

# **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

# Hidden 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, Hidden Analytical Inc., main office Michigan
- Office in Beijing, China
- Lifetime e-mail and telephone support with our systems



# Hidden Analytical Instruments are used in many countries worldwide

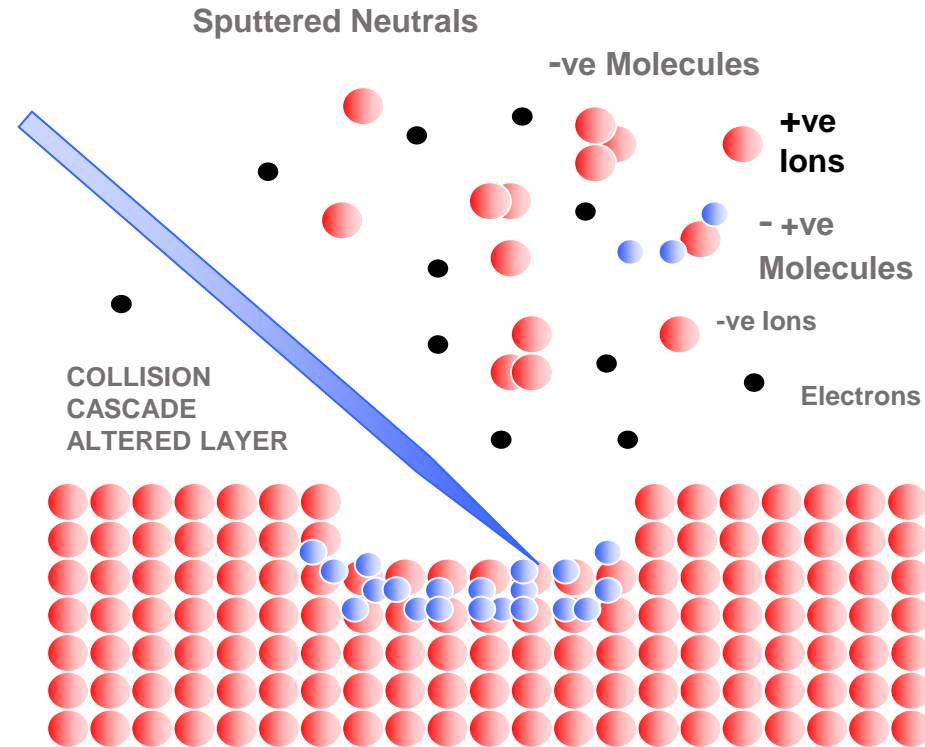


# Introduction to SIMS

- 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

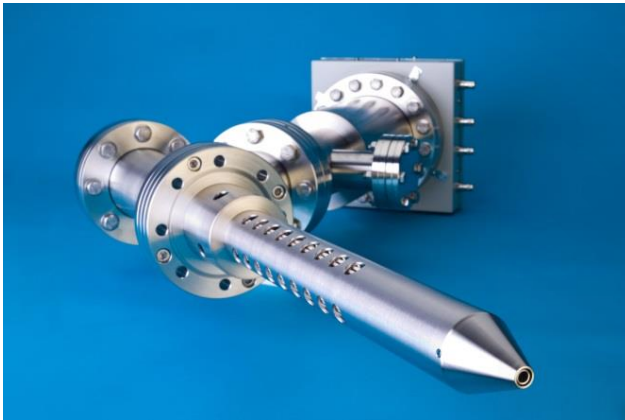
- Sputtered ions from milling are collected and analysed by mass spectrometry
- The sputtered ions are directed into the HIDEN SIMS detector by electric fields
- They then pass through an energy analyser\* (45 degree Electrostatic Sector Analyser or ESA) and then through a mass analyser (Quadrupole Mass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

\* Can assist in resolving interference masses

# Hidden 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**  
ANALYTICAL

Hidden 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.  $^{69}\text{Ga}$ ,  $^{71}\text{Ga}$ )

Elemental mapping and depth profiling

Detection of atomic and molecular ions (e.g.  $\text{Zr}^+$  and  $\text{ZrO}^+$ )

