An Introduction to Auger Electron Spectroscopy

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Contents

- Background
- An Auger microprobe
- The Auger process
- Auger spectroscopy
- Auger mapping
- Depth profiling

The Auger process

- Named after Pierre Auger
  - 1925
- Actually discovered by Lise Meitner
  - 1922
- Emission of an intial electron leads to the emission of a characteristic (Auger) electron.

Auger Electron Spectroscopy (AES)

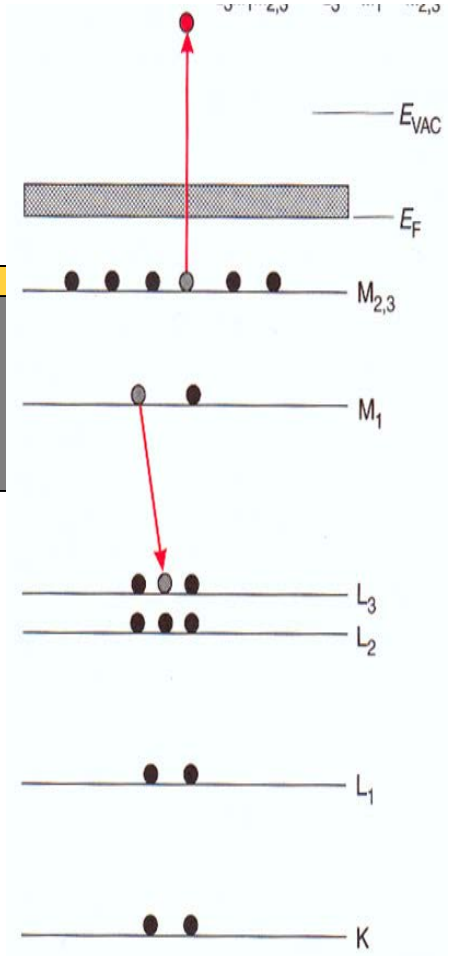
Signal
Electrons
(Spectrometer)

Exciting radiation
Electron beam (Scanning)

UHV vacuum

Analysis depth
(typically a few nm)

SAMPLE



Auger emission

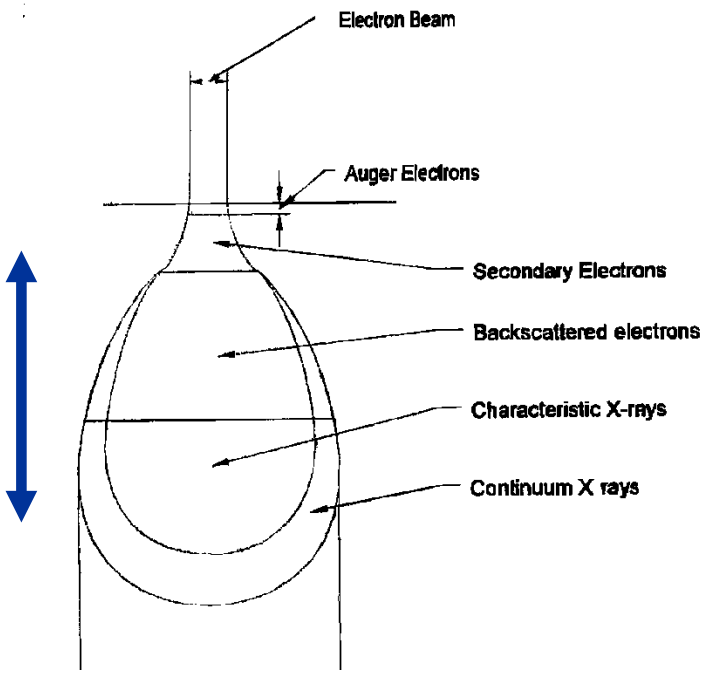
$E_{KL2,3\,L2,3} \approx E_K - E_{L2,3} - E_{L2,3}$

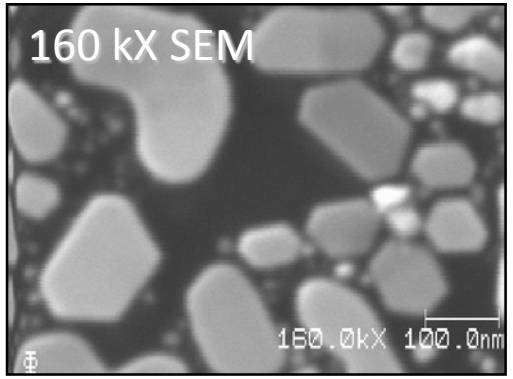
E = *energy of emitted electron*
E_K = *K-shell ionisation energy*
$E_{L2,3}$ = *L-shell electron energies*

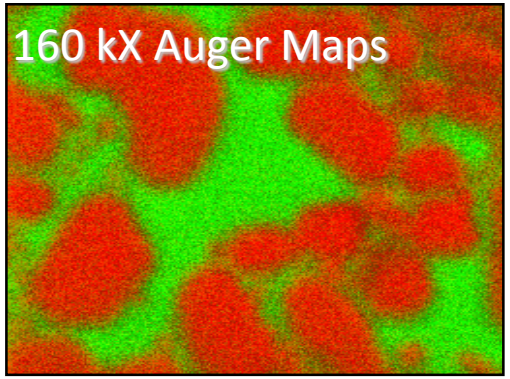
Auger - lateral resolution

4μm at 20kV

0,1μm 2kV







n.b. much better than EDS in bulk samples

Auger Electron Spectroscopy

- UHV Chamber
- FE-SEM quality electron column
- nm-scale depth resolution
- Depth profiling


The JEOL JAMP-9500F FE Auger Microprobe

Basic Specification

- 3nm SEI resolution
- 8nm probe diameter for Auger analysis
- Variable energy resolution from 0.05% to 0.6%
- Chemical state analysis in several 10nm areas
- Ion gun for sputter depth profiling
  Allows some charge neutralisation for analysis of insulating materials

Additional capabilities:

EDS system
- "Bulk" composition analysis.

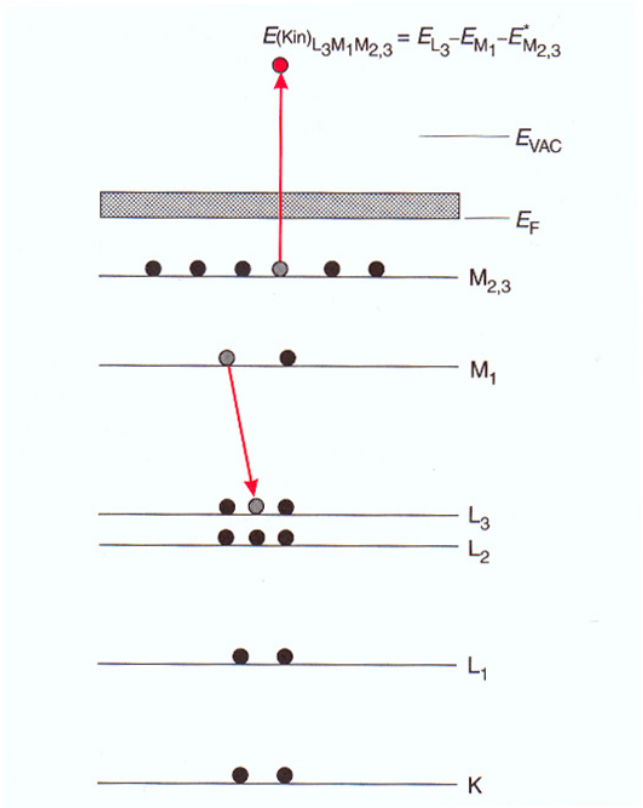
Backscattered electron detector
- Atomic number contrast.

Heating stage
- Diffusion experiments.

