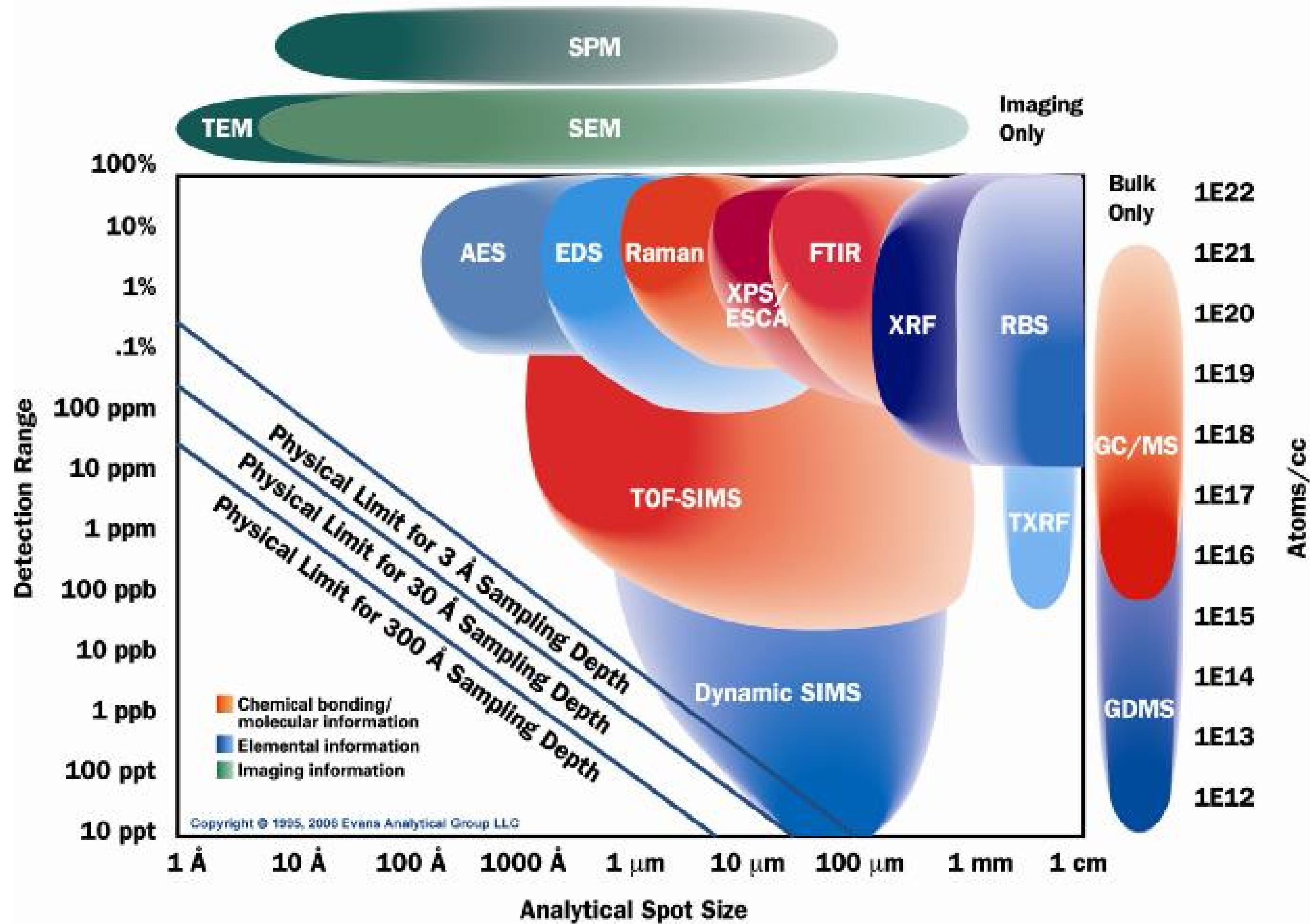
Comparing Analytical Techniques



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Excellent "first look" tool:
Energy Dispersive X-Ray Spectroscopy

(EDS)

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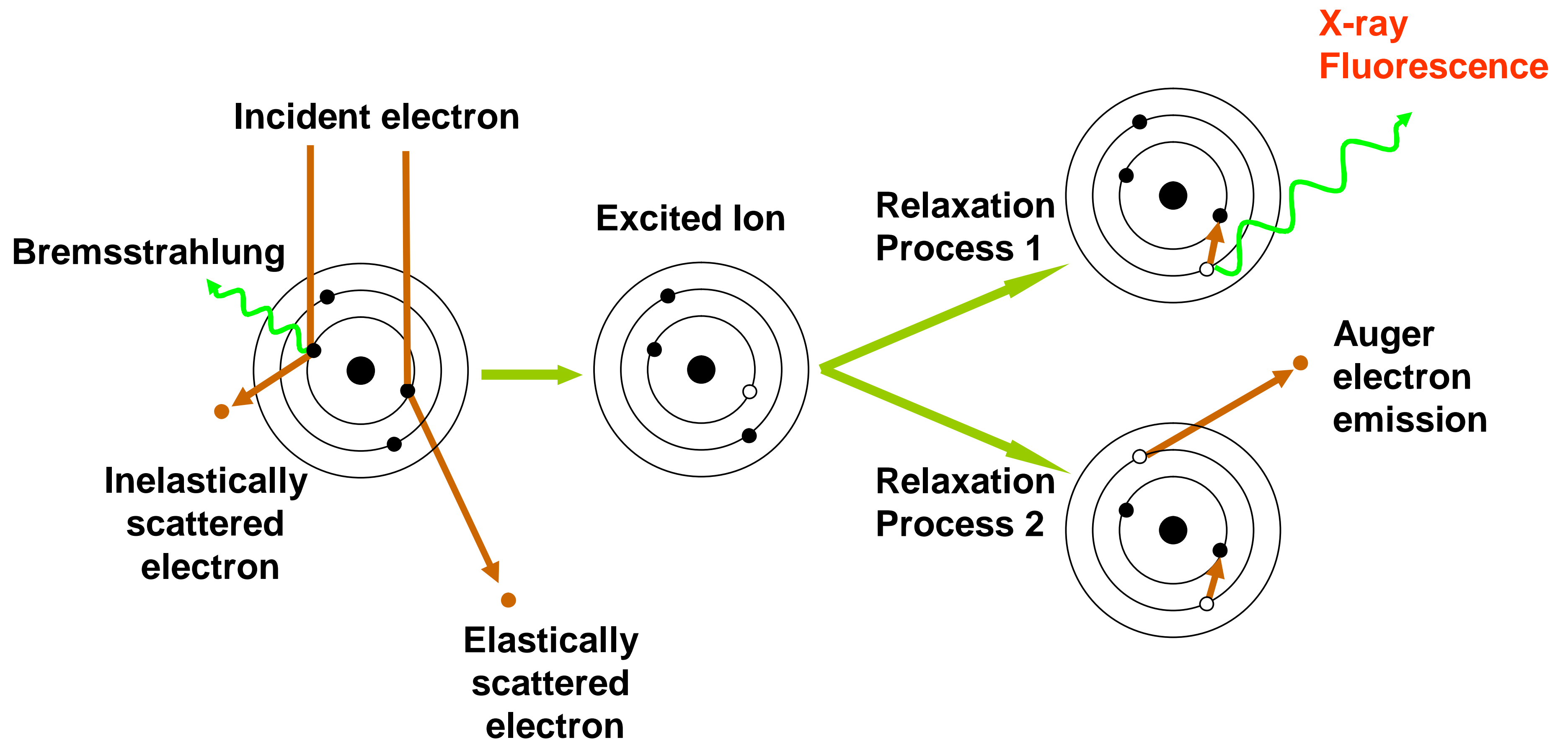
Physics of the Process



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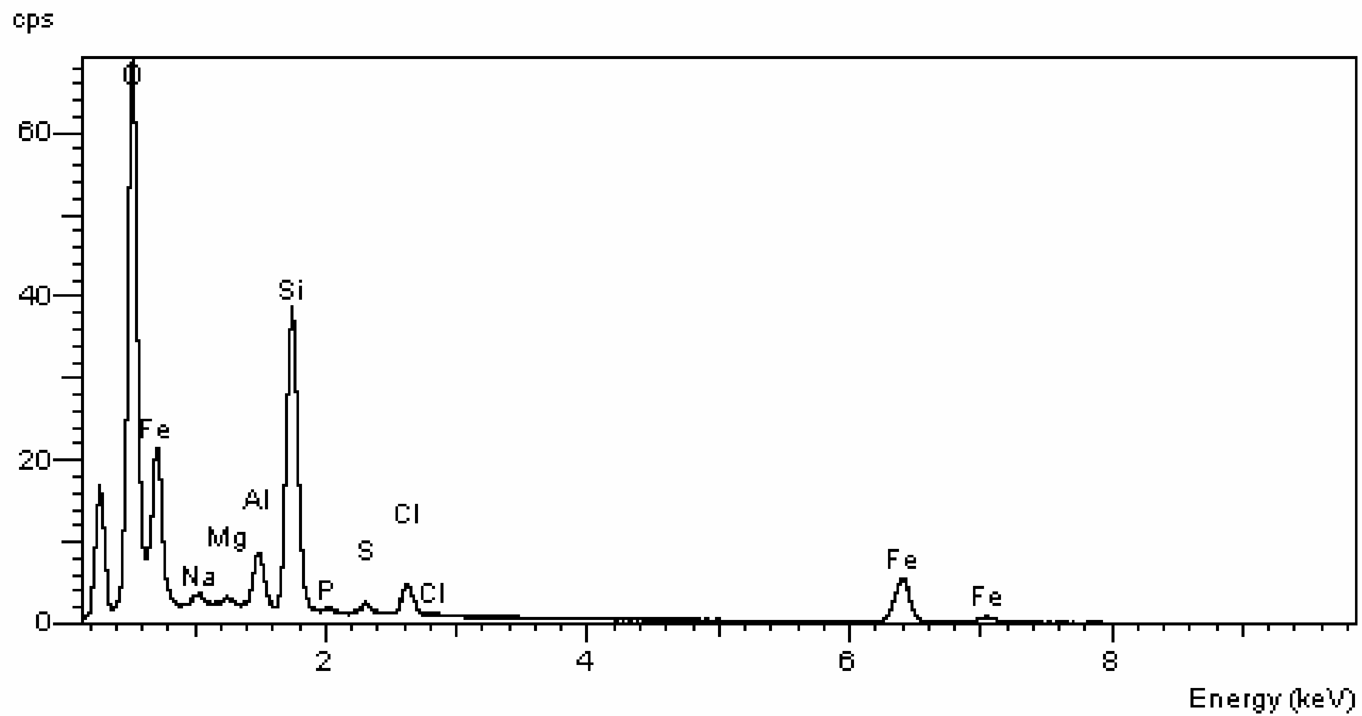
Typical EDS Data

Survey Spectrum of Particle



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Key Applications

- Near surface and bulk elemental information
  - Films > few hundred Å thick can be detected
  - Sampling depth to several microns can be achieved
  - Quantitative for some classes of samples
- Particles and small area analysis
  - Used primarily on particles > few thousand Å
  - Parallel detection makes elemental imaging fast
  - Quick ‘first look’ technique for failure analysis

Excellent “first look” tool

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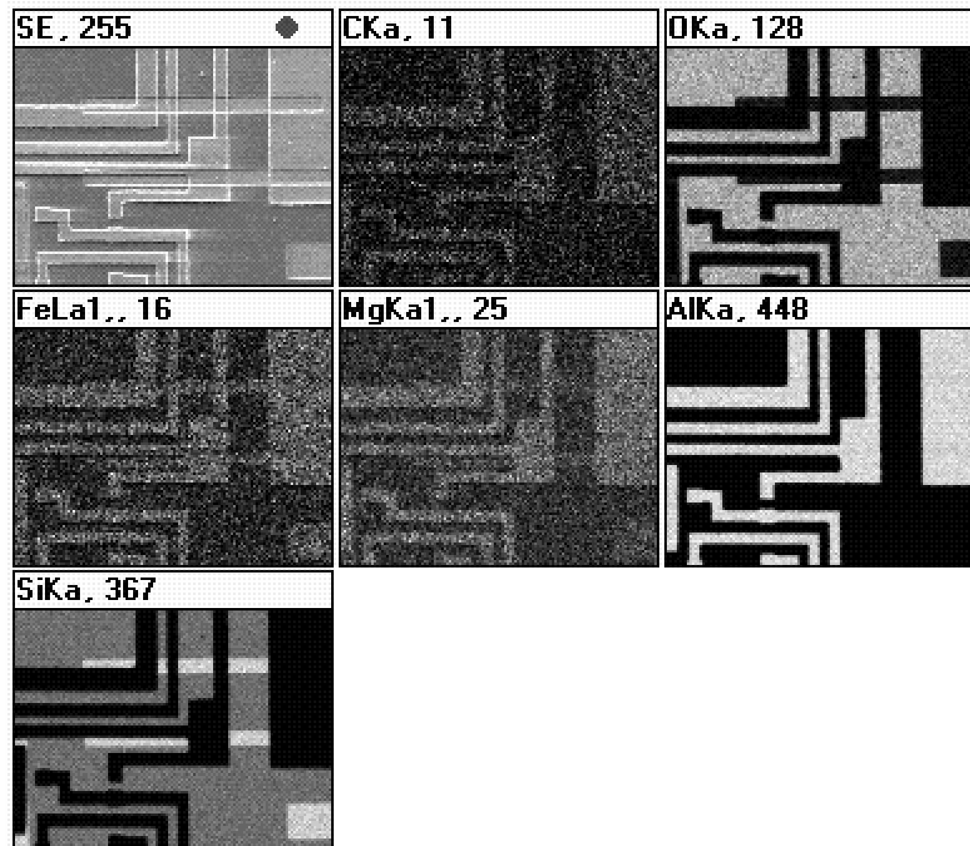
Typical EDS Data

Elemental Imaging



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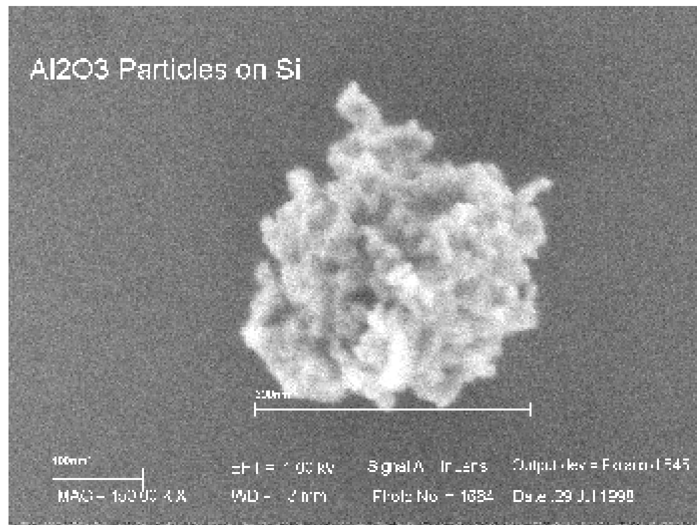
Small Particle Analysis



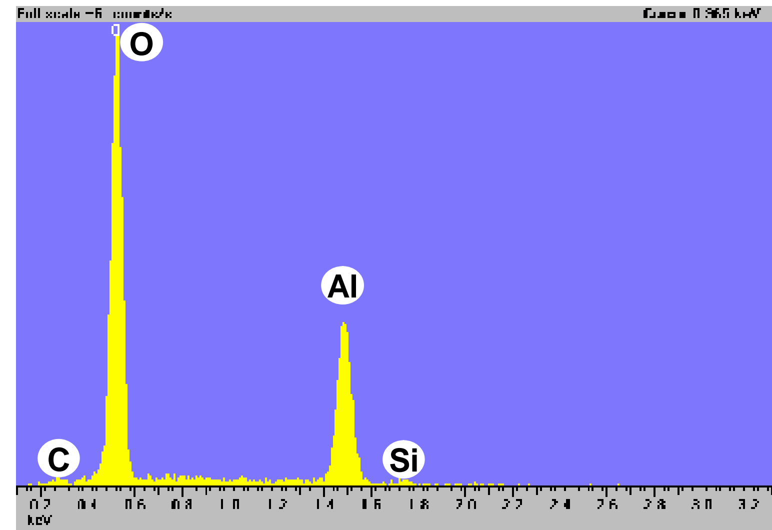


Image and EDS spectrum of 0.3 micron alumina particle on Si (3 keV beam)

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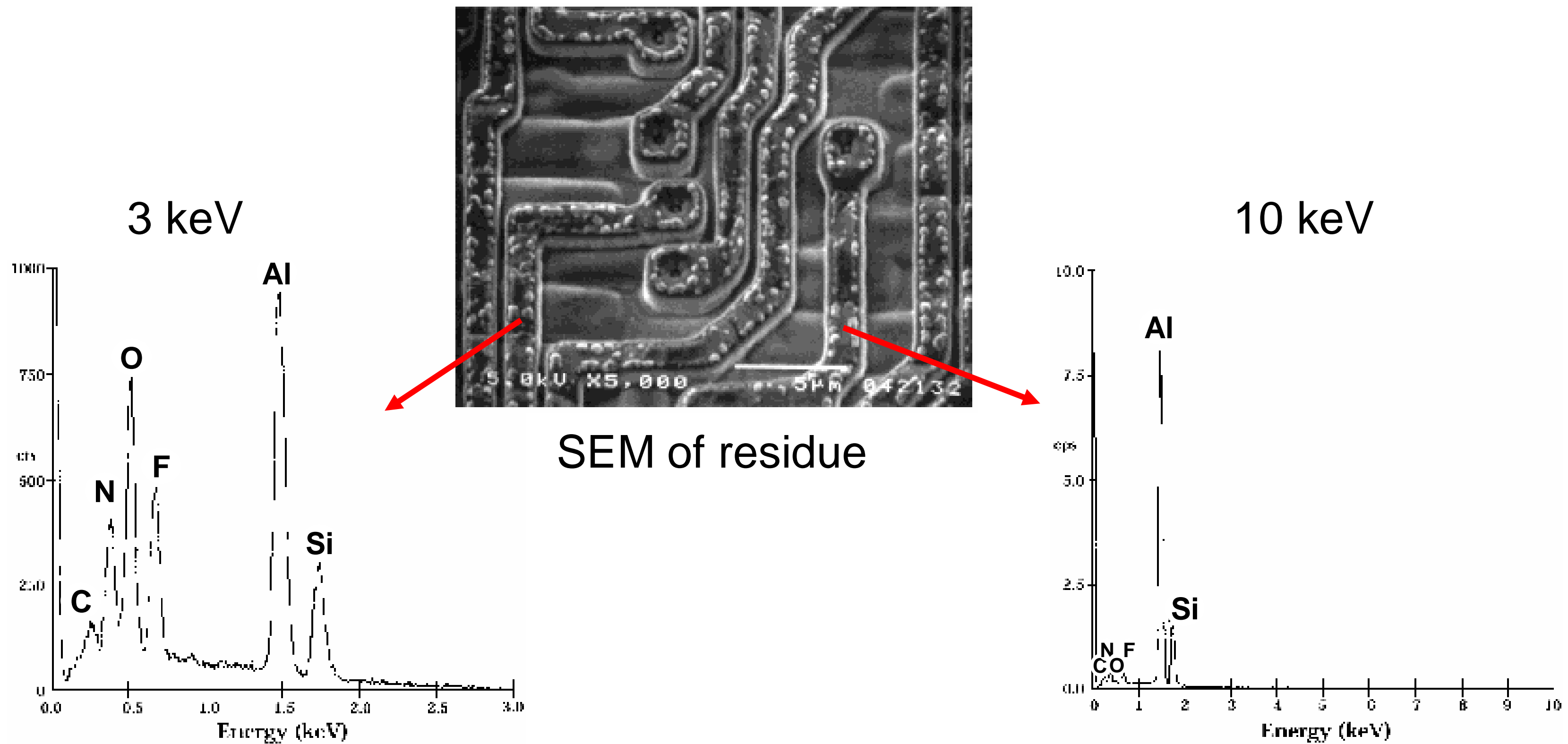
Thin Film Analysis

Higher Beam Energy Samples Deeper



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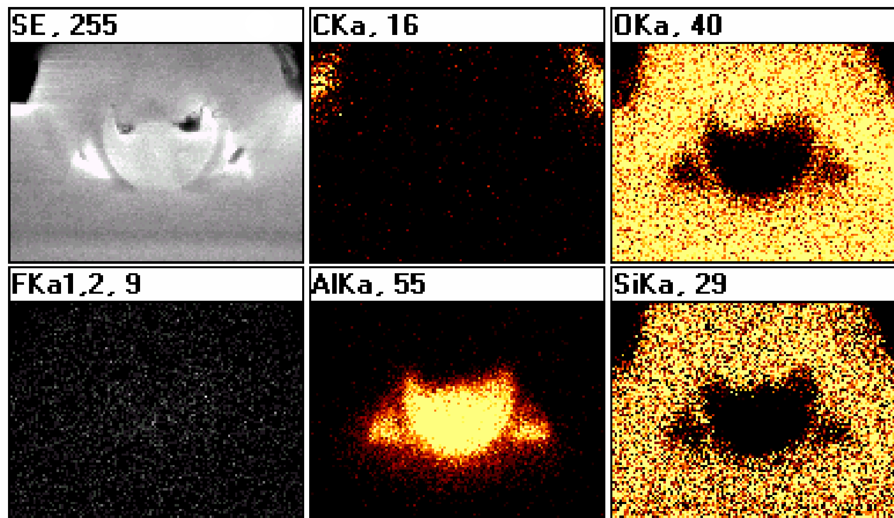
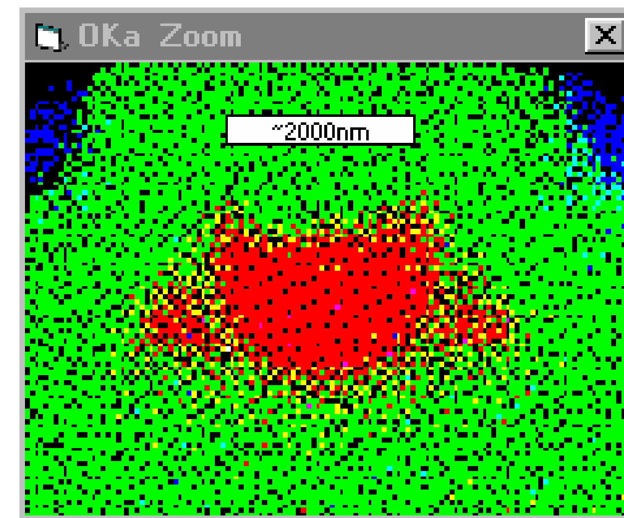
Elemental Imaging

Sub-Micron Resolution



Elemental images of Al inclusion in coating



Overlay of C, O and Al (red) images

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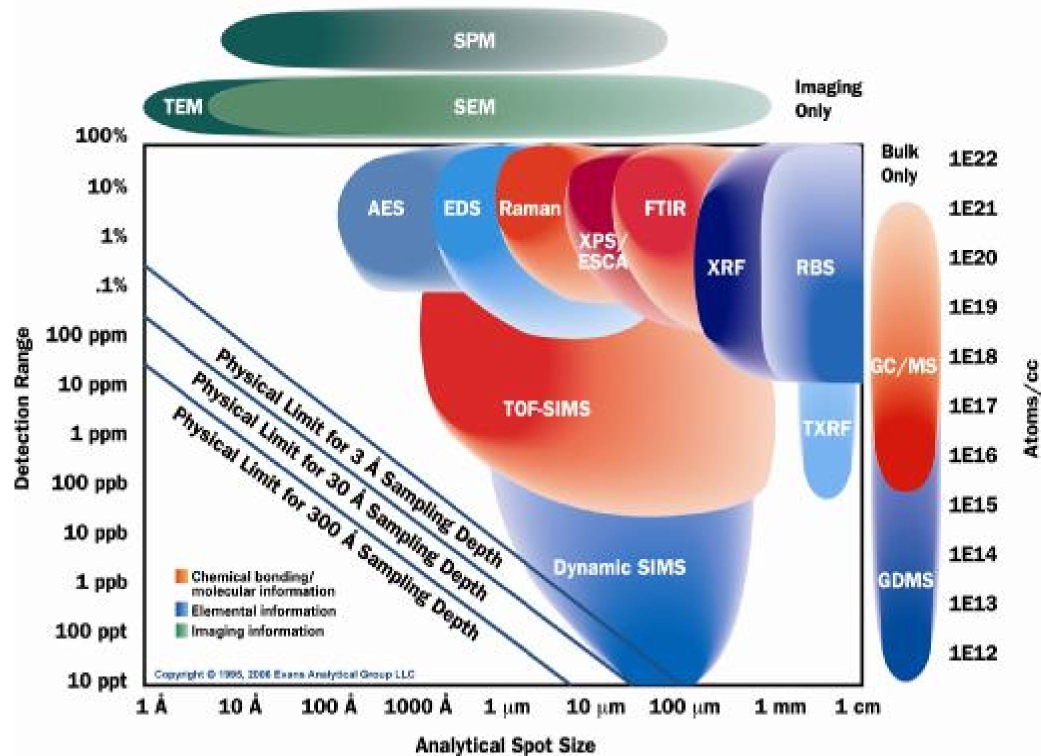
Technical Data Table



EDS

Quantitative	Yes	Destructive	No
Detection Limits	0.1-1.0at%	Lateral Resolution/ Probe Size	0.2-2µm
Chemical Bonding	No	Analytical Depth	0.5-3µm

Comment: Excellent “first look” tool

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Strengths and Weaknesses

- **Strengths**
  - Quick, ‘first look’ analysis
  - Versatile, inexpensive, and widely available
  - Quantitative for some samples (flat, polished, homogeneous)
- **Weaknesses**
  - Semi-quantification for samples that are not flat, polished & homogeneous
  - Size restrictions on samples
  - Samples must be vacuum compatible
  - Analysis (and coating) may spoil subsequent surface analysis
  - Poor low Z sensitivity
  - Not very surface sensitive
  - No depth resolution

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Surface Sensitive / Depth resolution: Auger Electron Spectroscopy

(AES)

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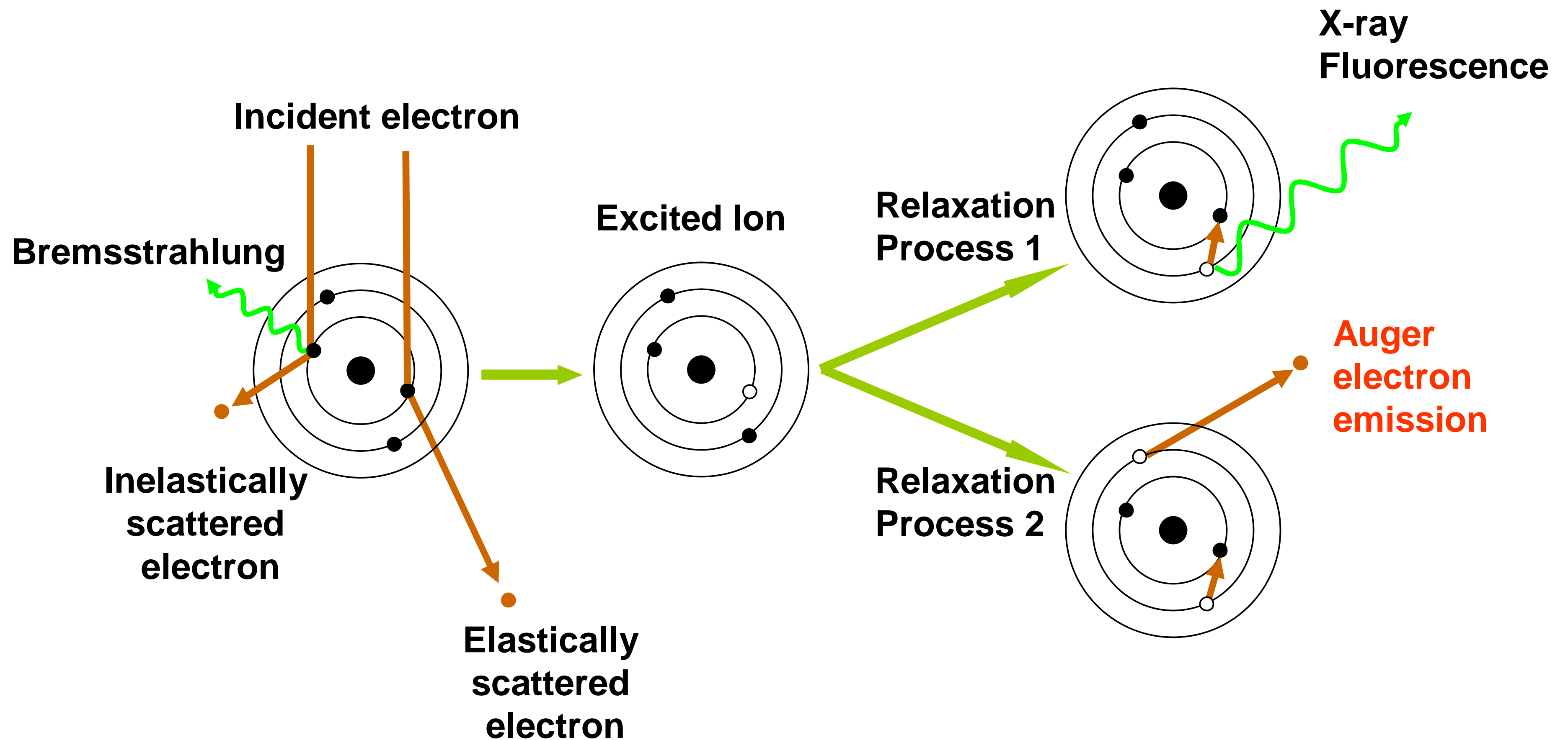
Physics of the Process



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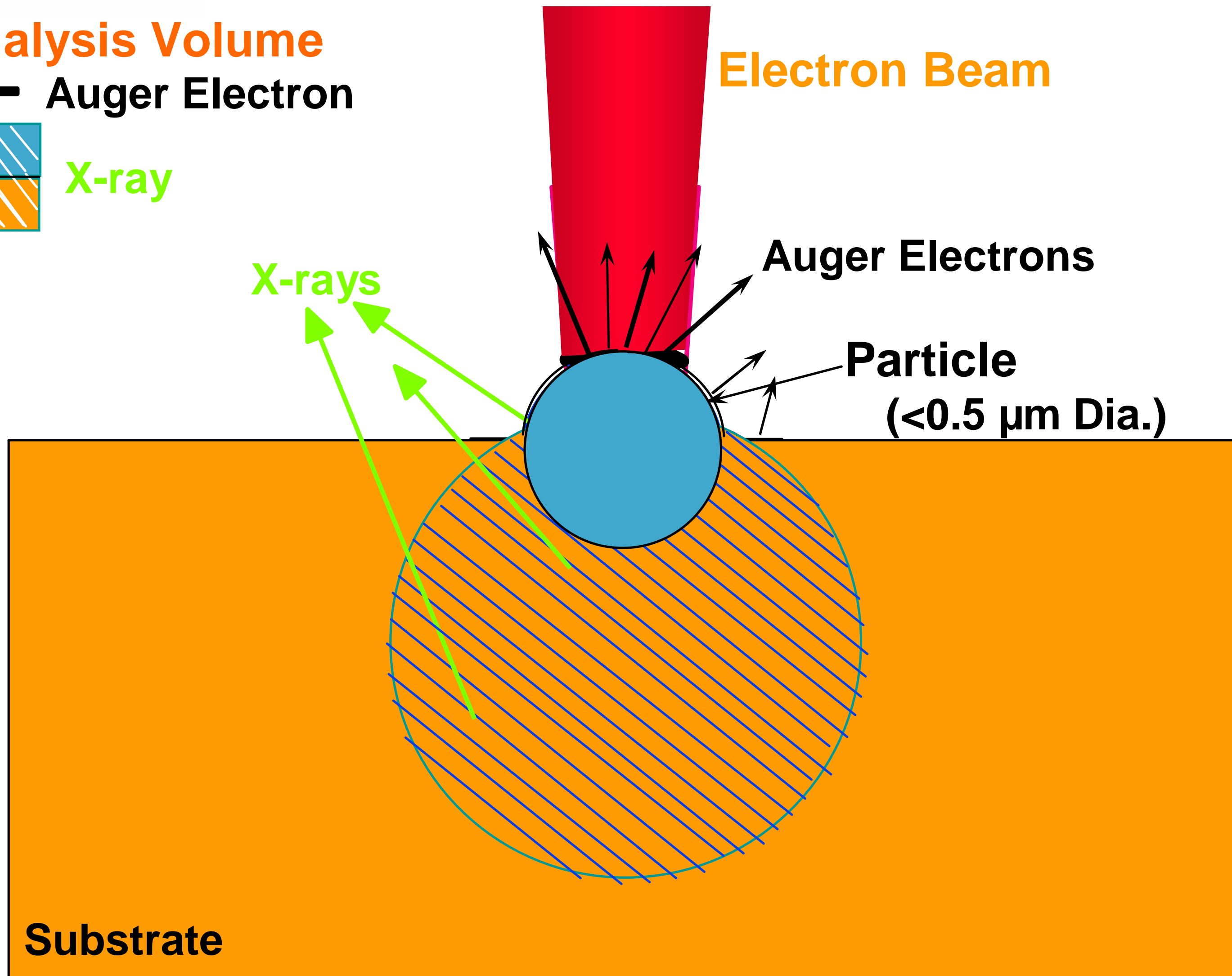
Particle Analysis