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

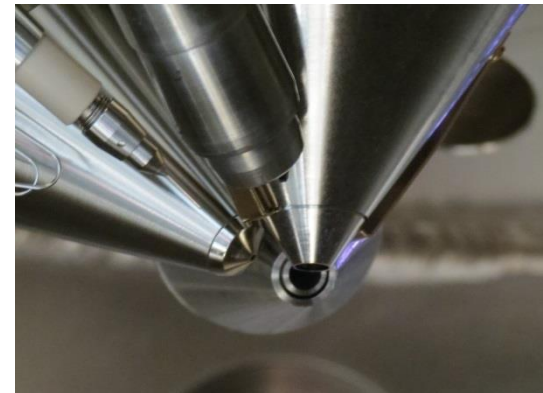
# FIB-SIMS Availability

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

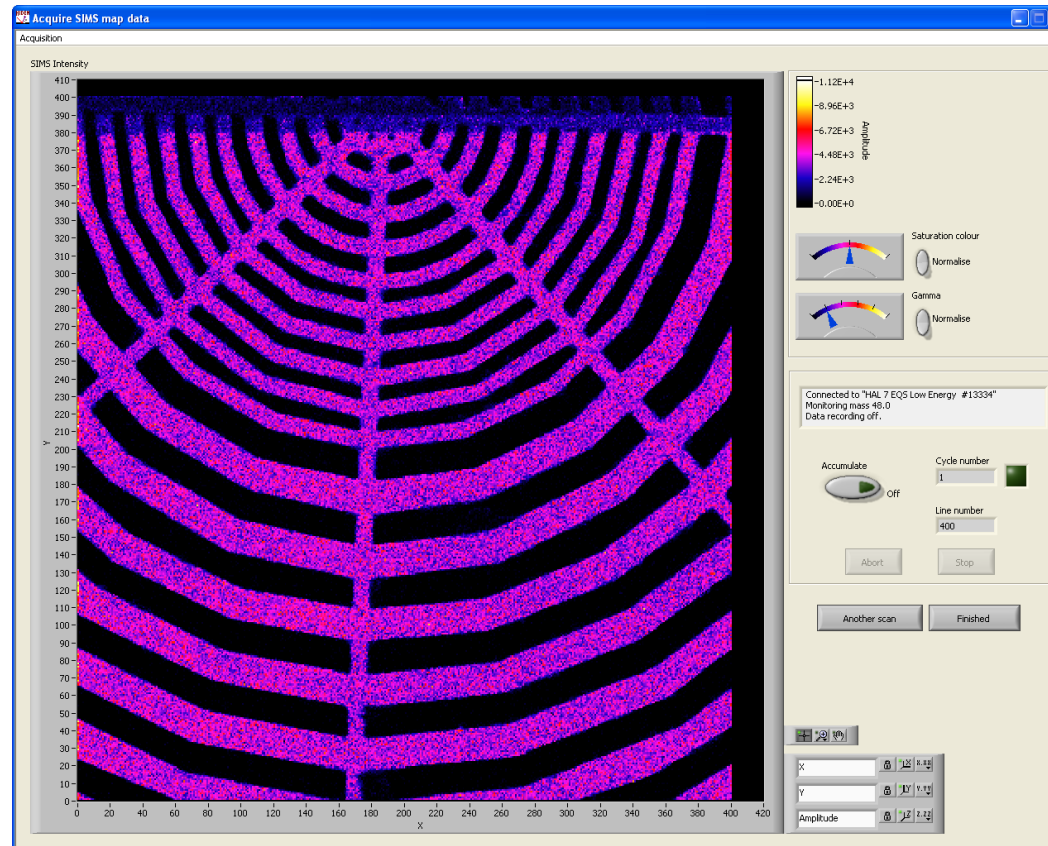


Zeiss Nvision X-beam FIB with EQS

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



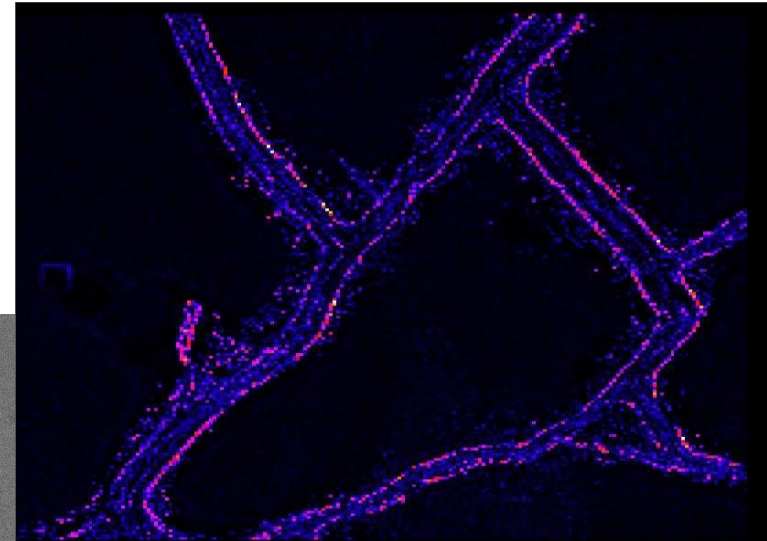
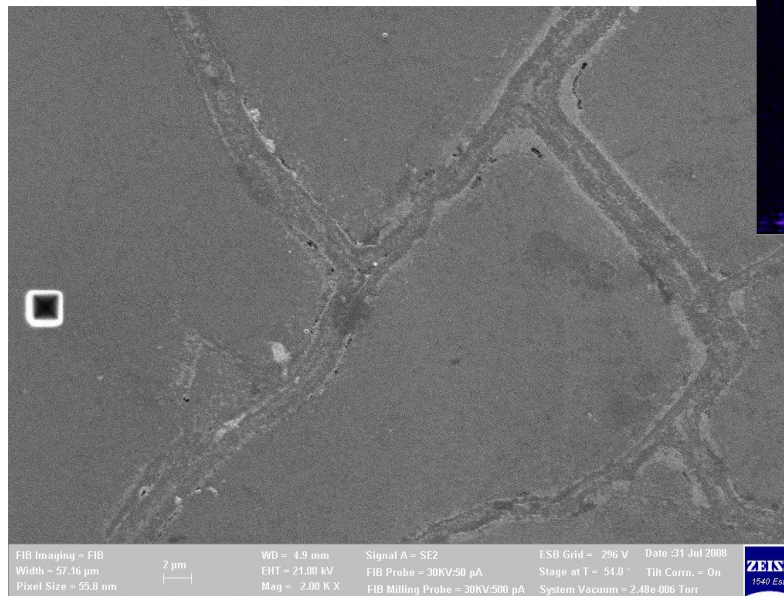
# FIB-SIMS Elemental Imaging