Liquid nitrogen fracture stage
- Grain boundary and interface studies.

Electron Beam Induced Current (EBIC)
- Recombination centre mapping in solar cells, etc.

Origin of Auger signal



Auger electron generation

LMM Auger electron emitted

Electron spectrometer

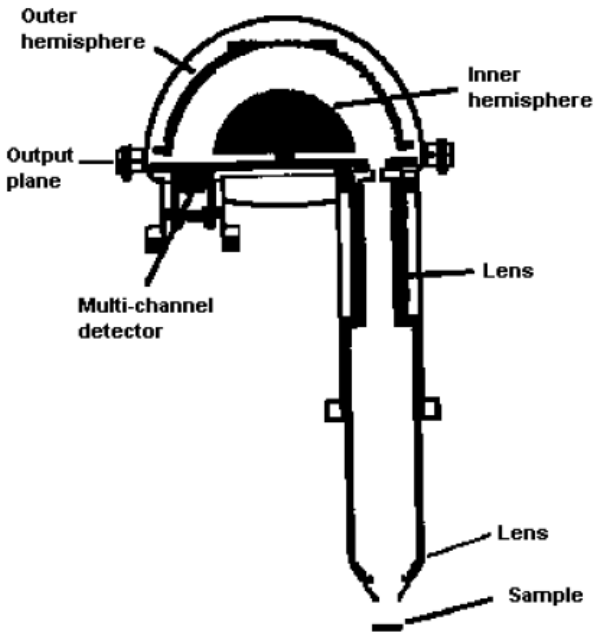


Figure 3.3: The hemispherical sector analyser, HSA [3].

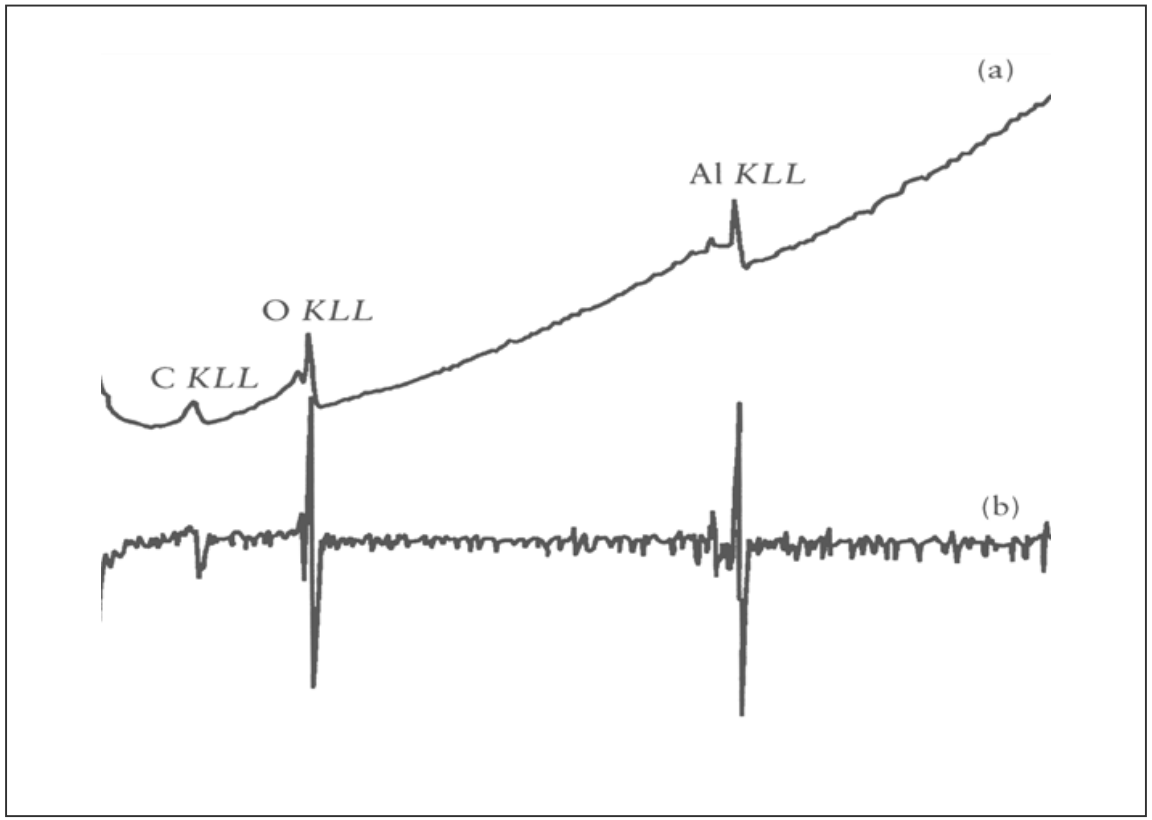
Hemispherical spectrometers often used.

Tend to be used in constant retard ration mode (CRR), as this supresses the strong low energy signal.

Constant analyser energy (CAE) also has uses.

Cylindrical mirror analyser used to be most common, still in use.

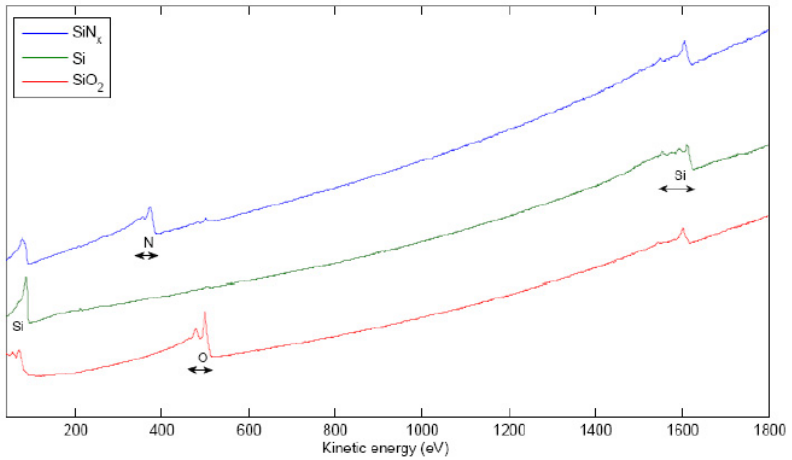
Auger spectrum - typical

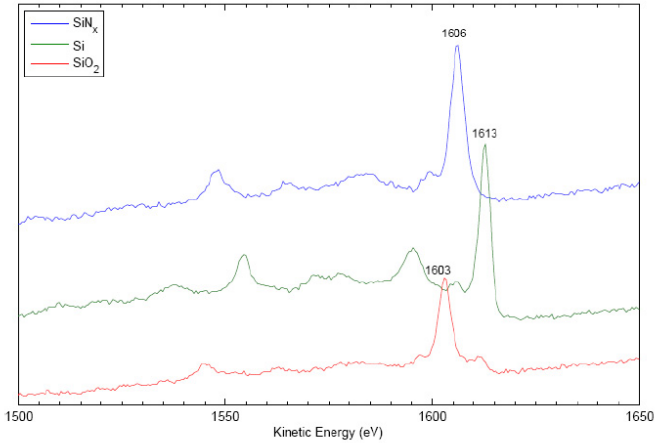


It is quite common to deal with the differential form of the spectrum.

