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HIDEN ANALYTICAL

FIB - SIMS

Focussed Ion Beam
Secondary Ion Mass Spectrometry

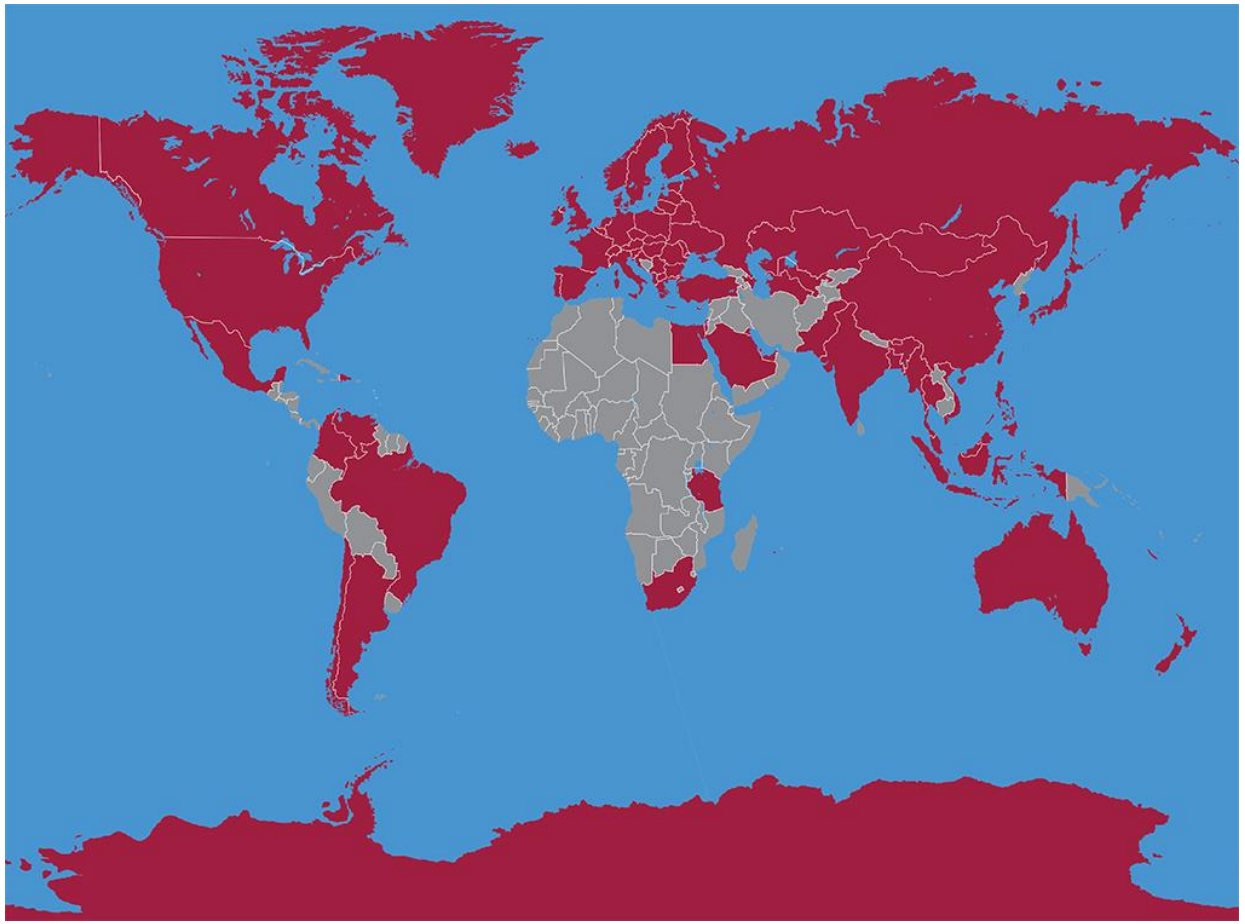
Outline

- Introduction to Hiden Analytical
- Introduction to SIMS
- FIB-SIMS - Introduction and key features
- FIB-SIMS - Applications data
- Hiden SIMS Software

Hiden Analytical Ltd.

- Privately owned manufacturing company. Company headquarters based in 23,000 sq ft factory in Warrington, England
- 80 employees work out of headquarters. 33 years in business
- US subsidiary, Hiden Analytical Inc., main office Michigan
- Office in Beijing, China
- Lifetime e-mail and telephone support with our systems

Hiden Analytical Instruments are used in many countries worldwide

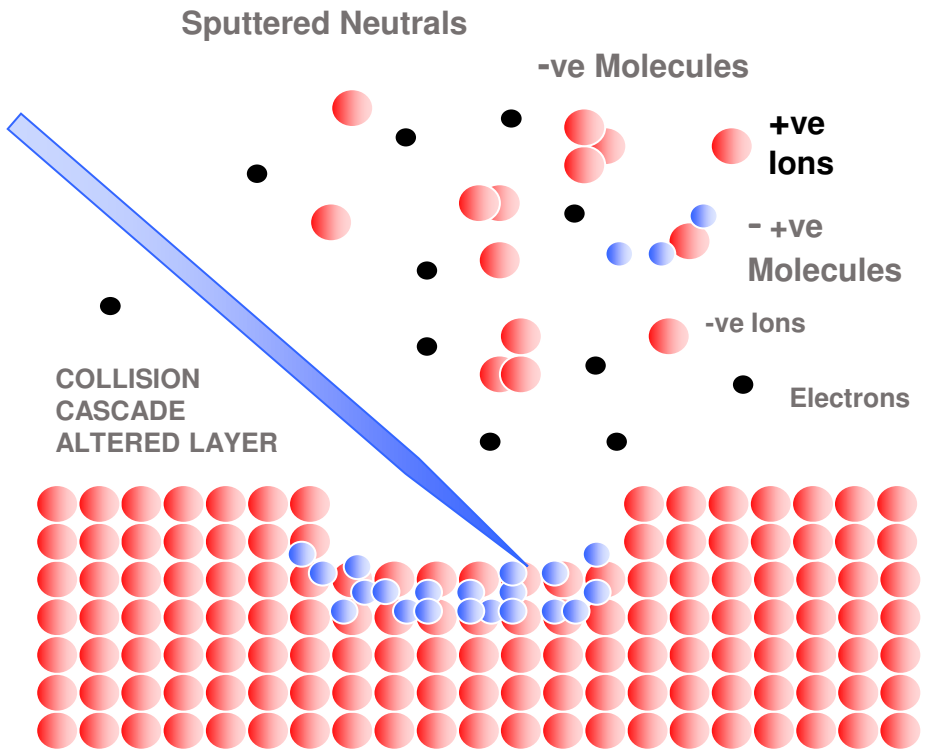


Introduction to SIMS

- SIMS is the acronym for Secondary Ion Mass Spectrometry
- SIMS is a surface sensitive technique, analysing the upper monolayers of the sample
- A primary beam of energetic ions, typically in the range of 500eV up to 30keV is used to sputter or erode the surface of the material under analysis
- In this sputtering process a number of ions are present in the sputtered material

Introduction to SIMS

Sputter Erosion of the Specimen



Introduction to SIMS

- Sputtered ions from milling are collected and analysed by mass spectrometry
- The sputtered ions are directed into the Hiden SIMS detector by electric fields
- They then pass through an energy analyser* (45 degree Electrostatic Sector Analyser or ESA) and then through a mass analyser (Quadrupole Mass Spectrometer or QMS)
- The Mass and Energy resolved ions then individually detected using a secondary electron multiplier

* Can assist in resolving interference masses

Hiden EQS

On-axis sampling, energy resolving mass spectrometer

- High efficiency screened extraction system – optimum ion collection
- 45° electrostatic sector energy filter – permits high dynamic count range (> 6 orders magnitude)

EQS-FIB 1000 SIMS on Zeiss FIB

Hiden SIMS on a Zeiss Auriga 60.