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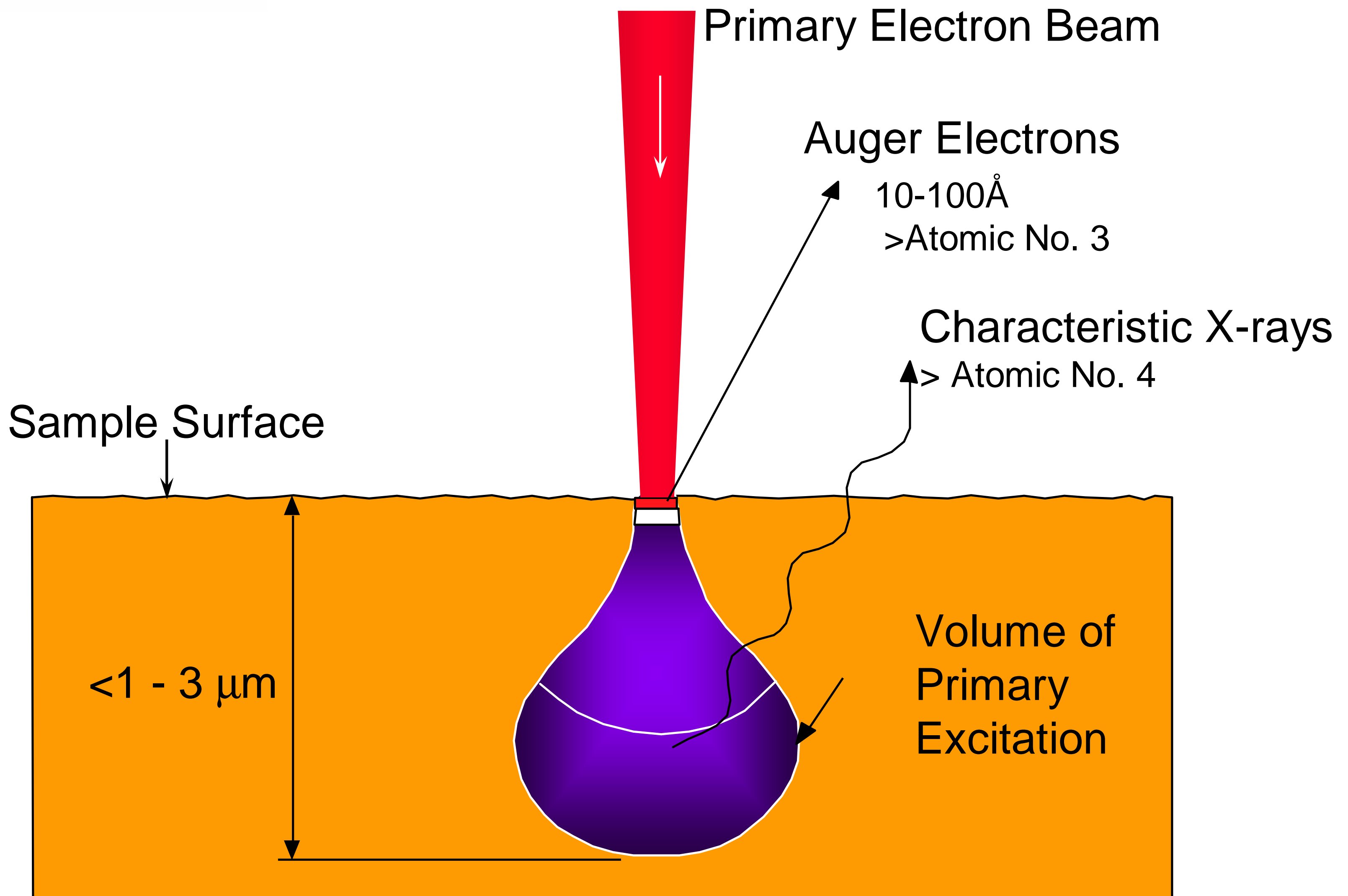
Electron Beam – Sample Interaction



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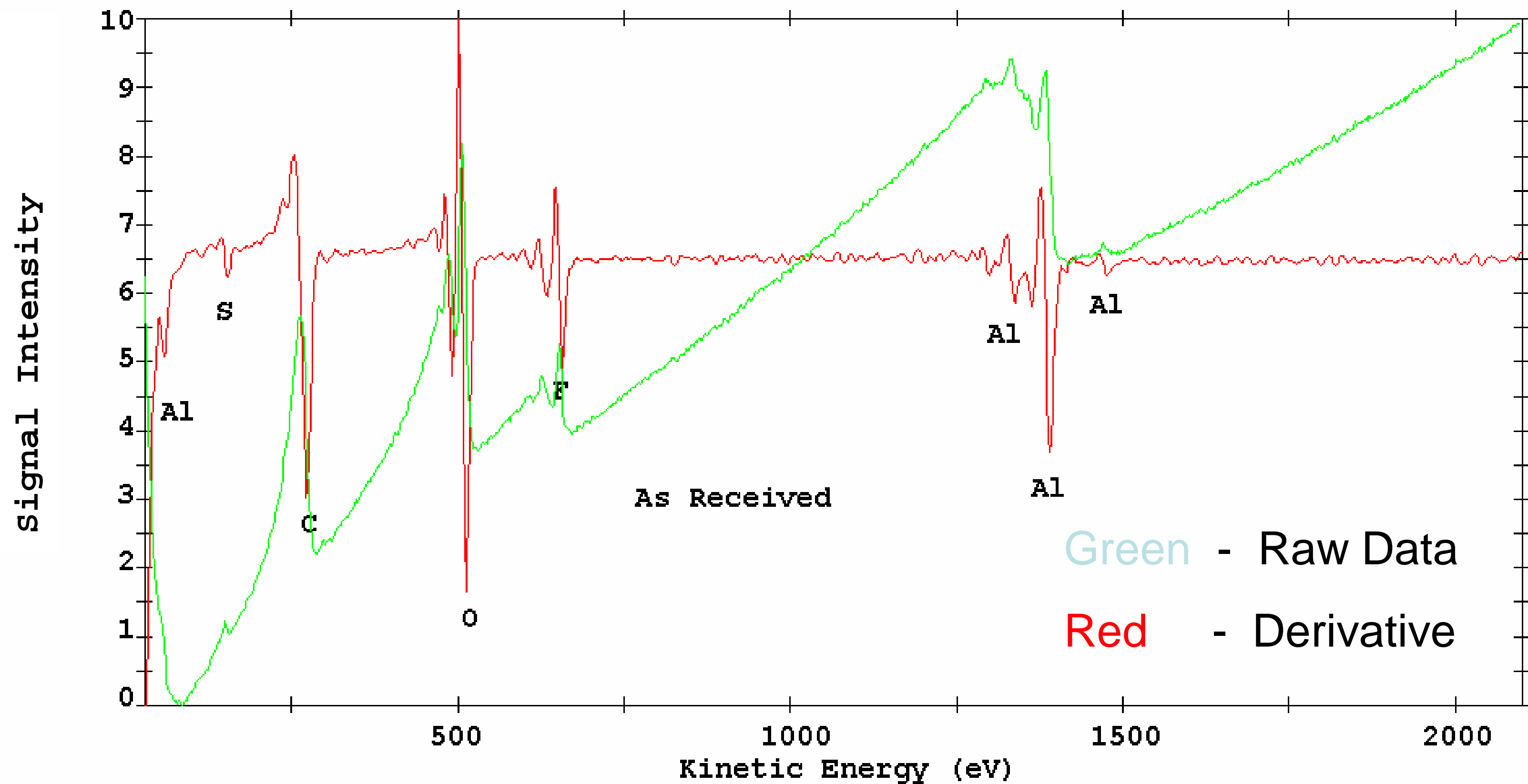
Typical Auger Survey Data

Derivatized data is used for peak identification



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Key Applications

- Particles and small area analysis
  - Particles as small as 25 nm can be analyzed
- Sub-monolayer analysis
  - Films too thin for EDS
- Thin film elemental analysis
  - Where high depth resolution is needed
  - Sampling depth to several microns with sputtering
  - Quantitative (best with standards)

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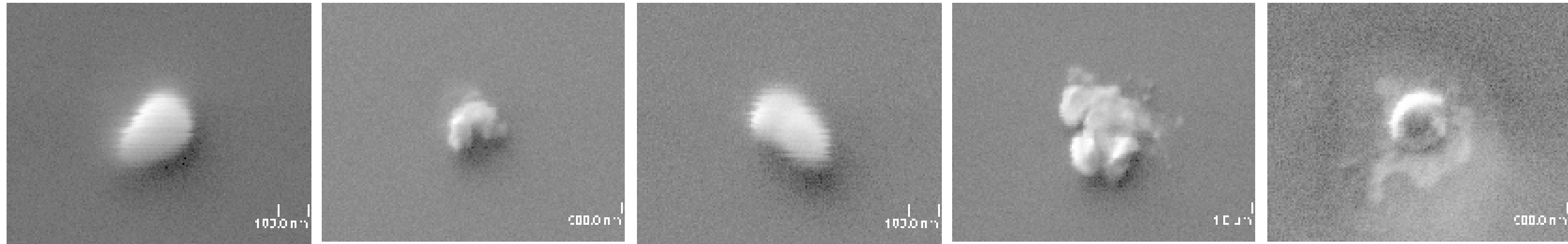
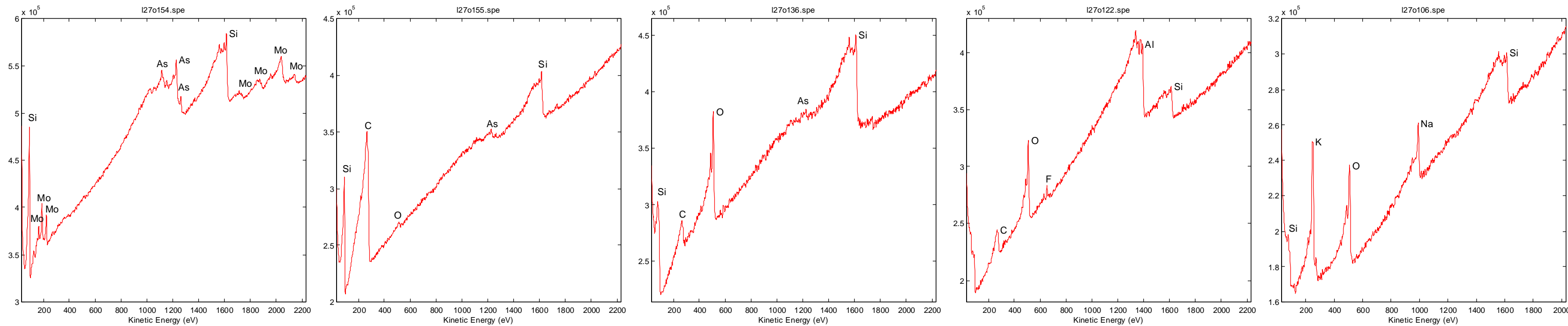
Particles on Ion Implanted Wafer

SEM



Auger Spectra



Elements

Mo, As | C, As | Si, O | Al, O, F | K, Na, O

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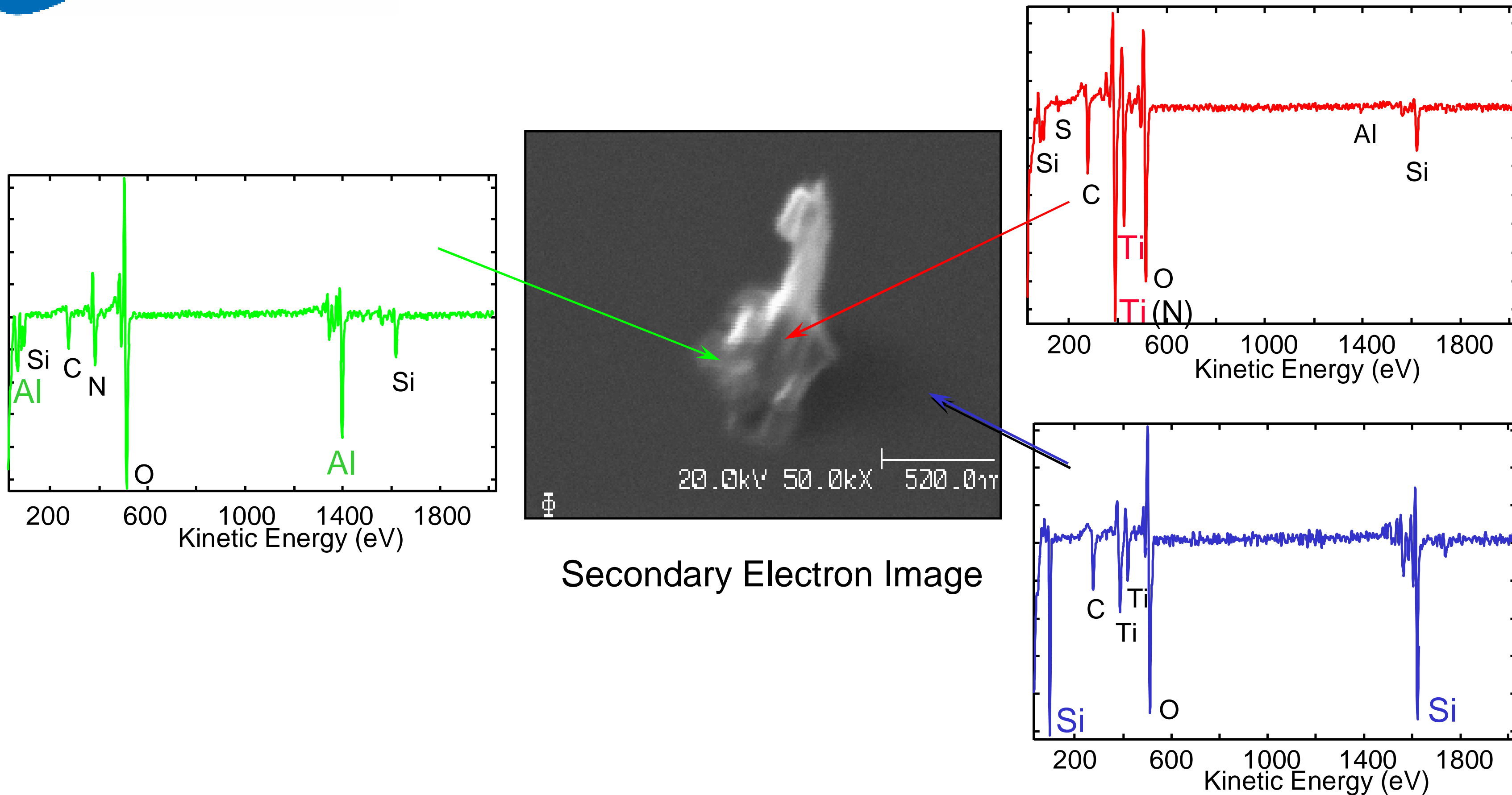
Particle Contamination from W Etch Back Process



Secondary Electron Image

- Auger spectra show the defect is composed of regions high in Ti or Al, and that the whole Si wafer is covered with a thin Ti film.

Y. Uritsky, et al., J. Vac. Sci. Technol. A 15(3), 1319 (1997)

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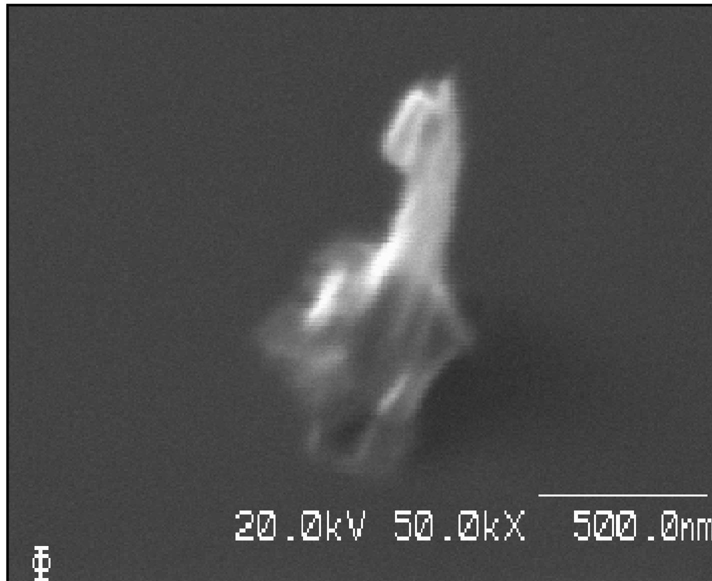
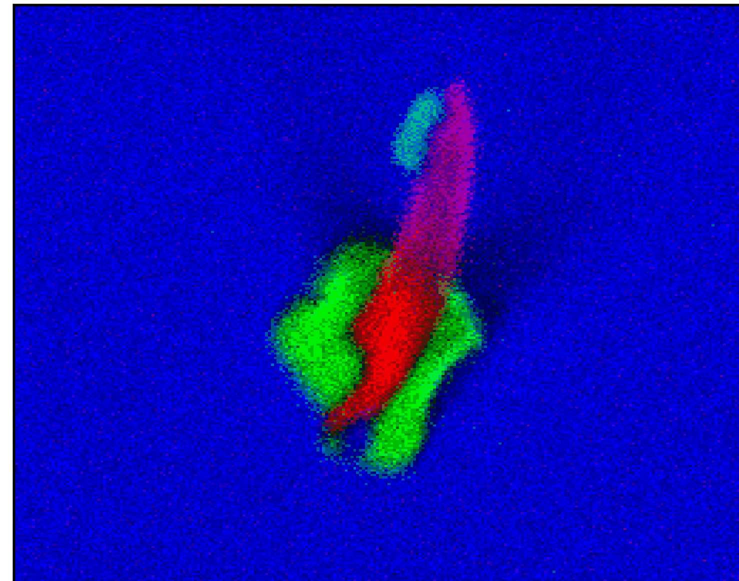
Particle Contamination from W Etch Back Process

Secondary Electron Image



Color Composite Auger Image



Red=Ti Green=Al Blue=Si

- Auger maps for Ti, Al and Si show the defect consists of separate phases of Ti and Al.

Y. Uritsky, et al., J. Vac. Sci. Technol. A 15(3), 1319 (1997)

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Identification of Thin Residue on Metal Lines

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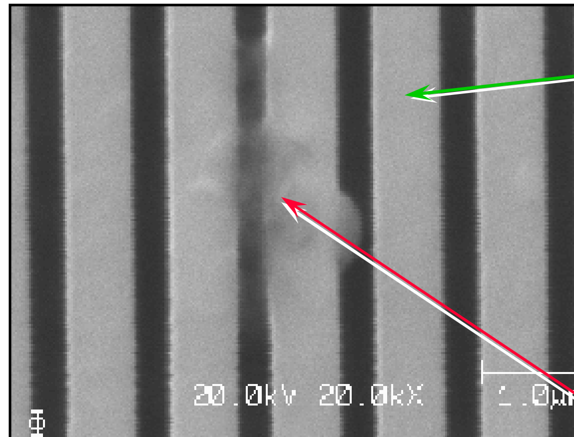
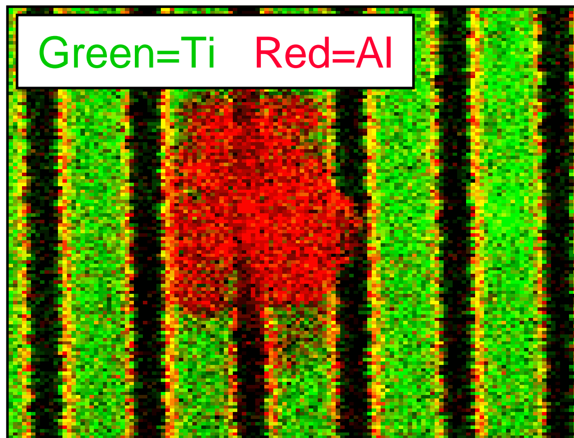
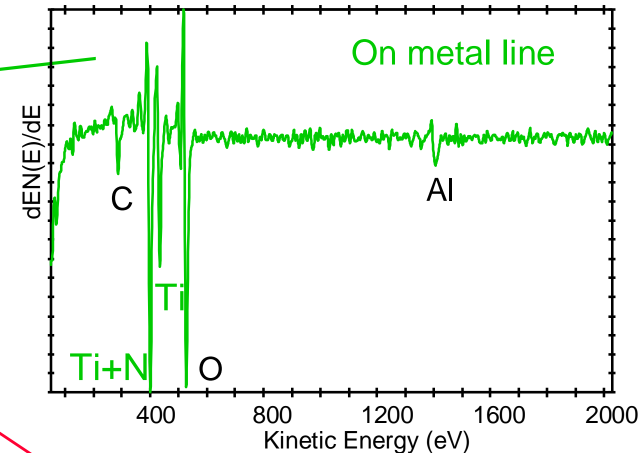
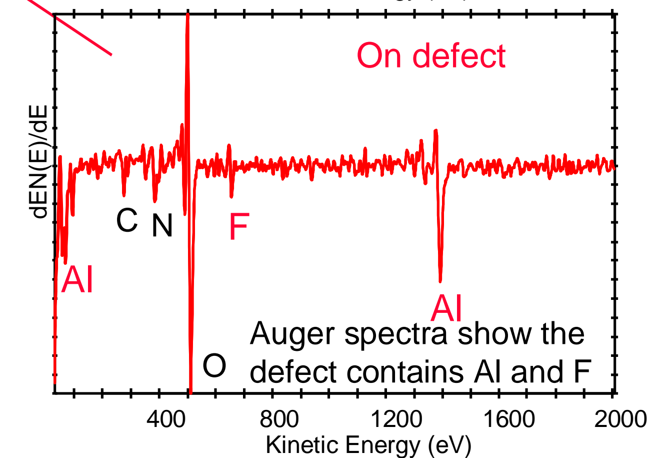
TiN/Al lines on SiO_2









- Auger analysis shows the thin residue is an Al flake, probably originating from the etch chamber.

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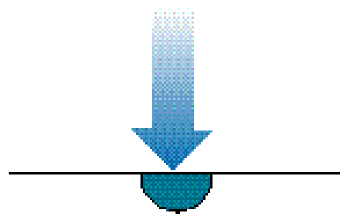
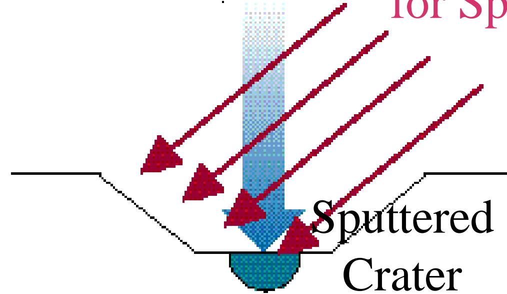
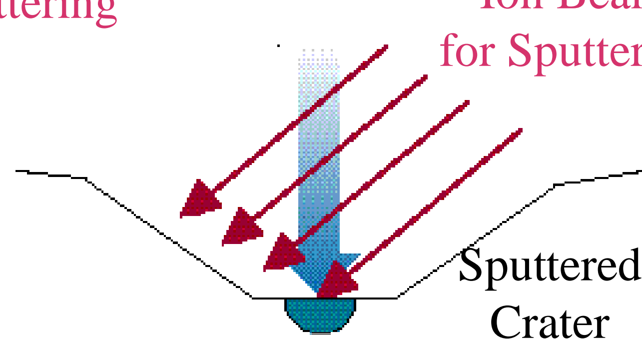
Compositional Depth Profiling

- Ion milling (sputtering) is used to remove material only!
- Auger analysis is performed on the surface at the bottom of the sputtered crater, and is independent of the sputtering process.







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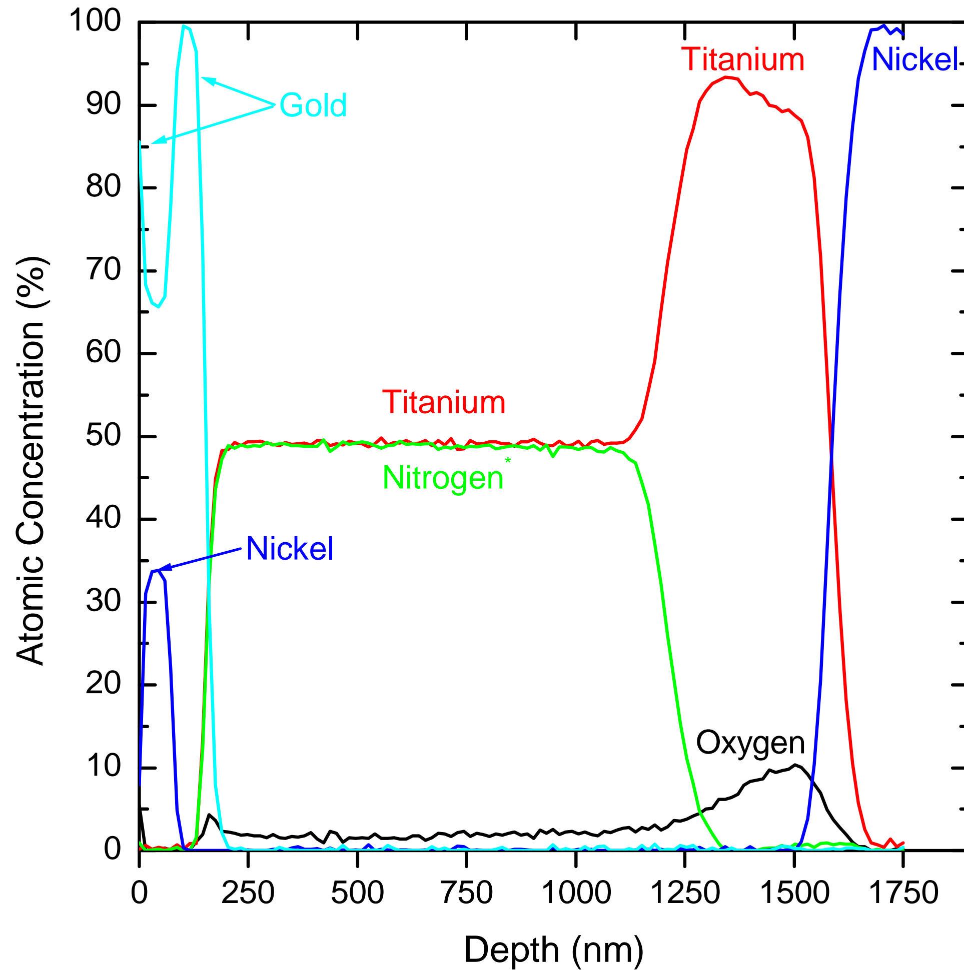
Thin Film Depth Profiling

Decorative Coating



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Auger Depth Profile of Bond Pad Failure

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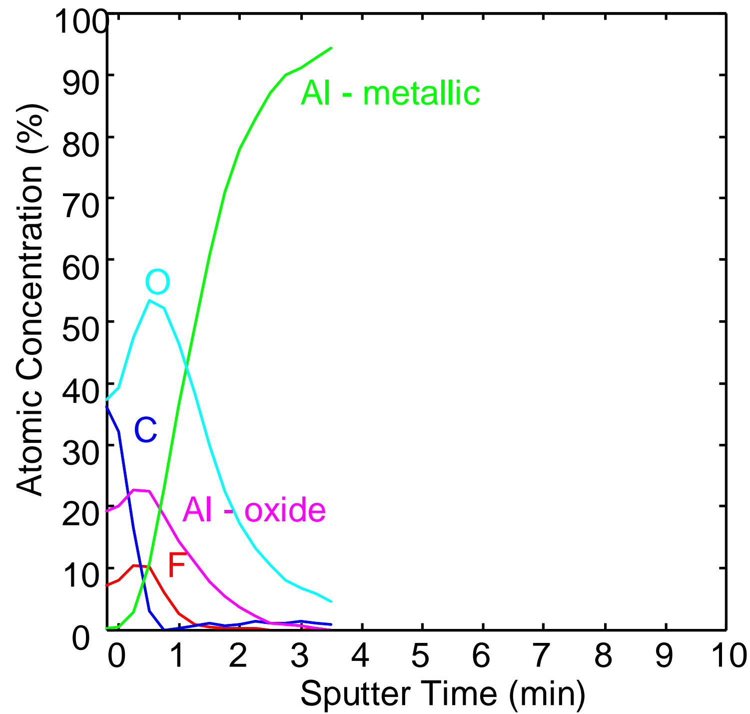
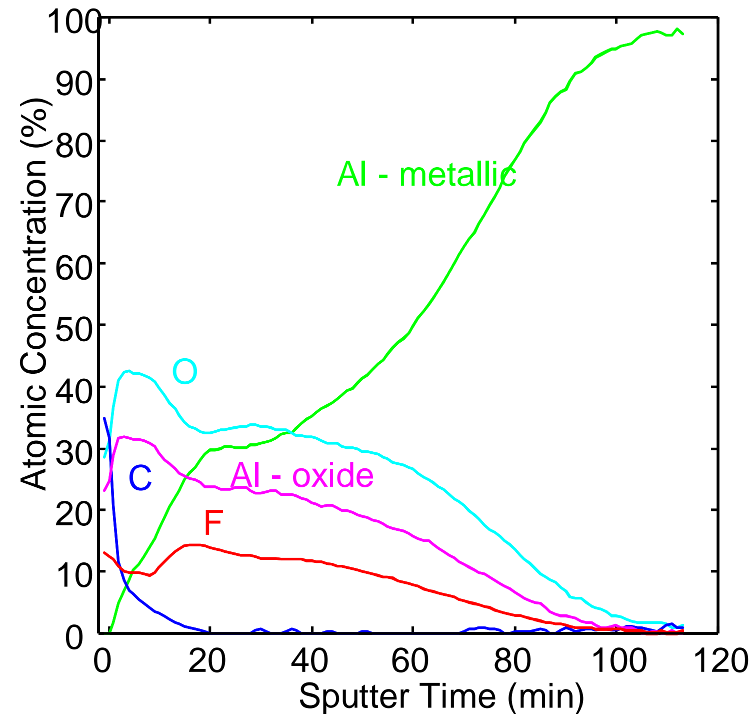
Good Bond Pad
Normal wire bond strength



Al oxide ~4 nm thick

Bad Bond Pad
Poor wire bond strength



Al oxide ~160 nm thick

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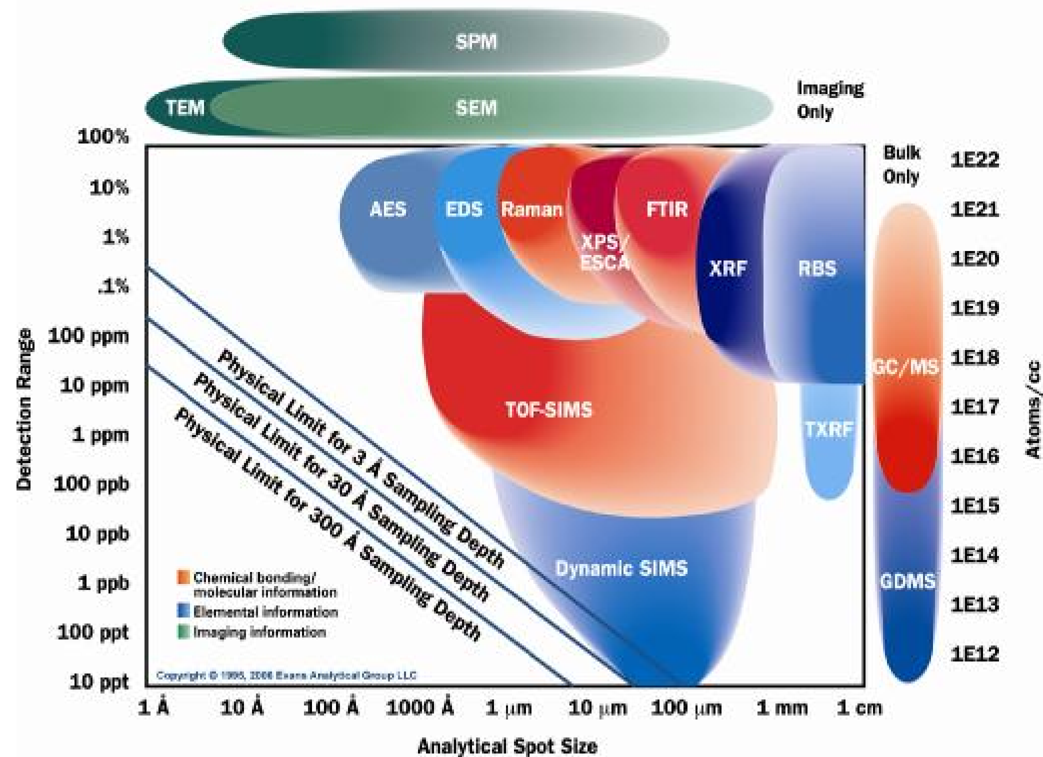
Technical Data Table

Analytical Resolution vs. Detection Limit



AES

Quantitative	Yes	Destructive	No
Detection Limits	0.1-1.0at%	Lateral Resolution/ Probe Size	0.01-2µm
Chemical Bonding	Some	Analytical Depth	10-60Å

Comment: Excels for small particles & features

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Strengths and Weaknesses

- Strengths
  - Surface sensitive
  - Good depth resolution
  - Often the only choice for small area analysis
- Weaknesses
  - Best quantification requires standards
  - Relatively low sensitivity (0.1 at%)
  - No or little chemical information
  - Insulators are difficult

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Chemical Bonding Information:
X-Ray Photoelectron Spectroscopy/Electron Spectroscopy for Chemical Analysis

(XPS/ESCA)