TiN spider web  
pattern on Si with  
1  $\mu\text{m}$  minimum  
feature size

TiN<sup>+</sup> with  $m/z = 62$   
was detected

# FIB-SIMS Imaging



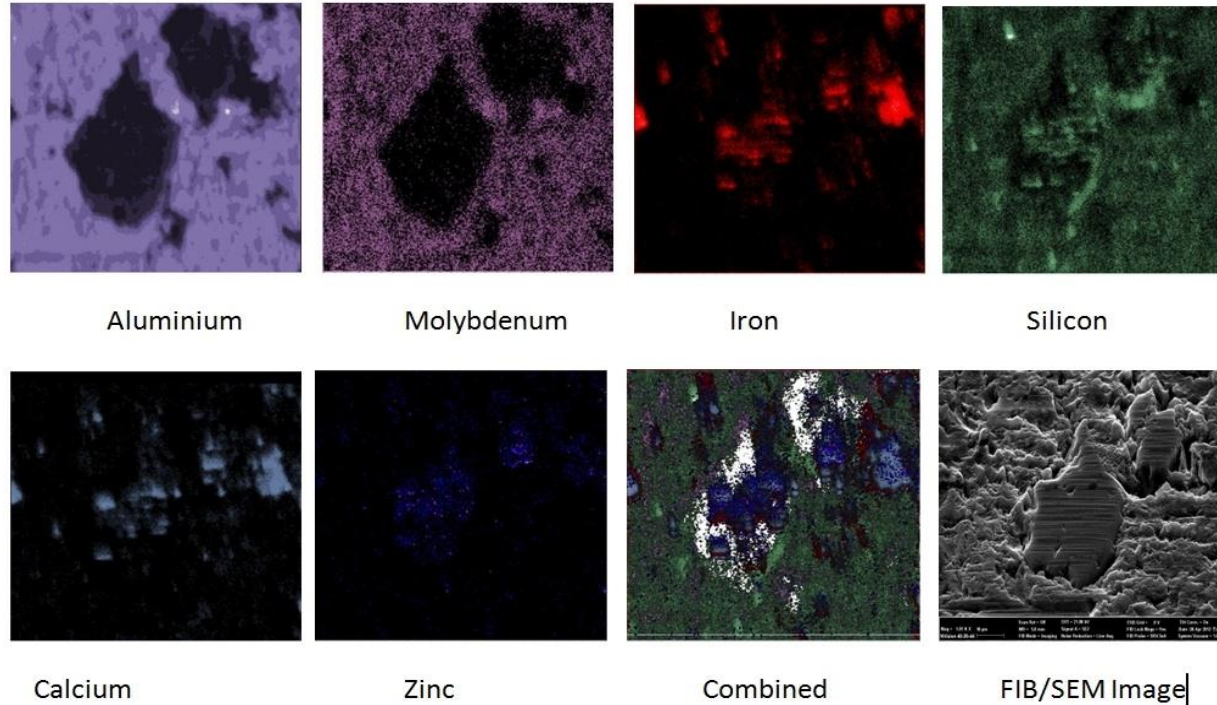
$^{27}\text{Al}^+$  image showing concentration at grain boundary  
LaSrCuFe oxide

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



# FIB-SIMS Elemental Imaging

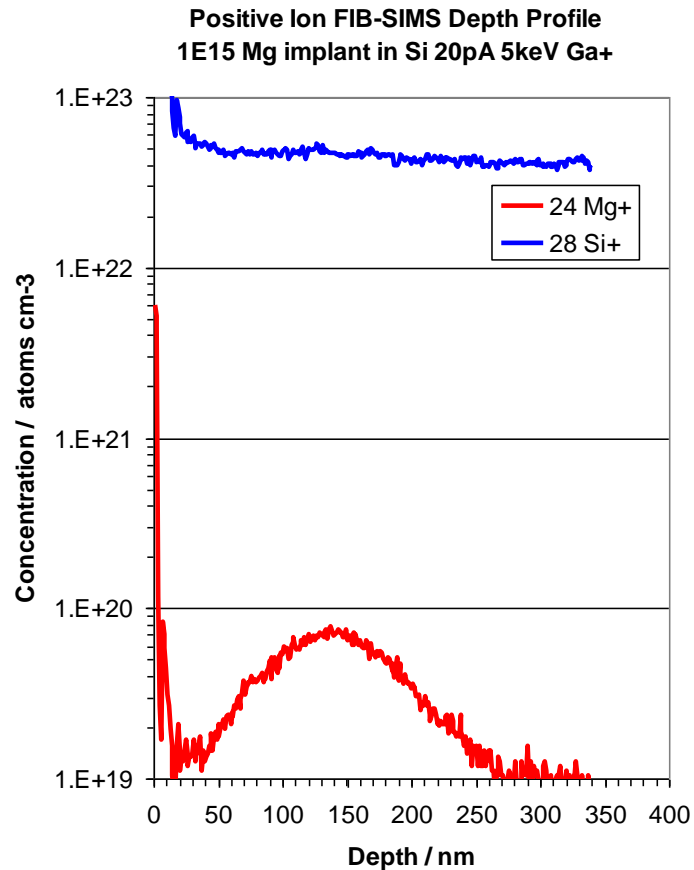
Multi Element Map showing the wear characteristics of an Alusil Engine liner<sup>1</sup>.