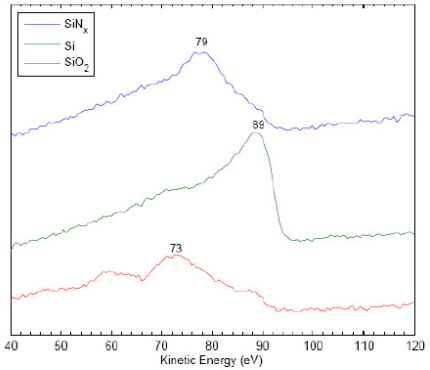
Chemical shift in Si compounds

Comparison of Si, SiN_x and SiO_2





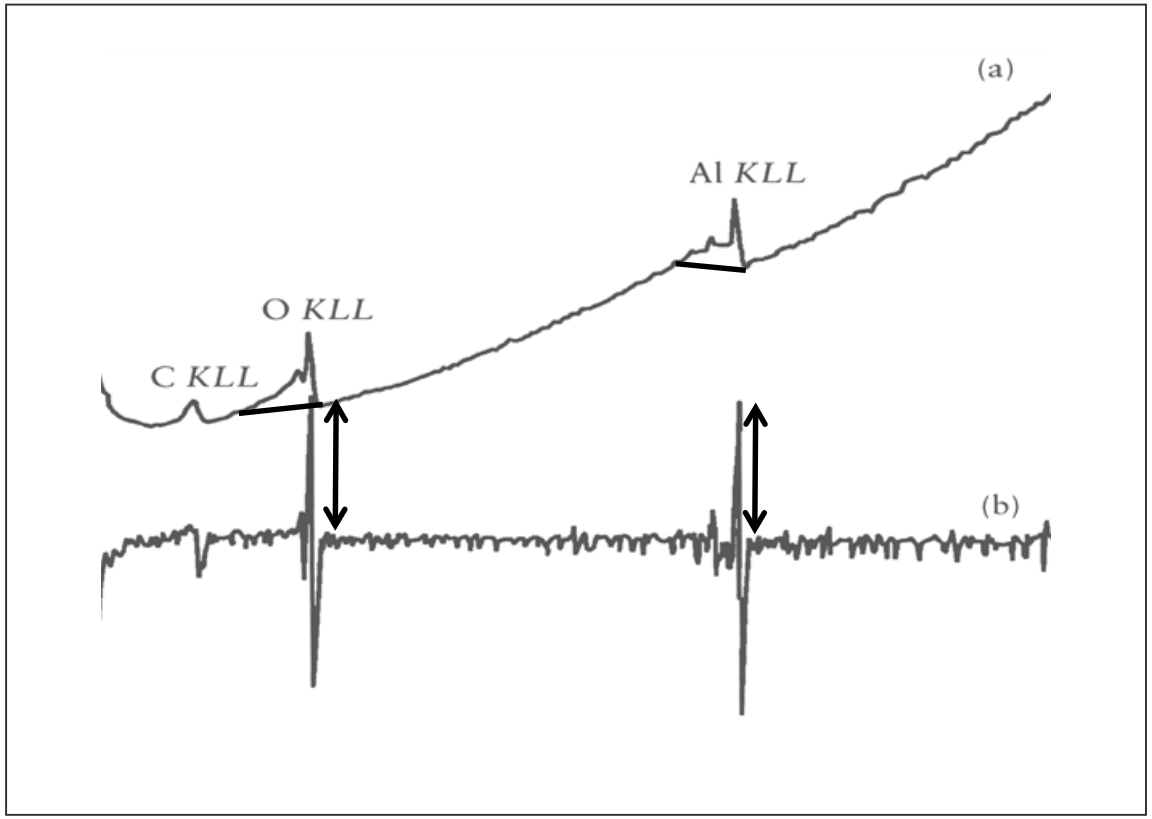
(b)



(c)

Figure 5.4: Auger energy spectra of Si, SiN_x and SiO_2. (a) shows the total spectra, whereas (b) gives the KLL-Auger peaks and (c) the LVV-peaks. Incident beam current $I_i \approx 6.5$ nA.

Auger spectrum - quantification



Can use peak areas or peak-to-background ratios

Auger spectrum - quantification

Concentration of element N_A is:

$$N_A = I_A/(I_A + F_{AB}I_B+F_{AC}I_C+....)$$

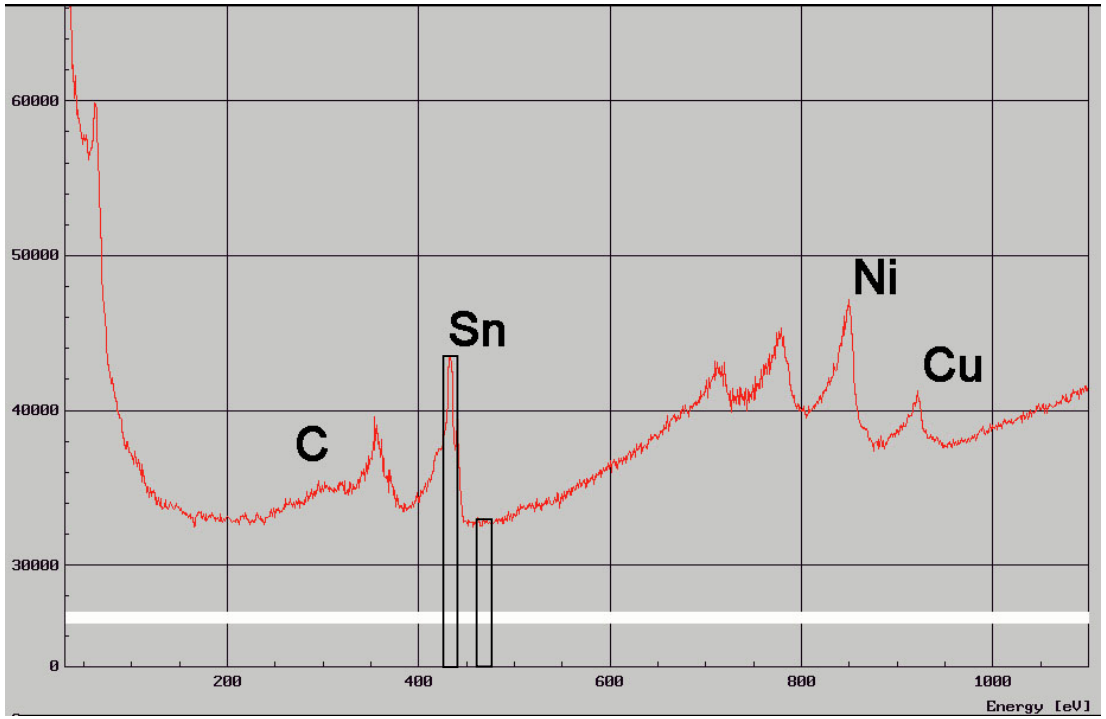
I is the element intensity

F is a sensitivity factor determined from binary standards such that:

$$F_{AB} = (I_A/N_A/I_B/N_B)$$

This is highly simplified but can work reasonable well.