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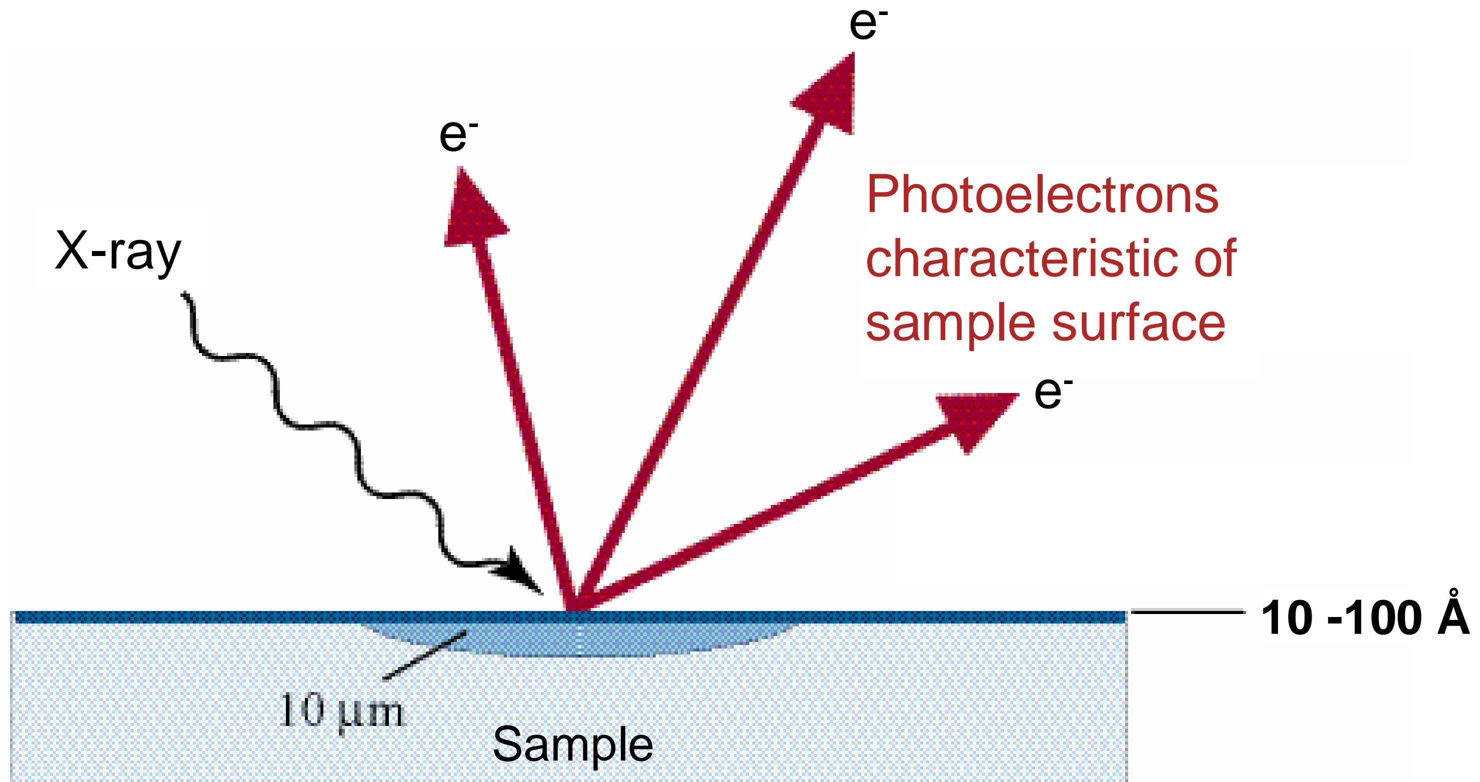
XPS Process



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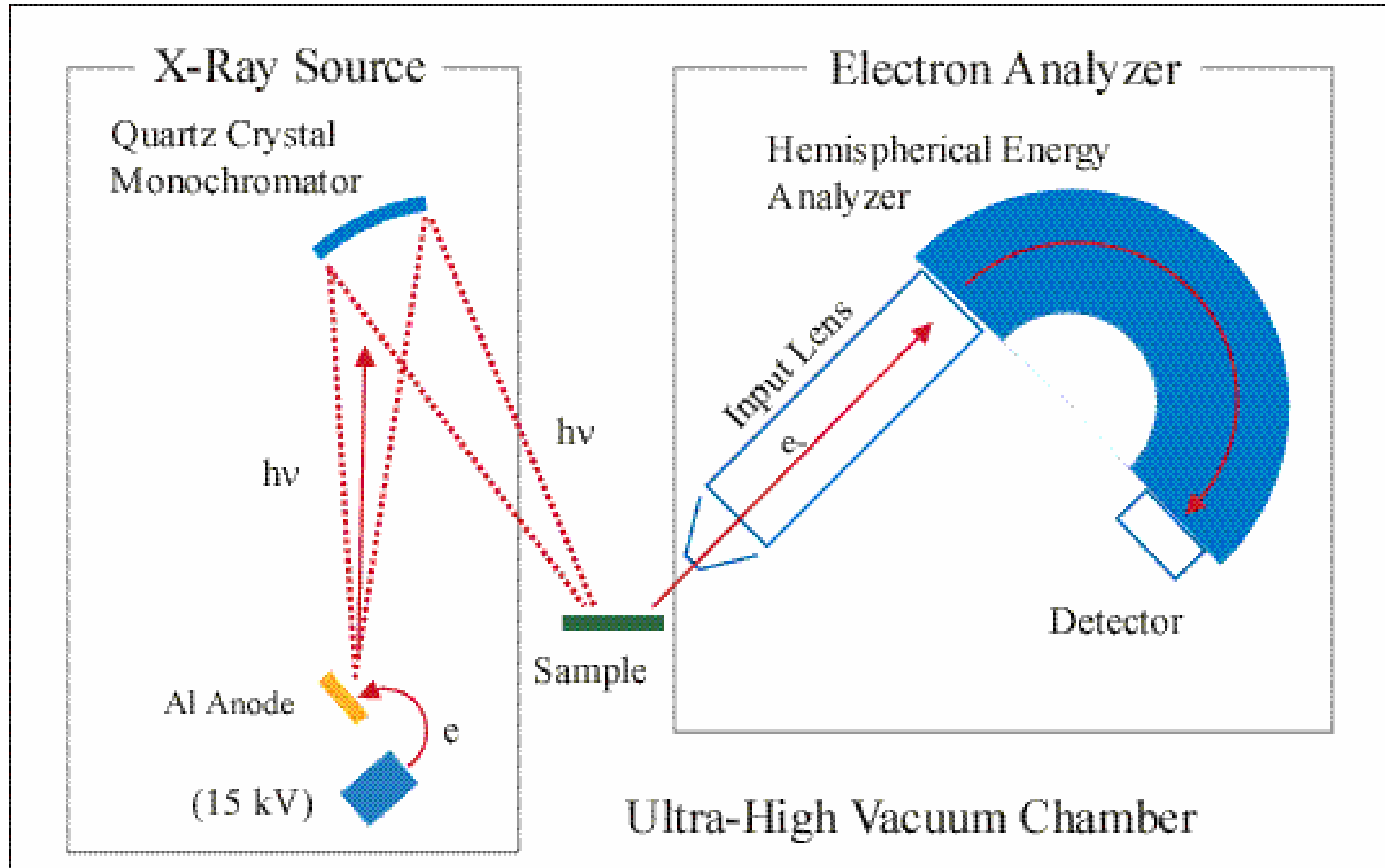
X-Ray Photoelectron Spectrometer



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XPS Technique

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- Measure kinetic energy (KE) of photoelectrons ejected from sample
- Calculate photoelectron binding energy (BE) in electronvolts, eV

$$BE = h\nu - KE - \phi + \delta$$

$h\nu$ = excitation x-ray energy (fixed energy)
ϕ = electron spectrometer work function
δ = net surface charge

- Survey spectrum: identifies elements at surface
- High resolution spectrum: identifies chemical state from peak position and peak shape

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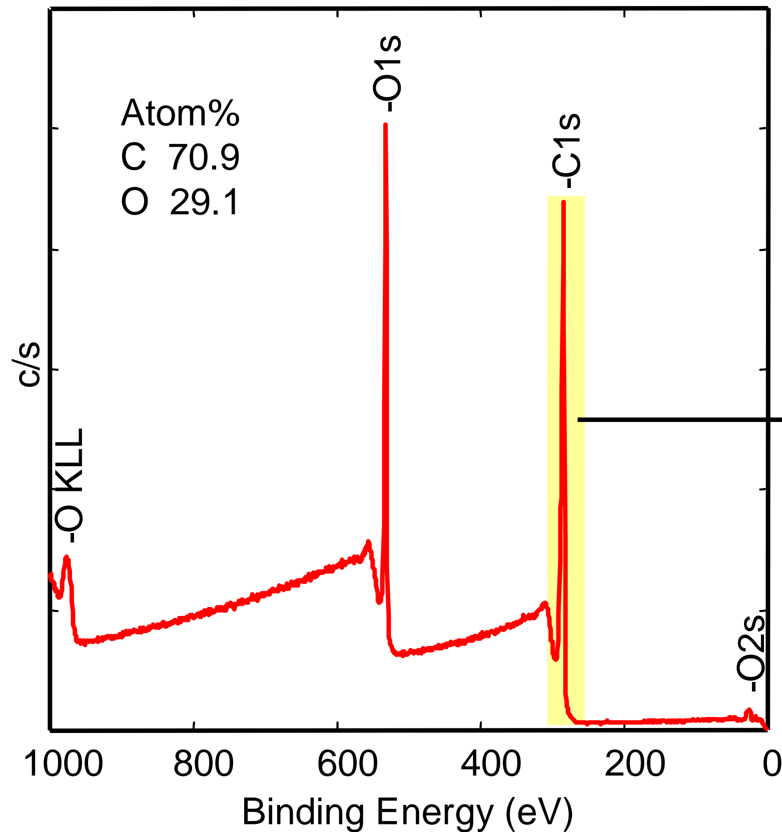
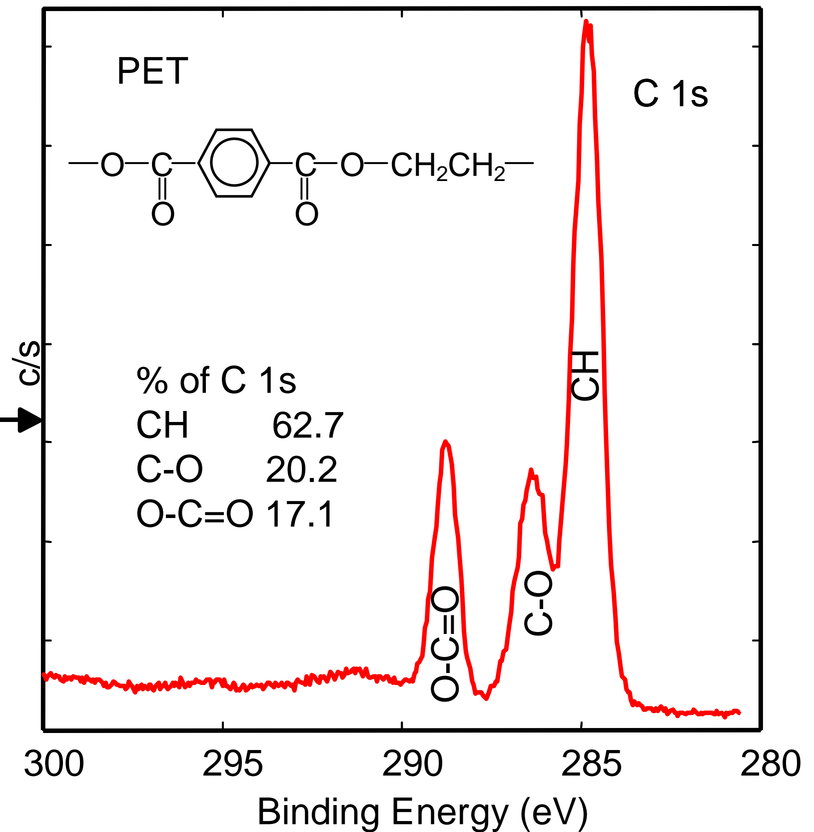
Typical Data

Poly(ethylene terephthalate)





XPS survey spectra provide quantitative elemental information

High resolution XPS spectra provide quantitative chemical state information

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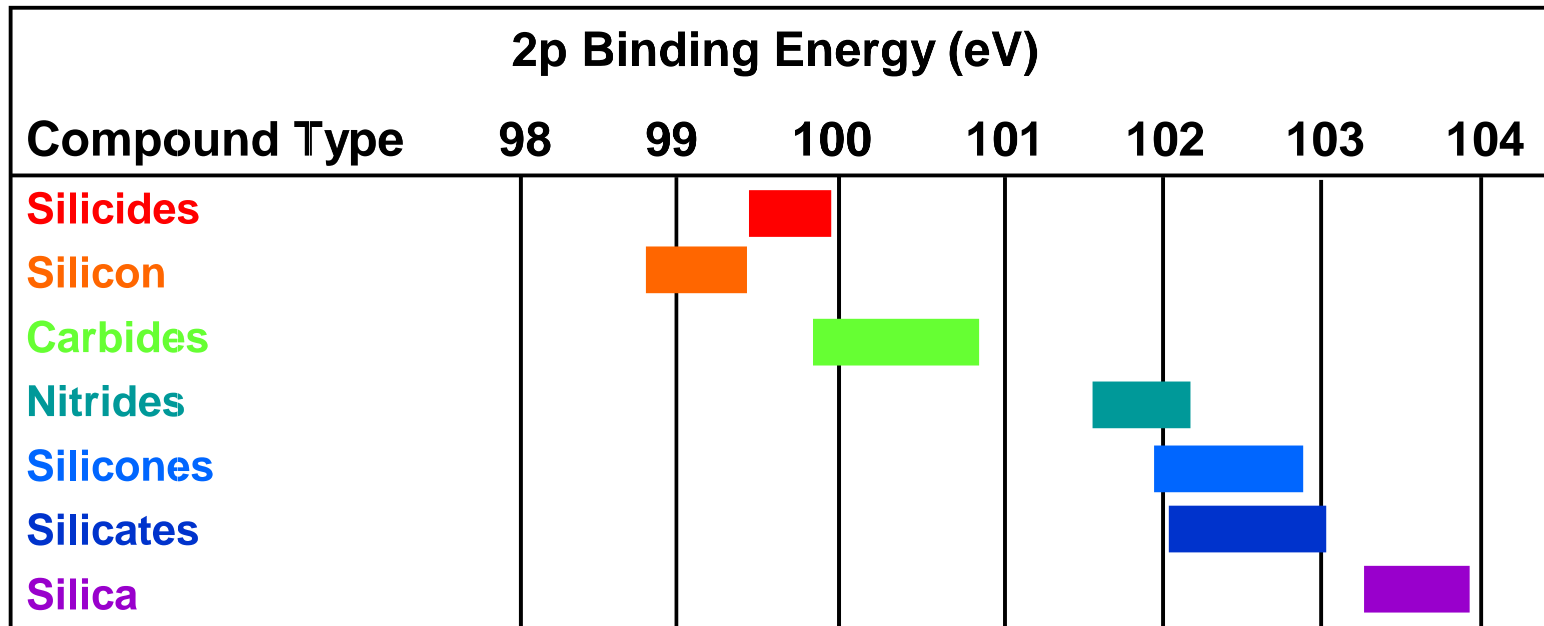
Silicon Chemical State Table



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Typical III-V Applications

Measurement of **oxide chemistry** as a function of processing
- thickness
- Ga°, GaN, Ga_2O_3, As°, As_2O_3, As_2O_5, etc
- Ga-rich vs As-rich vs. stoichiometric
- impurities (fluorides, chlorides from etch)

Issues related to **metallization**
- interface chemistry
- metals oxidation
- interdiffusion

Cleaning residues
- quantitative organic and inorganic contamination with sub-monolayer sensitivity

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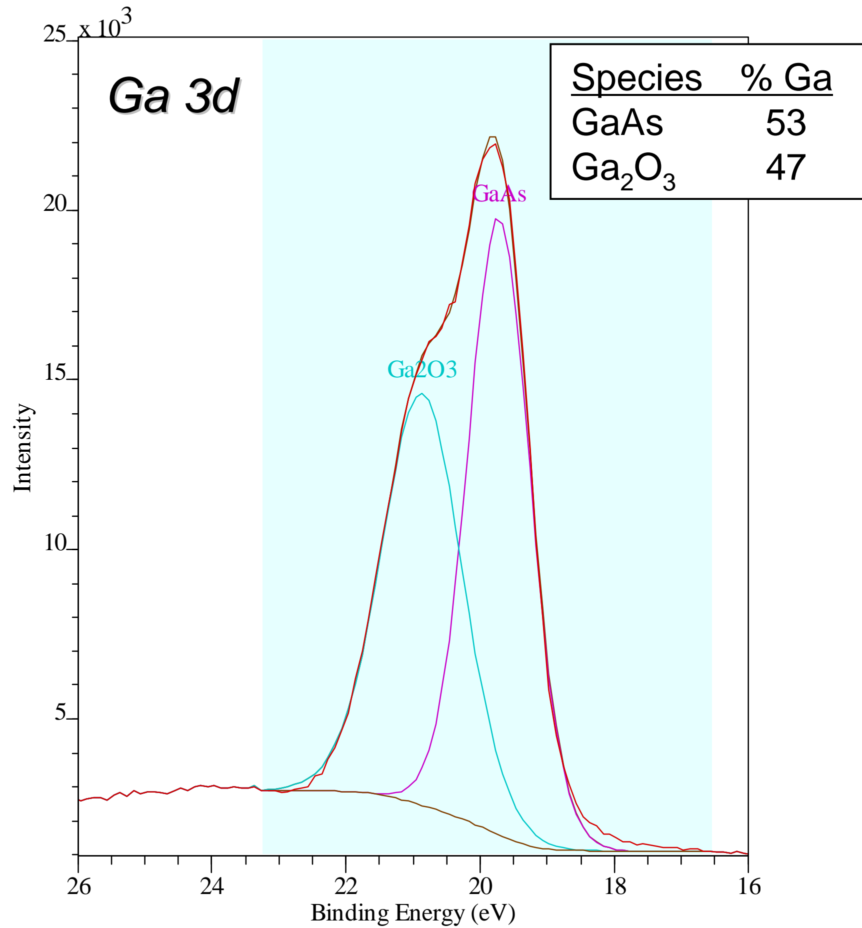
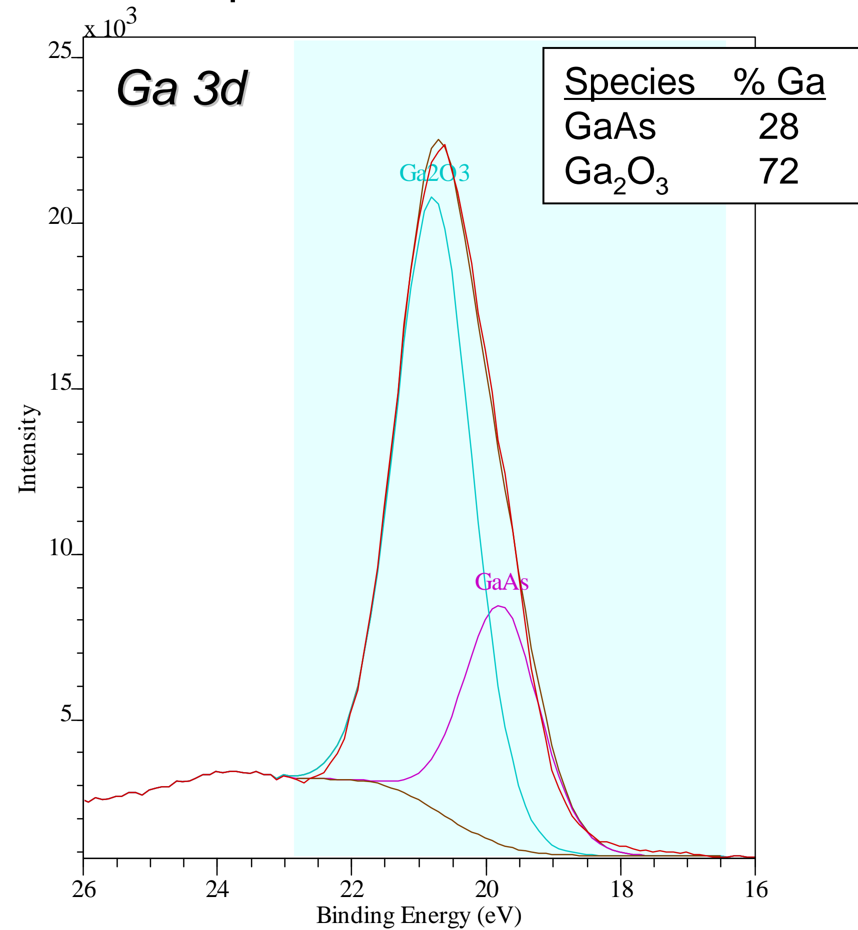
Oxidation of GaAs under different conditions

Sample 1



Sample 2



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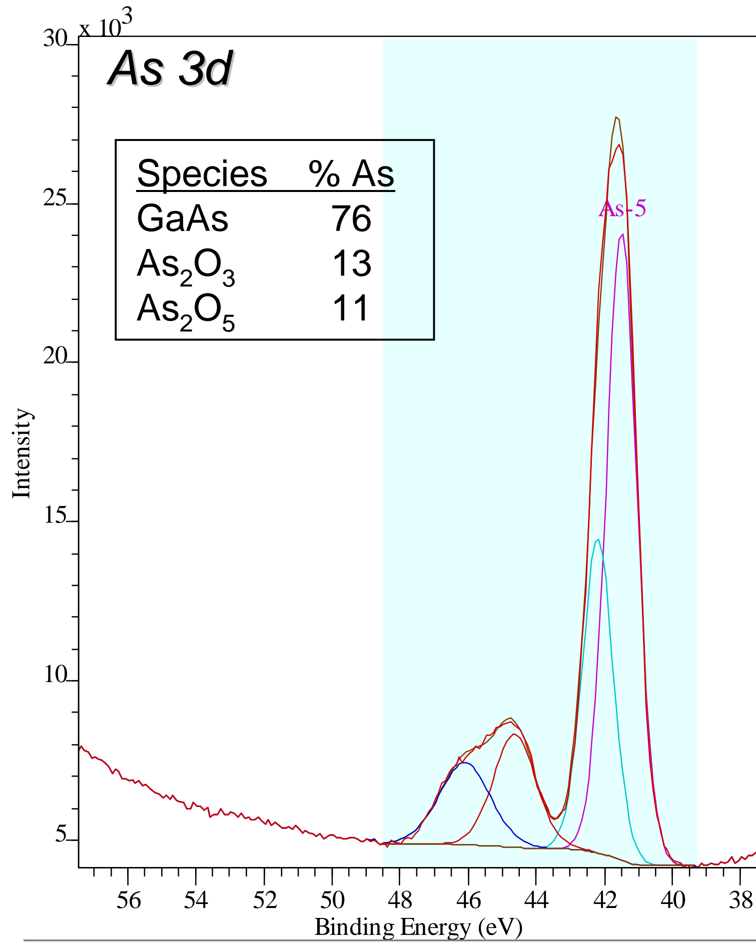
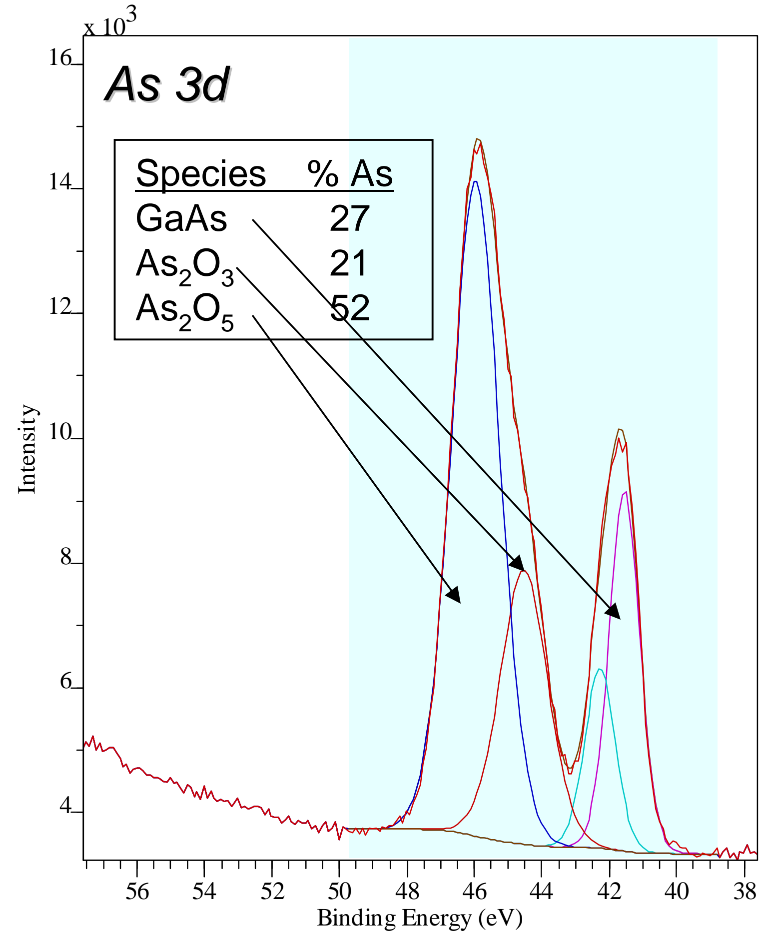
Oxidation of GaAs under different conditions

Sample 1



Sample 2



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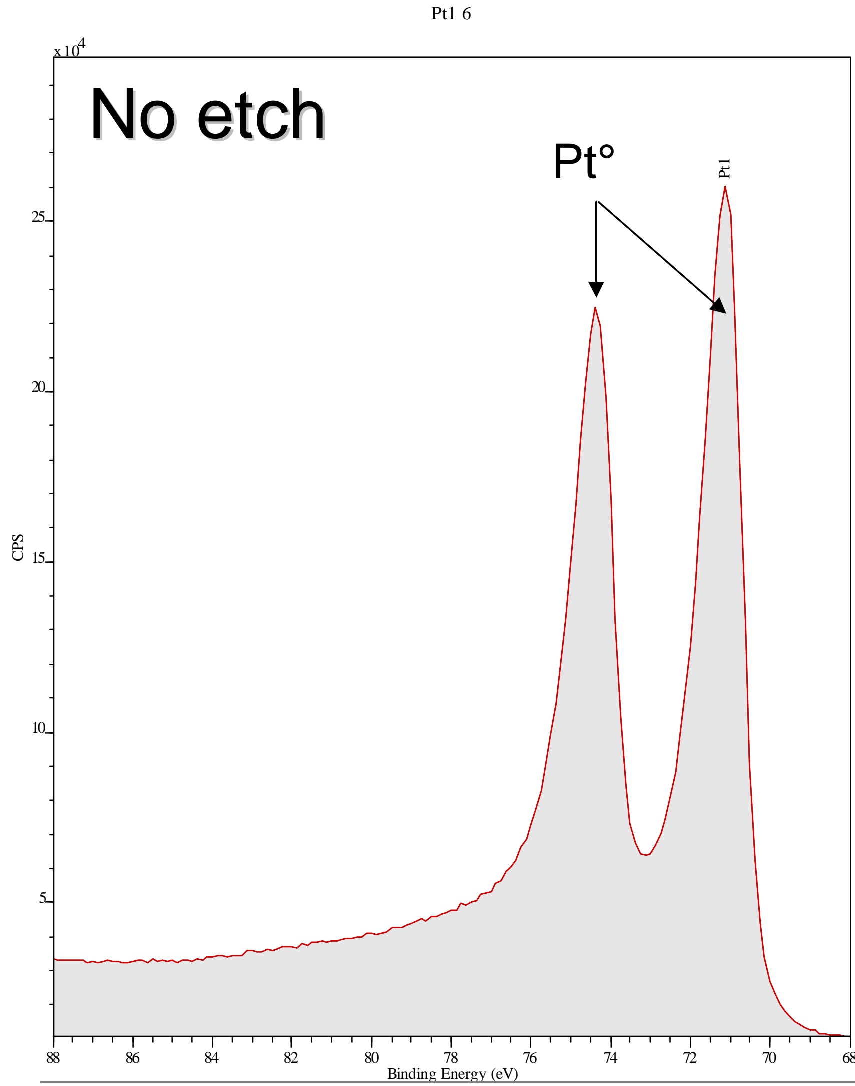
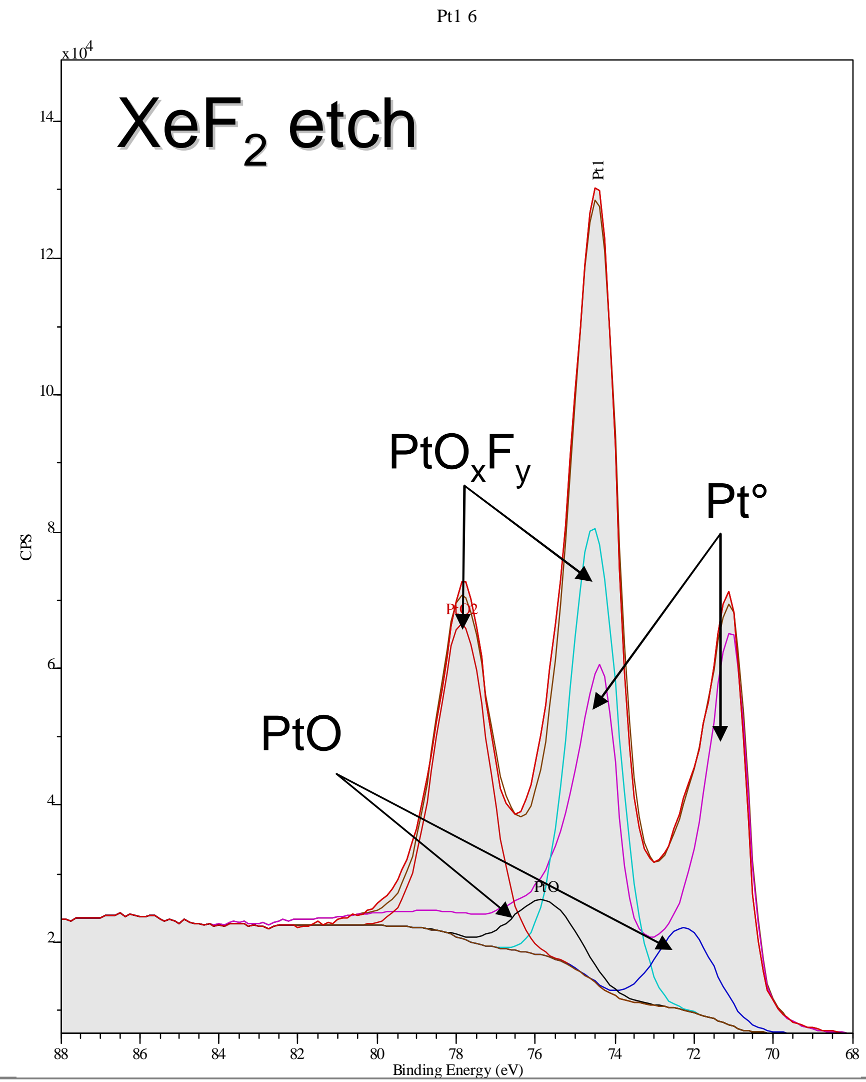
Plasma etching residue on Pt contacts

High resolution Pt 4f spectra





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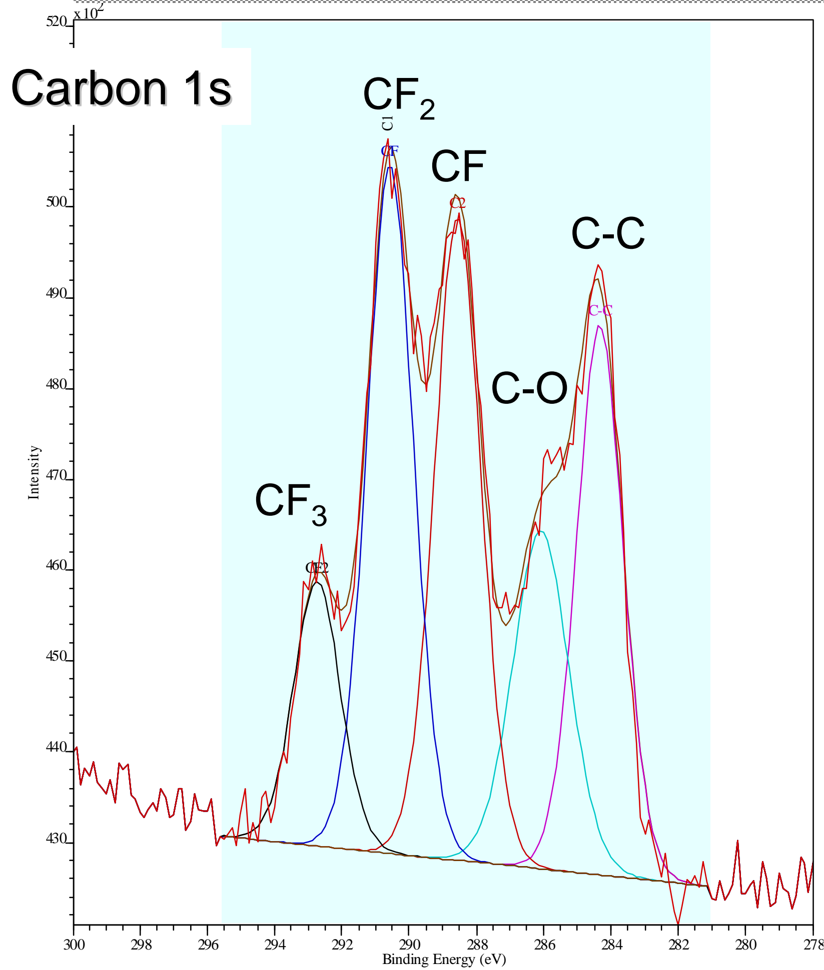
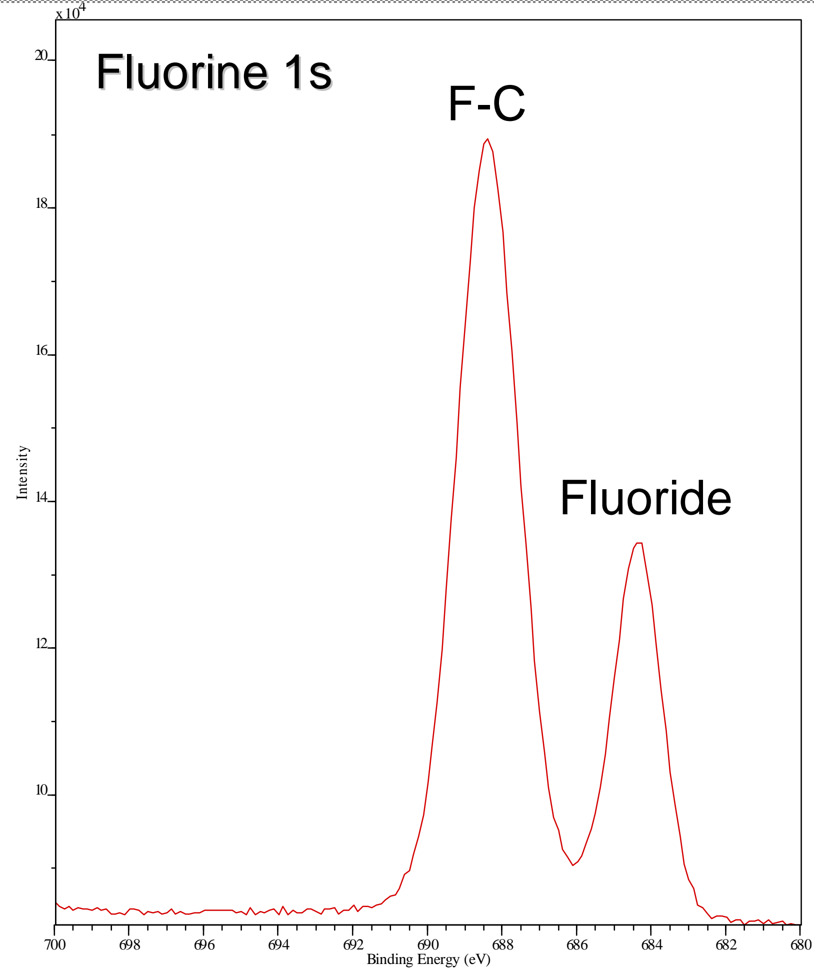
Plasma etching residue on Pt contacts

C 1s and F 1s spectra from XeF_2 sample





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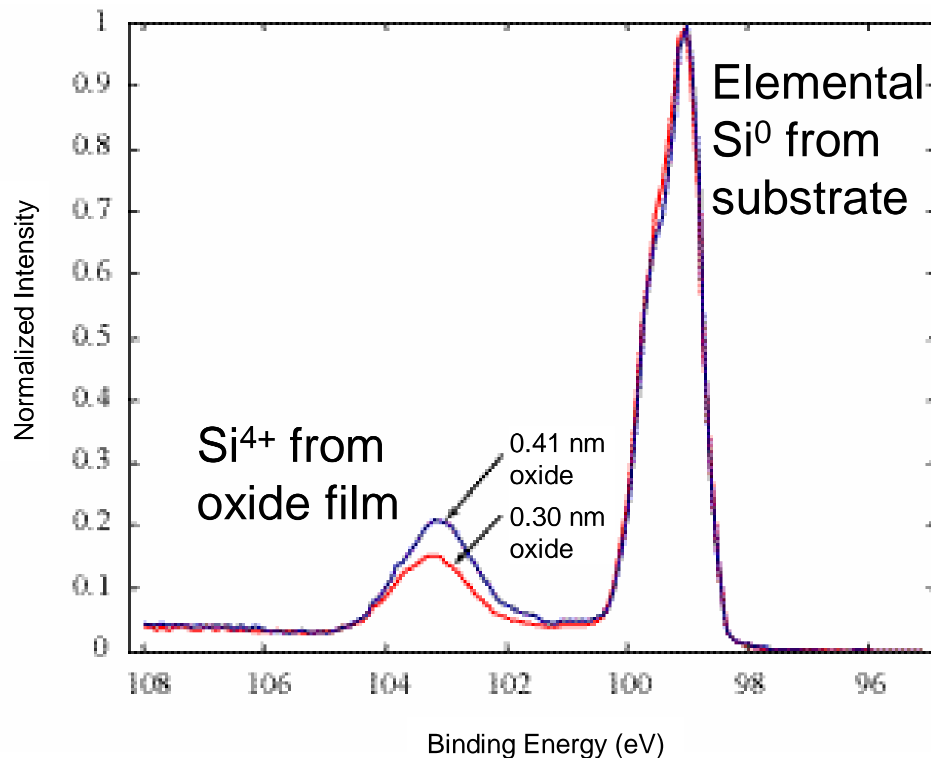
Silicon Oxide/Oxynitride Film Thickness



- Objective: Determine the thickness of thin SiO_2 layers on Si.
- Using intensities of film and substrate peaks along with parameters derived from standard samples, film thicknesses determined with sub-Angstrom precision.