FIB-SIMS – Key Features

A powerful surface analytical technique, especially for high sensitivity nano-scale materials analysis

Analysis of trace elements down to ppm levels (thin films, semiconductors, solar cells)

Isotope detection (e.g. ^{69}Ga, ^{71}Ga)

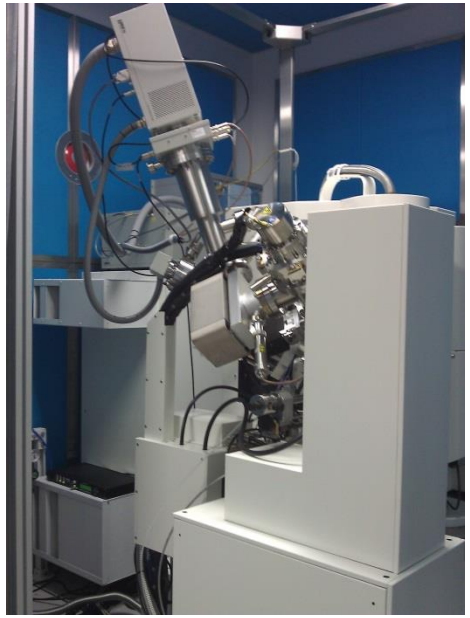
Elemental mapping and depth profiling

Detection of atomic and molecular ions (e.g. Zr^+ and ZrO^+)

50 nm lateral analytical resolution possible (better than conventional SIMS)

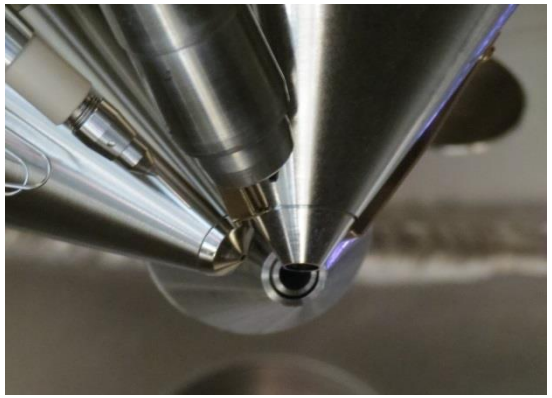
FIB-SIMS Availability

The EQS-FIB SIMS is available for the following Carl Zeiss microscopes:

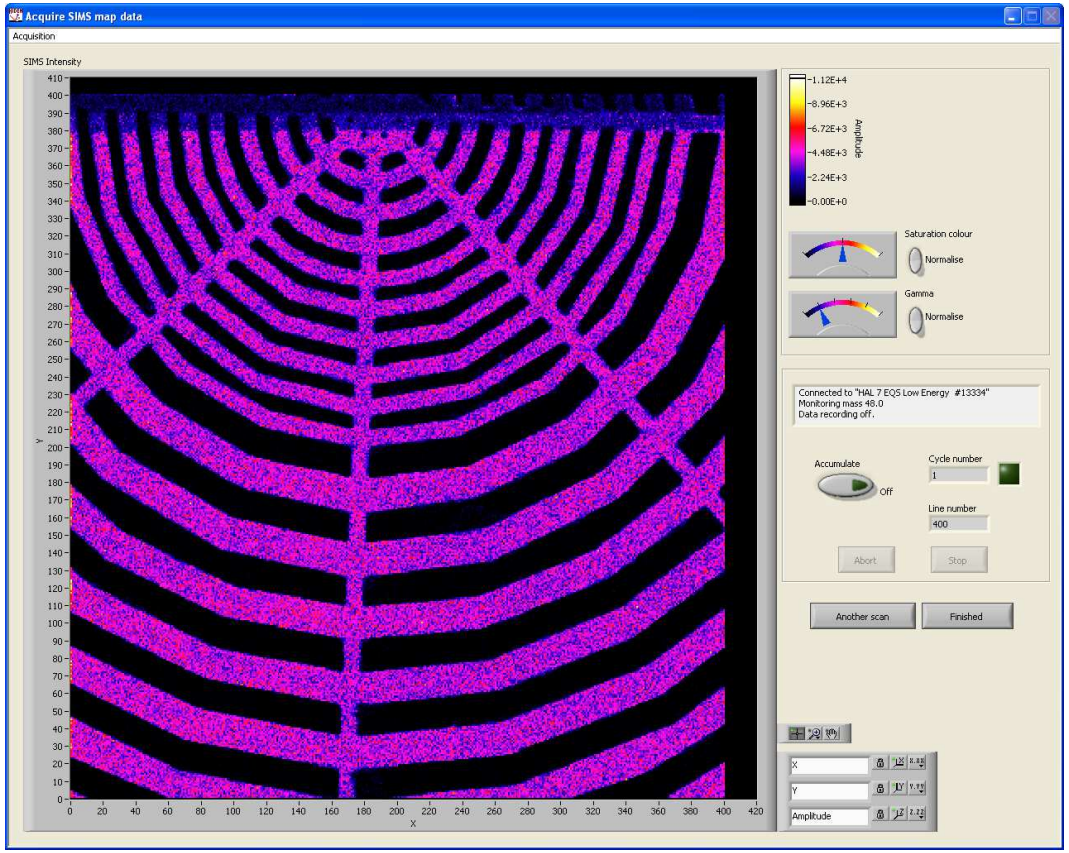


Zeiss Nvision X-beam FIB with EQS

- AURIGA series
- NEON series
- 15xx series
- XB 340, XB 540



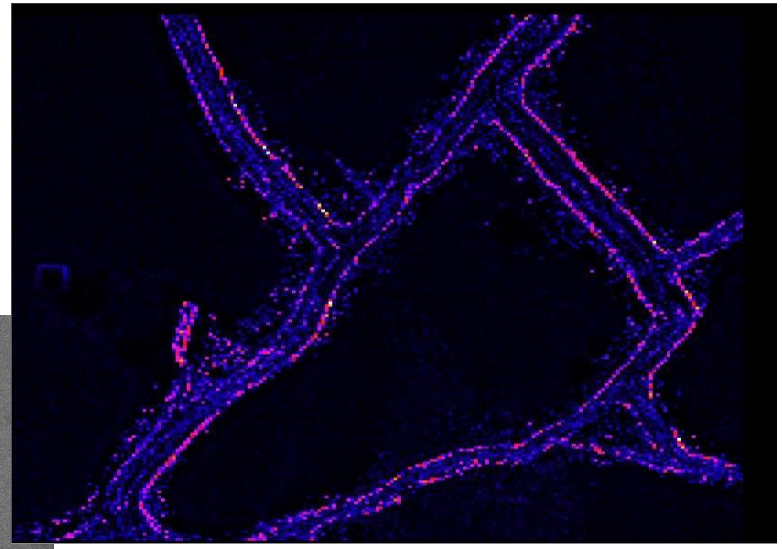
FIB-SIMS Elemental Imaging

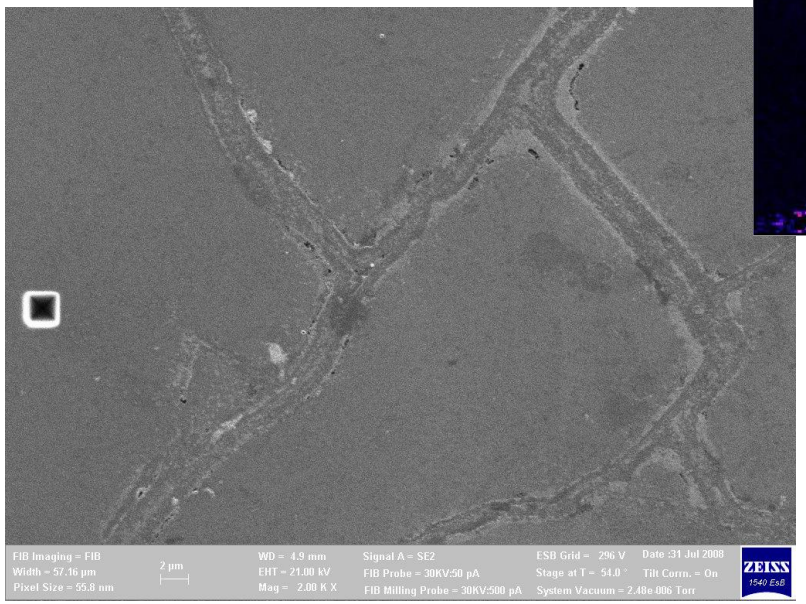


TiN spider web pattern on Si with 1µm minimum feature size

TiN^+ with m/z = 62 was detected

FIB-SIMS Imaging