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

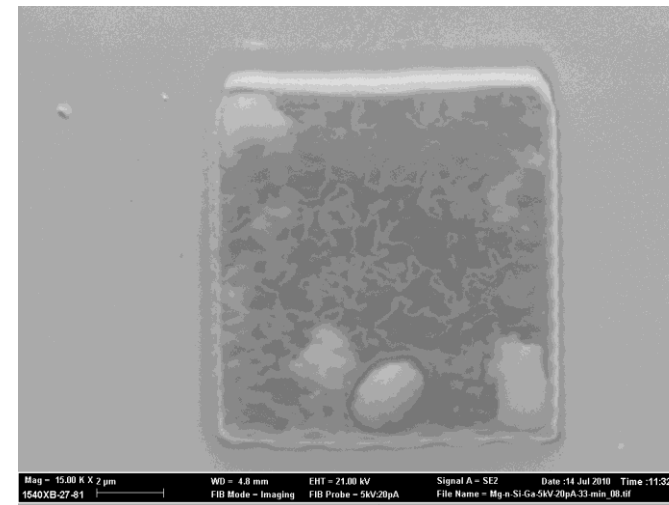
# FIB-SIMS Depth Profiling



Depth Profile of <sup>24</sup>Mg<sup>+</sup> dopant  
in bulk Si

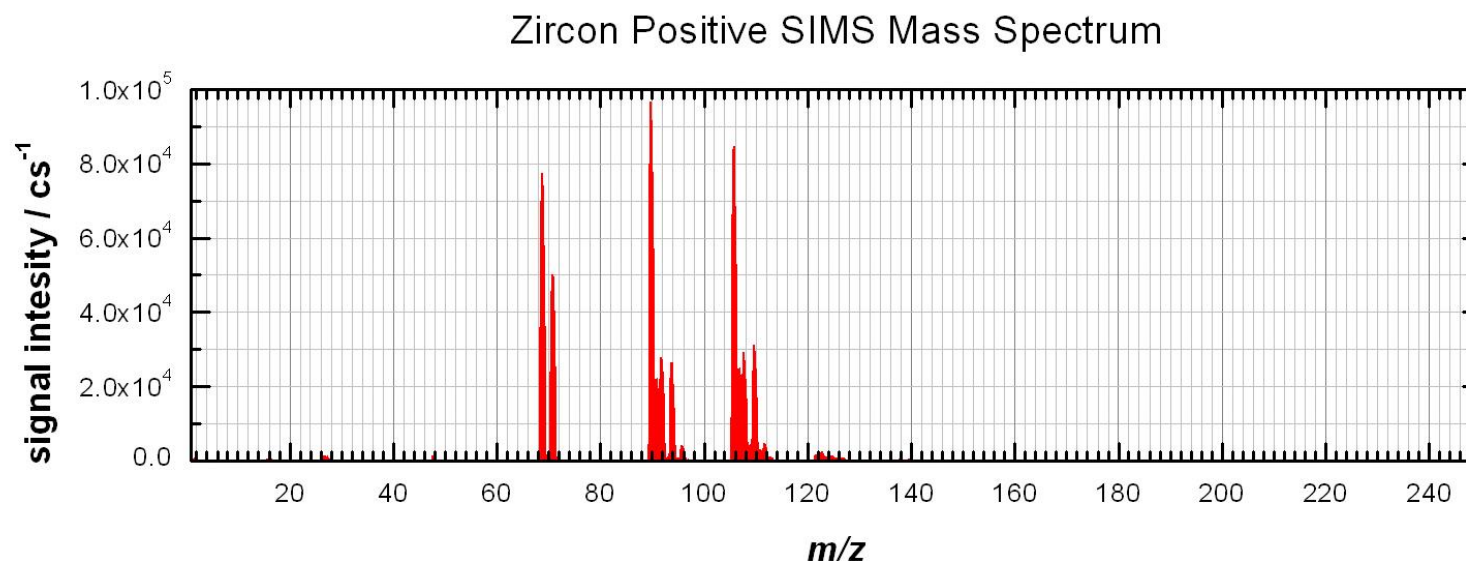
Peak of <sup>24</sup>Mg<sup>+</sup> implant is  $7 \times 10^{19}$   
atoms cm<sup>-3</sup>

~ 0.15% atomic



# FIB-SIMS Mass Spectrum - Zircon

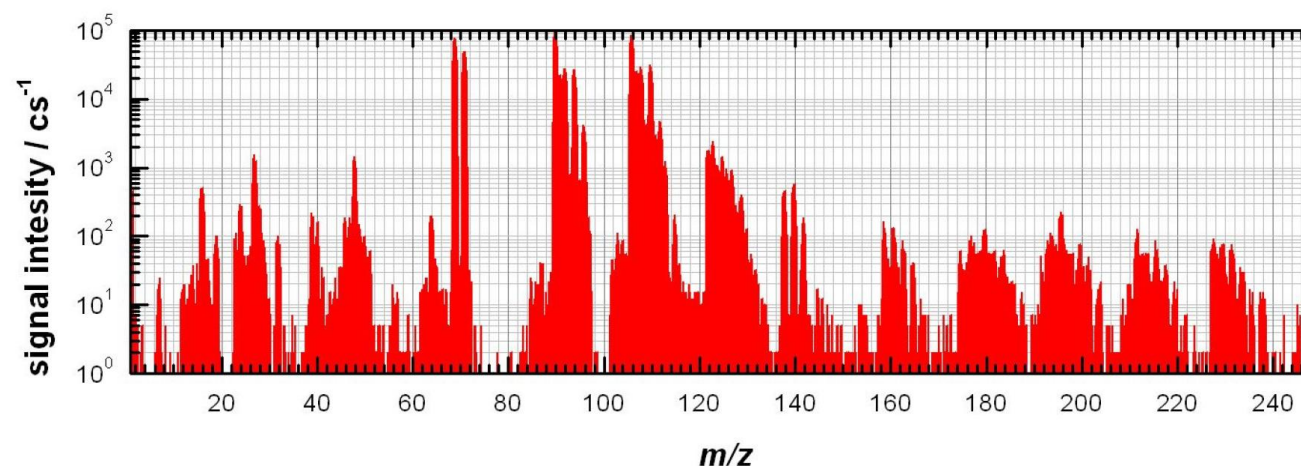
Analysis of individual zircon grains in a possible meteoritic rock sample  
- can FIB SIMS determine if it is actually likely to be extra-terrestrial in origin?