Auger mapping



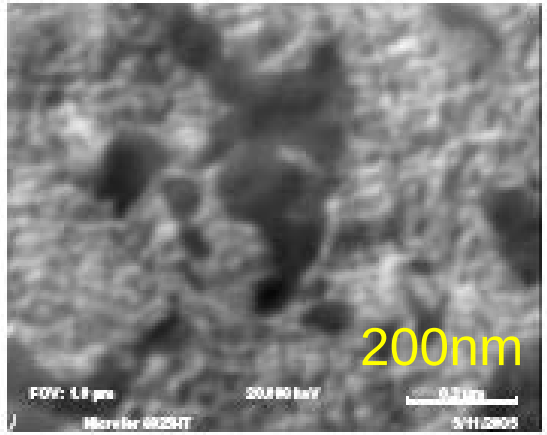
Pixel-by-pixel, typically calculate;

(peak-background)/background

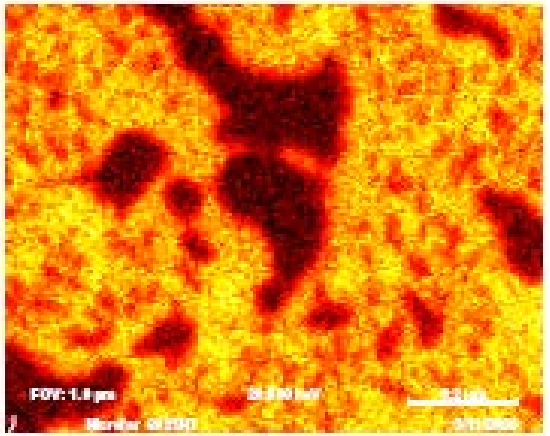
Combined SEM/Auger analysis

Carburised alloy

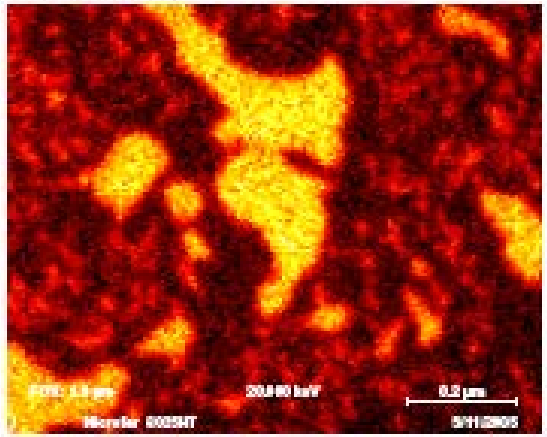
Nicrofer 6025HT



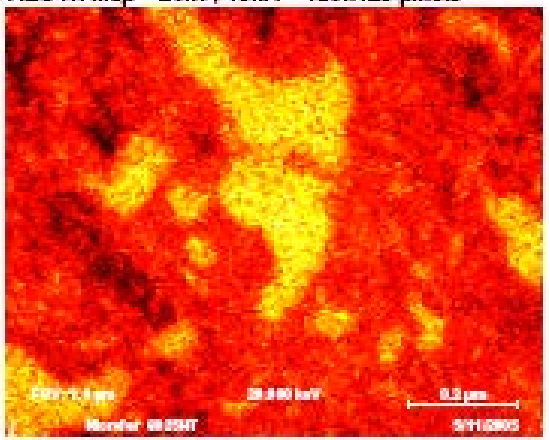
SEI 20kV; 10nA



AES Ni Map 20kV; 10nA 128x128 pixels

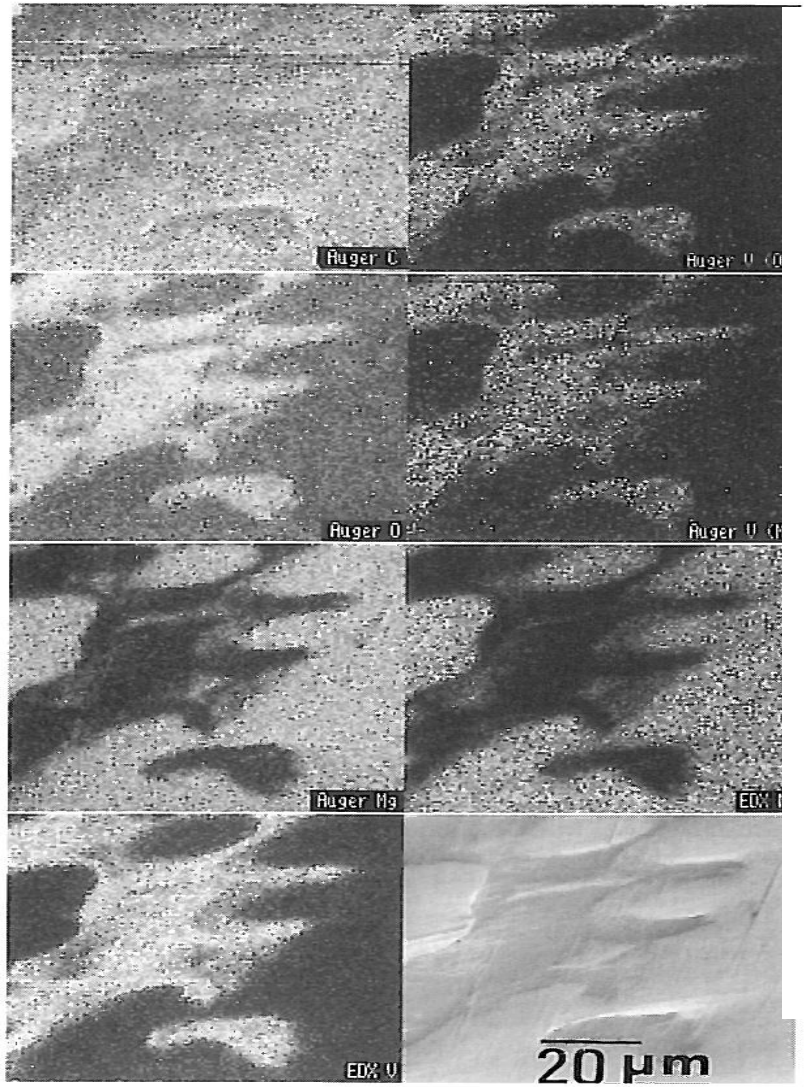


AES Cr Map 20kV; 10nA 128x128 pixels

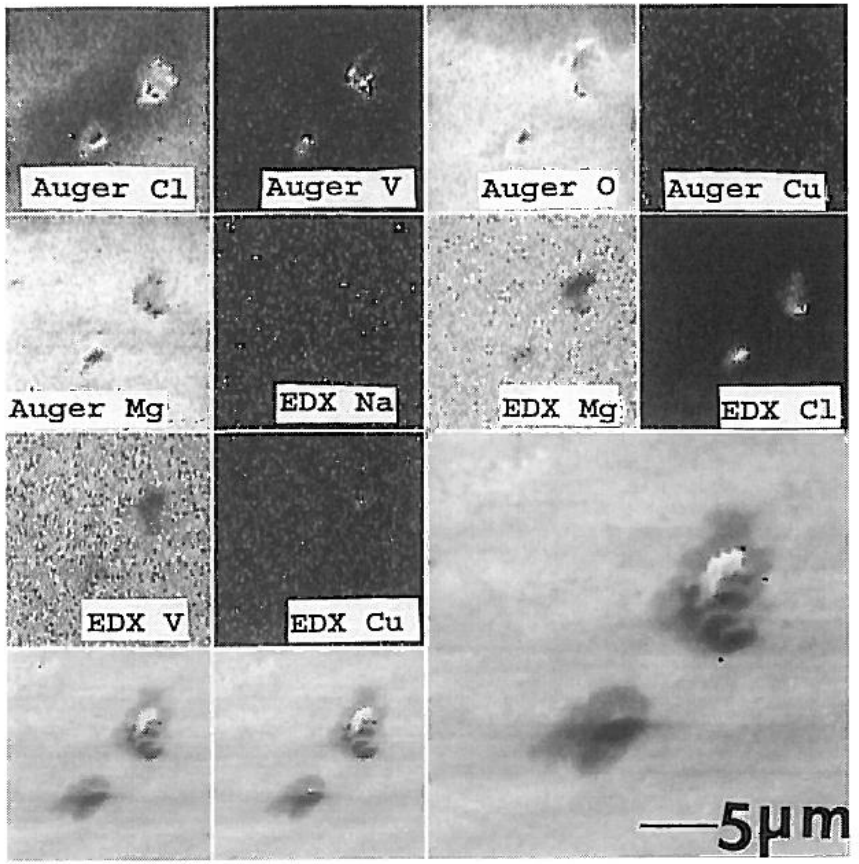