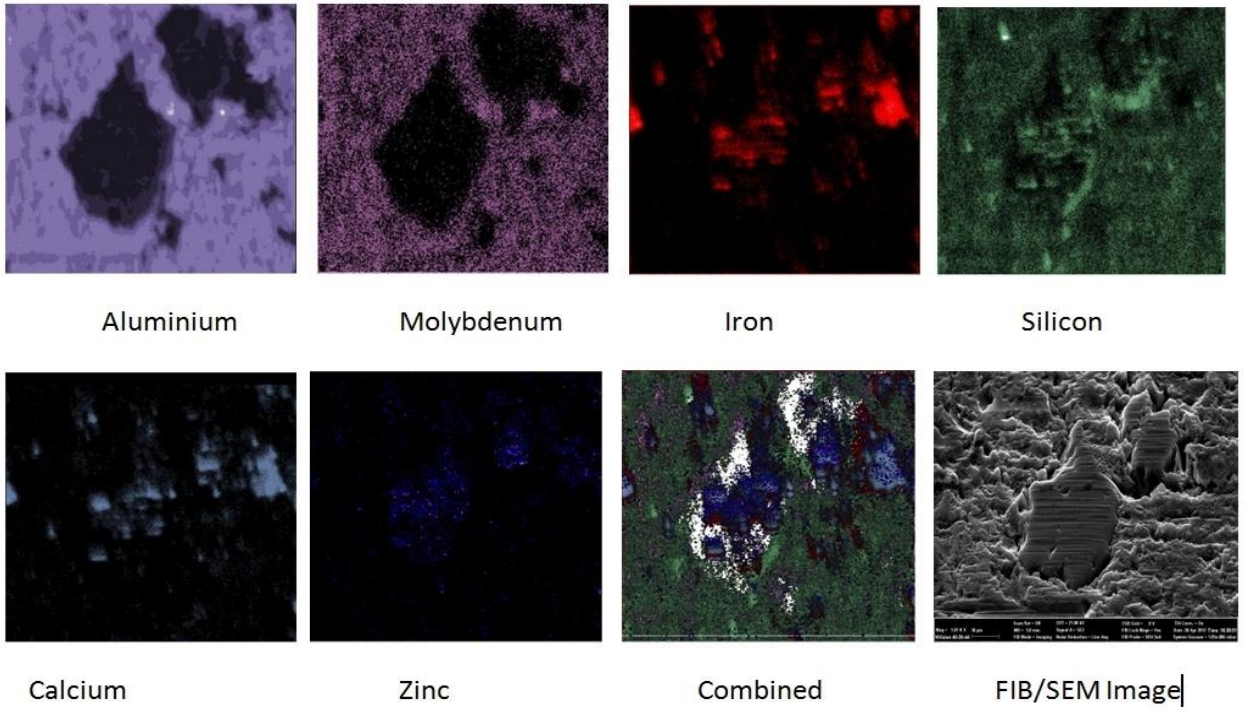


$^{27}Al^{+}$ image showing concentration at grain boundary
LaSrCuFe oxide

Sample: Richard Chater, Imperial College London, UK
Instrument: Zeiss Neon, Hiden EQS

FIB-SIMS Elemental Imaging

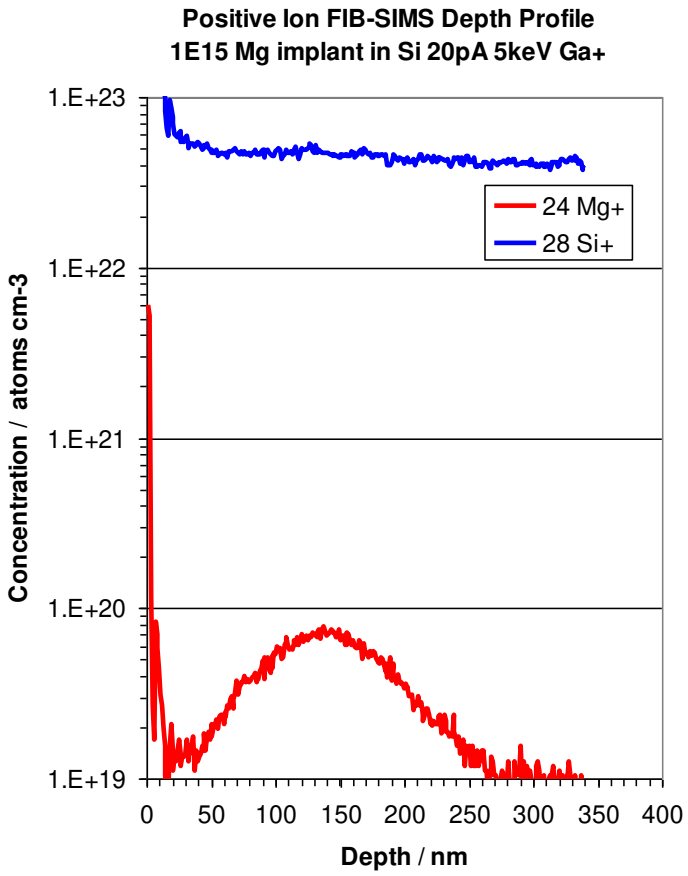
Multi Element Map showing the wear characteristics of an Alusil Engine liner[1].