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Depth Profile Analysis

- Destructive (Organic information typically lost)
  - Energetic ion beam sputtering
  - Sputter rates 10 to 500 Å/min
- Non-destructive
  - Angle resolved analysis
  - Sampling depths 10 to 100 Å
  - May also employ different x-ray sources

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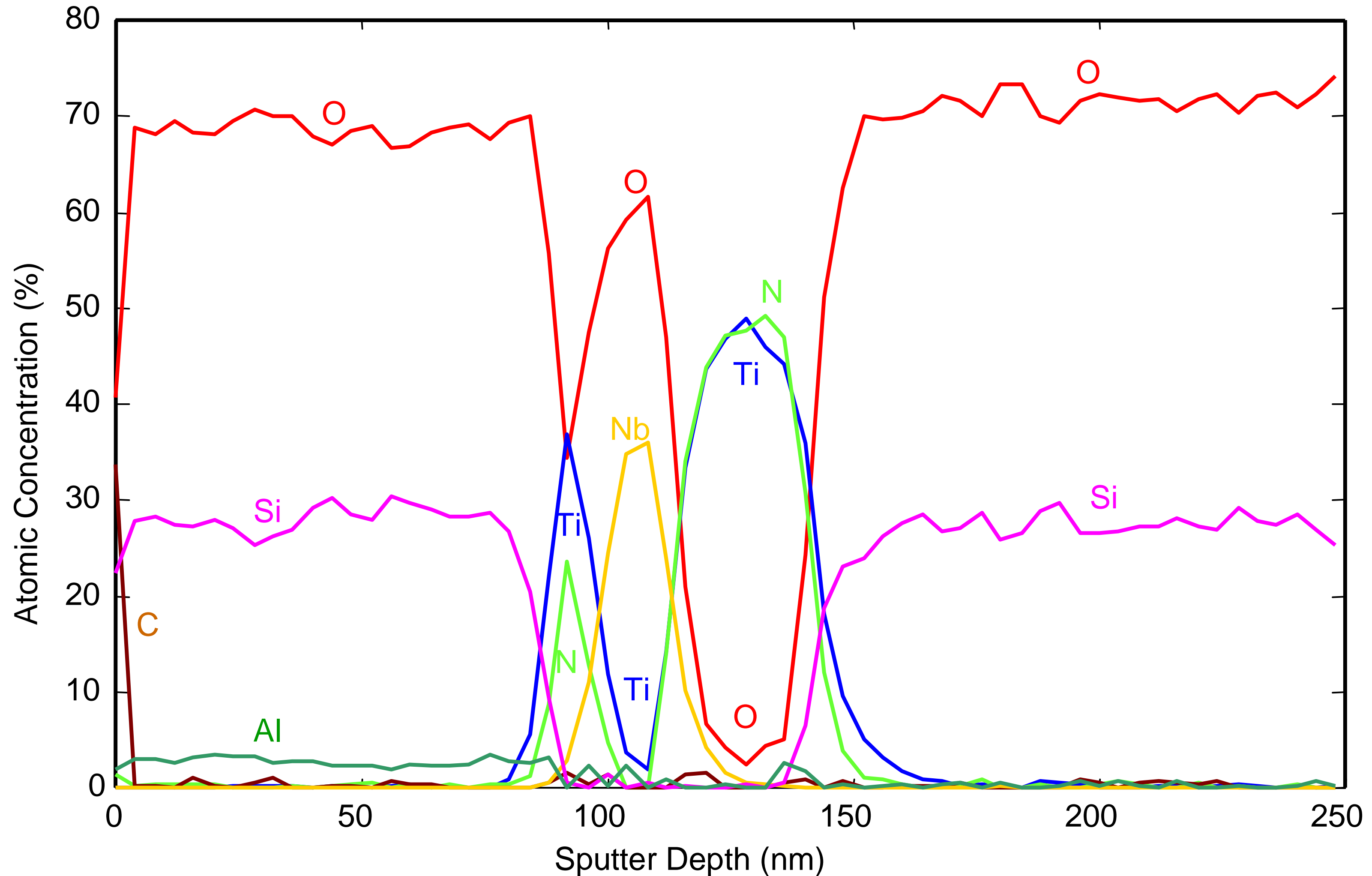
Sputter Depth Profiling of Insulating Materials



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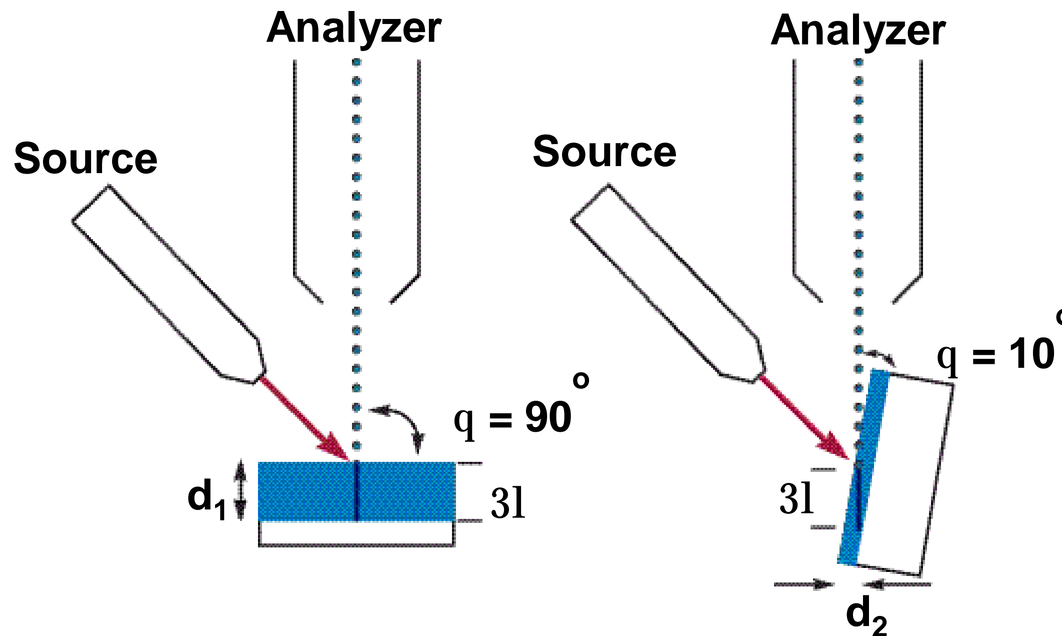
Non-destructive: Angle Resolved Analysis

- Sample is tilted with respect to x-ray source and analyzer
- Angle determines sampling depth (d)



d = Sampling Depth = ~3l sinq
$d_2 < d_1$ even though l is constant

Plasma Treated Contact Lens, Angle Resolved Analysis

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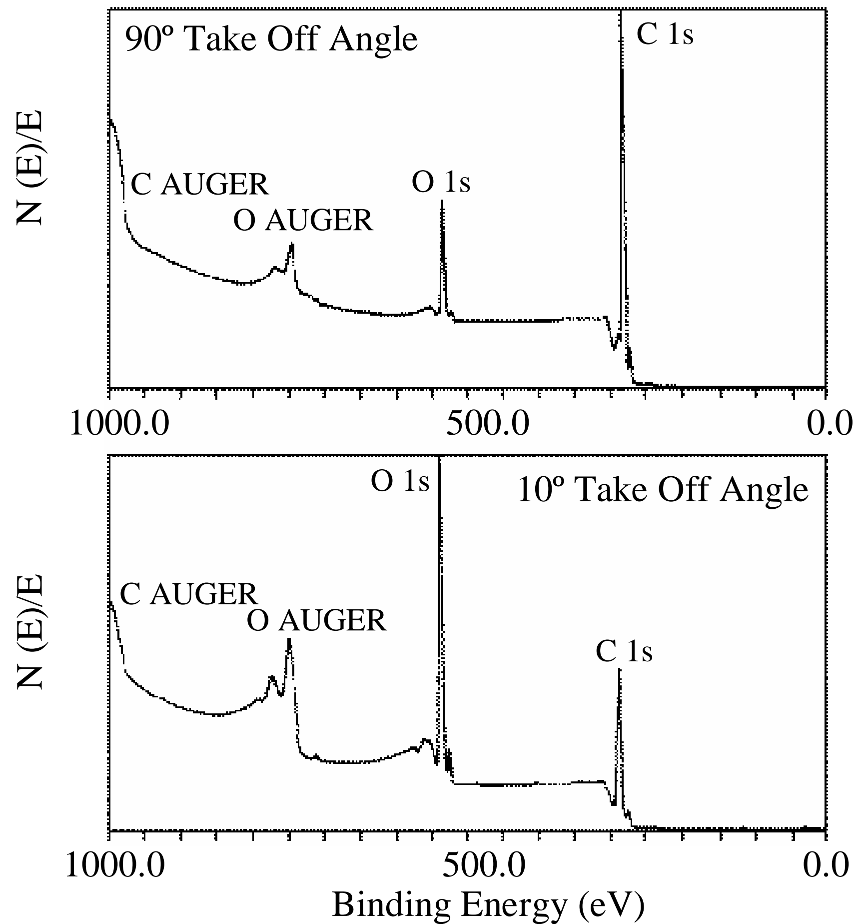
High Sensitivity Mode



Greater sampling depth

More surface sensitive

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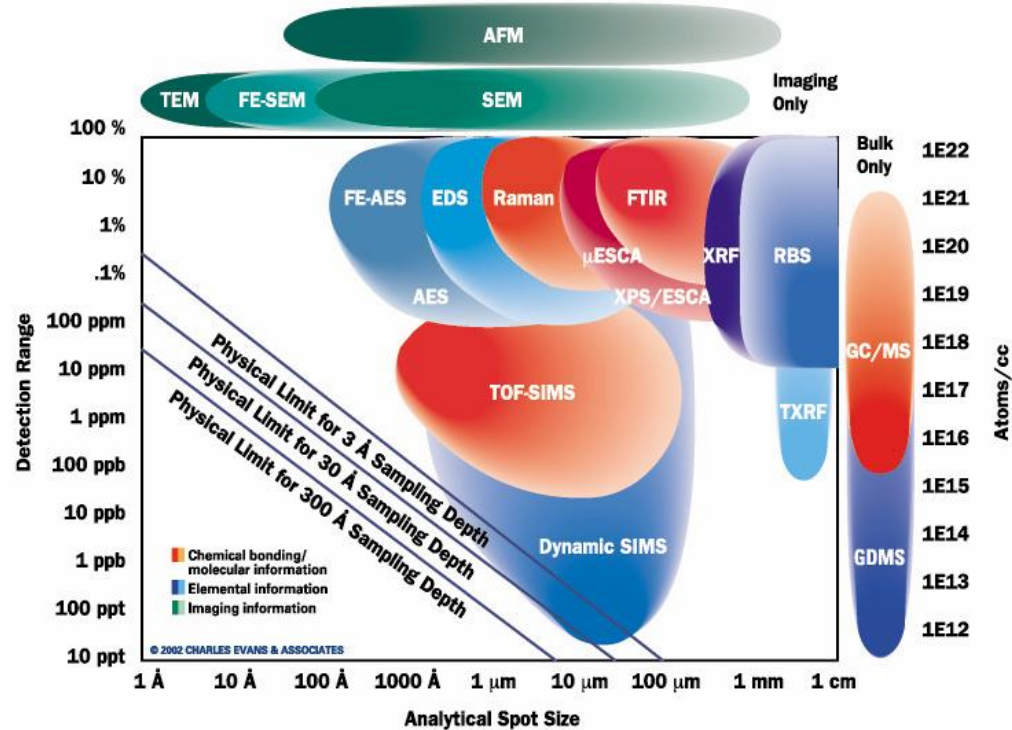
Technical Data Table



XPS

Quantitative	Yes	Destructive	No
Detection Limits	0.05-0.5at%	Lateral Resolution/ Probe Size	10µm
Chemical Bonding	Yes	Analytical Depth	1-10nm

Comments: Provides bonding information for organic and inorganic species

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High Sensitivity:
Secondary Ion Mass Spectrometry

(Dynamic and Static SIMS)

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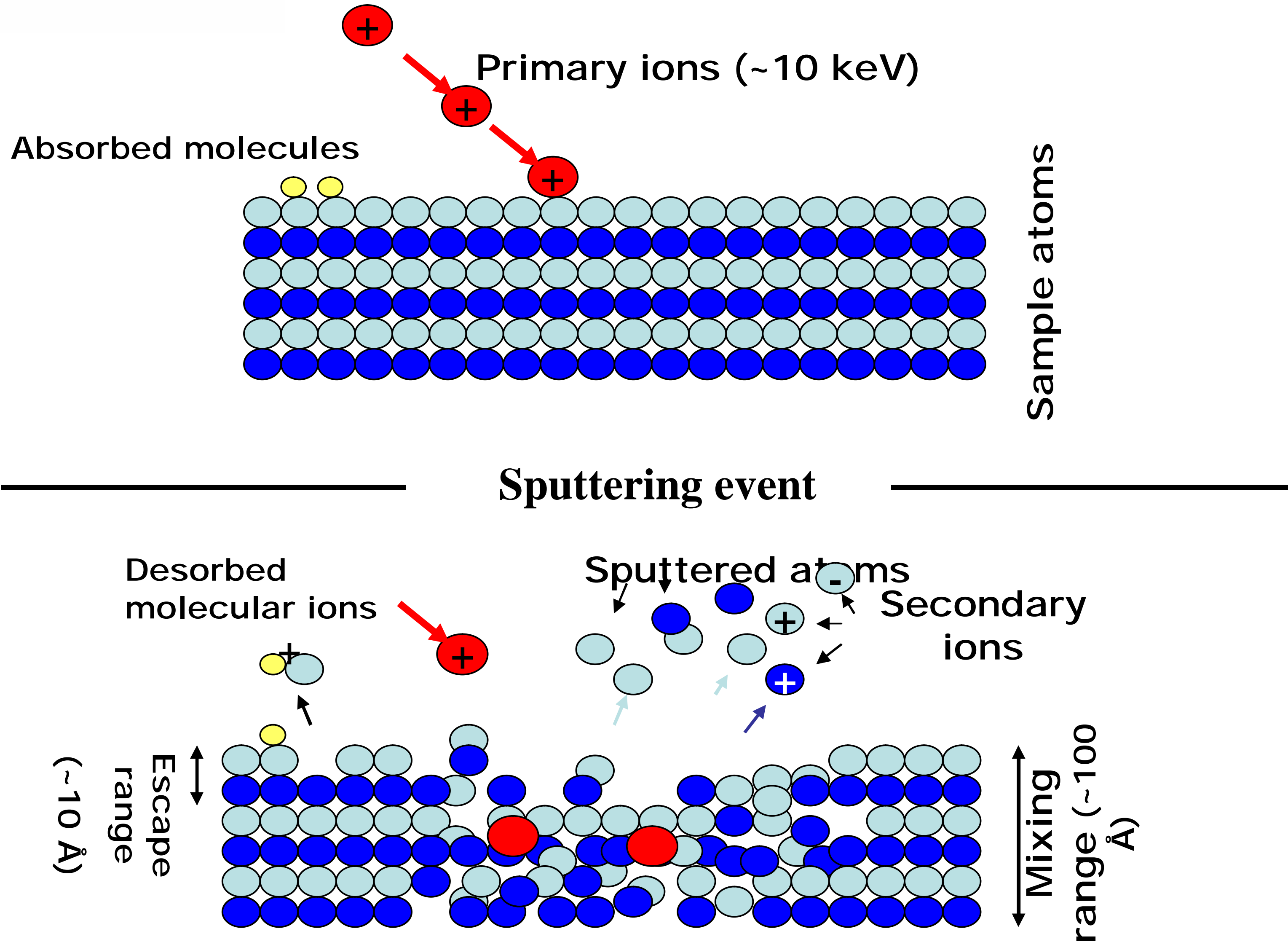
Basic Sputter Process in SIMS



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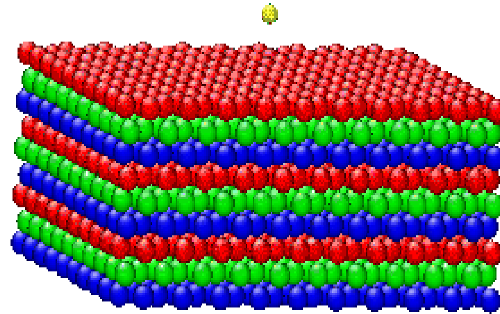
Ion Surface Interaction

3 keV Ar^+->Ni{001}



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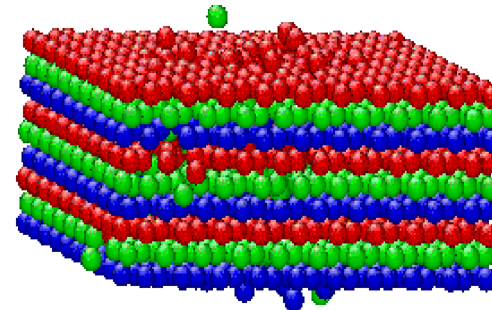

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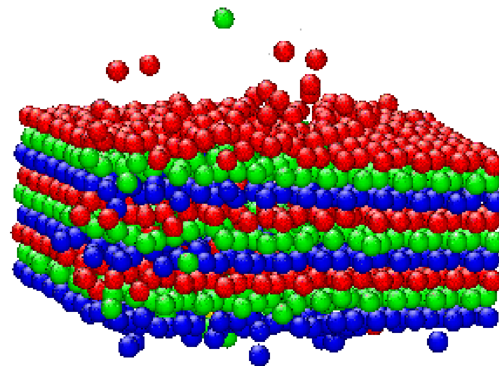

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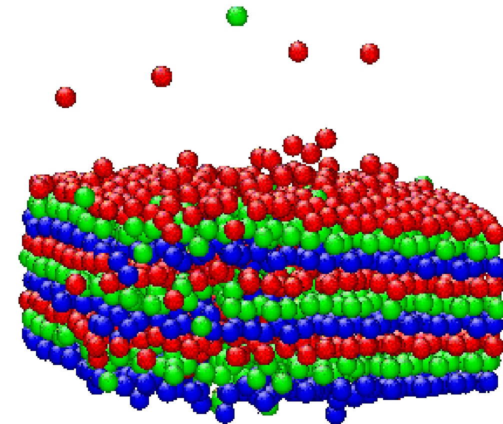

120 fs

Data Courtesy of
The Pennsylvania State University

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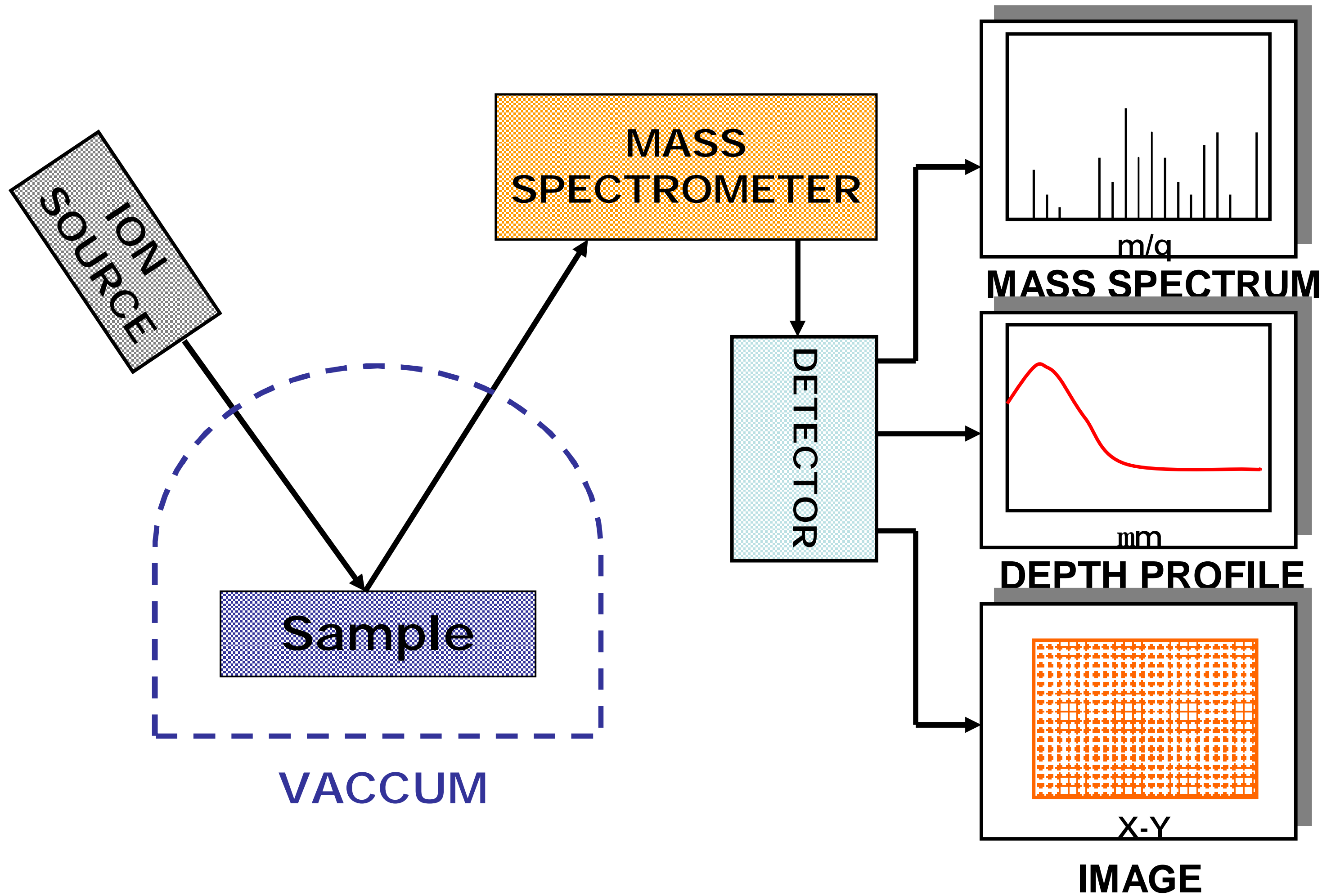
SIMS Technique Schema



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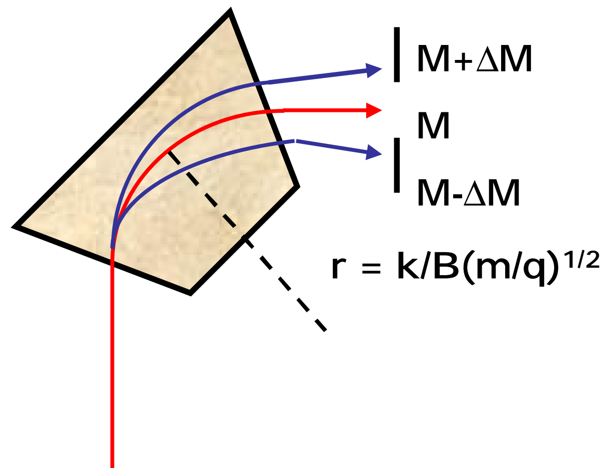
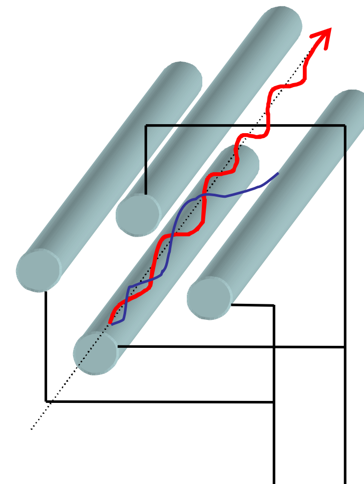
SIMS Instrument Type

Magnetic sector

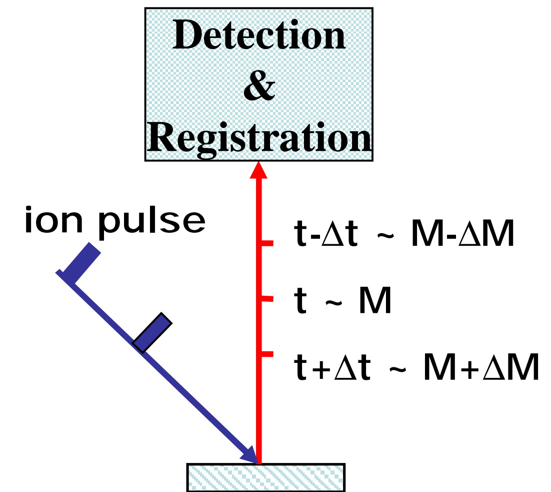
Quadrupole

Time of Flight





Vo(t) = Vc+Vs cosωt



m/q ~ B

m/q ~ V(f)

m/q ~ t

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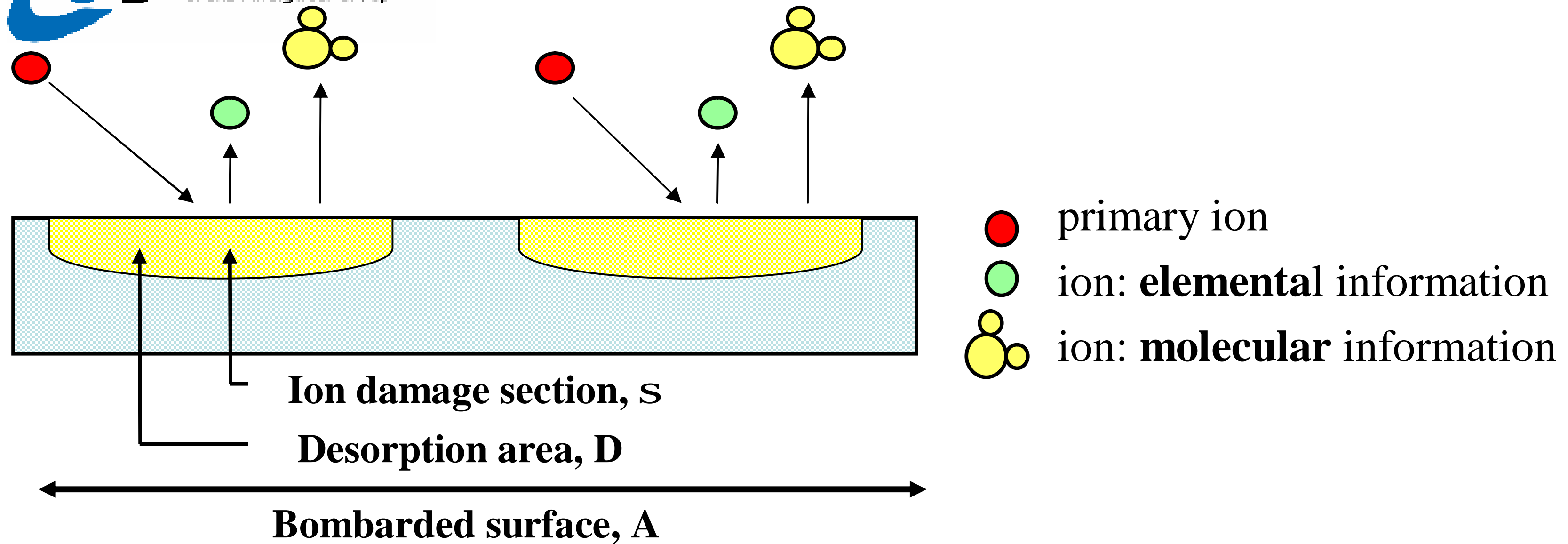
Dynamic vs. Static SIMS



	Ss << A Static SIMS	Ss = A Dynamic SIMS
Primary ion dose	<1E12 ions/cm2	>1E12 ions/cm2
Information	Chemical	Elemental
Analysis	Only surface	Depth profile
Instrument	TOF	Magnetic & Quad

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Organic information: Time-of-Flight Secondary Ion Mass Spectrometry

(TOF-SIMS)