AES C Map 20kV; 10nA 128x128 pixels

Combined Auger EDS mapping

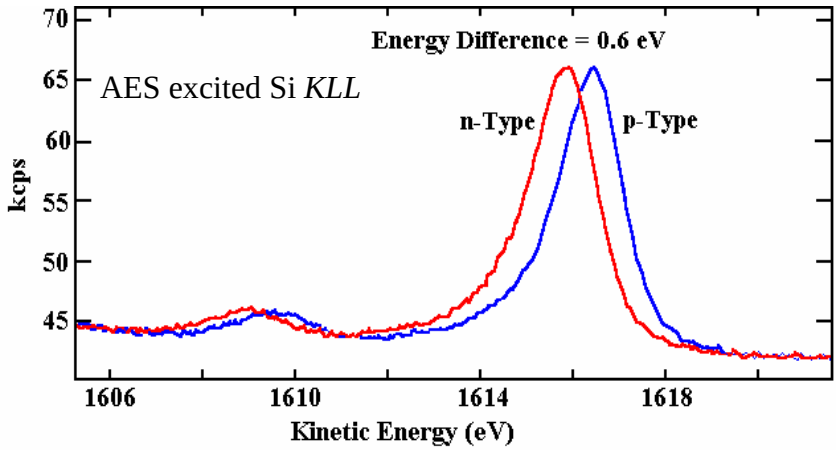


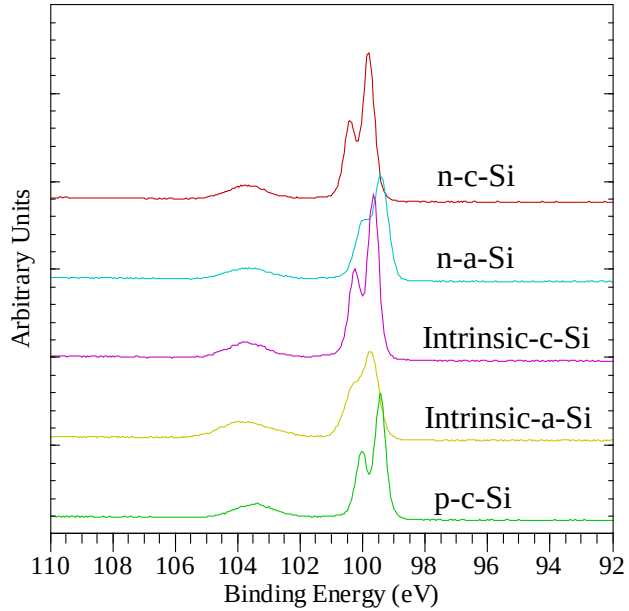
12 Auger and EDX elemental maps of oxidised surface of Mg–27V alloy: oxidation occurred during grinding/polishing of surface

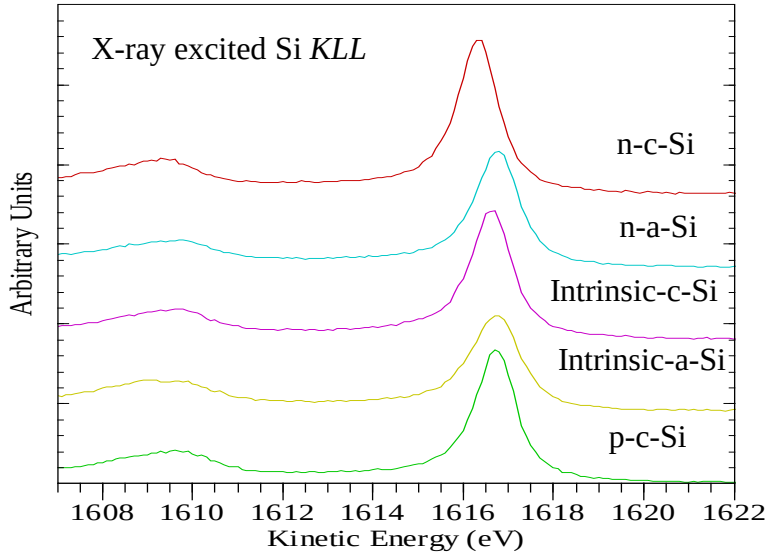


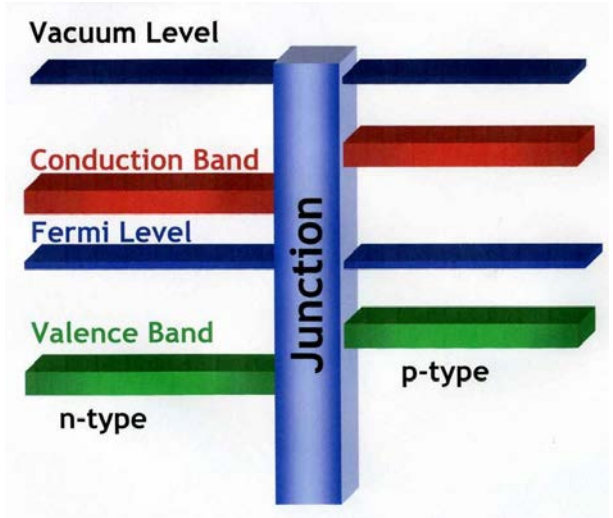
13 Auger and EDX elemental maps of corroded surface of Mg–27V alloy after 1 min immersion in 3 wt-%NaCl solution