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

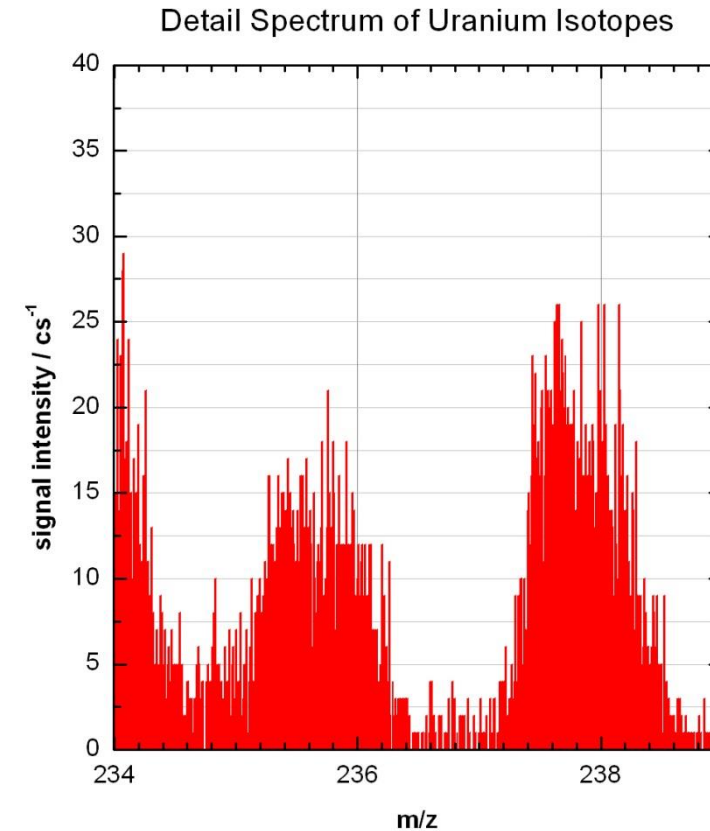
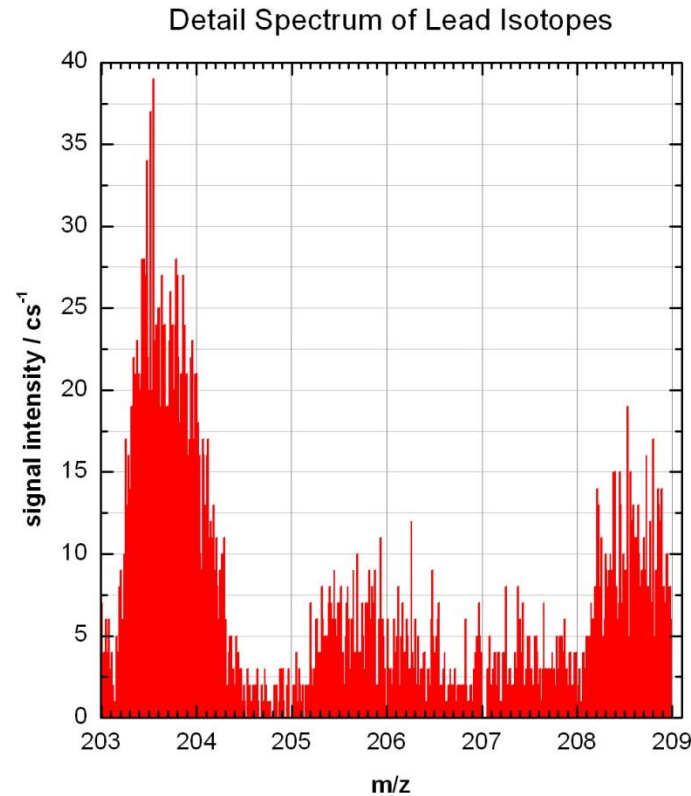


Positive SIMS Mass Spectrum showing high dynamic range

Plotting the same spectrum on a logarithmic axis reveals a more complex chemistry, with significantly more information. The  $\text{ZrO}_2^+$  peaks are now more prominent, and  $\text{Ga}_2^+$  show clearly at 138, 140 and 142.

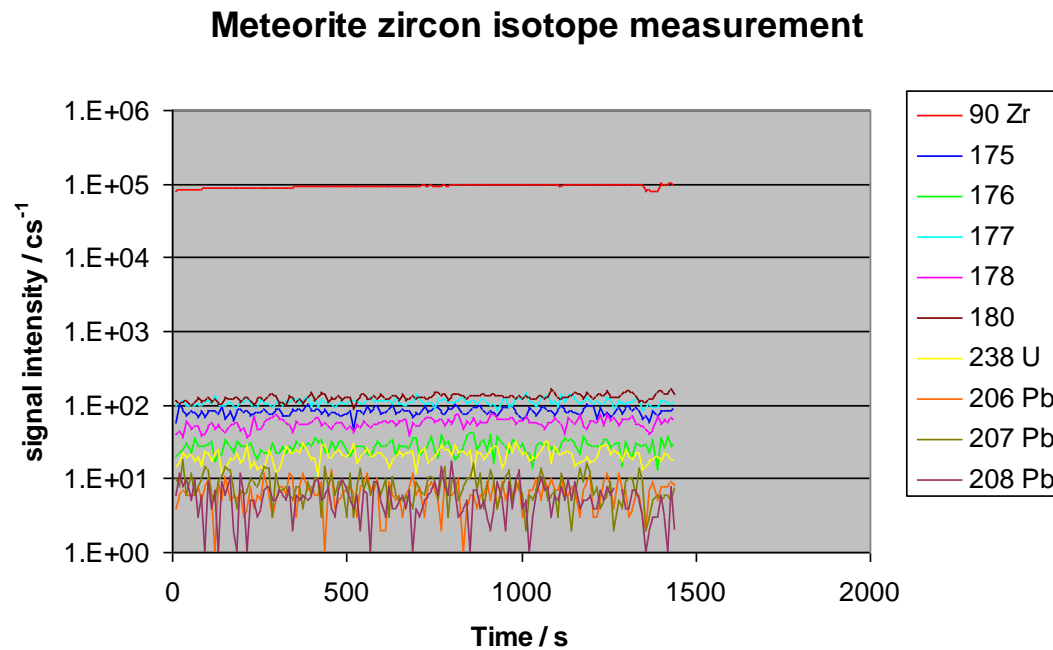
Lower in the mass range Ti and TiO are observed at 48 and 64 with small quantities of Mg (24) and Al (27) also present. Oxygen, although present in large quantities, is a relatively weak emitter of positive ions leading to the signals at 16 and 32.