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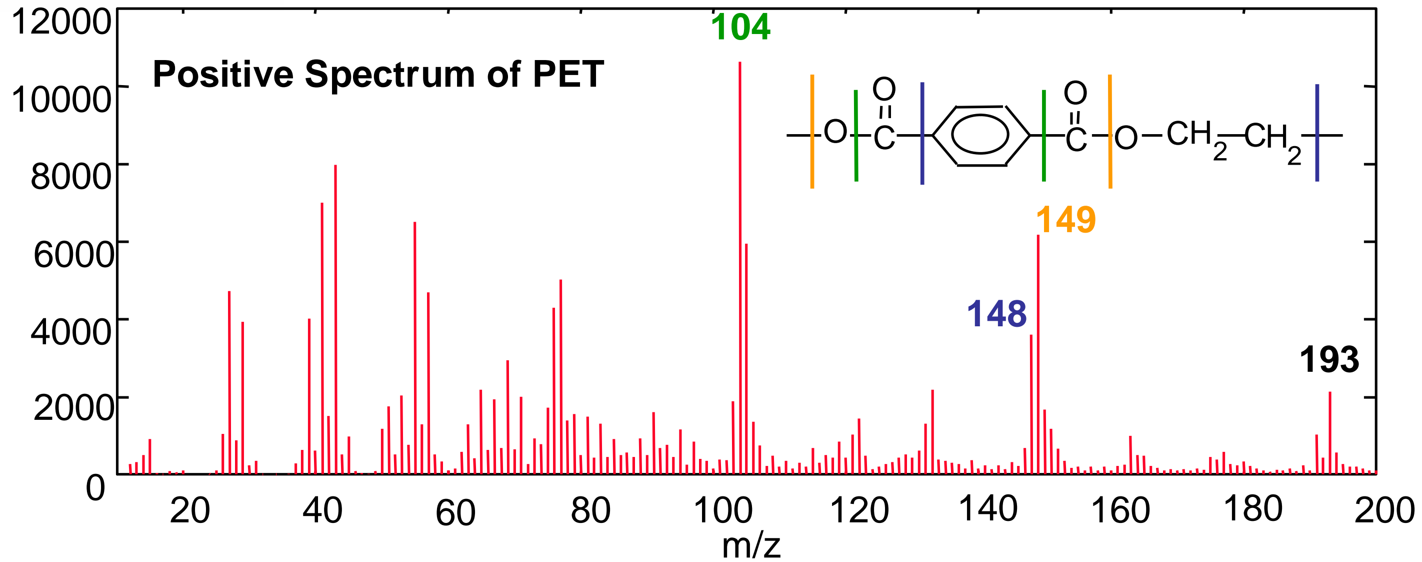
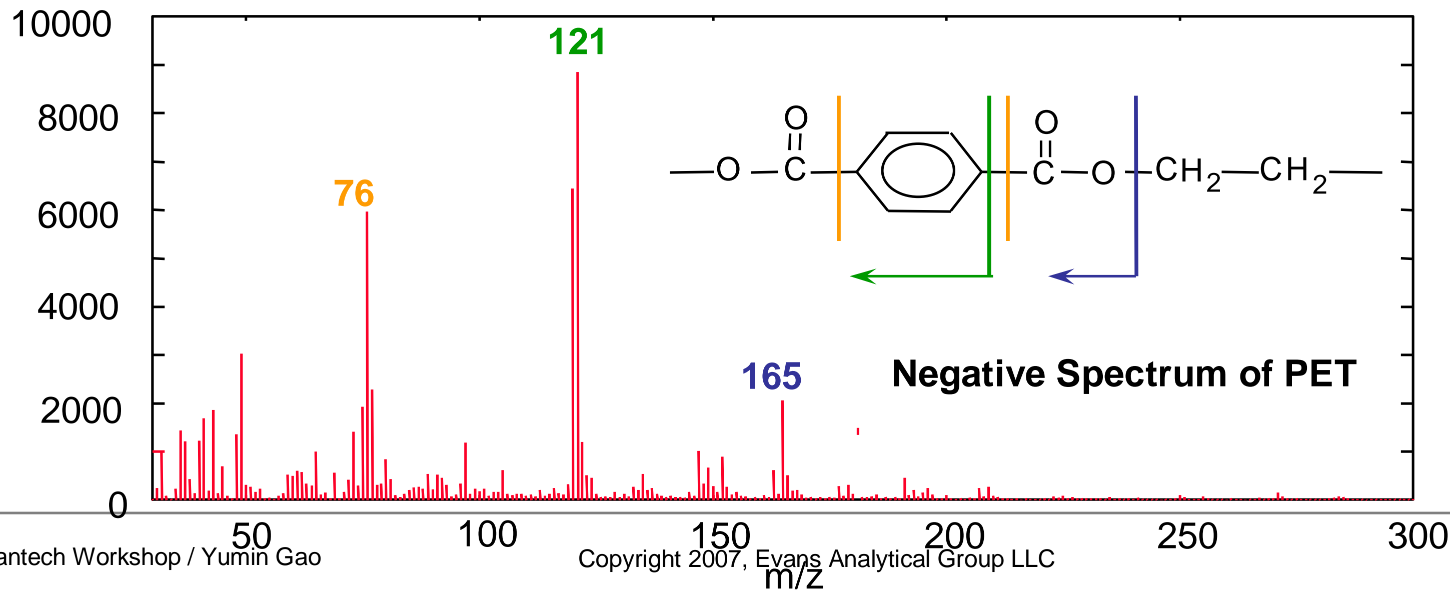
Typical Data





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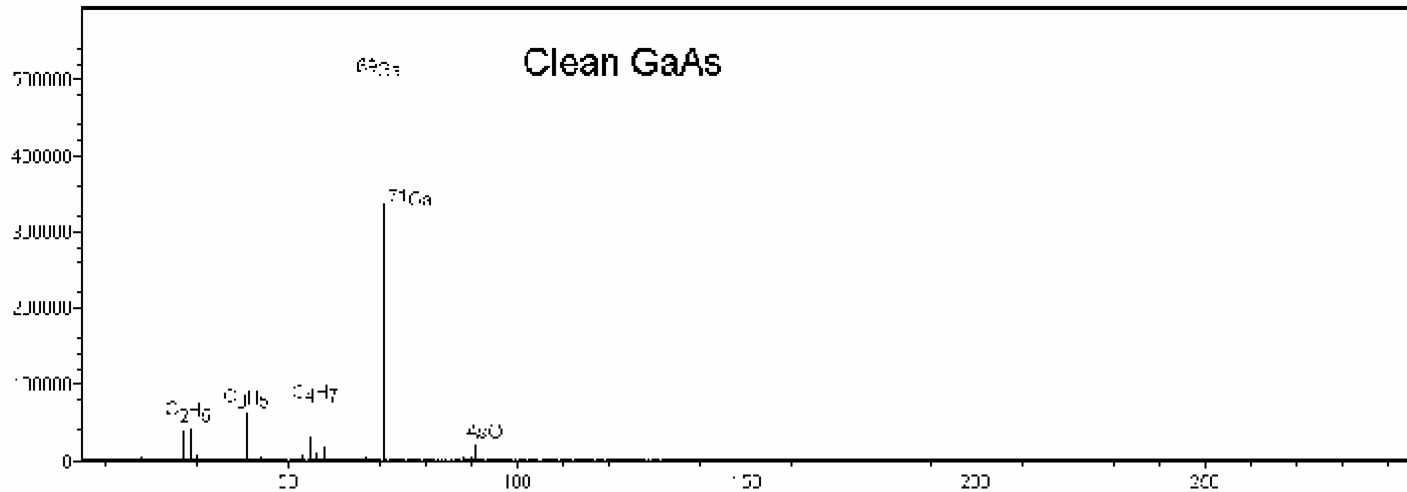
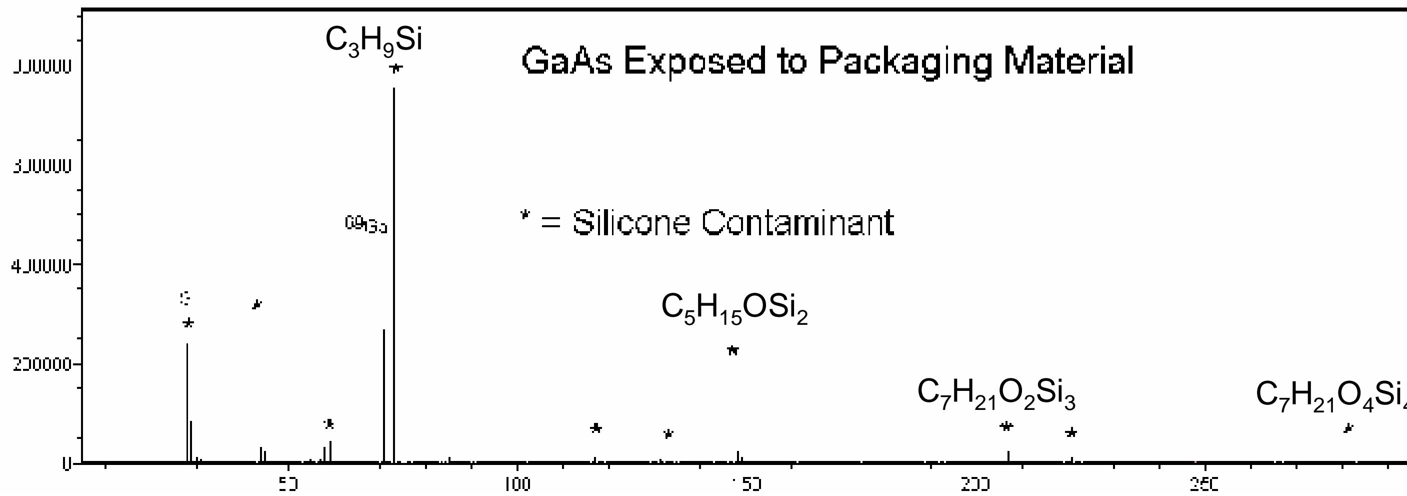
Contamination on GaAs





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Key Applications

- Surface characterization of organic and elemental materials
- Mapping distributions of surface species
- Contaminant identification ($\leq$ ppm)
  - Elemental
  - Molecular
- Failure analysis
  - Adhesion
  - Bond Pads
  - Coatings
- Evaluation of cleaning processes (QA/QC)
- Identification of stains, discolorations, and hazes
- Metal quantification on GaAs

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Strengths and Weaknesses

- Strengths
  - Can provide specific molecular information on thin (submonolayer) organic films/contaminants
  - Survey analysis allows more complete characterization of a surface
  - Excellent detection limits (ppm) for most elements
  - Probe size ~0.2 µm for imaging
  - Can analyze insulators and conductors
- Weaknesses
  - Usually not quantitative
  - For some samples, organic information can be limited
  - UHV technique (though cold stage can be used)
  - At times, too surface sensitive

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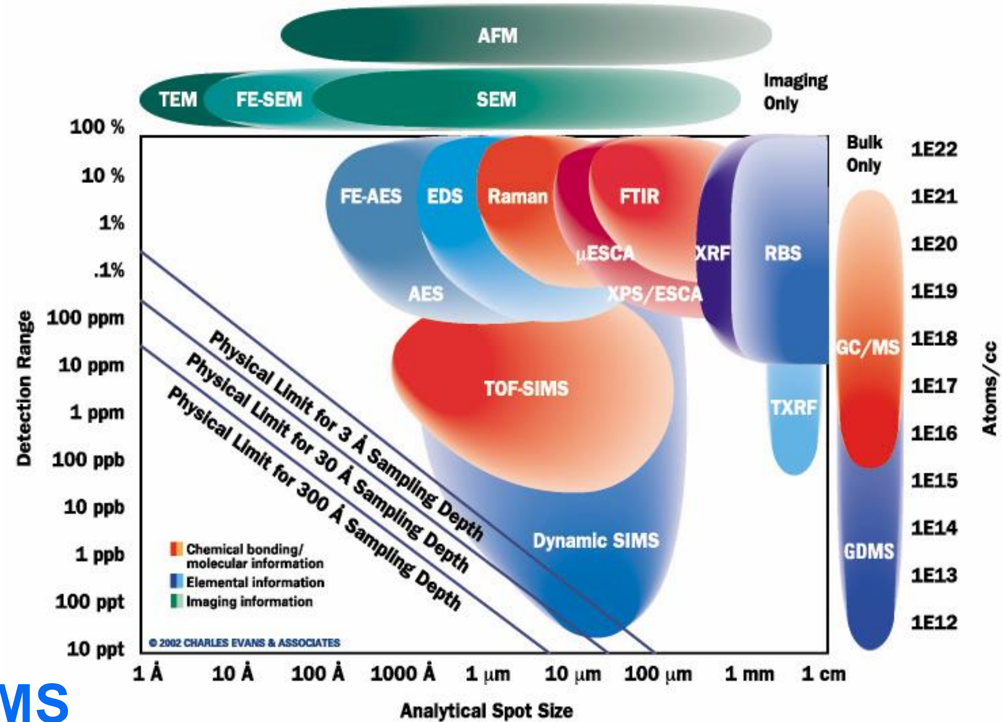
Technical Data Table



TOF-SIMS

Quantitative	Limited	Destructive	No
Detection Limits	10^{7}-10^{11} at/cm^{2}	Lateral Resolution/ Probe Size	0.2µm
Chemical Bonding	Yes	Analytical Depth	1-5 monolayers

Comments: Can identify specific organic compounds

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High Sensitivity/ Depth resolution: Dynamic Secondary Ion Mass Spectrometry

(Depth profiling-SIMS)

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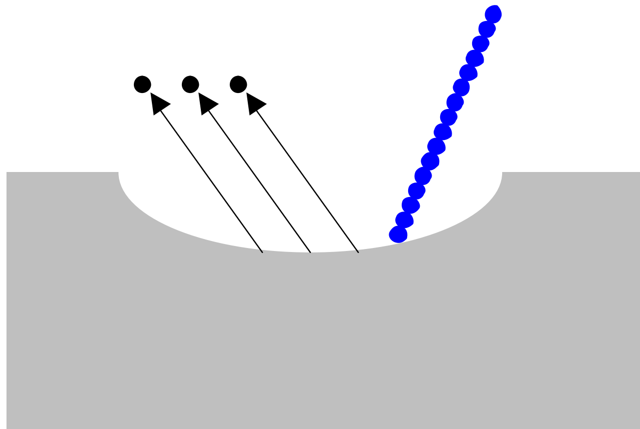
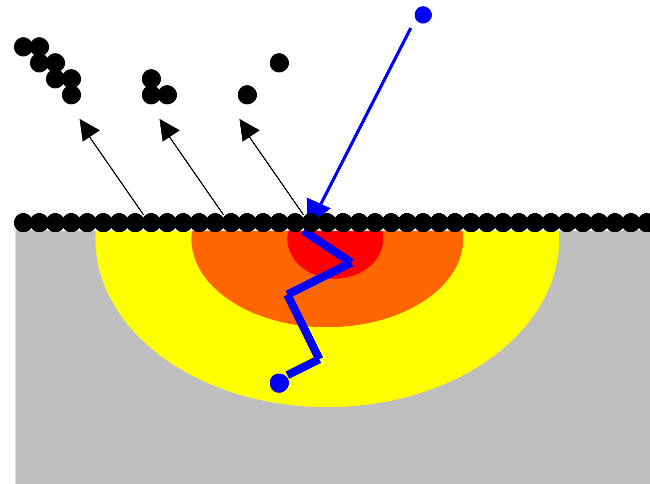
Analytical Modes of SIMS

Dynamic SIMS



- Material removal
- Elemental analysis
- Profiling

Static SIMS



- Ultra surface analysis
- Elemental or molecular analysis
- Analysis complete before significant fraction of molecules destroyed

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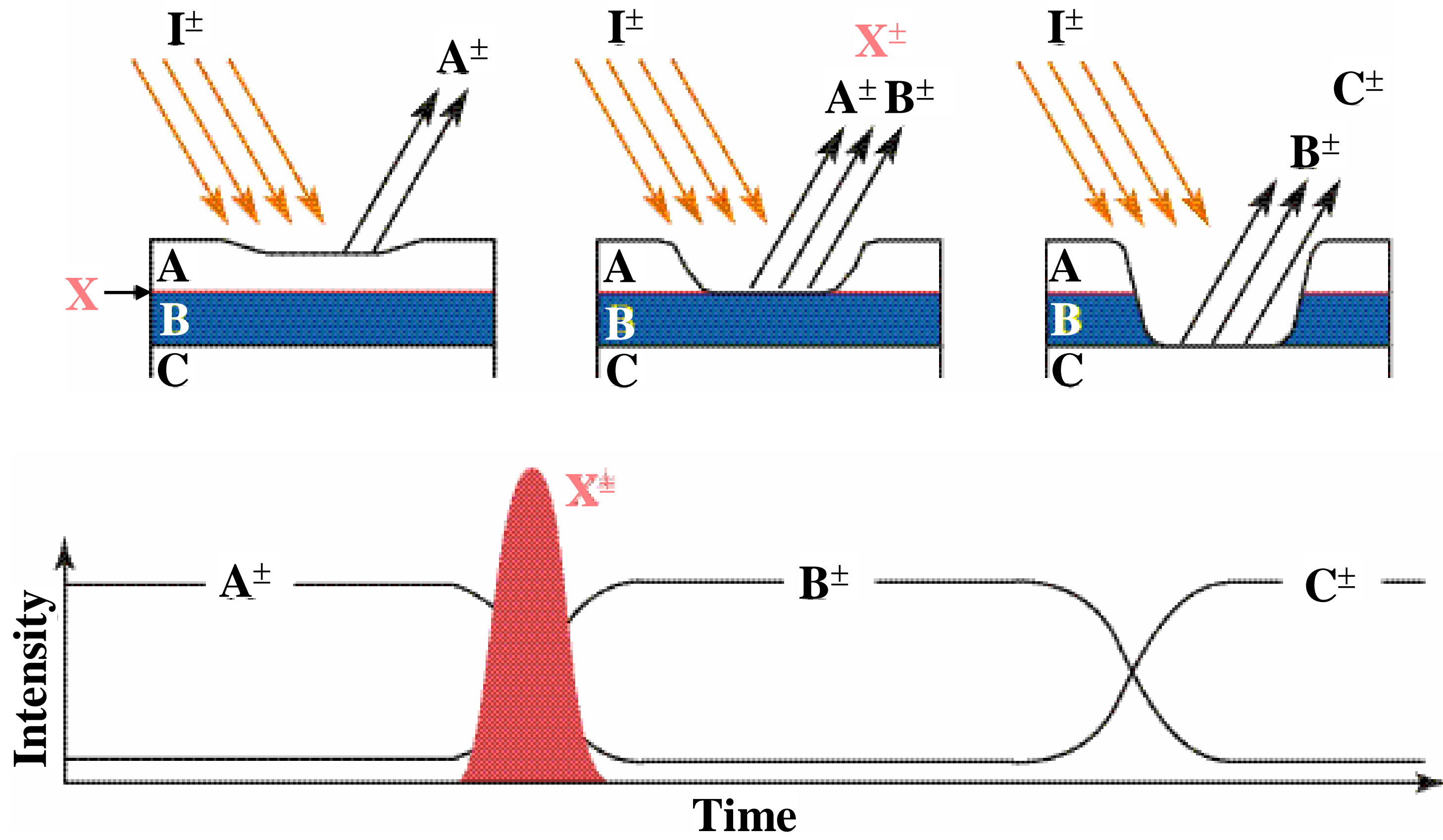
Dynamic SIMS for Depth Profiling



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Instrumentation and Capabilities

Mass Separation	Manufacturers	Strengths	Weaknesses
Magnetic sector	Cameca	• High transmission(~40%) • High mass resolution (M/ΔM ~10,000)	• Slow peak switching (magnet hysteresis effect.
Quadrupole mass filter	PHI, Atomika/Cameca	• Low primary beam energy (down to 100 eV) • Effective charge compensation for electrically insulating samples • Fast peak switching	• Low mass resolution (M/ΔM ~200) • Low transmission (~1%)

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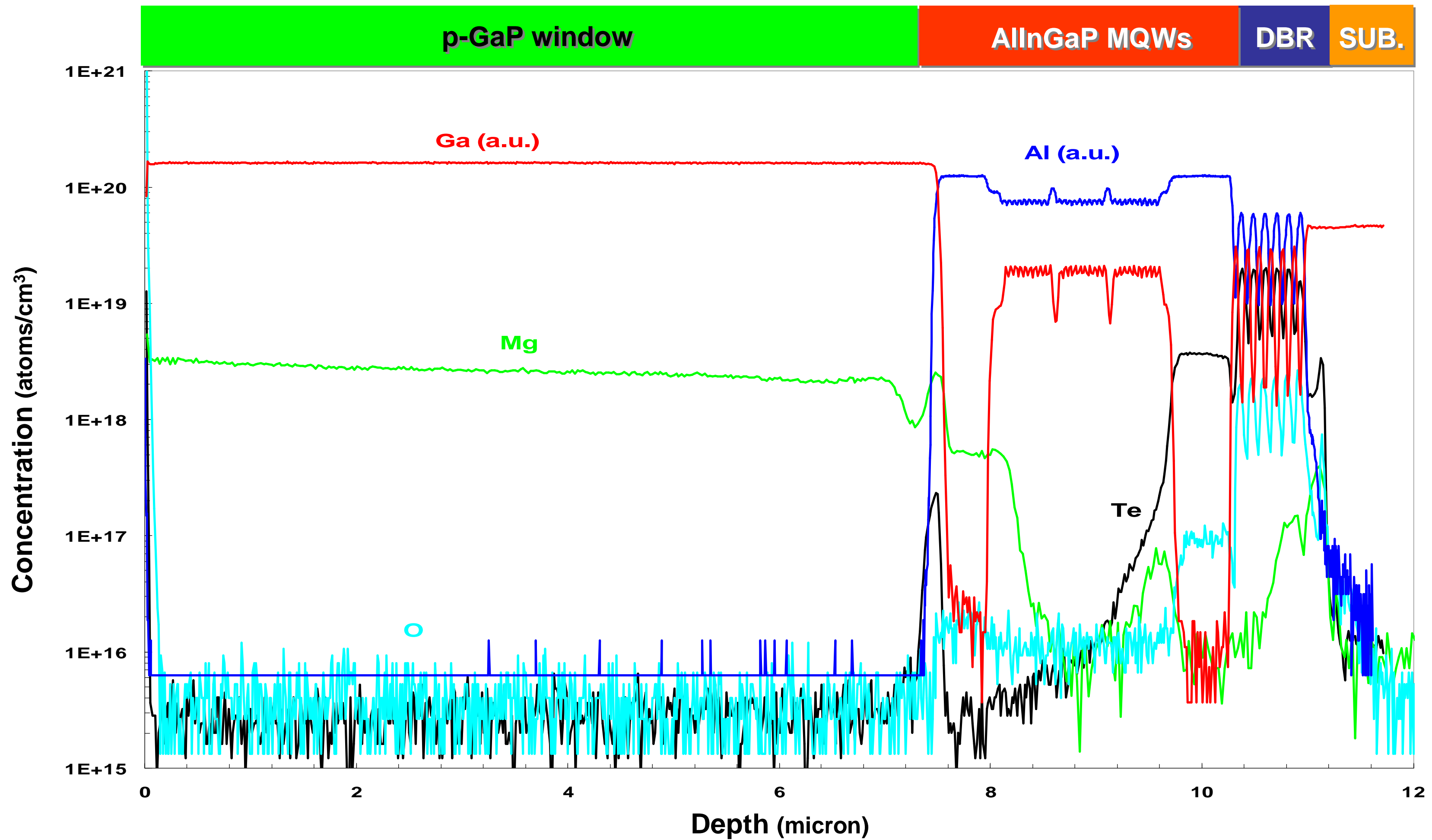
A Typical III-V SIMS Profile: AlInGaP LED



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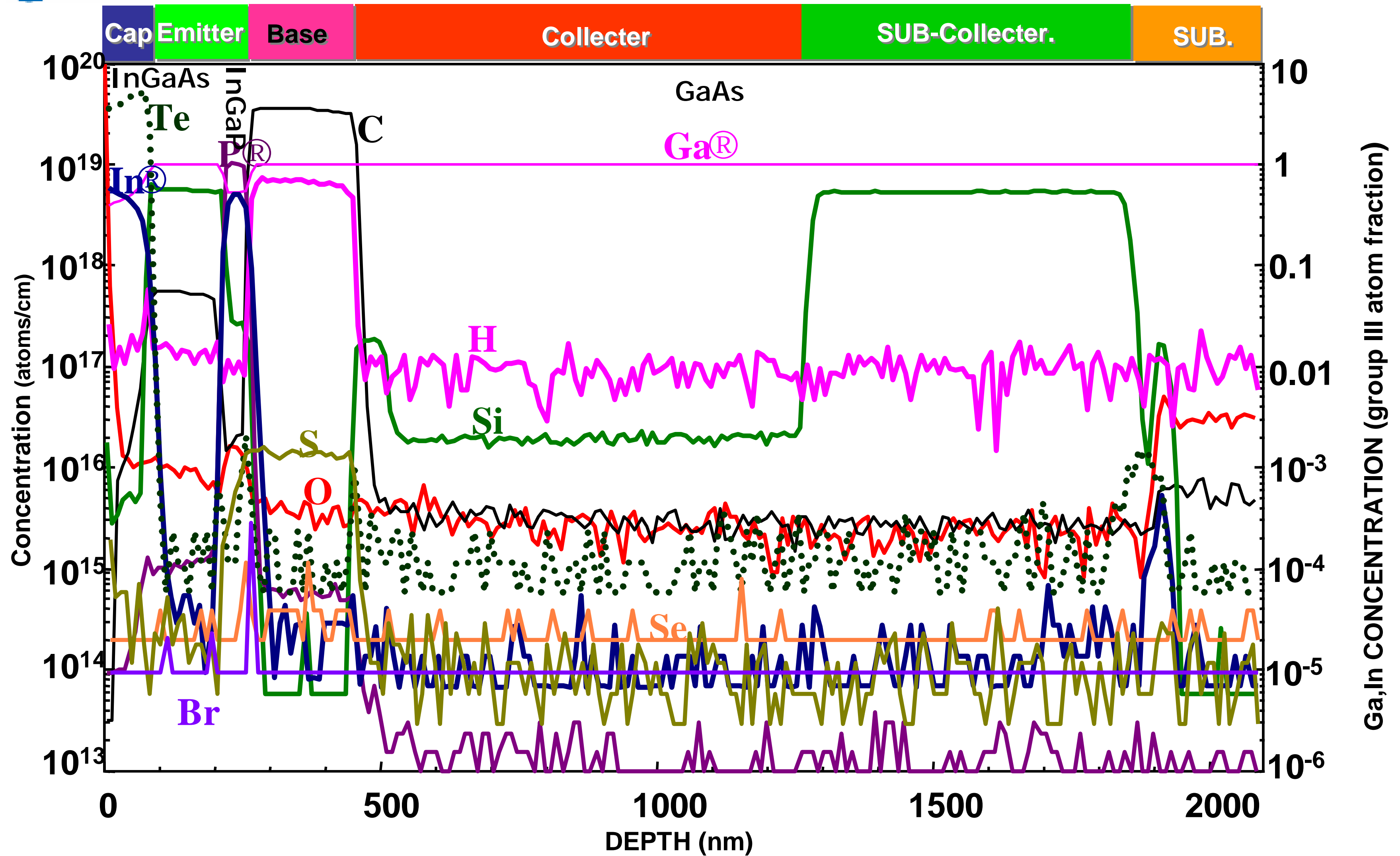
A Typical III-V SIMS Profile: InGaP HBT



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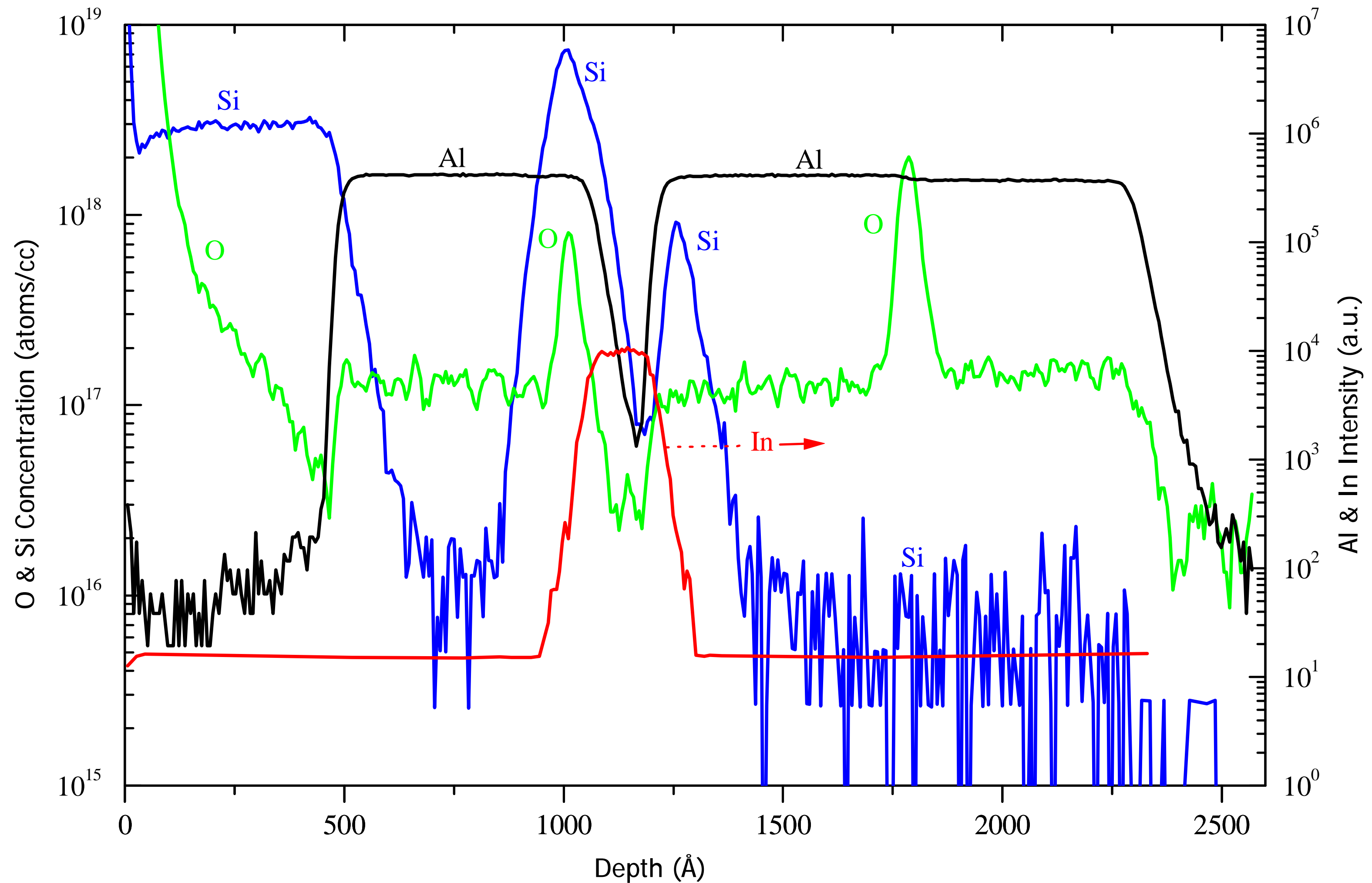
AlGaAs/InGaAs HEMT structure 1 keV Cs bombardment



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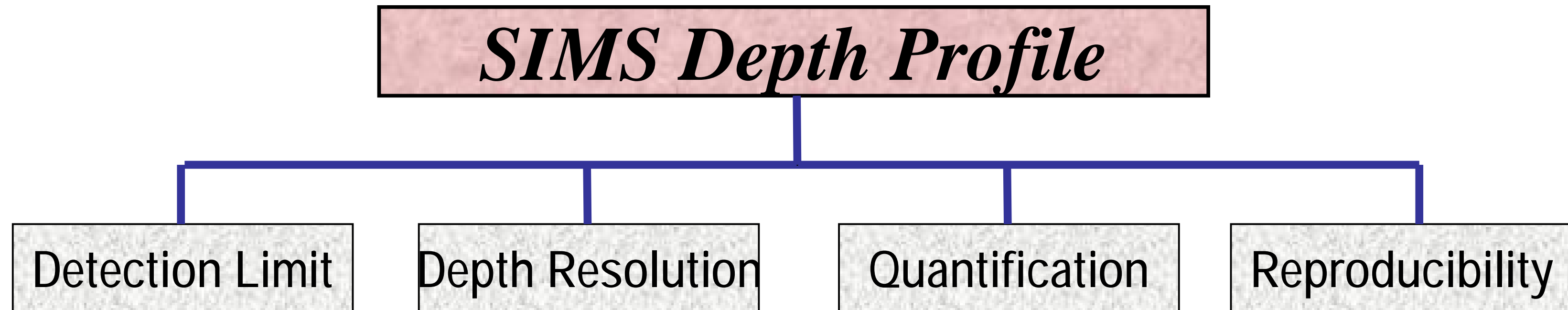
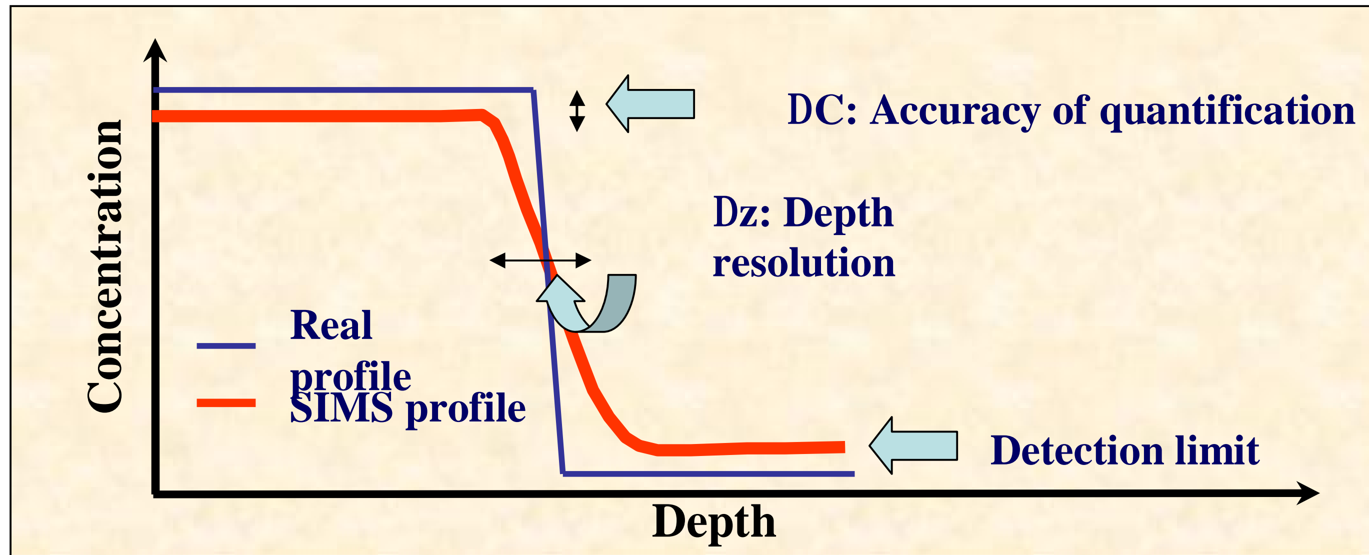
Some SIMS Key Aspects