Images are courtesy of Dr John Walker, National Centre for Advanced Tribology, University of Southampton, UK. Instrument is ZEISS NVision/Hiden EQS

1. Walker, J. C., T. J. Kamps, and R. J. K. Wood. "The influence of start-stop transient velocity on the friction and wear behaviour of a hyper-eutectic Al-Si automotive alloy." Wear, 2012, In Press.

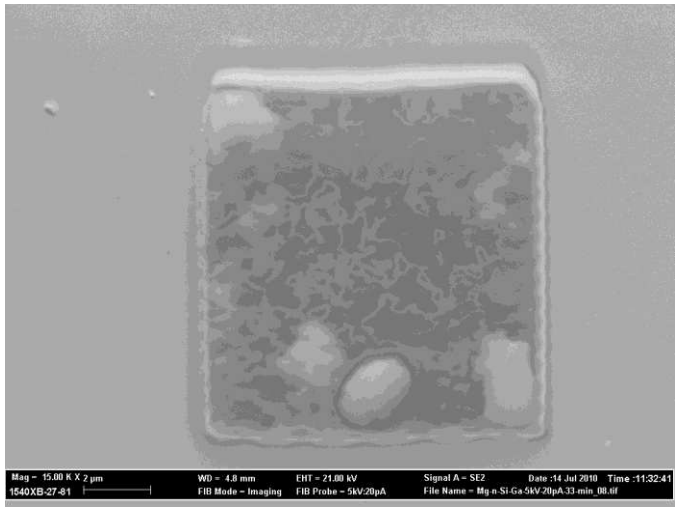
FIB-SIMS Depth Profiling



Depth Profile of $^{24}Mg^{+}$ dopant in bulk Si

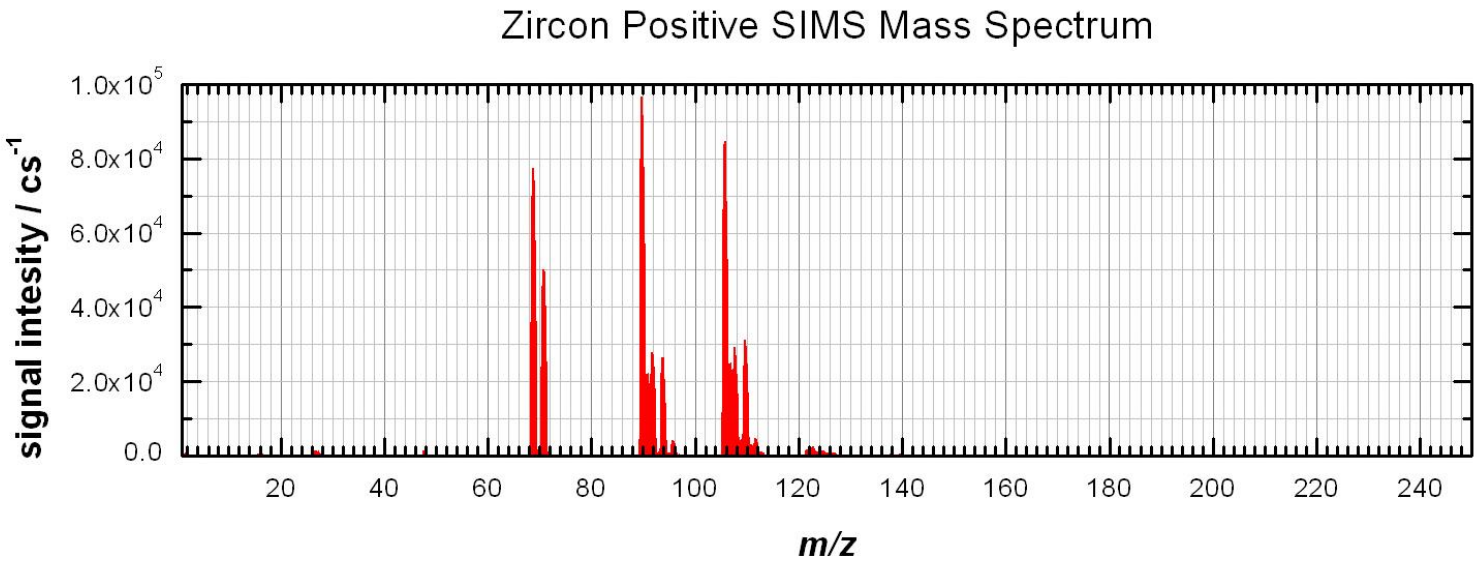
Peak of $^{24}Mg^{+}$ implant is $7x10^{19}$ atoms cm^{-3}

~ 0.15% atomic



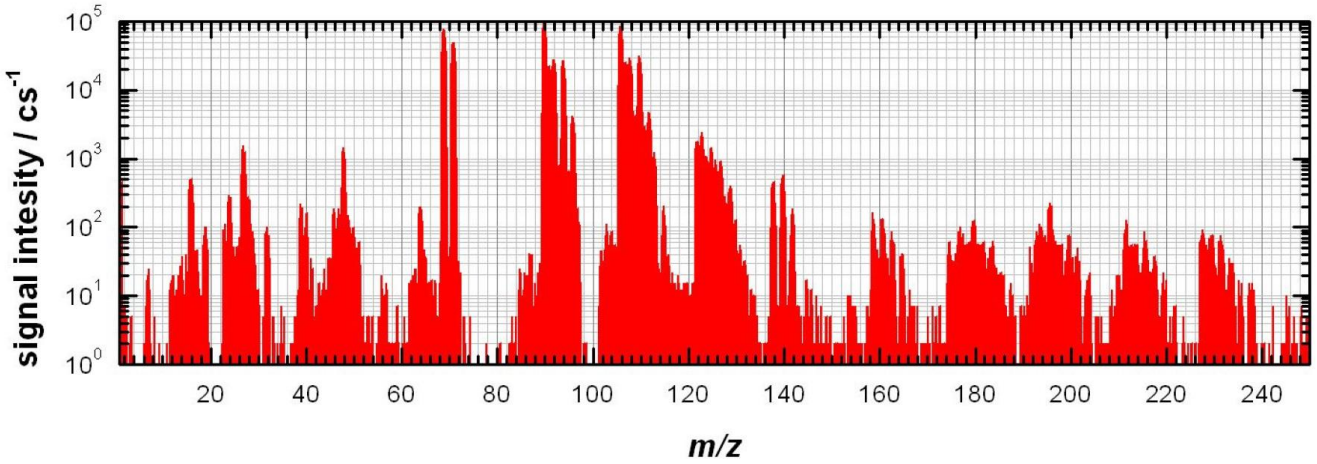
FIB-SIMS Mass Spectrum - Zircon

Analysis of individual zircon grains in a possible meteoritic rock sample
- can FIB SIMS determine if it is actually likely to be extra-terrestrial in origin?