Auger and XPS spectra of Si: p and n Type

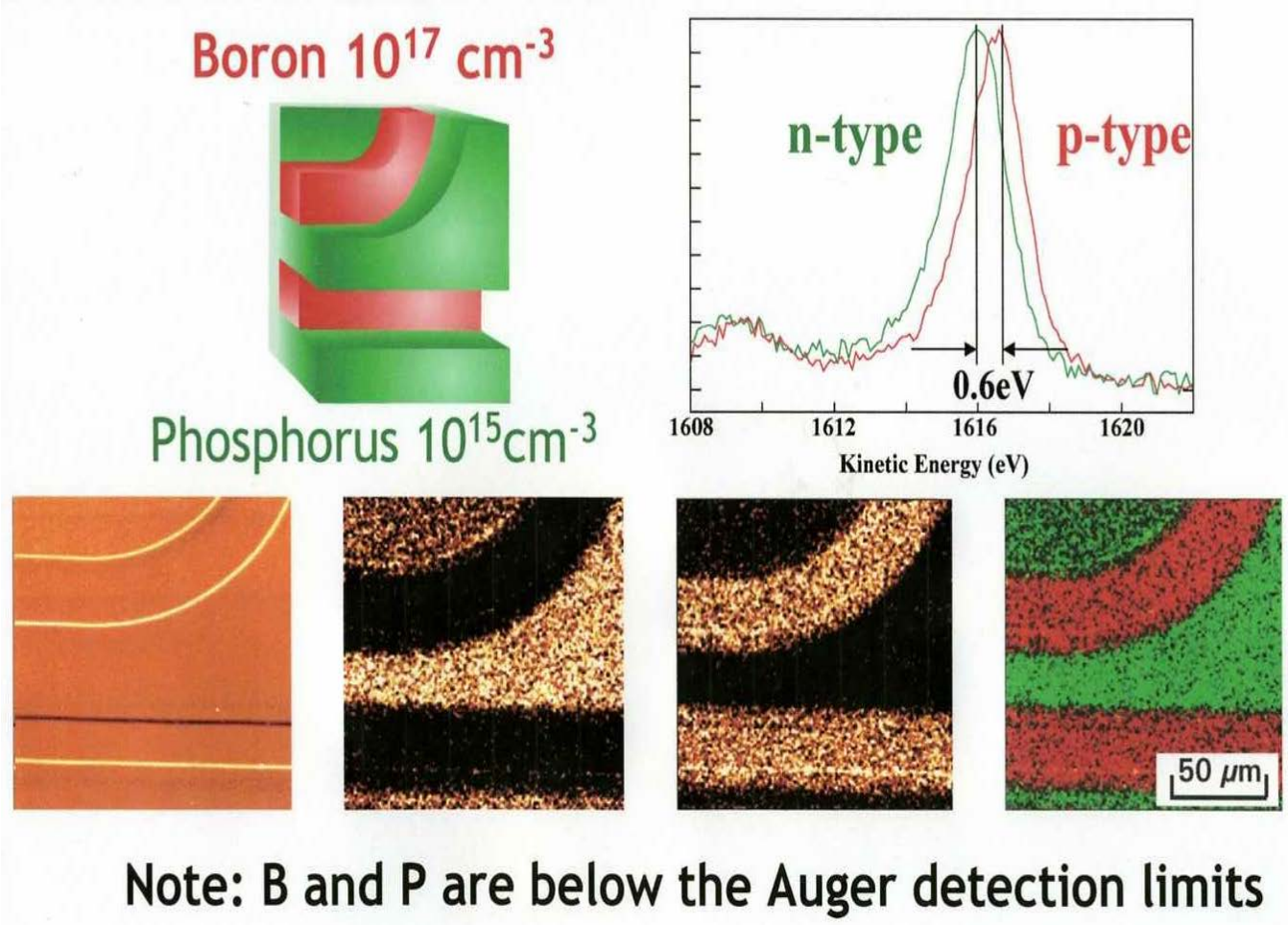






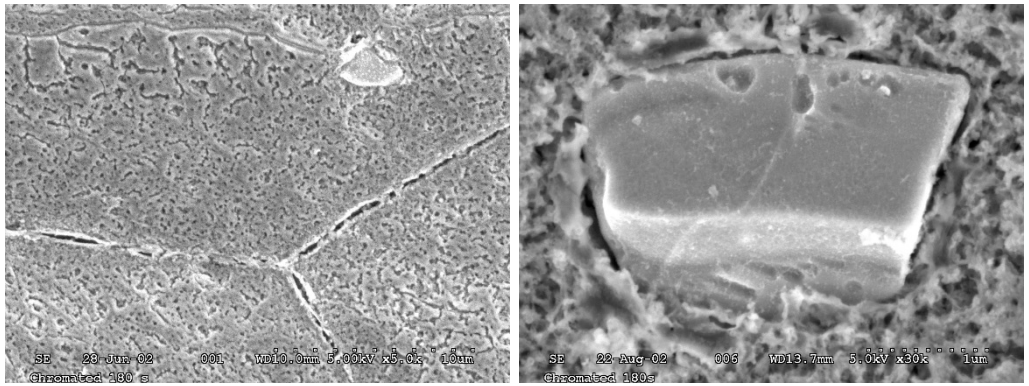


Auger mapping of Si p and n type

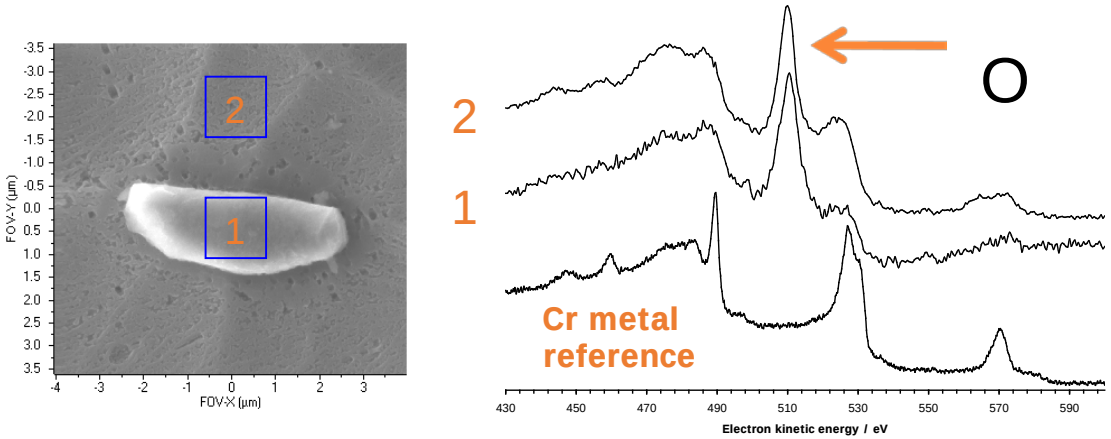


AES – nm lateral and depth resolution

Chromated aluminium alloy surface



- Chromating pretreatment
  - corrosion protection
  - before coating, painting, bonding, etc
- Does the chromate ”passivate” intermetallic particles?

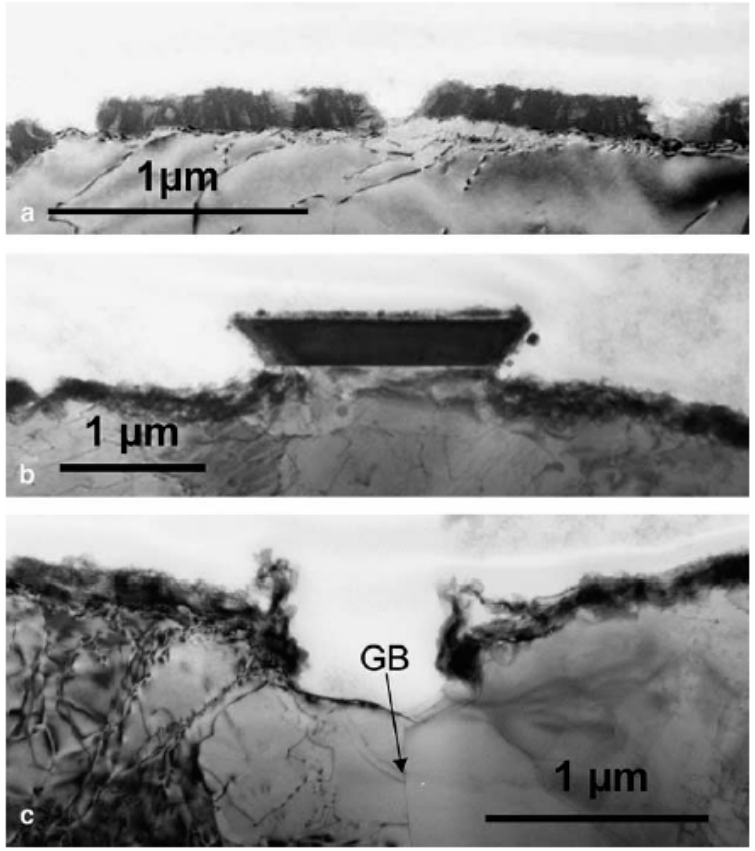


- Auger spectra show that the intermetallic particles are covered by a thin layer of Cr-oxide that is invisible in the SEM images.