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Methods to Optimize Detection Limits

- Use oxygen or cesium bombardment
  – Enhance positive or negative ion production
- Monitor molecular vs. atomic ion species
- Optimize instrument conditions
  – High mass resolution
  – Background subtraction
  – Energy filtering

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Elemental Detection for O_2^+ and Cs^+ Primary Ion Sources

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- Optimal analysis by O_2^+SIMS (positive ions detected)
- Optimal analysis by Cs^+SIMS (negative ions detected)
- Cs^+ cluster ions

Transition Metals

IA		IIIA	IVA	VA	VIA	VIIA	VIIIA			IB	IIB	IIIB	IVB	VB	VIB	VIIB	VIII
1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac	104 (Rf)	105 (Ha)	106												

For III-V:

- p-type dopant (except C in GaAs): O/positive ions& Cs/positive ions
- n-type dopants: Cs/negative ions
- C, H, O in Cs/negative ion
- Metal: O/ O/positive ions

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Typical Detection Limits in InP, GaAs, GaN

For electropositive elements

Element	M^+ (O_2^+)	M^- (Cs^+)
Li	3E13	1E16
Be	3E14	1E20
B	1E15	3E15
Na	3E14	2E17
Mg	1E14	1E20
Al	2E15	1E17
K	2E14	2E18
Ca	3E14	1E20
Ti	2E14	1E18
V	1E14	1E17
Cr	1E15	2E17
Mn	3E14	1E18
Fe	1E15	3E17
Ni	1E16	5E17
Cu	3E16	1E16
Zn	1E16	1E20
Sr	5E15	1E20
Y	1E17	1E20
Zr	1E15	4E17
Nb	1E16	1E18
Mo	1E16	1E18
Cd	5E16	1E21
In	3E15	3E17

For electronegative elements

Element	M^- (Cs^+)	M^+ (O_2^+)
H	2E17	2E18
C	1E16	2E18
N	5E15 (NGa-)	5E18
O	1E16	1E20
F	2E14	5E16
P	2E15	1E16
Si	2E15	1E16
S	1E15	1E19
Cl	3E15	2E17
Ge	5E15	2E16
Se	5E14	2E17
Br	5E13	1E17
Te	1E15	2E17
Ag	2E16	2E16
Au	1E15	1E17

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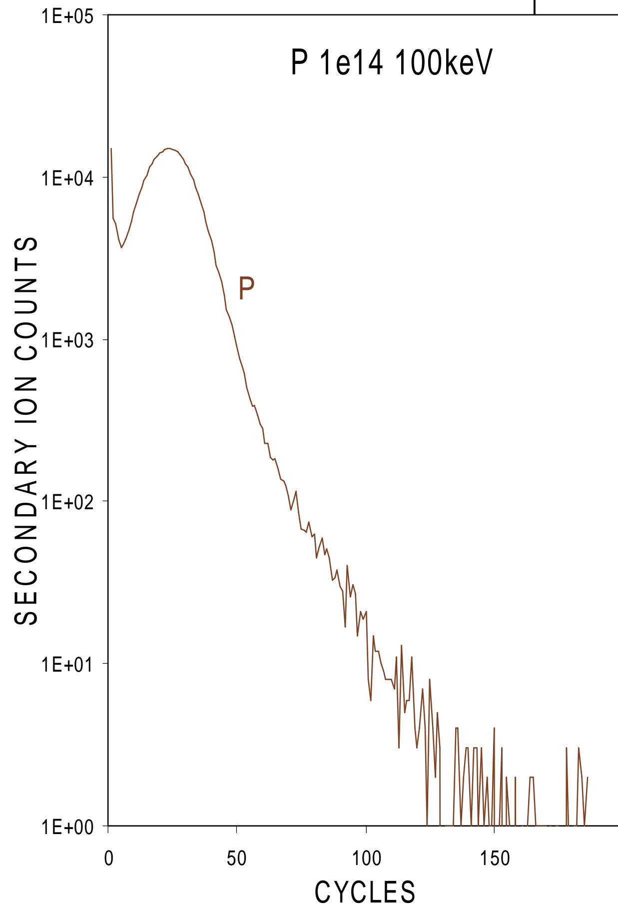
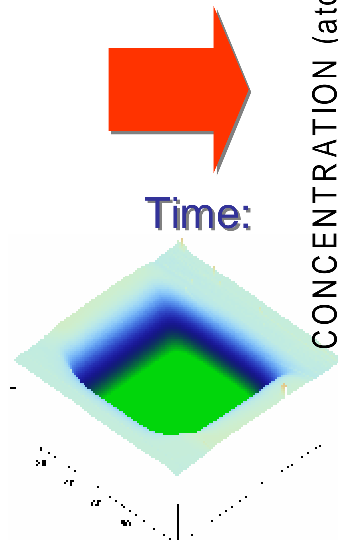
SIMS Quantification

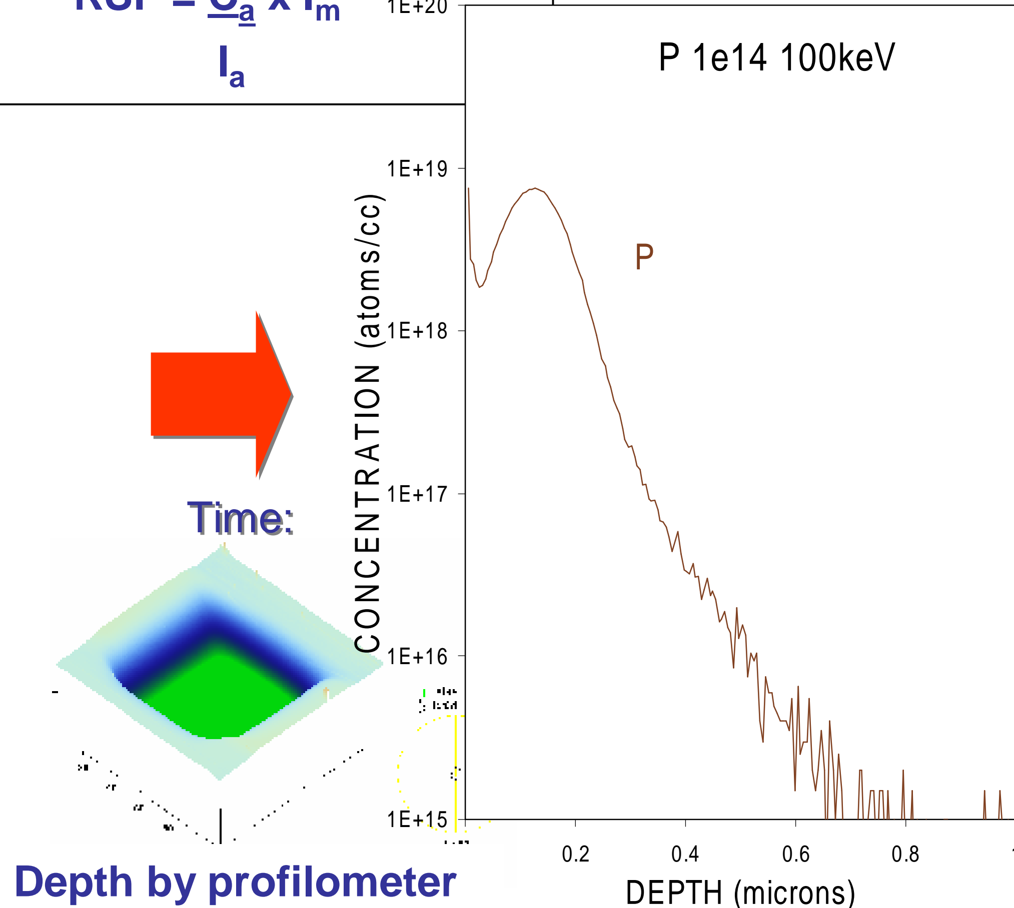
With a STANDARD of known composition:

$$RSF = \frac{\underline{C_a} \times I_m}{I_a}$$





Depth by profilometer



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SIMS Accuracy and Precision

- Accuracy with standard 10%-20%
  - Implant standard 5%-10% Different labs have different standards
  - Depth measurement 5%
  - Precision (Reproducibility) 10% in the same run
- Accuracy without standard: 100-200% or high
  - Knowledge on the matrix effect

Precision: reproducibility between run

- 10% routinely by an experienced analyst or following a good protocol
- 2% can be achieved using the HPIC protocol

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Depth Resolution

- Definition:
  - The width within which a measured analytical signal decreases from 84% to 16% of its maximum plateau value when sputtering through a sharp interface between two media[1]
- Depth resolution depends on
  - element/matrix combination
  - impact conditions (primary ion species, ion energy and angle of incidence)
  - sputter depth (ion bombardment induced topography)
  - artifacts from non-perfect sample and from non-perfect shape of crater bottom
  - crater edge rejection
- Optimum depth resolution is obtained by controlling all of the above

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Benefits of Reducing Primary Ion Beam Energy

- Reduces recoil mixing, which arises from direct collision of primary ions with sample atoms.
- Reduces cascade mixing, which results from motion and collisions of sample atoms with each other.

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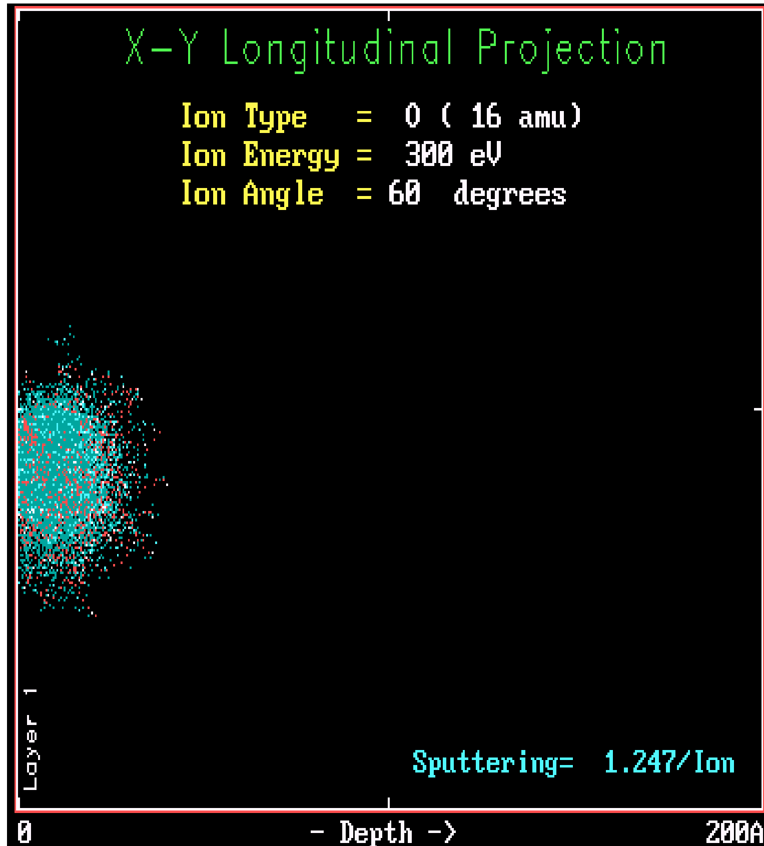
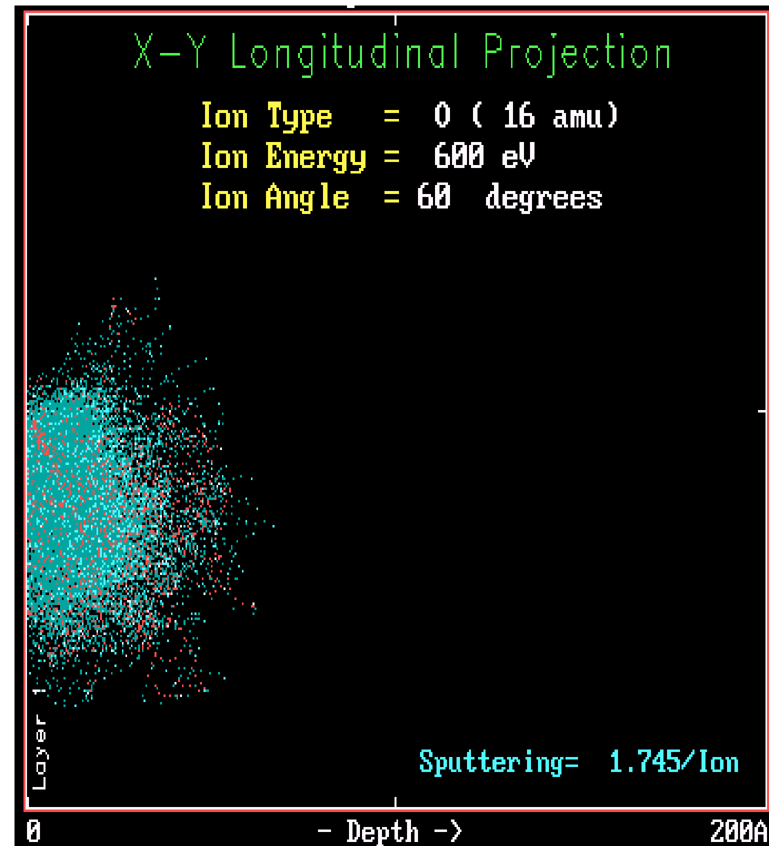
Comparison of Primary Energy to Mixing Depth





Simulations from TRIM (Transport of Ions in Matter)

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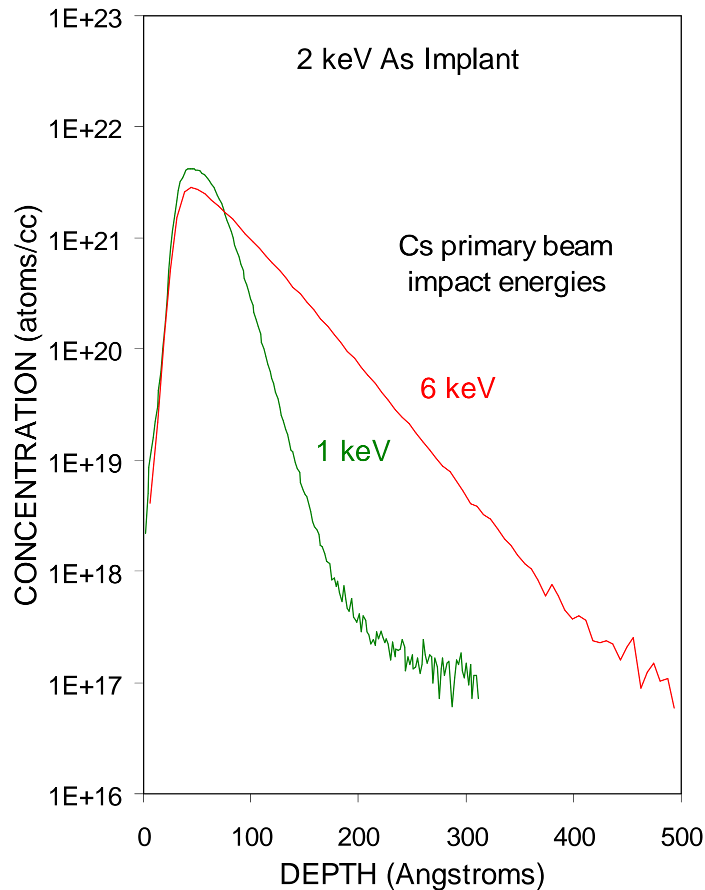
Improved with Lower Energy



- Improved depth resolution is seen as impact energy is decreased.
- The 6 keV profile shape is entirely a product of ion beam mixing.
- The 1 keV profile shows dramatically improved depth resolution that reveals much of the true profile shape.

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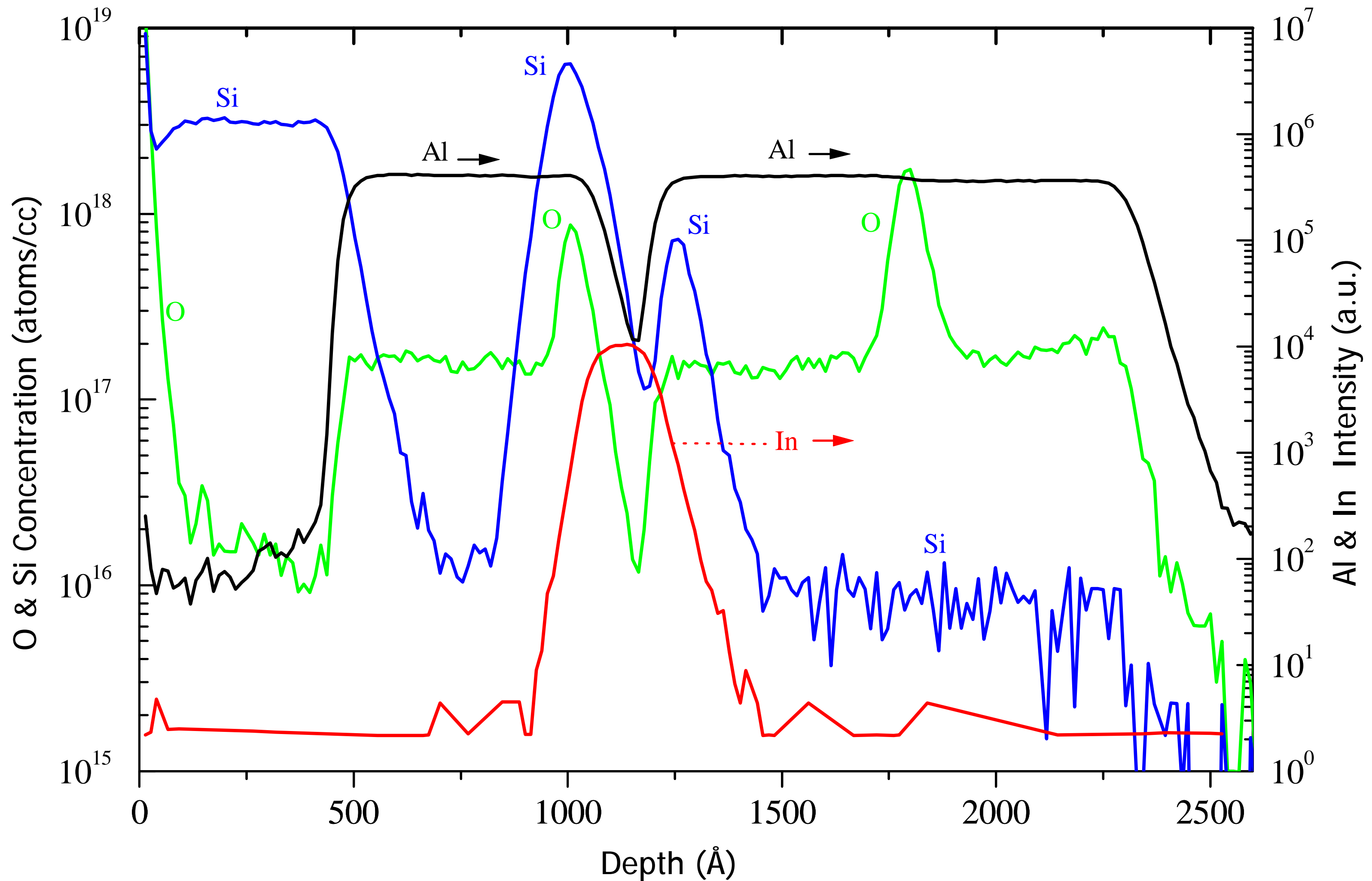
AlGaAs/InGaAs HEMT structure
2 keV Cs bombardment



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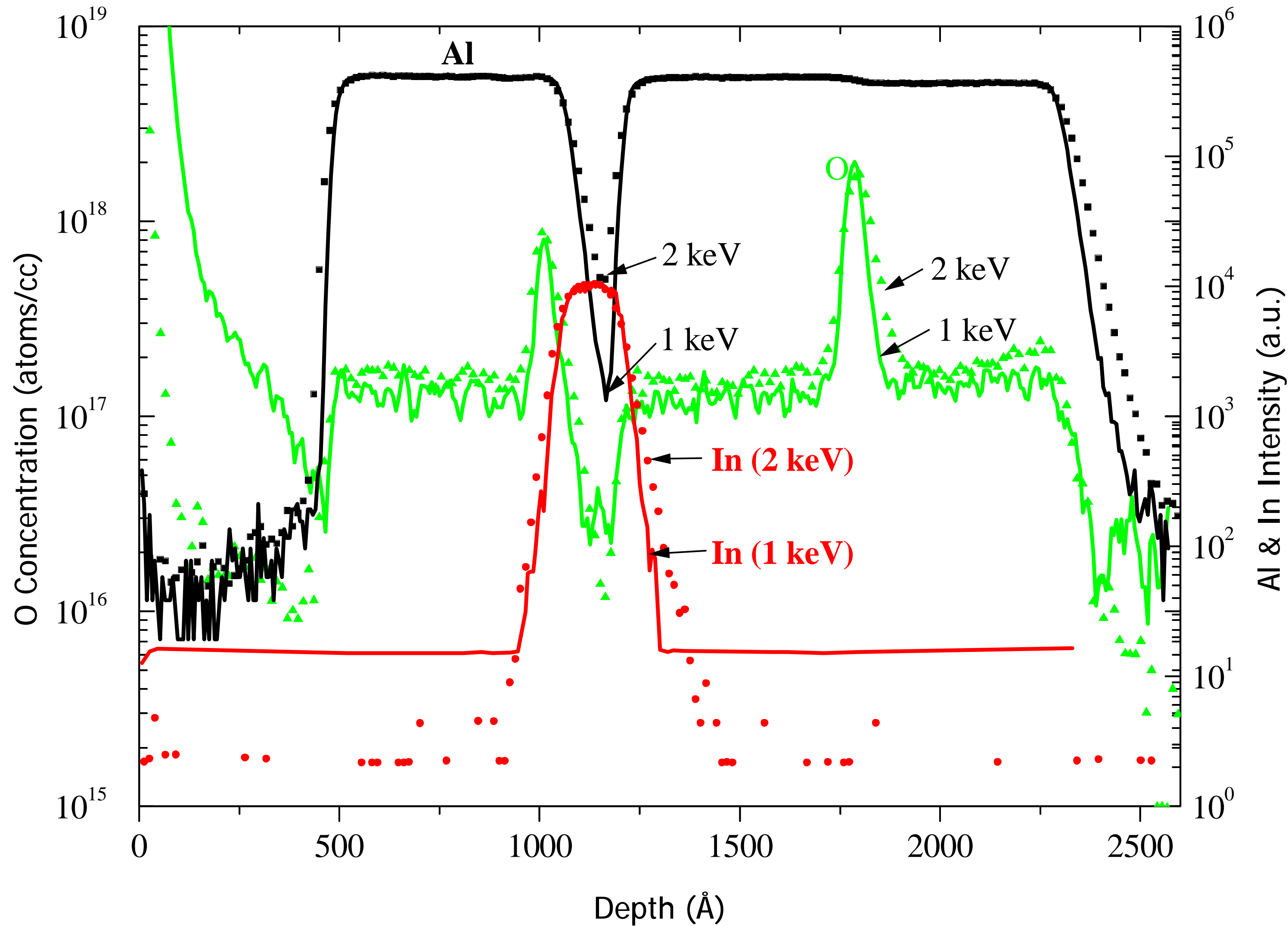
AlGaAs/InGaAs HEMT structure 2keV vs 1keV Cs bombardment



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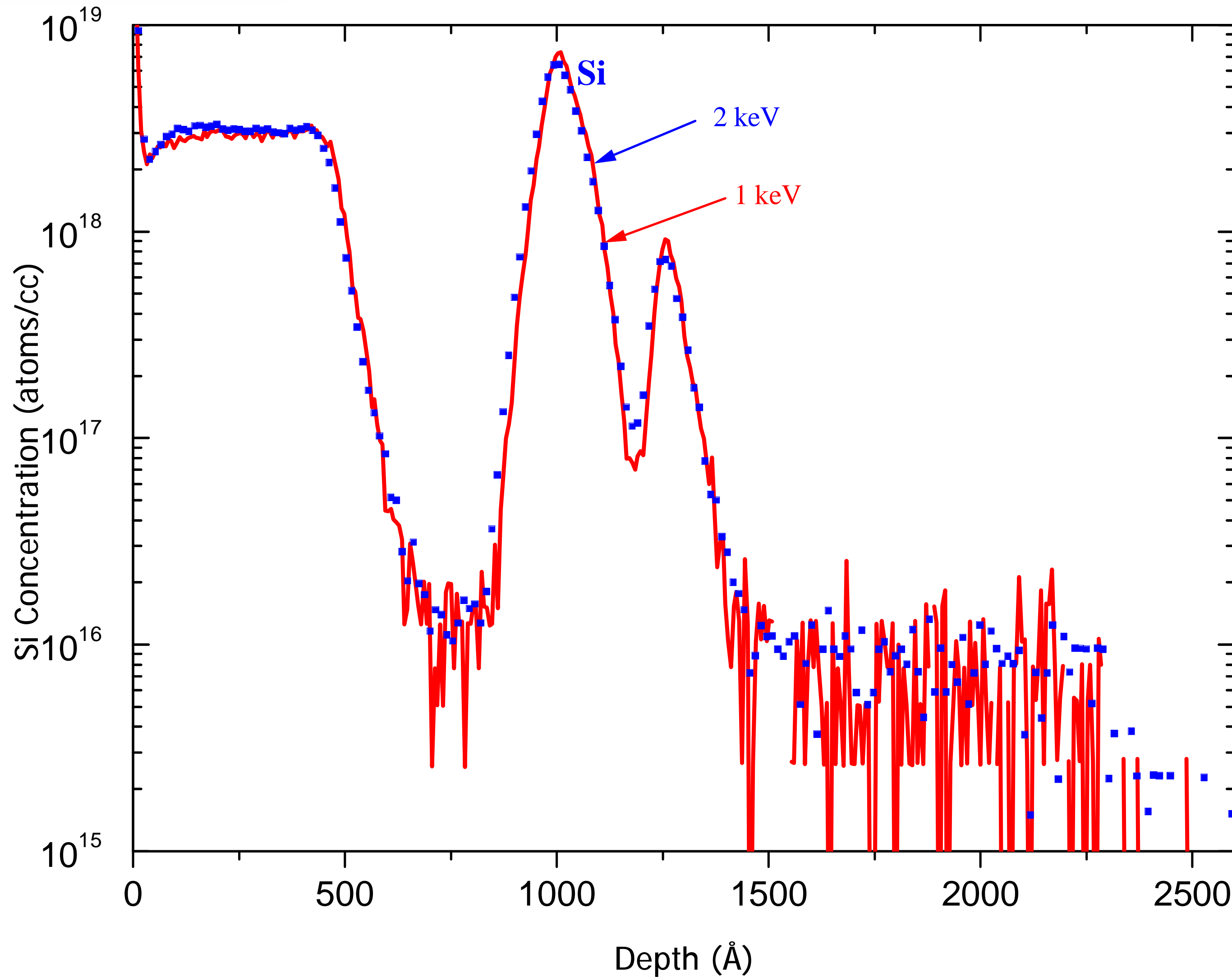
AlGaAs/InGaAs HEMT structure 2keV vs 1keV Cs bombardment



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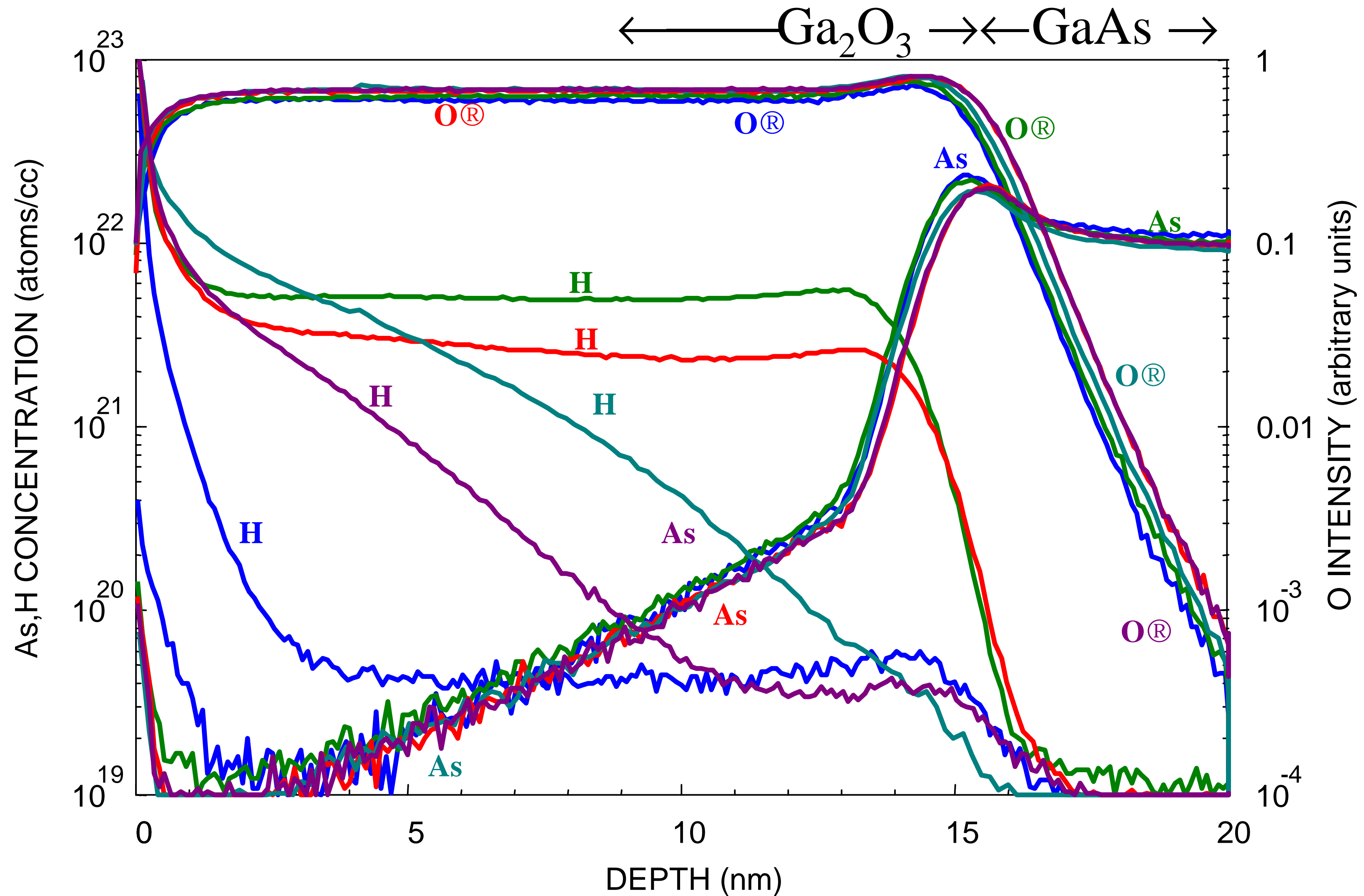
Hydrogen treated Ga_2O_3 15nm gate dielectric



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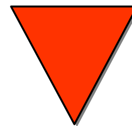
SIMS Strengths

- Strengths
  - Excellent detection sensitivity (ppm-ppm) for dopants, impurities and known contaminants
  - Can detect all elements and isotopes, including H
  - Inherent sputtering of layered structures



- Powerful analysis tool
  - Depth profiling dopants and impurities in III-V heterostructures
  - Surface, layer, substrate and interface
  - Stoichiometry in some cases

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Key Applications for III-V Analyses

- Dopant calibration (Be, C, Mg, Si, Zn, Se, etc.)
- Impurity measurements (H, C, O, etc.)
- Compositional analyses ($Al_xGa_{(1-x)}As$, $In_yGa_{(1-y)}As$, etc.)
- Process development
- Process monitoring
- Process transfer
- Epi equipment evaluation

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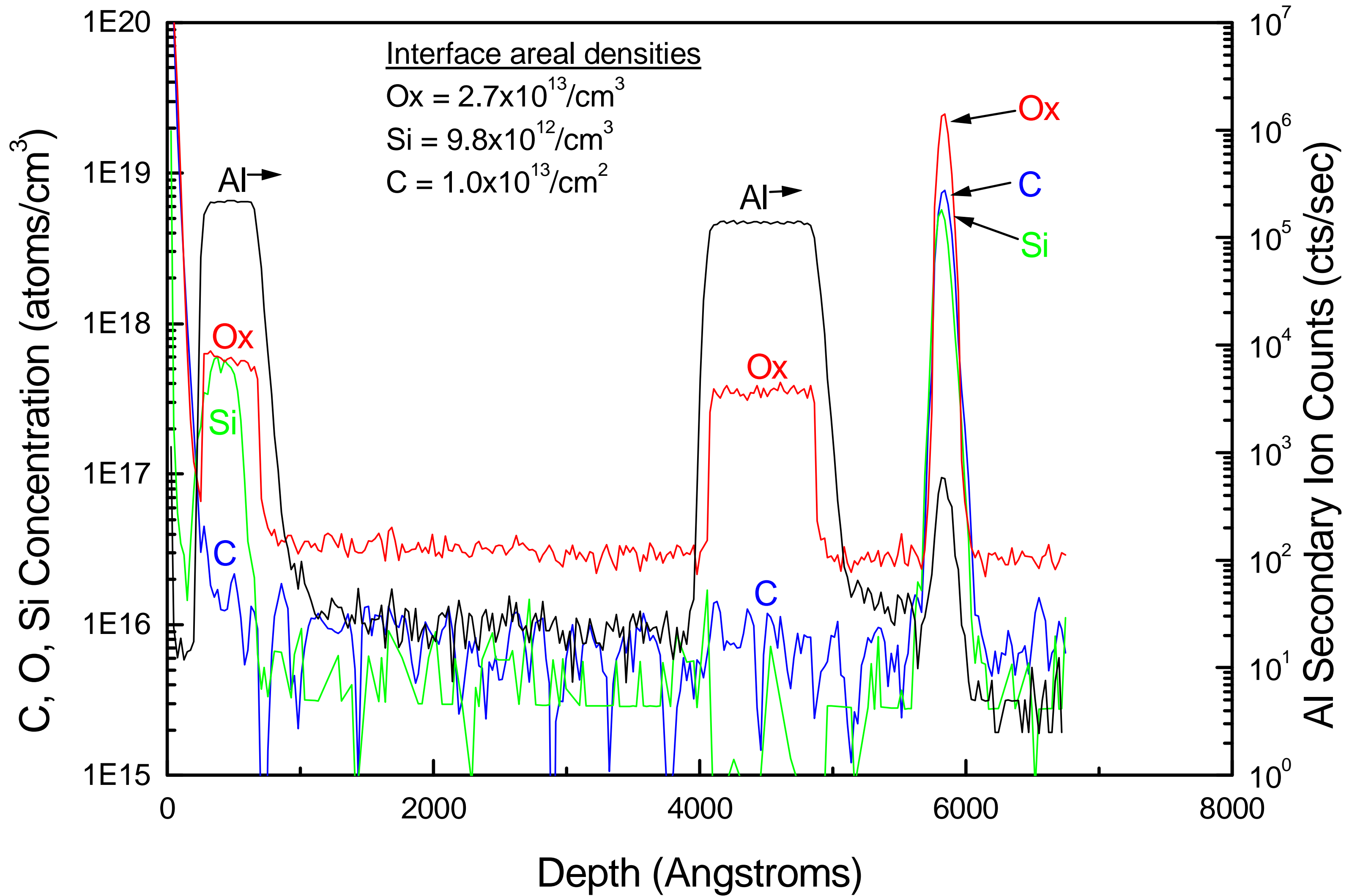
Interface Contamination: HEMT