The spectrum above (plotted on a linear intensity scale) shows three significant groups. Ga from the ion probe is visible at *m/z*= 69 and 71 with Zr and ZrO isotopes appearing from 90 and 106 respectively. A small signal caused by ZrO_2 is also discernable from 122.

Zircon Mass Spectrum – High Dynamic Range

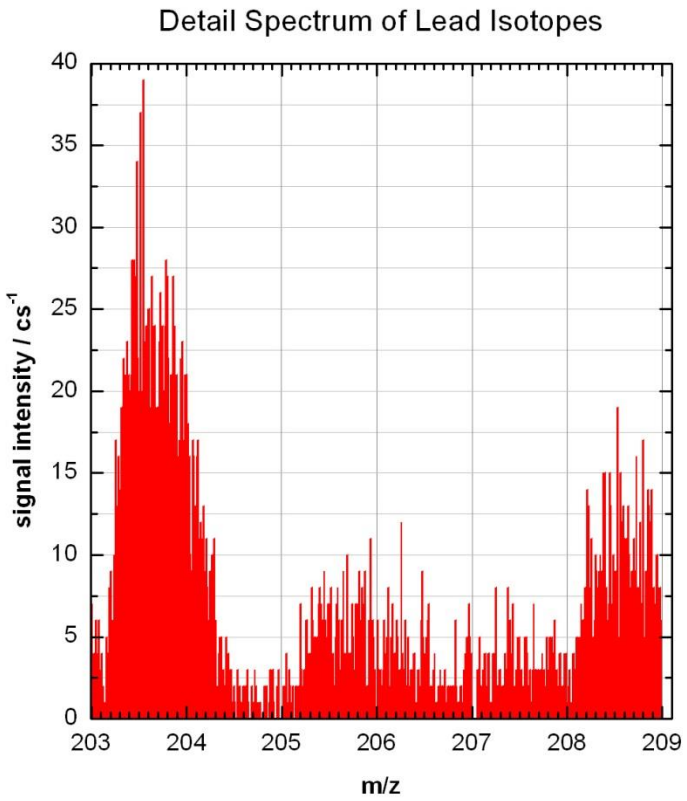


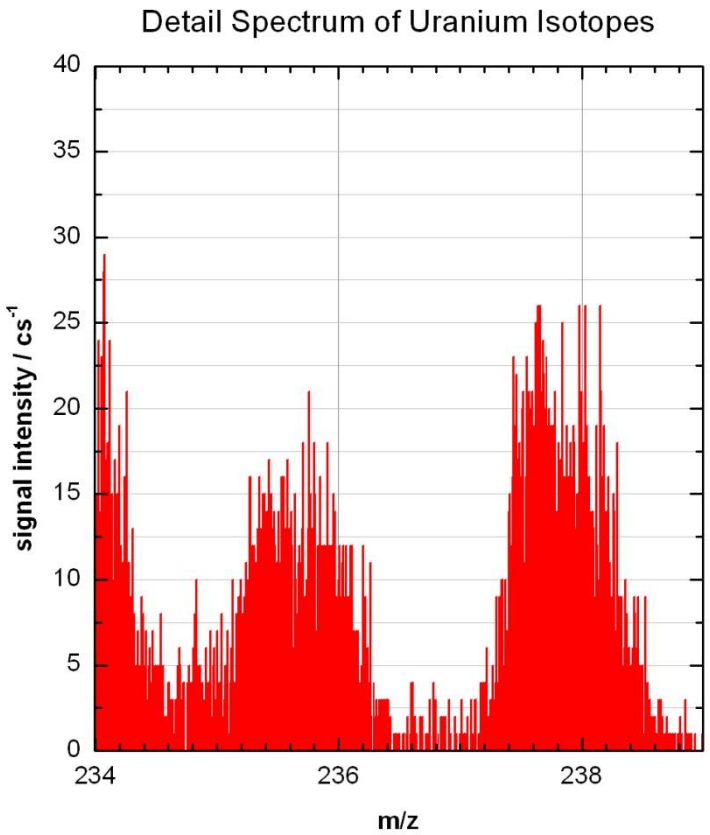
Positive SIMS Mass Spectrum showing high dynamic range

Plotting the same spectrum on a logarithmic axis reveals a more complex chemistry, with significantly more information. The ZrO_2^+ peaks are now more prominent, and Ga_2^+ show clearly at 138, 140 and 142.

Lower in the mass range Ti and TiO are observed at 48 and 64 with small quantities of Mg (24) and Al (27) also present. Oxygen, although present in large quantities, is a relatively weak emitter of positive ions leading to the signals at 16 and 32.

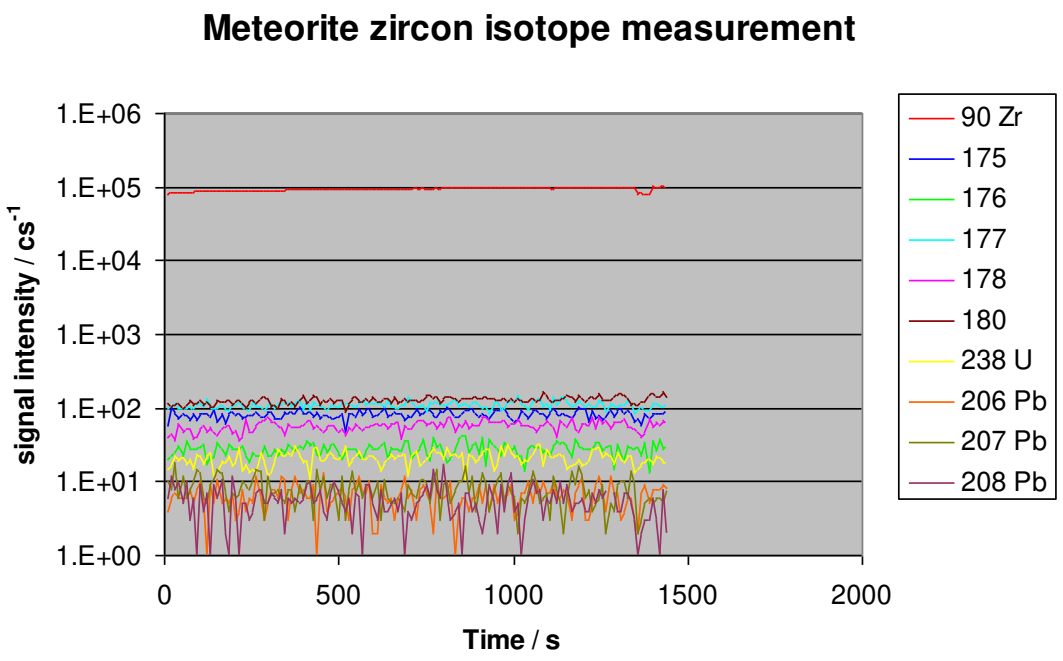
FIB–SIMS Mass Spectrum – Zircon





SIMS can be used to analyse the isotopic abundance of elements, in this case low levels of Pb and U in the Zircon sample.