O. Lunder [a,b,*], J.C. Walmsley [a], P. Mack [c], K. Nisancioglu [b]

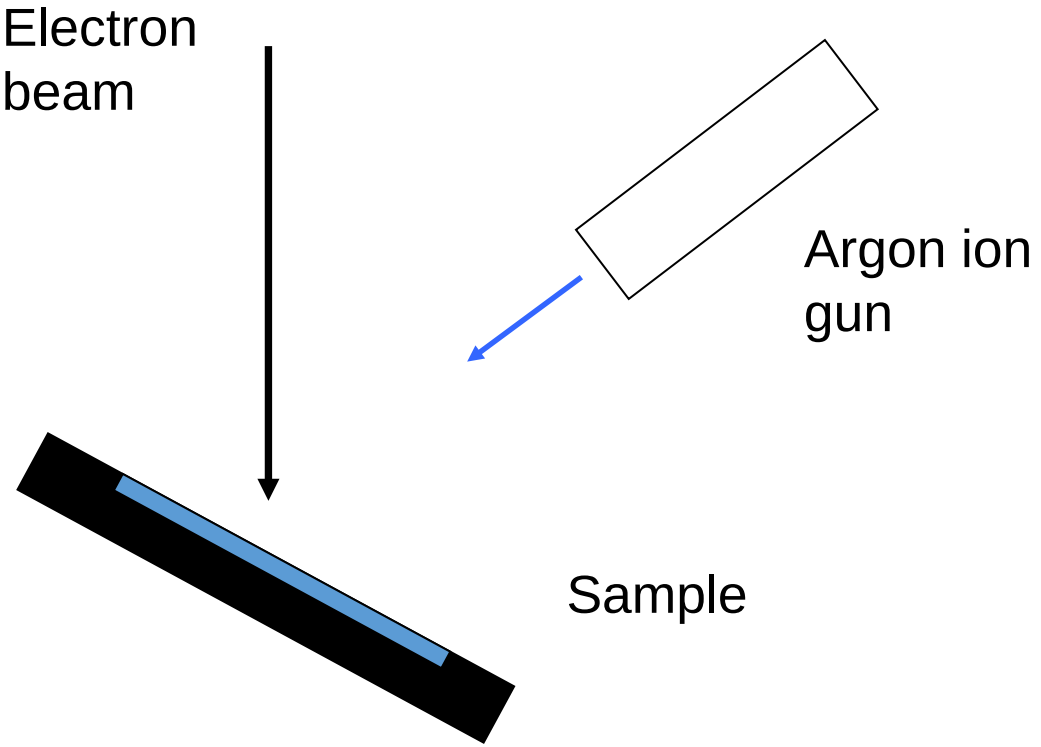
Corrosion Science 47 (2005) 1604–1624

Combined SEM/TEM/Auger analysis



TEM analysis

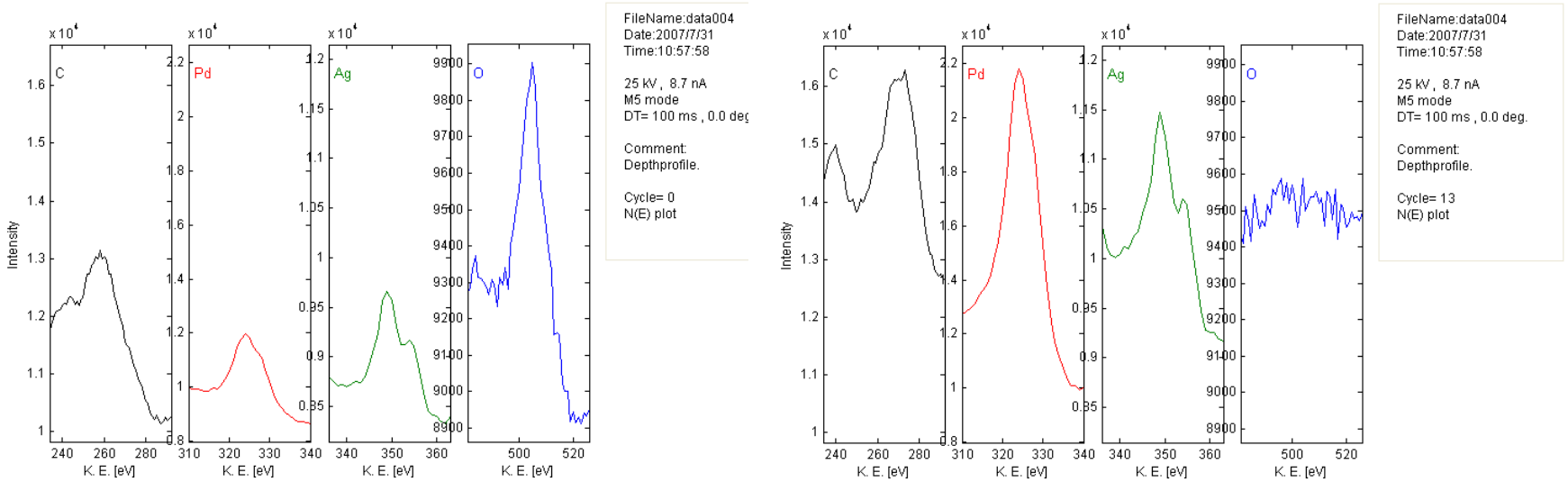
Depth profiling - schematic



Process is fully automated, sample can be rotated (Zalar rotation).

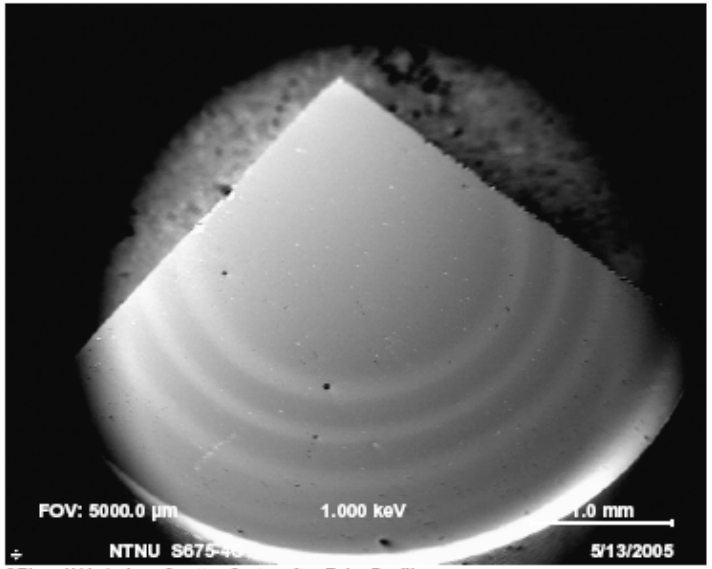
N.B. Can also perform angle-resolved analysis.