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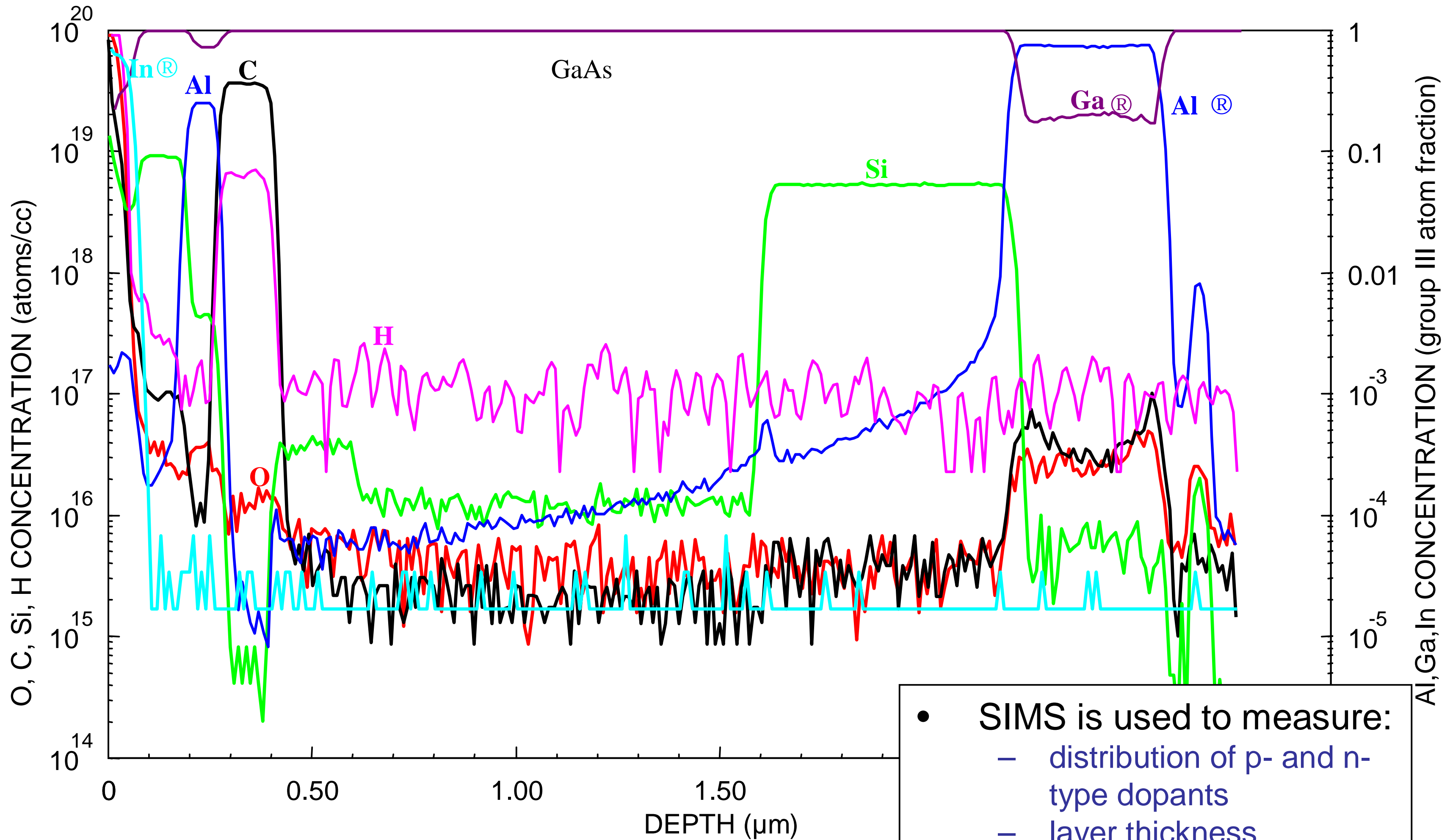
SIMS for III-V High Speed Electronics

InGaAs/AlGaAs/GaAs HBT SPC



- SIMS is used to measure:
  - distribution of p- and n-type dopants
  - layer thickness
  - p-n junction

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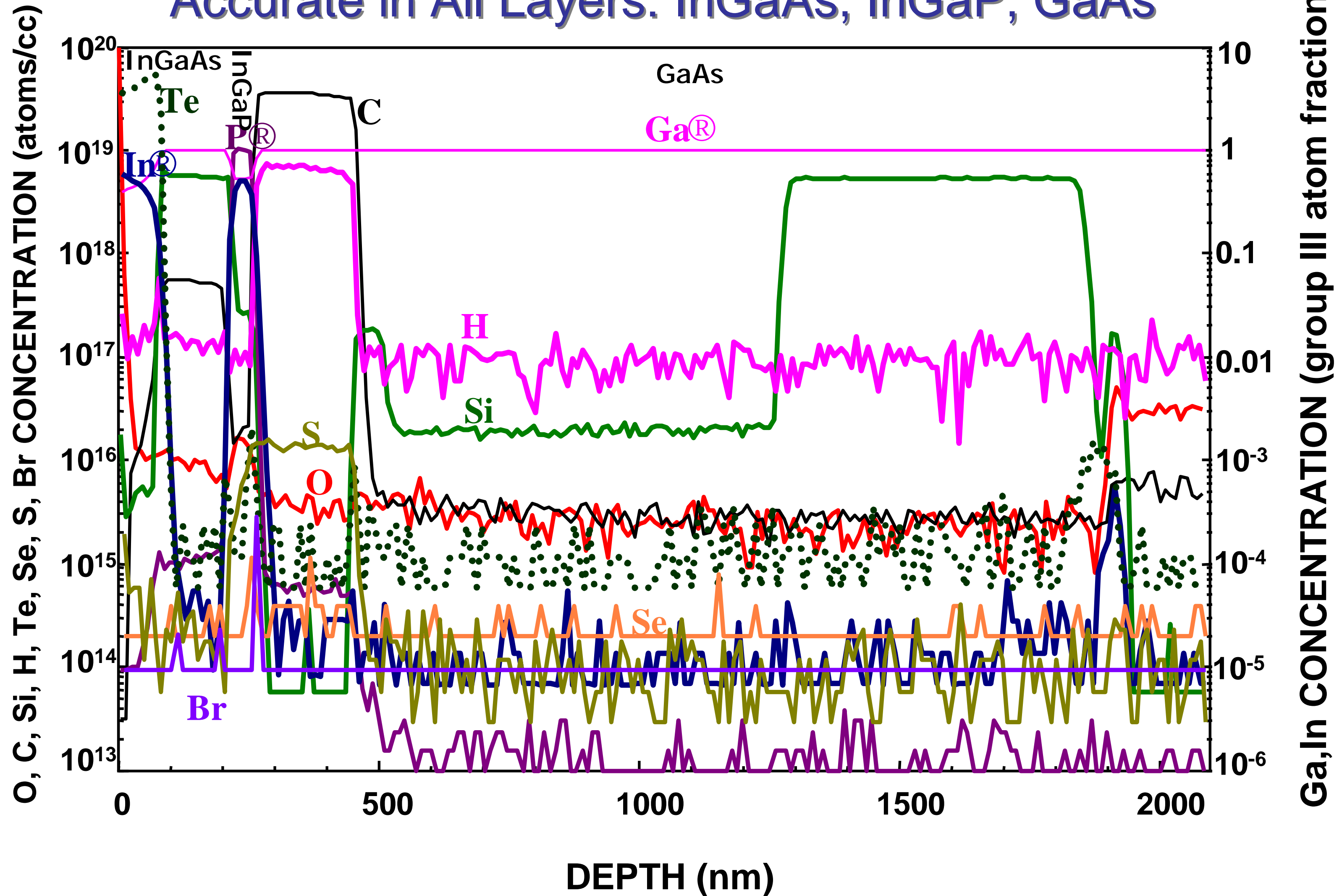
State-of-the-Art InGaP Emitter HBT



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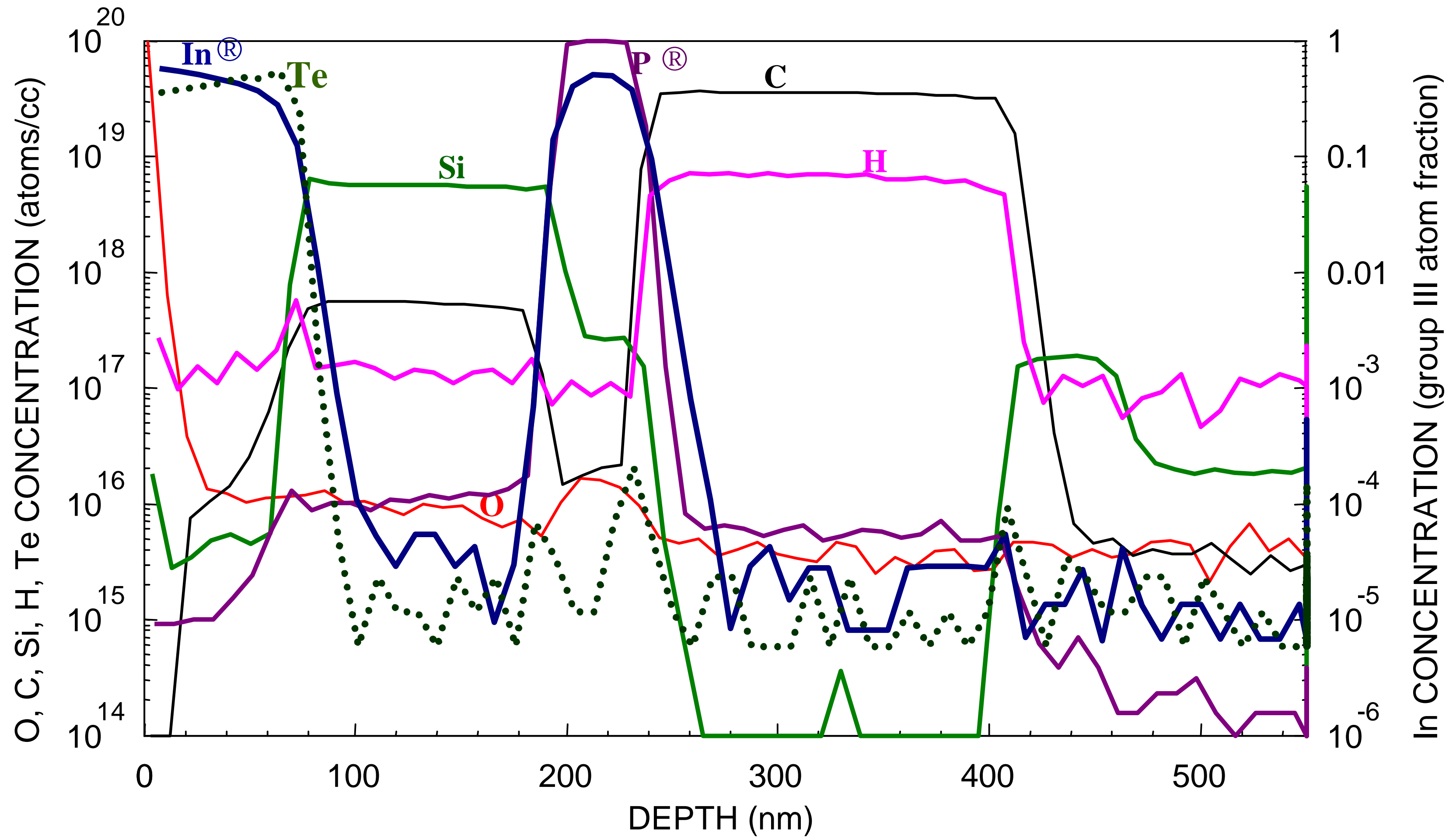
InGaP HBT



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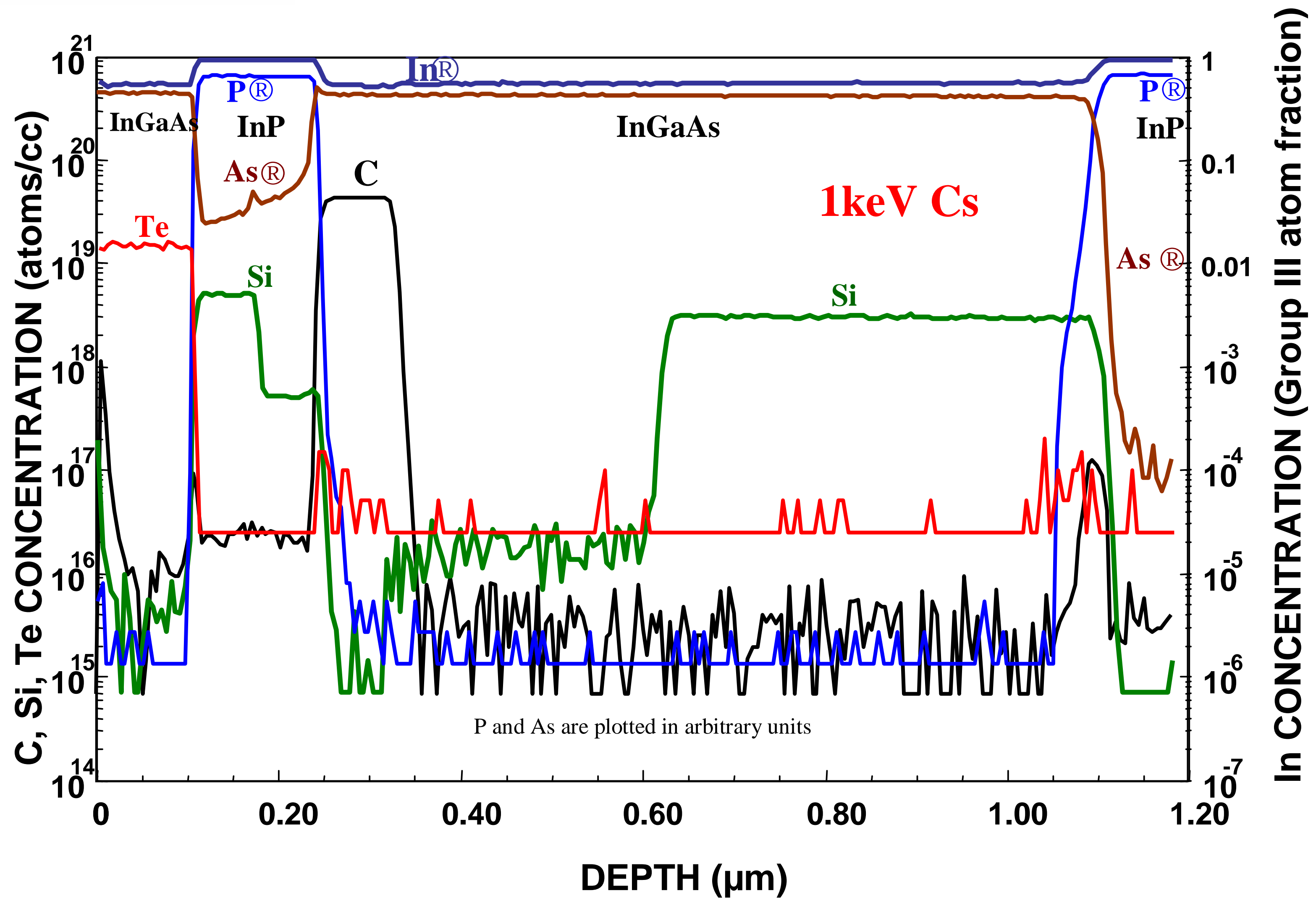
InP HBT



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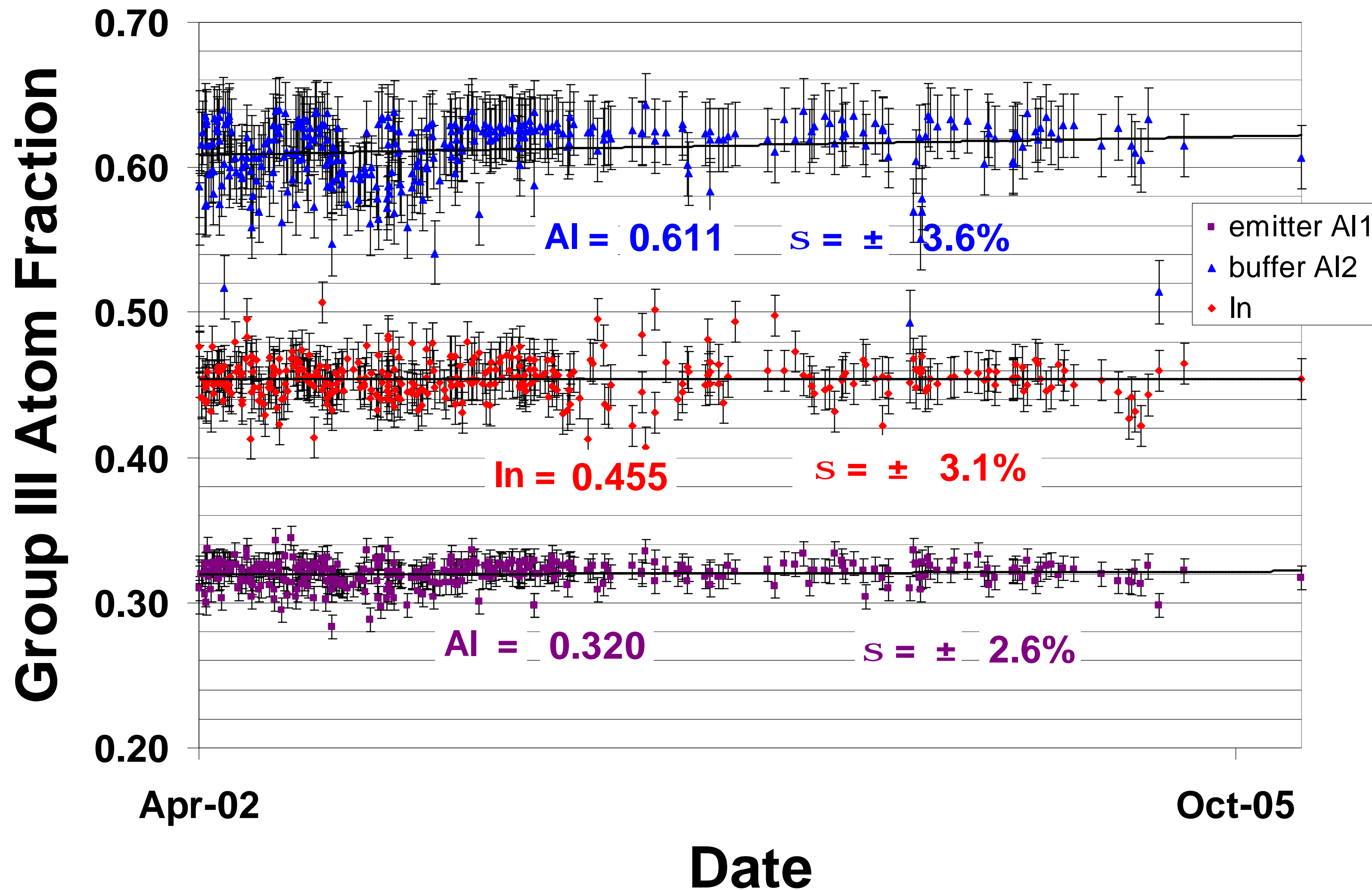
SIMS Measurement Long Term Statistics

3 – 5% Precision over 3 year span



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SIMS Measurement Long Term Statistics

Concentration (atoms/cm³)

4.E+19
3.E+19
2.E+19
1.E+19
2.E+17

Carbon doping = 4.09E+19 s = ± 4.5%

GaAs Base C
Contact Si2
sub-coll Si5

Si doping = 5.43E+18 s = ± 3.7%

Si doping = 4.02E+18 s = ± 3.8%

Apr-02
Oct-05
Date

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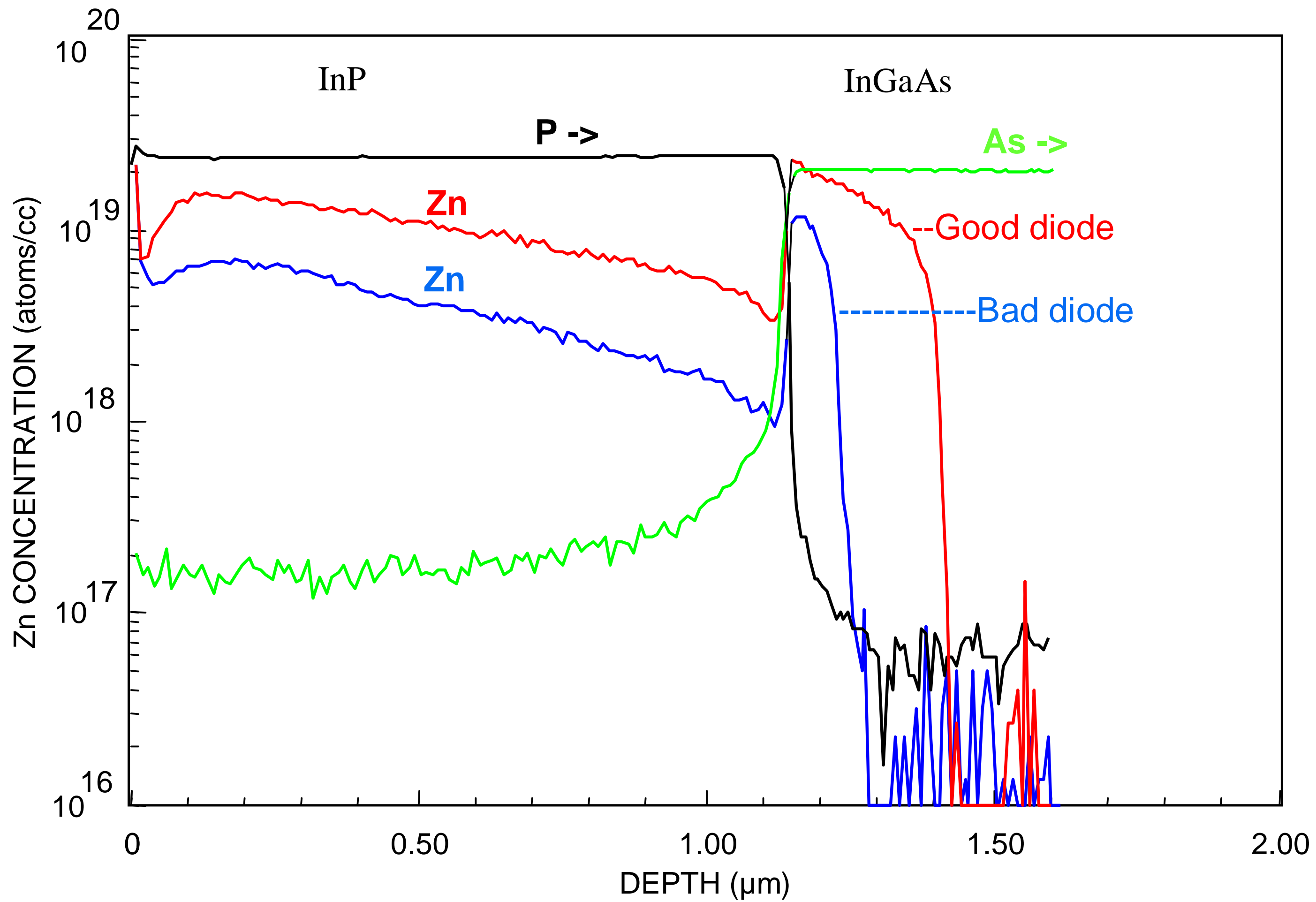
SIMS for III-V Optoelectronics

Zn Diffusion in Good vs. Bad Photodiodes



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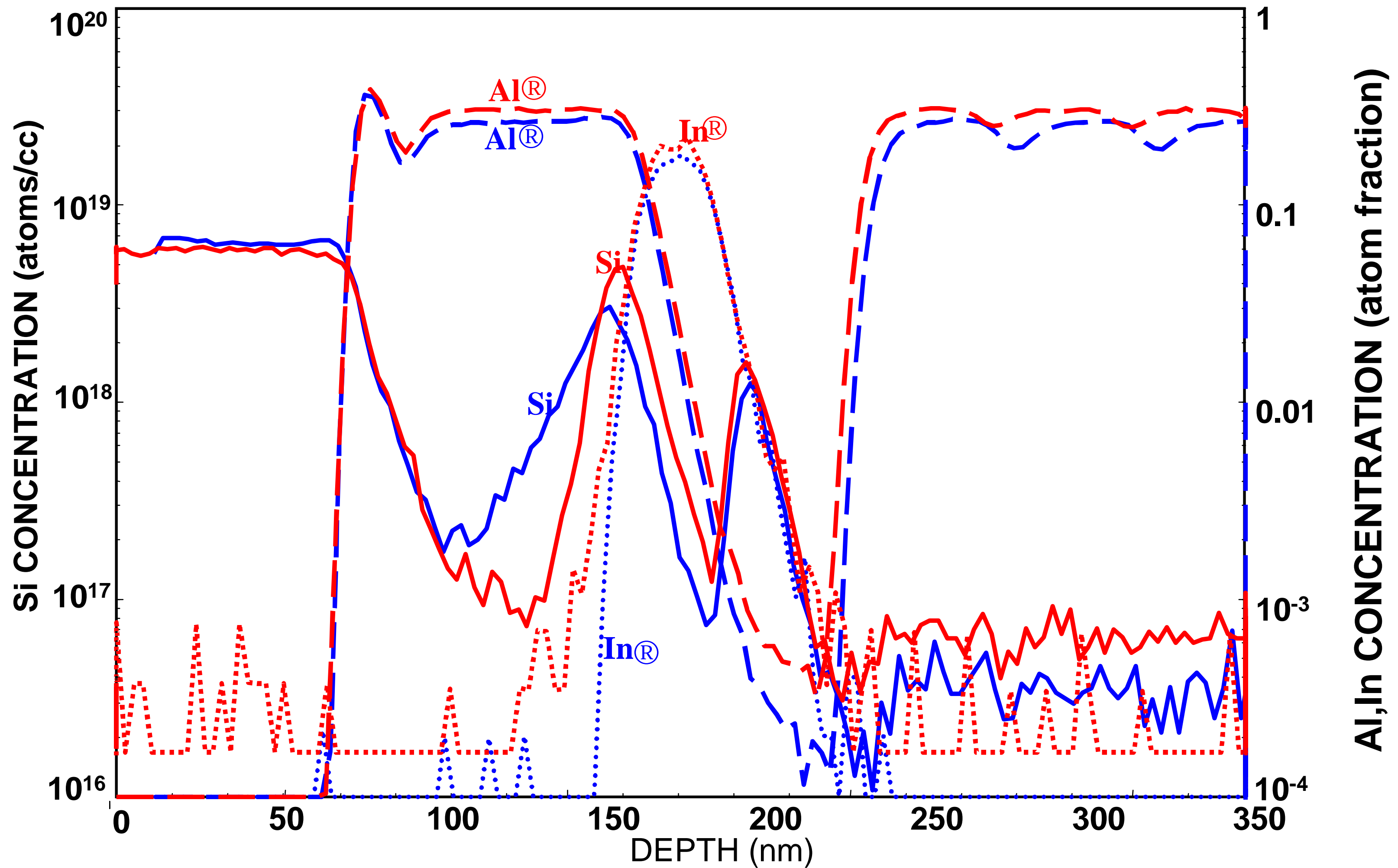
Good and Bad PHEMT Comparison



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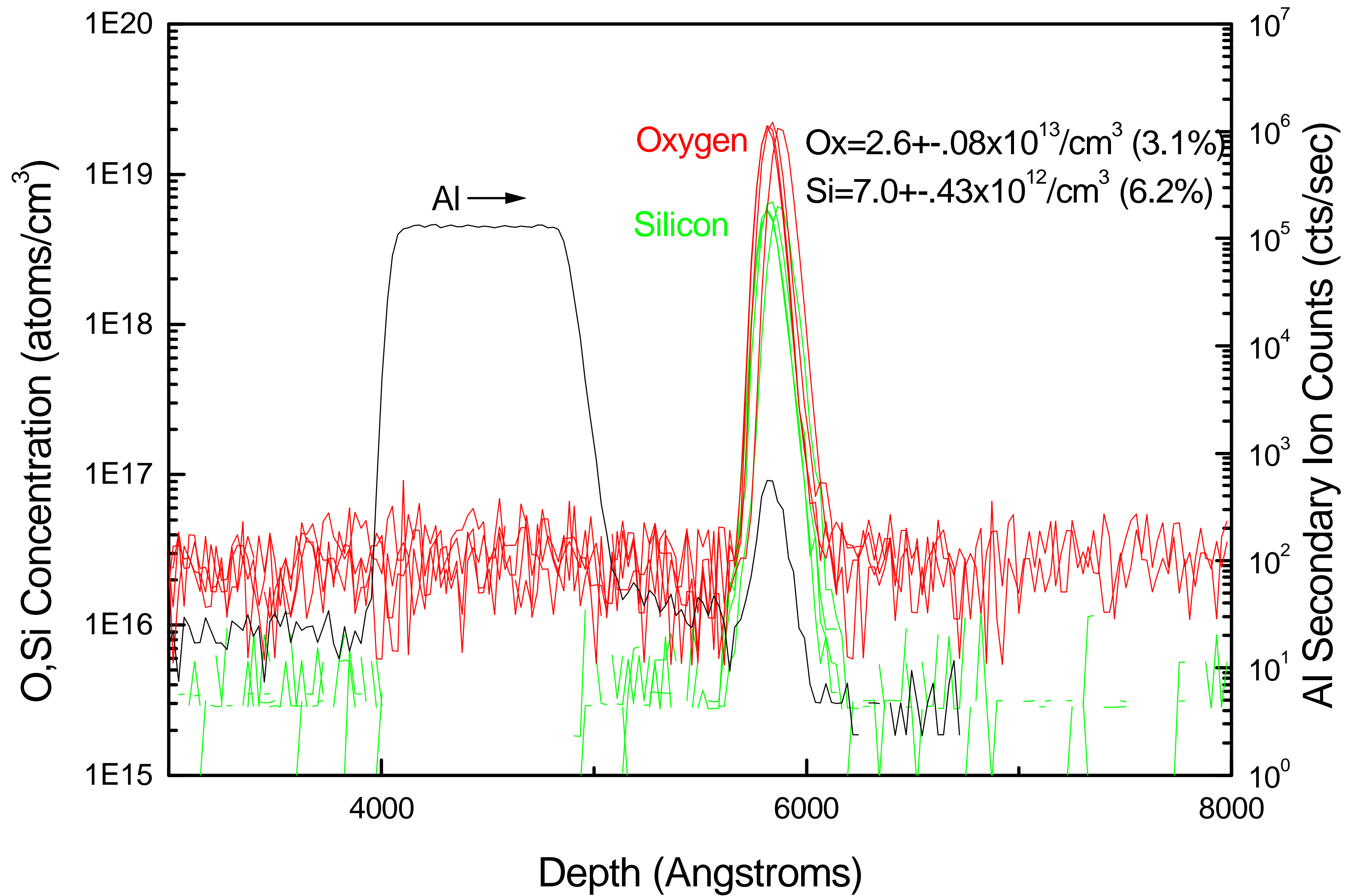
Interface Contamination: HEMT