FIB–SIMS Mass Spectrum – Zircon



Integrating specific isotopes over time allows greater statistical significance.

206 Pb = 945 counts (±3.3%)
207 Pb = 1141 counts (±3%)

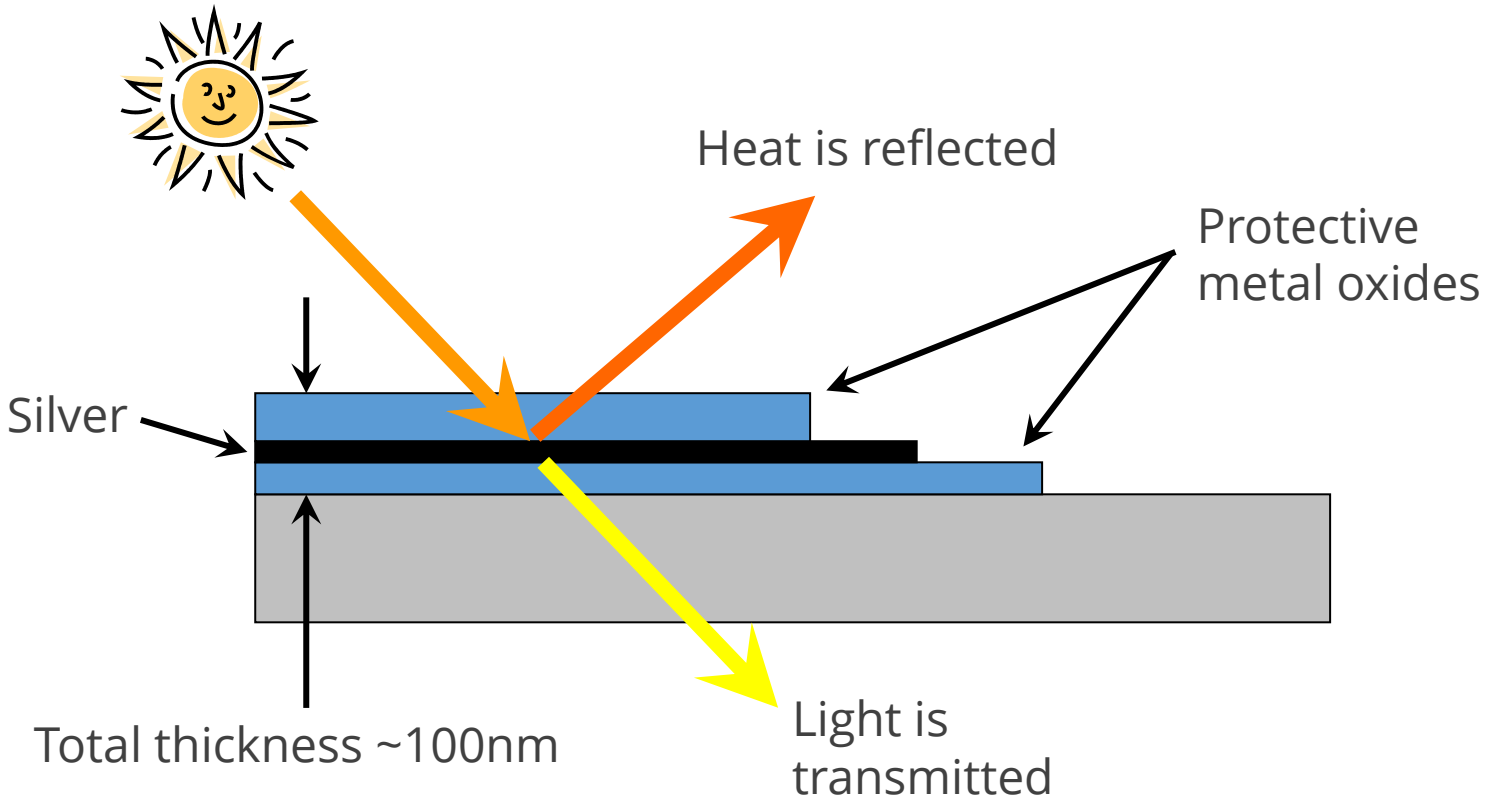
Ratio 206/207 = 0.83± 0.05

Primordial ratio – 0.9
Present day crust ratio – 1.2

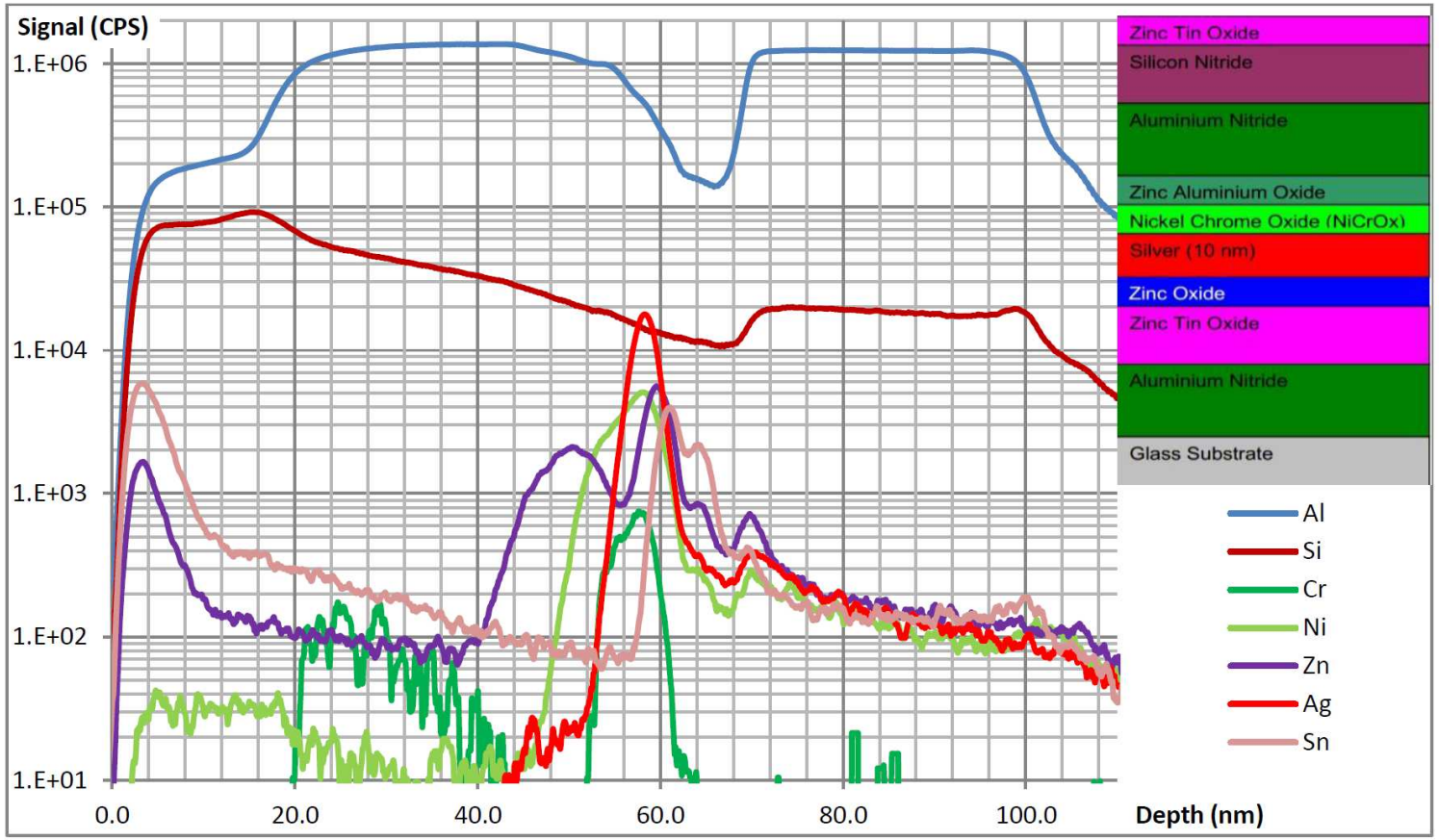
The sampled zircon is thus representative of the oldest material and therefore a good candidate for meteoritic origin.

Case Study – Glass Coating (low-e glass)