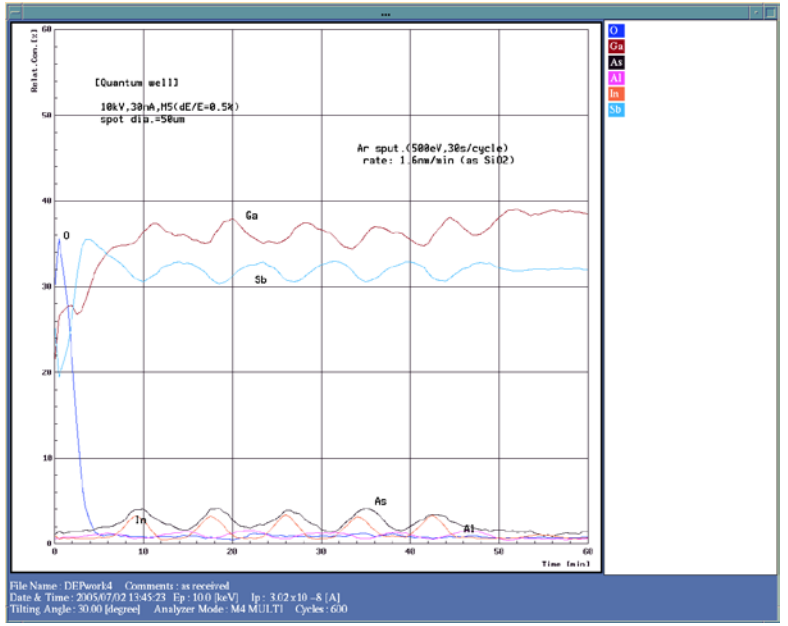
Depth profiling – raw data



Surface and after sputtering – as grown Pd/Ag membrane

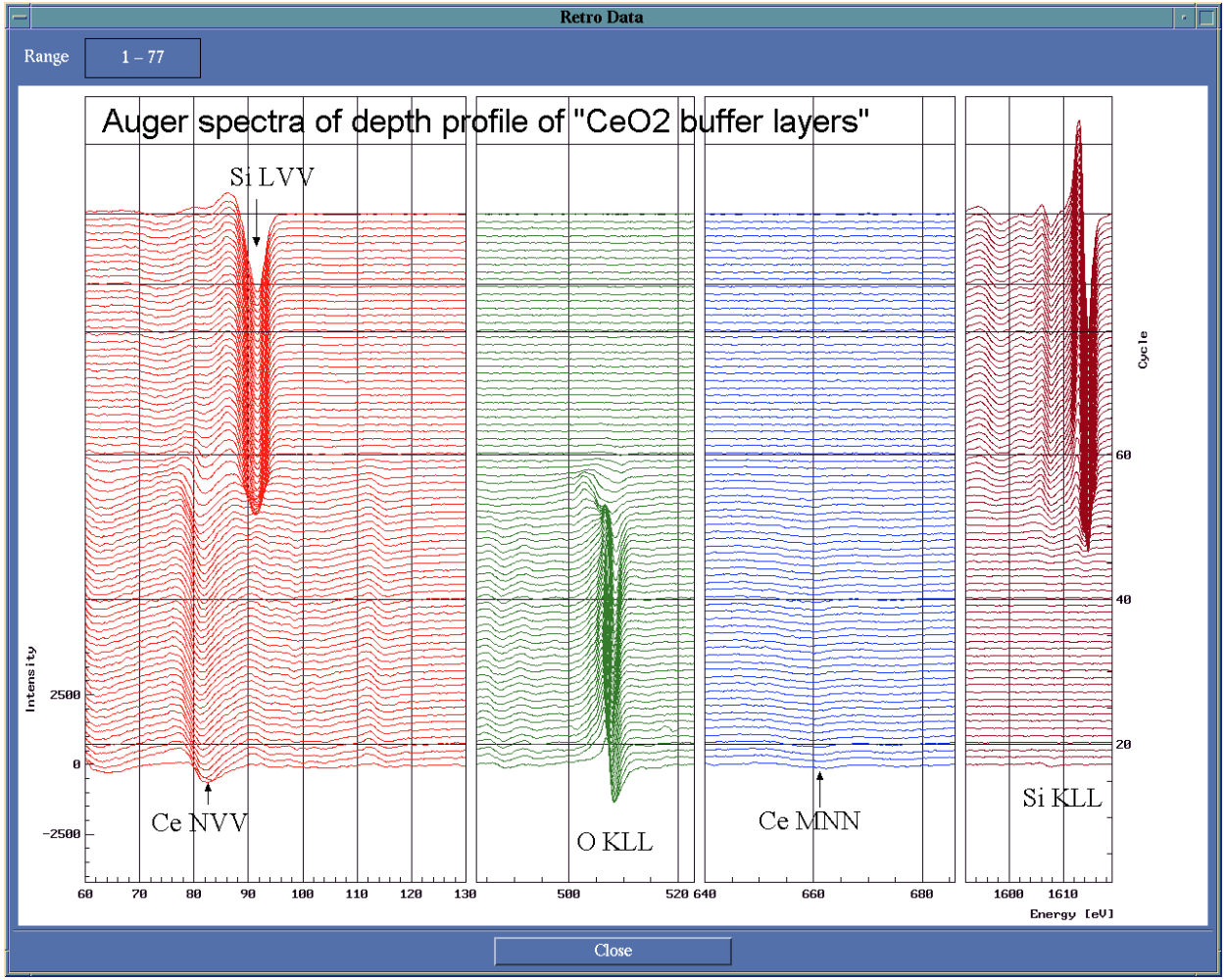
Depth profiling - example



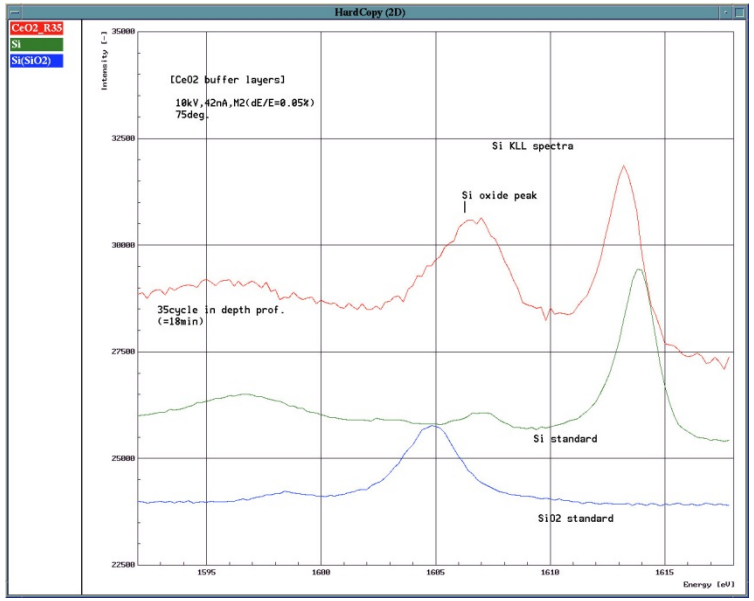


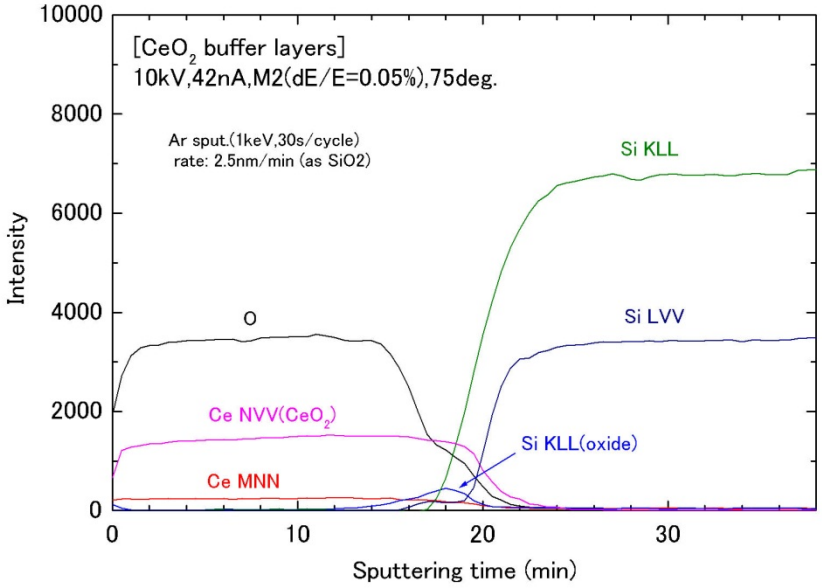
Multiple quantum well structures

Depth profiling CeO_2 buffer layer



Depth profiling CeO_2 buffer layer





Potential Auger Applications

- Conducting / semiconducting materials
- Corrosion studies
- Coatings
- Depth profiling
- Catalysis
- Carbon/other material fibres
- Metallurgy
  - Grain boundaries in steels
- Can be extended to low conductivity materials
  - Use low energy ion gun to flood surface
  - Probably not polymers

Summary

- Combine SEM and electron spectroscopy
  - High lateral and depth resolution
- Chemical state information
  - Need to explore the possibilities
- Depth profiling
  - ”Bread and butter” applicaion.
- Segregation and interface studies
  - Surfaces and internal interfaces
- Complementart with other techniques
  - XPS/TEM……

References

- *Scanning Auger Electron Microscopy;*

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- *Practical Surface Analysis by Auger and Photoelectron Spectroscopy;*

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