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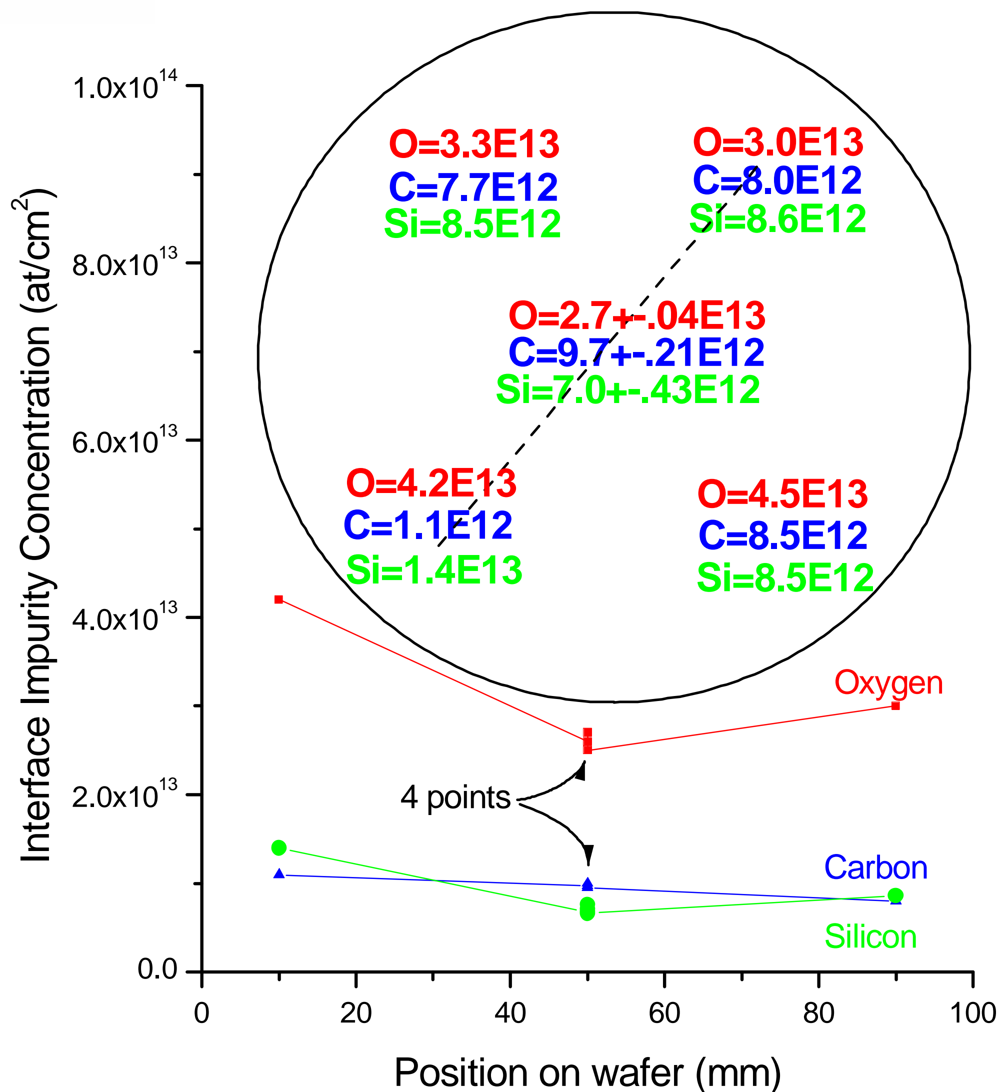
Variation of Interfacial Contamination across Wafer



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Sample Preparations for SIMS

Sample Preparations:

- **Chemical Etching**
- **Mechanical Polishing**
  - Thinning sample
  - Smoothing surface

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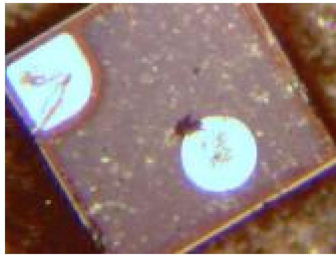
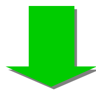
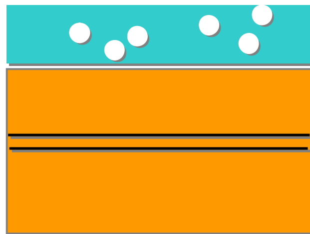
Sample Prep for GaN LED

Finished GaN LED die



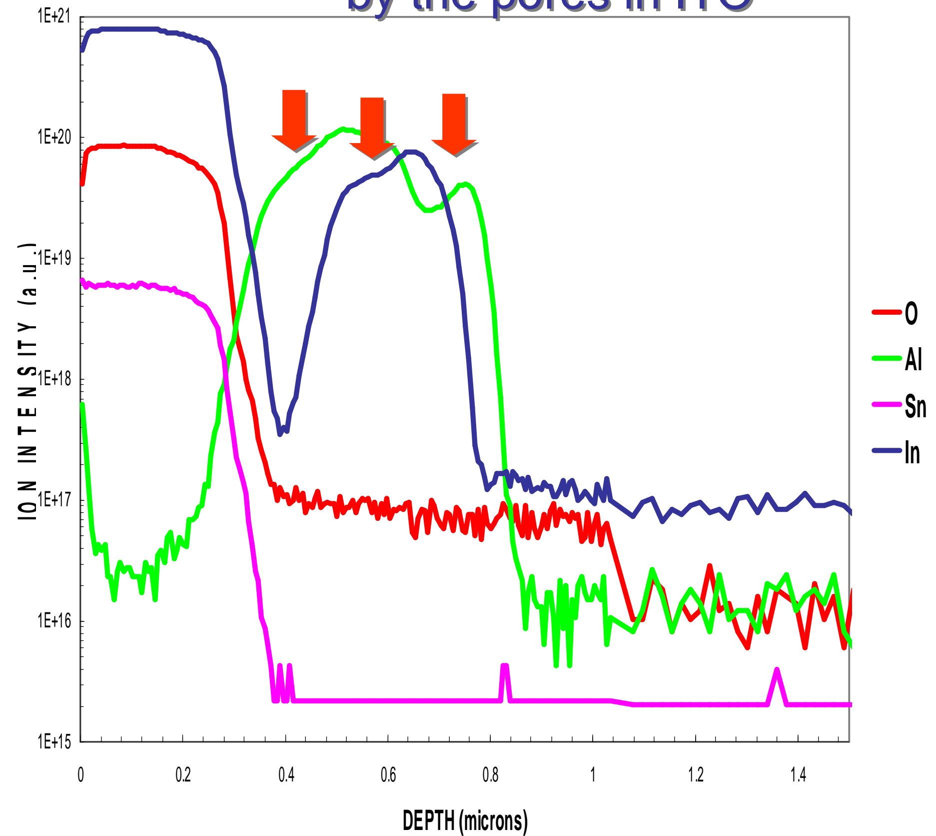




ITO (porous)

GaN Epi

Al and In Forward tails caused by the pores in ITO



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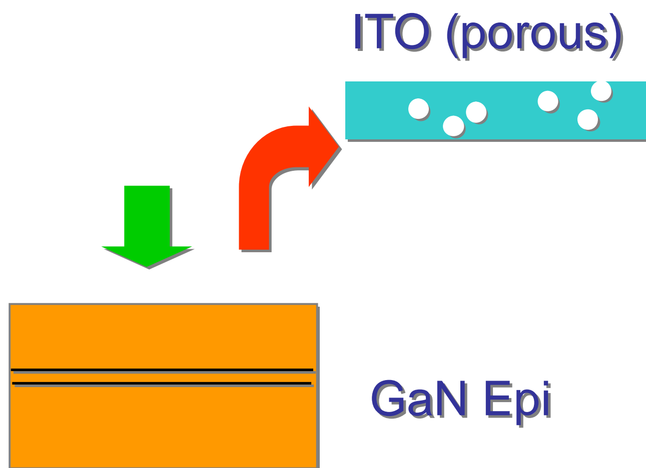
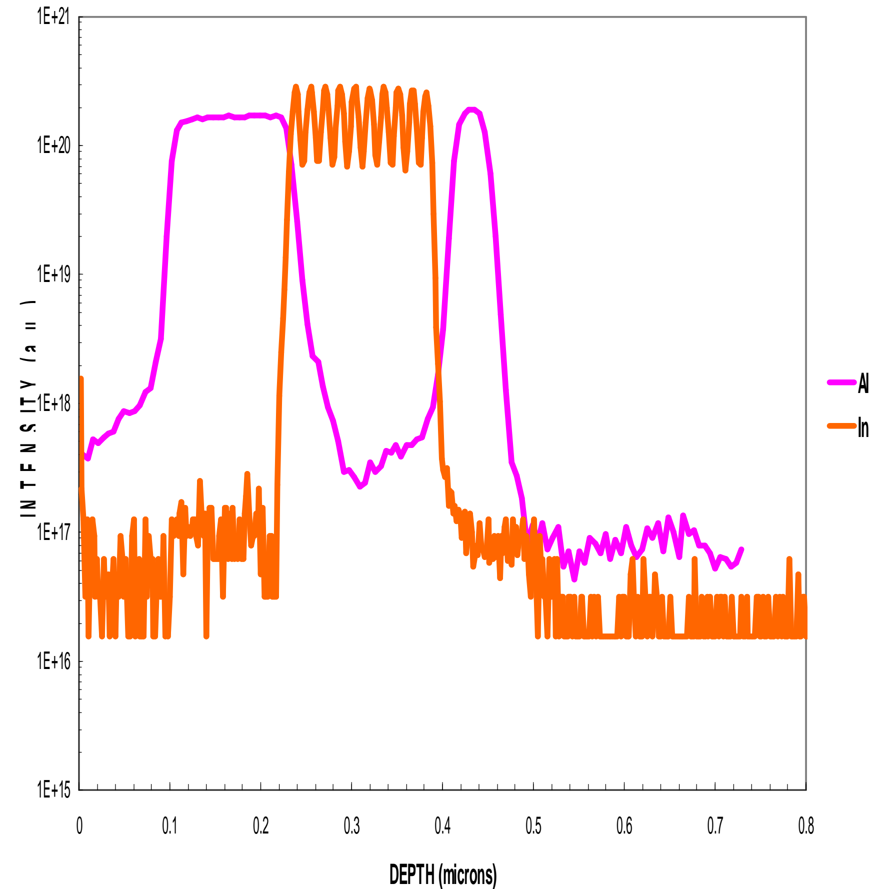
Sample Prep for GaN LED

Finished GaN LED die

Remove ITO by Chemical Etching





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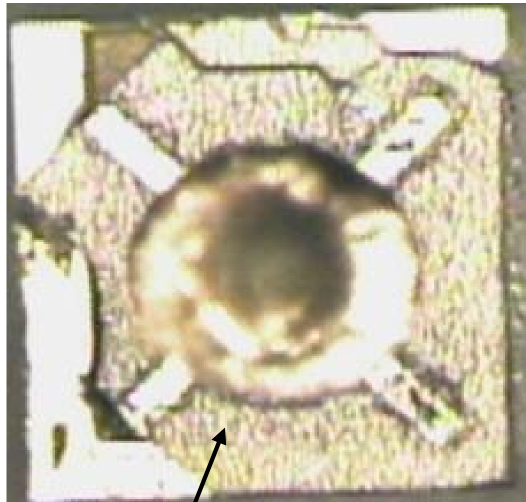
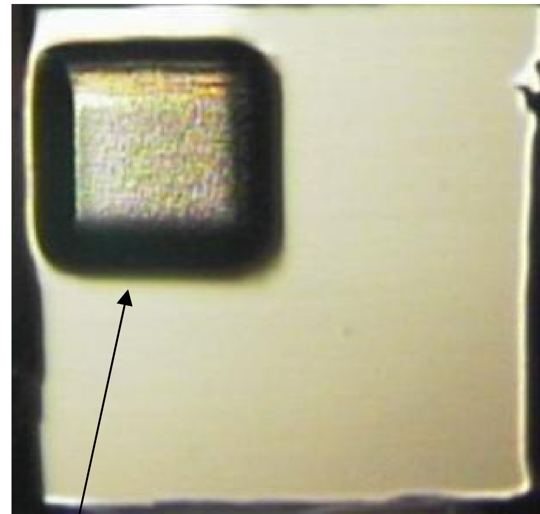
AlInGaP LED: Polishing

300 x 300 micron chip

Before **After**





Ball bond

SIMS crater

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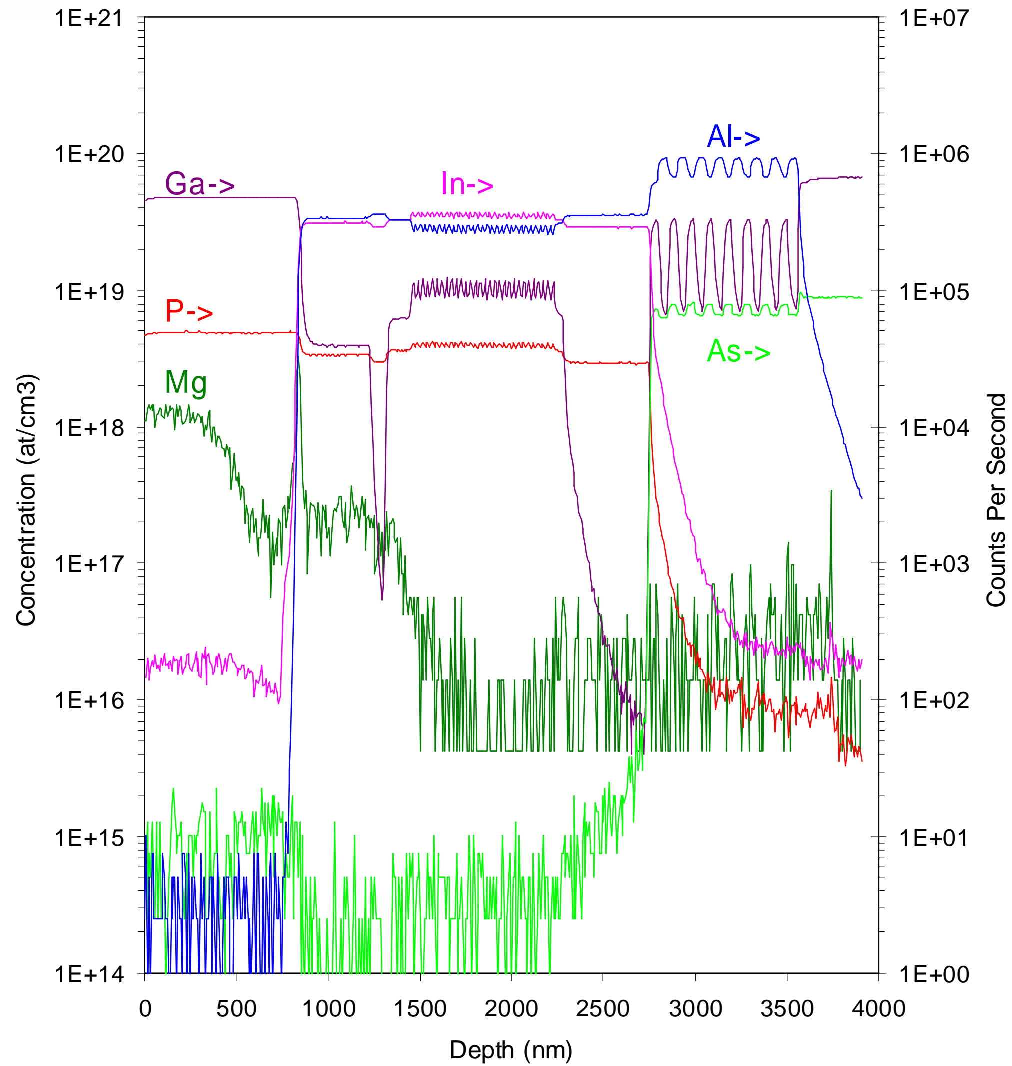
AlGaInP LED wafer after front side polishing



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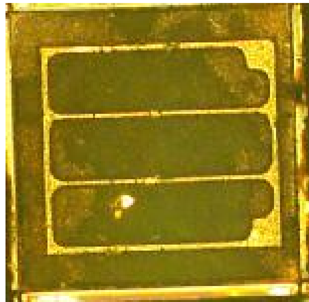
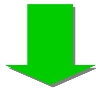
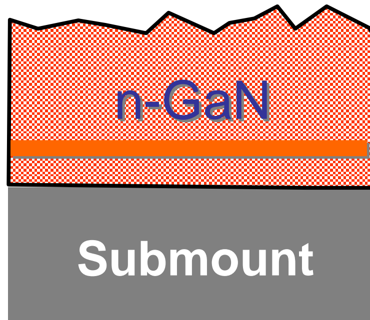
Reverse Engineering

Flip GaN: n-GaN up







Cannot resolve the MQW area

CONCENTRATION (atoms/cc)

1E+21
1E+20
1E+19
1E+18
1E+17
1E+16
1E+15

Secondary Ion Intensity (a.u.)

0 0.5 1 1.5 2 2.5 3 3.5 4 4.5 5

DEPTH (microns)

Mg
Si
Al->
Ga->
In->

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Back Side Polishing

Difference in the film thickness* within analytical area 30 µm

1mm, 0.6Å/1µm

9Å

0.5mm 0.2Å/µm

3Å

2Å

200µm
0.125Å/µm

*The film thickness from center to edge

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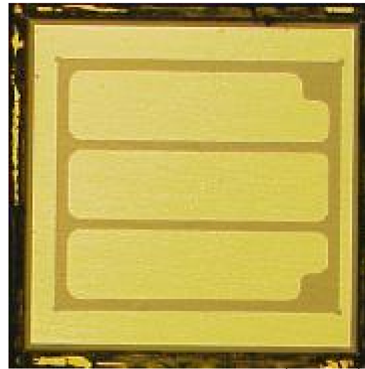
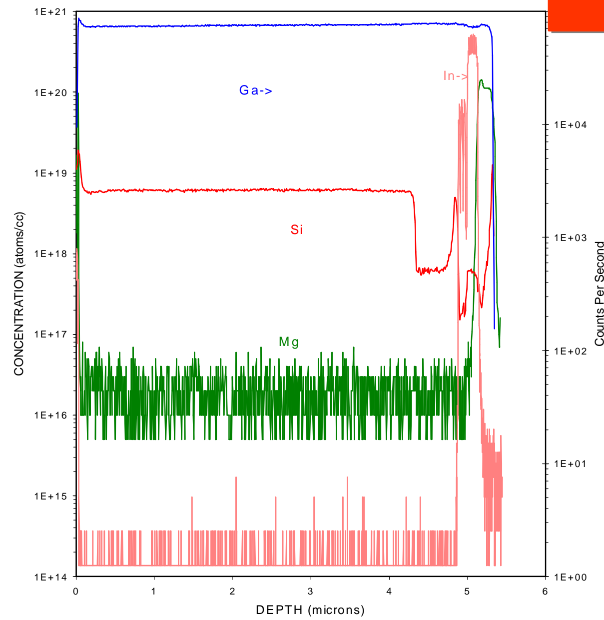
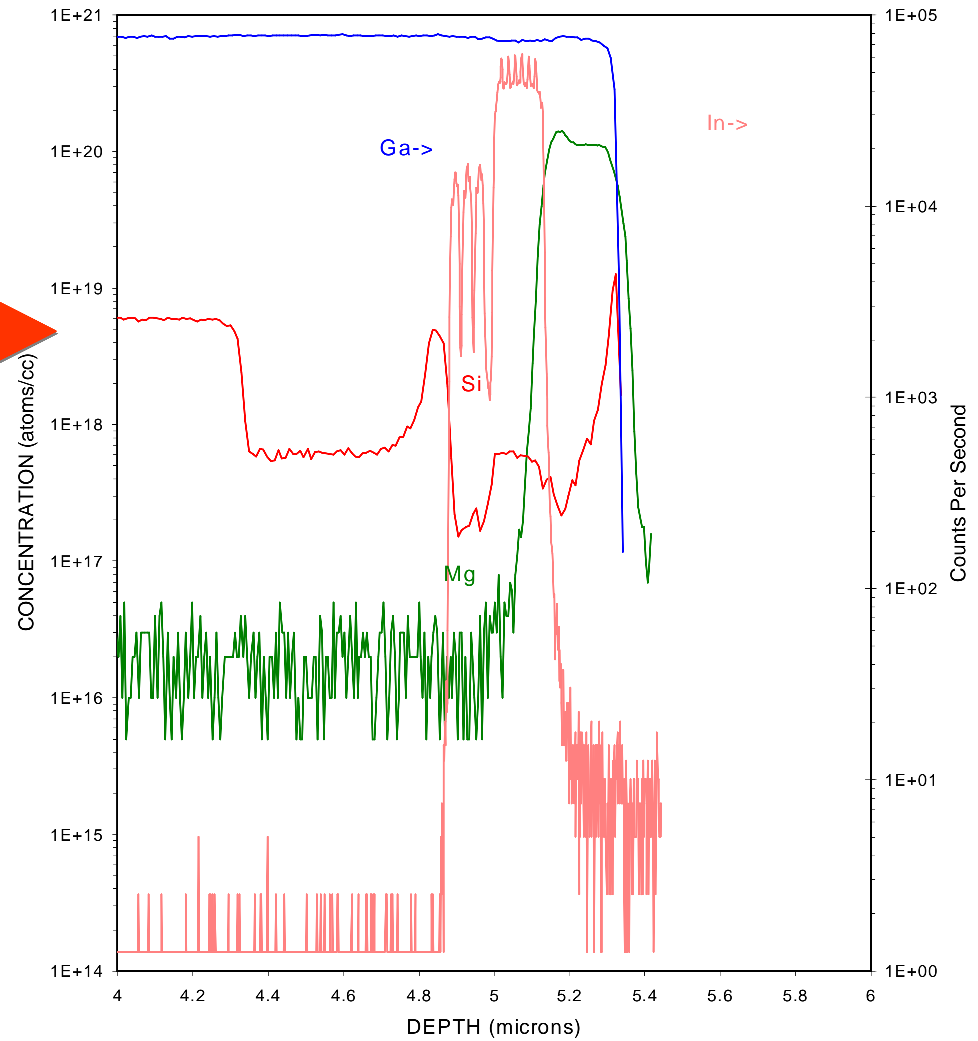
Sample Preparations for a GaN rough surface chip







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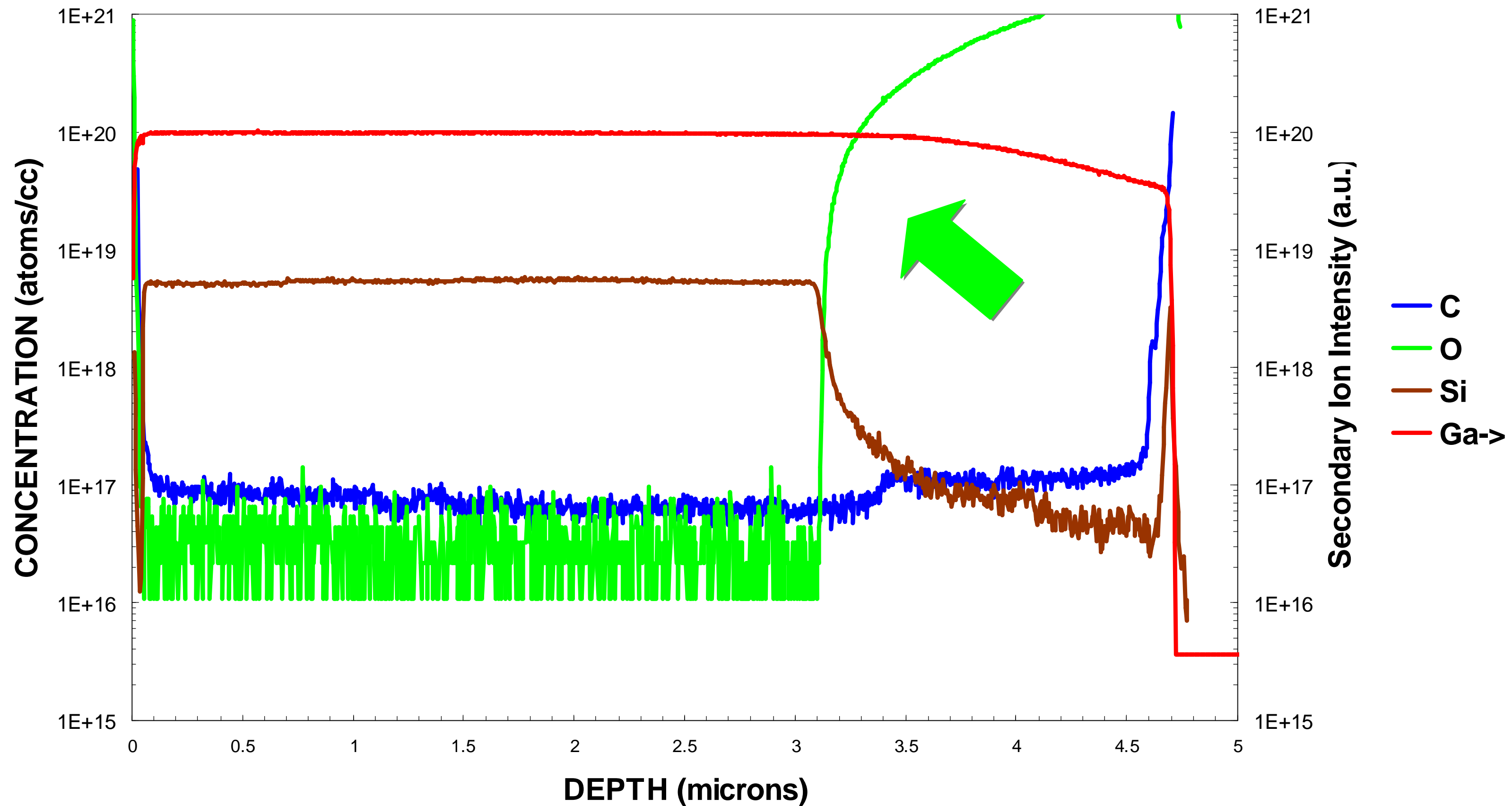
SIMS Artifact: Defects (pinholes) on SIMS profiling

Oxygen forward tail from the Al2O3 substrate



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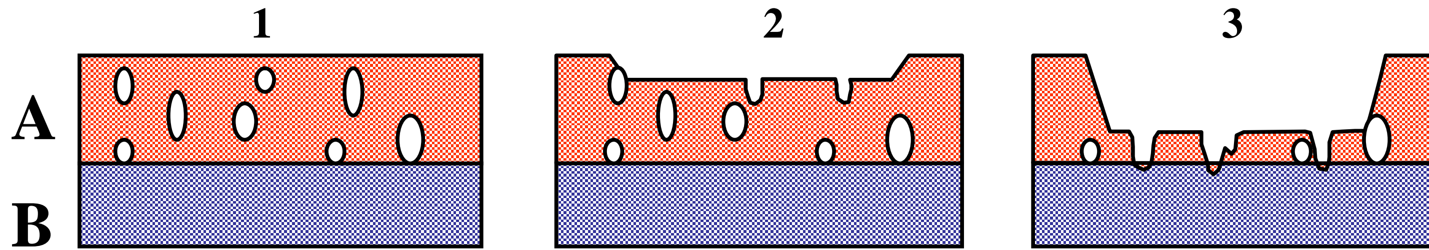
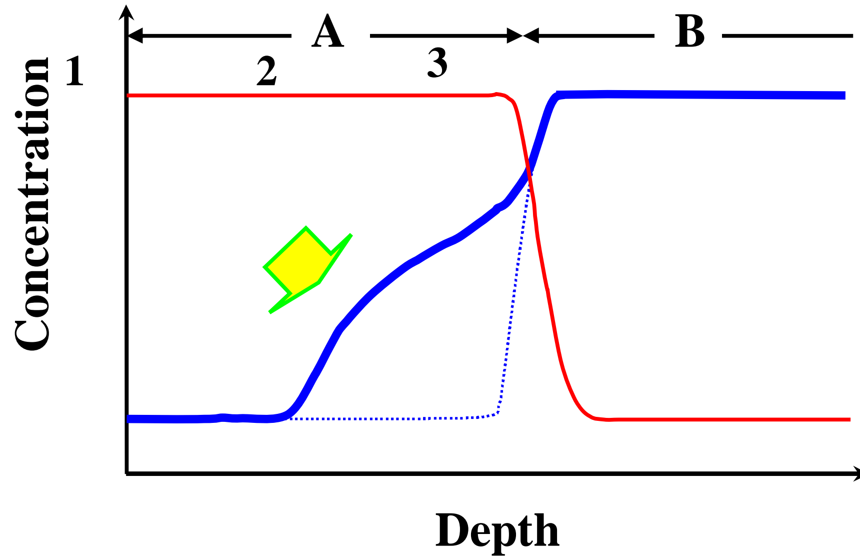
Defects on SIMS profiling

Pinholes: dislocations





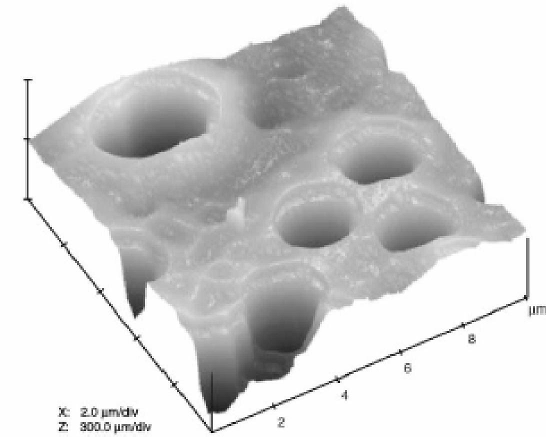


Fig. 4. AFM images of the surface after the SIMS sputtering was stopped in the middle of the GaN layer (#924). The image area is $10 \times 10 \mu m^2$.

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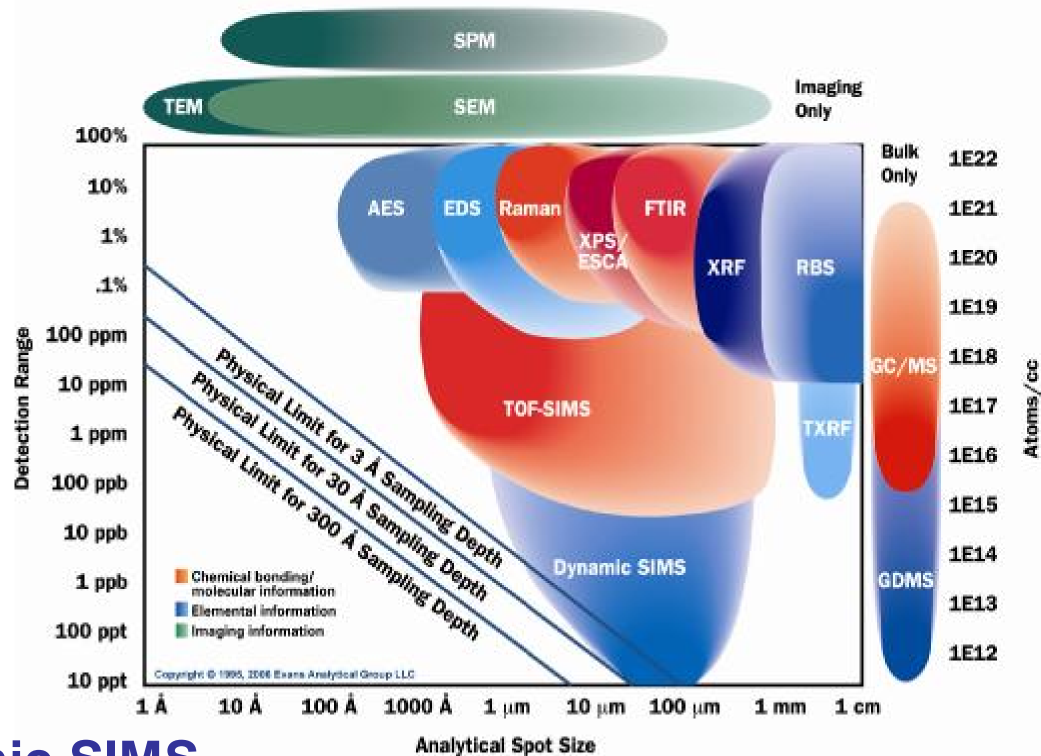
Technical Data Table



Dynamic SIMS

Quantitative	Yes	Destructive	Yes
Detection Limits	10^{12}-10^{16}at/cc	Lateral Resolution/ or Probe Size	≥10µm
Chemical Bonding	No	Depth Resolution	1-20nm

Comments: Detects all elements/isotopes, depth profiling technique, excellent detection limits

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Strengths and Weaknesses

- Strengths
  - Excellent detection sensitivity for dopants, impurities and known contaminants
  - Depth profiles of layered structures
  - Can detect all elements and isotopes, including H
  - Stoichiometry in some applications
- Weaknesses
  - Destructive
  - Element specific (poor survey technique)
    - Difficult to find unknown contaminants
  - No chemical information
  - Limited surface information

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Summary of Microanalysis in III-V Compound Semiconductors

- **Substrate**
  - Surface cleanness:
    metals: SEM-EDS, FE-AES, XPS, TOF-SIMS (trace)
    organic: μ-FTIR, XPS, TOF-SIMS (trace)
  - Stains and discoloration: SEM-EDS (first look);
    XPS; TOF-SIMS;
  - Particles: <10 μm: SEM-EDS, FE-AES, TOF-SIMS, Raman
    >10 μm: above, as well as μ-FTIR, μ-XPS
  - Surface oxide: XPS
  - Bulk Impurities: SIMS, GDMS

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Summary of Microanalysis in III-V Compound Semiconductors

- **Epitaxy and Implantation**
  - Compound composition : AES; SIMS; RBS; LEXES
  - n and p Doping Control: SIMS;
  - Impurities such as H, C and O metals: SIMS
  - Thickness: SEM; TEM (QWs); SIMS

  - Stains and discoloration: SEM-EDS (first look);
    XPS; TOF-SIMS;
  - Particles: <10 μm: SEM-EDS, FE-AES, TOF-SIMS, Raman
    >10 μm: above, as well as μ-FTIR, μ-XPS

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Summary of Microanalysis in III-V Compound Semiconductors

- **Metallization; Etching and Passivation**
  - Metal diffusion: AES; SIMS; RBS;XPS
  - Impurities such as C, O and metal (Cu for example): SIMS
  - Interface studies: AES; SIMS; RBS;XPS
  - Residues: SEM-EDS (first look); XPS; TOF-SIMS;
  - Particles: <10 μm: SEM-EDS, FE-AES, TOF-SIMS, Raman
    >10 μm: above, as well as μ-FTIR, μ-XPS

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