Low Emissivity Architectural Glass

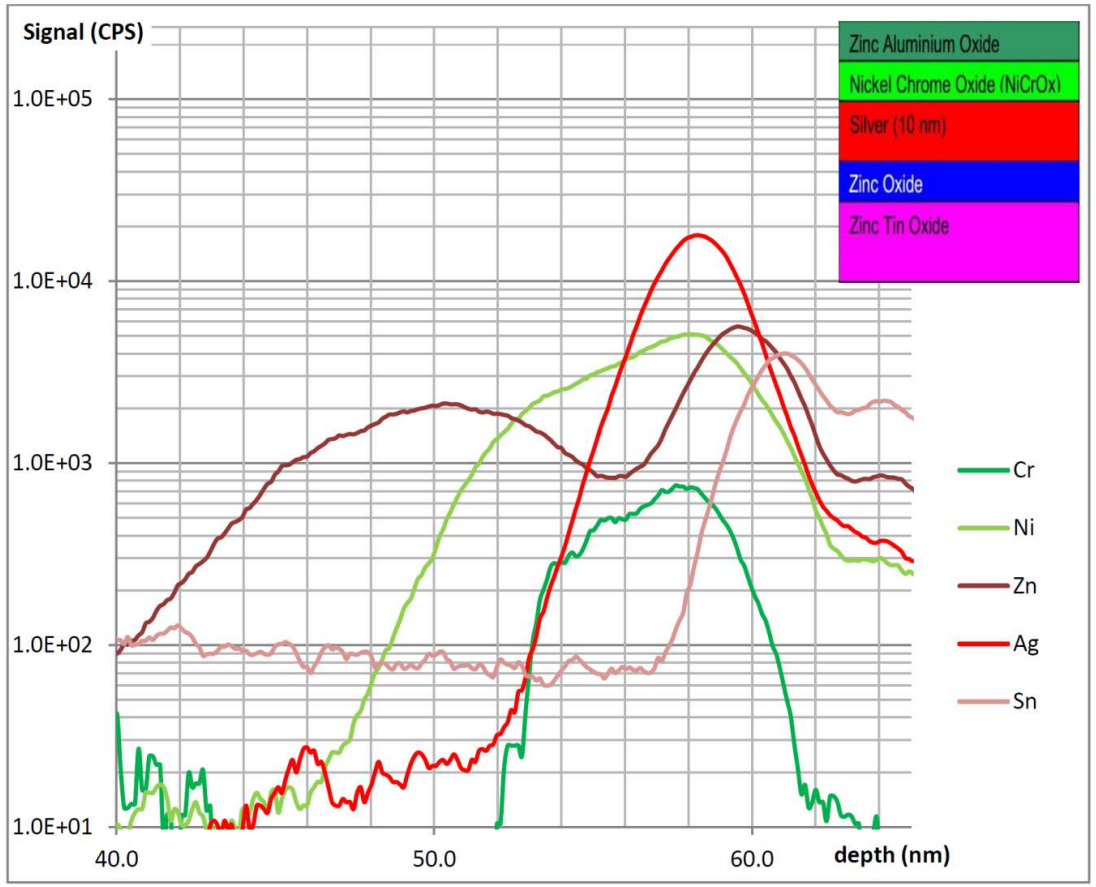


FIB-SIMS Depth Profile of Low Emissivity Float Glass



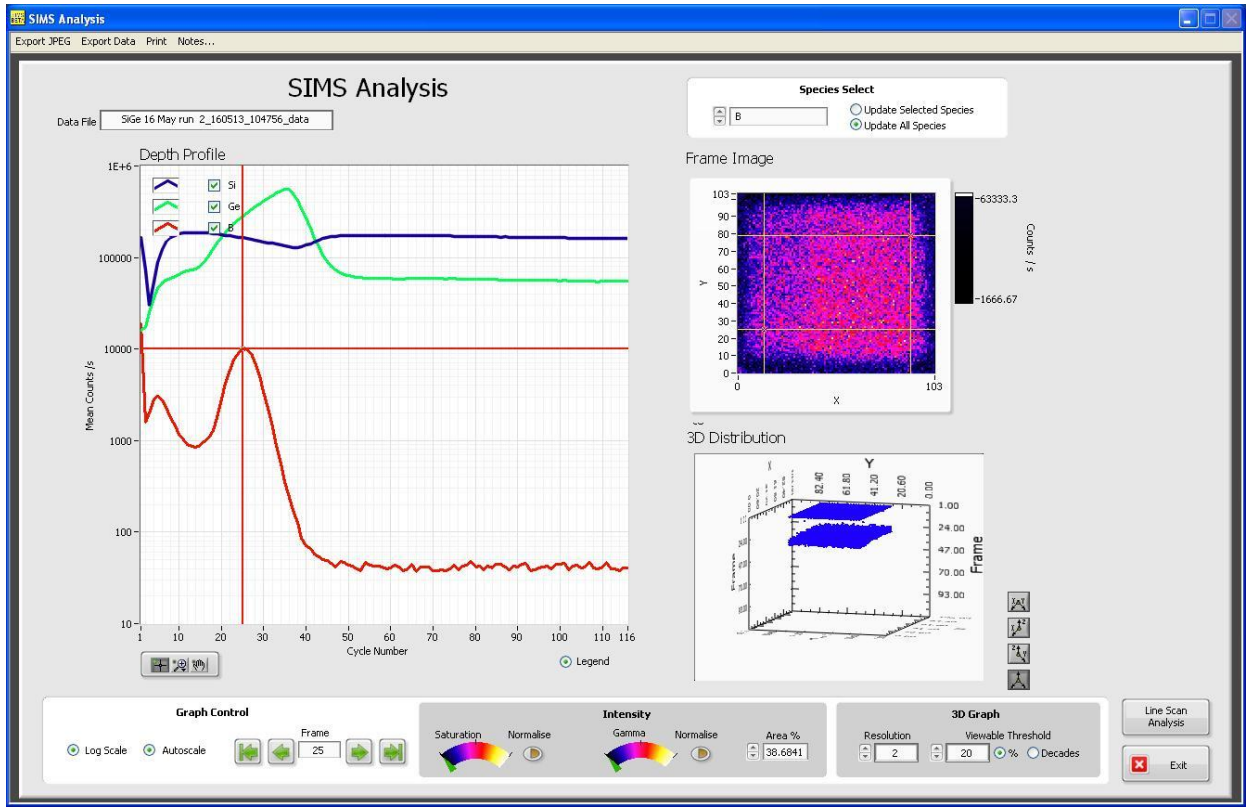
Full depth profile 85 µm x 85 µm x 0.1 µm

FIB-SIMS Depth Profile of Low Emissivity Float Glass



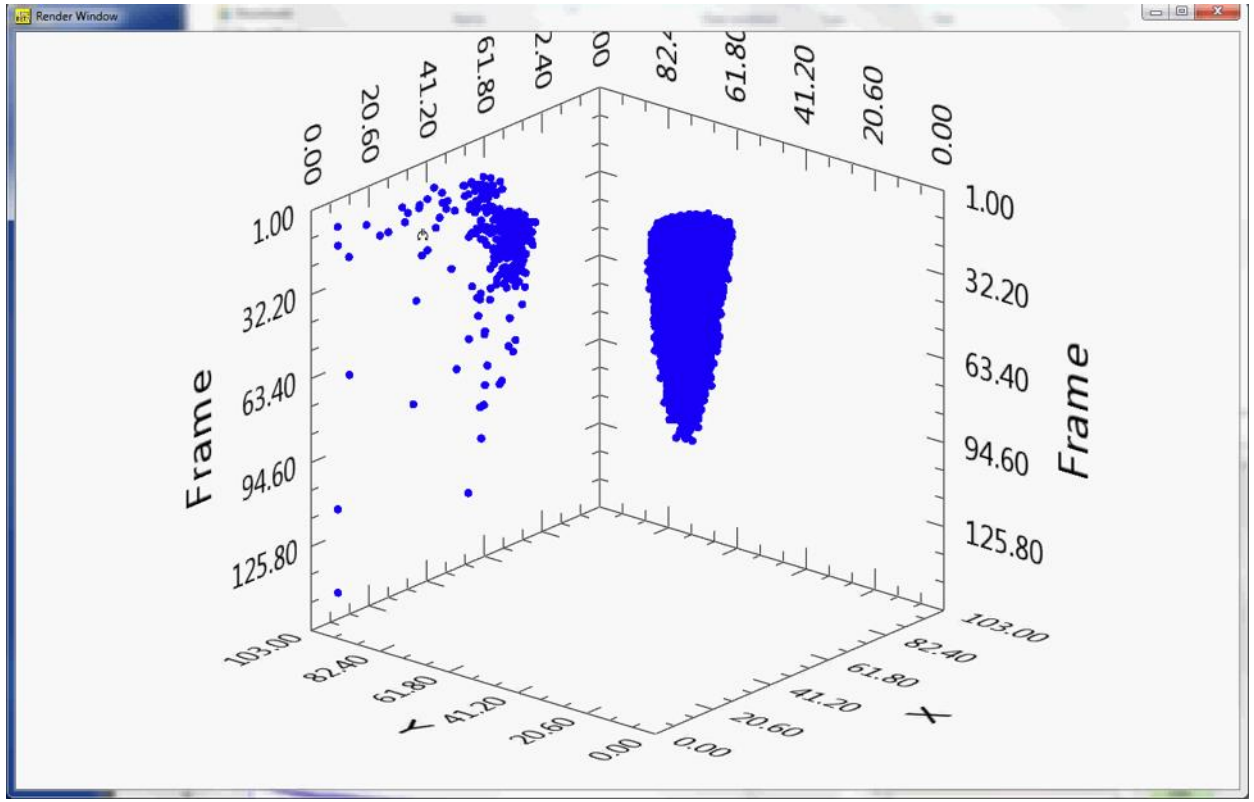
Detail of Silver layer over 25 nm depth

New Hiden SIMS Software Suite



During analysis the analysis window displays the depth profile, image data and a 3D representation of the distribution. It also controls the electronic gating.

3D Profiling by SIMS



The video shows the mass resolved aluminium signal arising from aluminium oxide grit particles embedded in the work-piece after a grinding operation. Volume is 800 µm square x 35 µm deep.

Comparison with TESCAN

Function	TESCAN	Hiden EQS series 1000 (Auriga FIB-SIMS)
Analyser type	Orthogonal ToF	Triple filter Quadrupole
Mass Range	Typically m/z 1-500	m/z 1-300 (with m/z 1-510 and 1-1000 options possible)
Mass resolution m/Dm	> 500	> 500
Energy Filter/Measurement*	No	Yes (cps over energy at selected mass)
Detection limit	1ppm	~1ppm
Dynamic range	10^5	10^7
Sensitivity	$4x10^5$ cps / ?	$>5x10^5$ cps/nA Ga @30keV on Al @ $1x10^{-6}$ torr
Lateral resolution	~50nm	~50nm
Depth resolution	20nm	<20nm (for low energy primary ion)
RGA Facility**	No	Yes
Sample extraction field	Low	typically 100V
Elemental mapping capability	Yes	Yes

*Assists in resolving mass interferences
** Can provide data on precursor gas quality, vacuum quality and be used for system leak checking etc.

- www.HidenAnalytical.com
- The Hiden website is an excellent resource with product pages, brochures, catalogues, product pages with some application notes, presentation and other information.
- Contact +44 1925 445225 for direct support.