# 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 ( $\pm 3.3\%$ )

207 Pb = 1141 counts ( $\pm 3\%$ )

**Ratio 206/207 =  $0.83 \pm 0.05$**

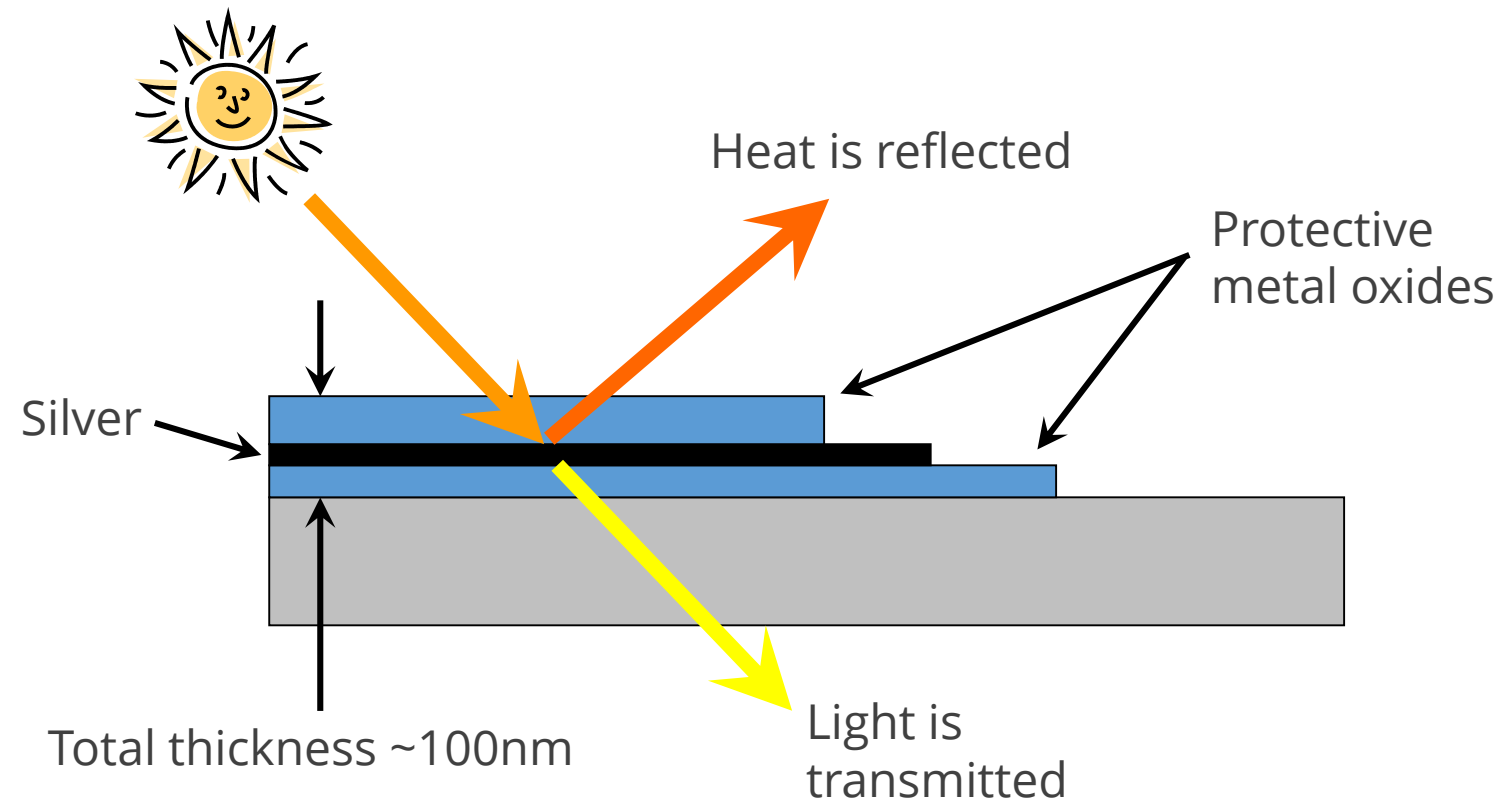
Primordial ratio – 0.9

Present day crust ratio – 1.2

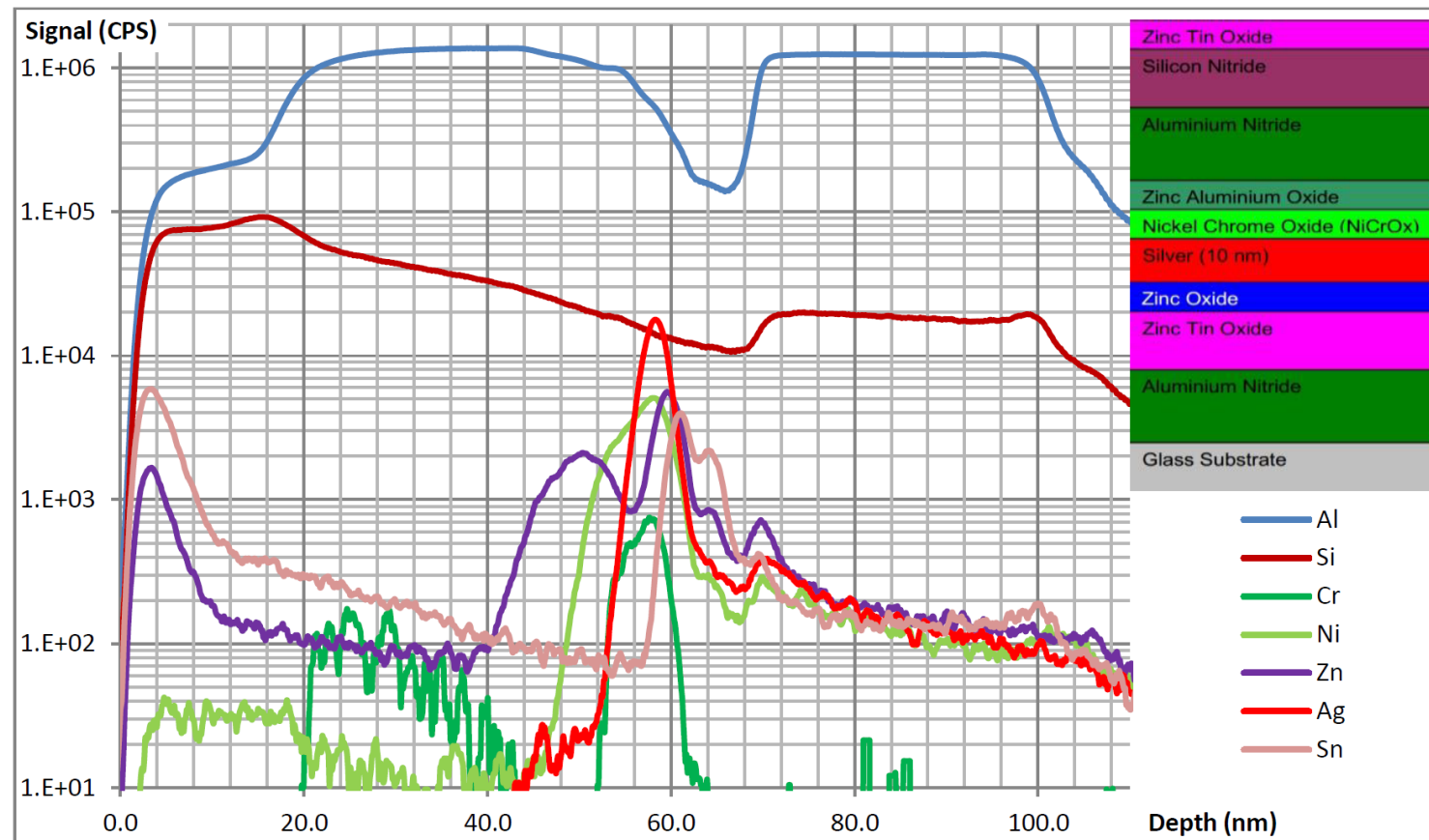
The sampled zircon is thus representative of the oldest material and therefore a good candidate for meteoritic origin.

# Case Study – Glass Coating (low-e glass)

Low Emissivity Architectural Glass

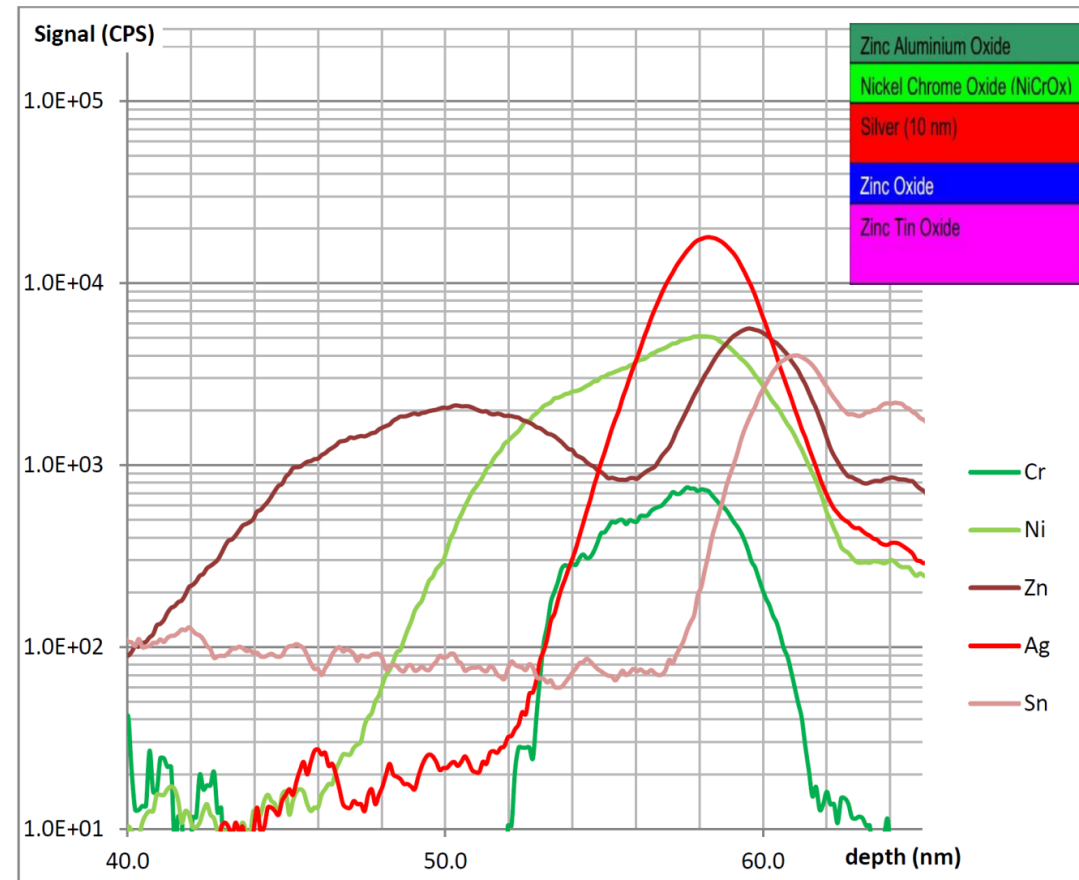


# FIB-SIMS Depth Profile of Low Emissivity Float Glass



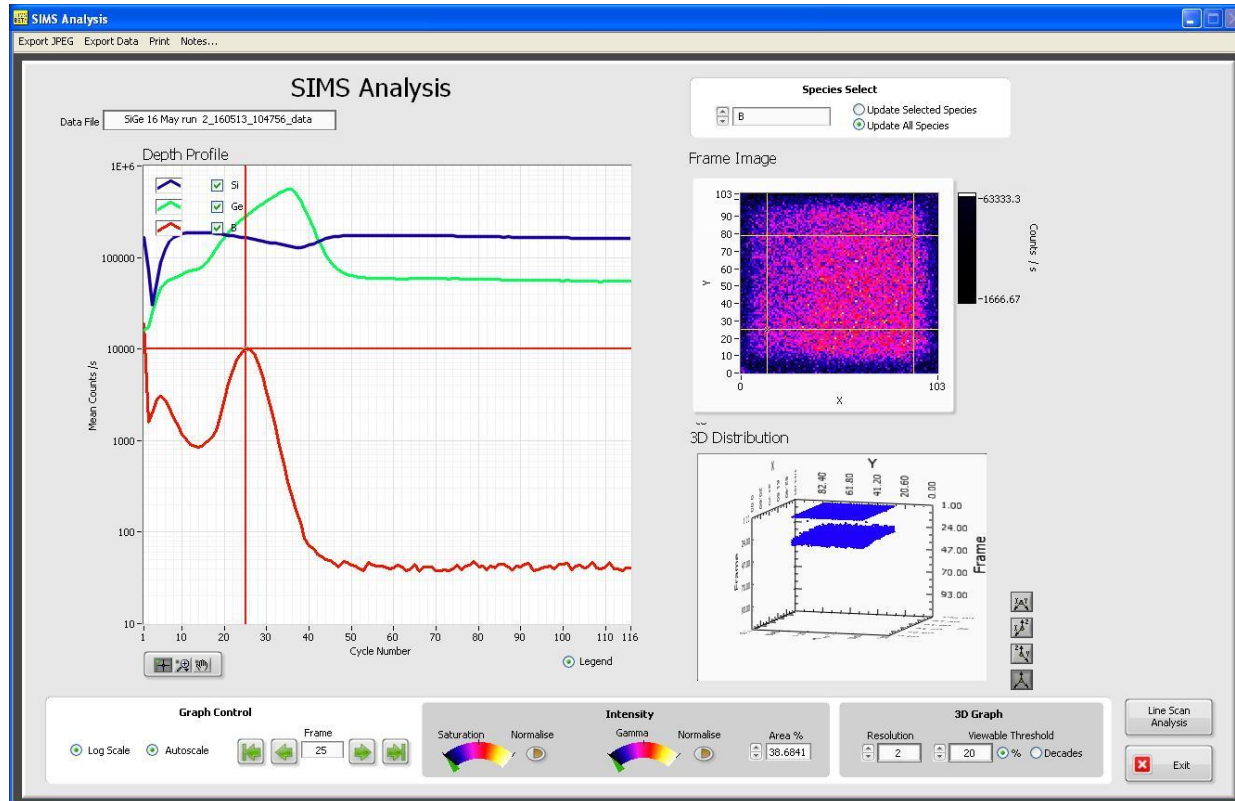
Full depth profile 85  $\mu\text{m}$  x 85  $\mu\text{m}$  x 0.1  $\mu\text{m}$

# FIB-SIMS Depth Profile of Low Emissivity Float Glass



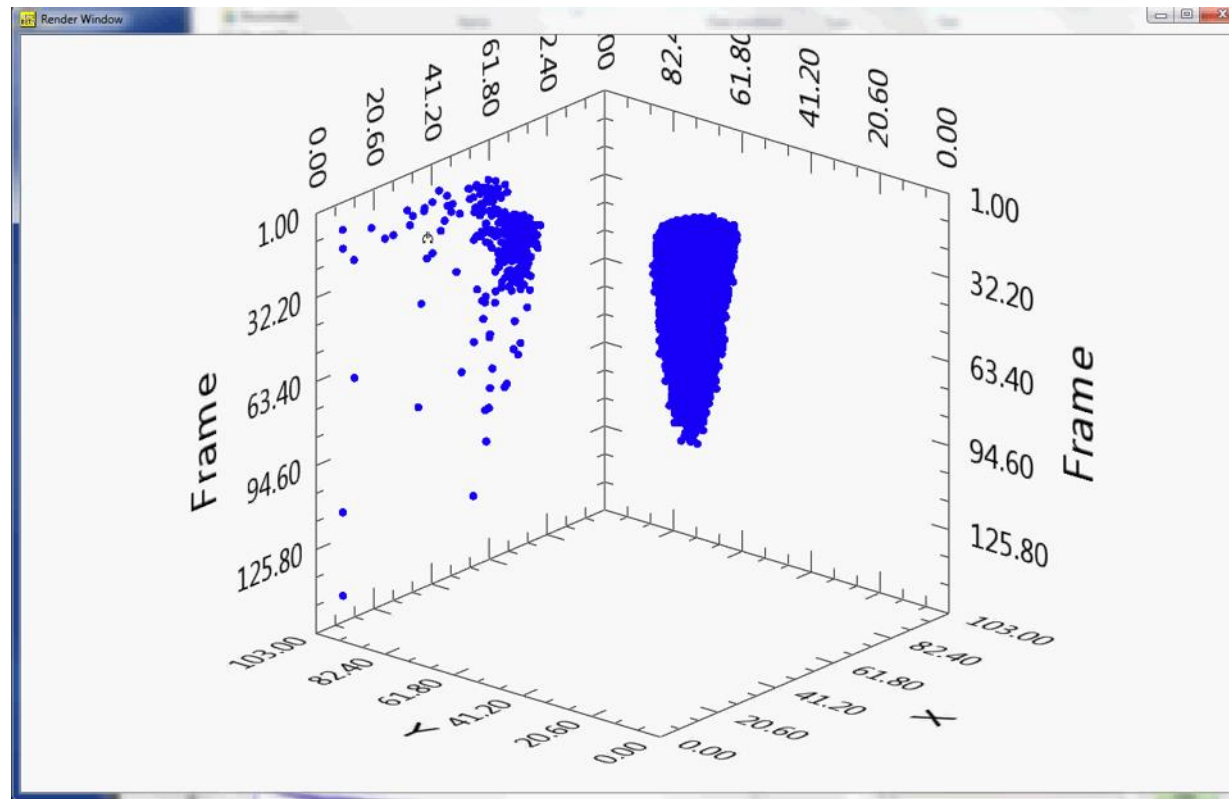
Detail of Silver layer over 25 nm depth

# 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  $\mu\text{m}$  square x 35  $\mu\text{m}$  deep.



# Comparison with TESCOAN

| Function                     | TESCAN                    | Hidden 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 <sup>5</sup>           | 10 <sup>7</sup>   |
| Sensitivity                  | 4x10 <sup>5</sup> cps / ? | >5x10 <sup>5</sup> cps/nA Ga @30keV on Al @ 1x10 <sup>-6</sup> 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.



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