Differentially pumped quadrupole SIMS probe on FIB-based and two-beam microscopes

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Motivation

- Ultra-high spacial resolution SIMS in combination with electron microscopy, EDS, EBSD.

Instrumentation

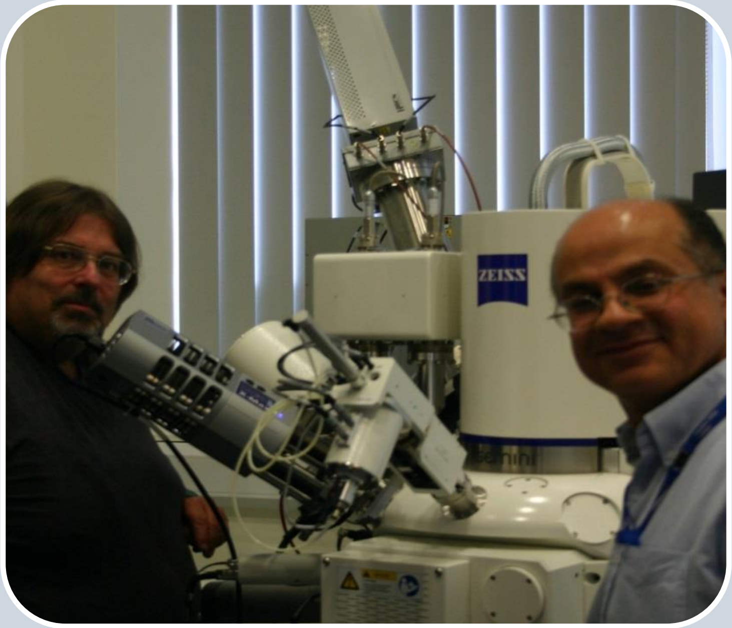
- A Hiden EQS 1000 SIMS detector, separately pumped has been attached to a two-beam Zeiss Auriga SEM with Orsay gallium ion gun.
- A single beam gallium ion microscope, FEI FIB200 with an FEI built SIMS detector has been fitted with a second SIMS detector, Hiden EQS 1000 which is also separately pumped.

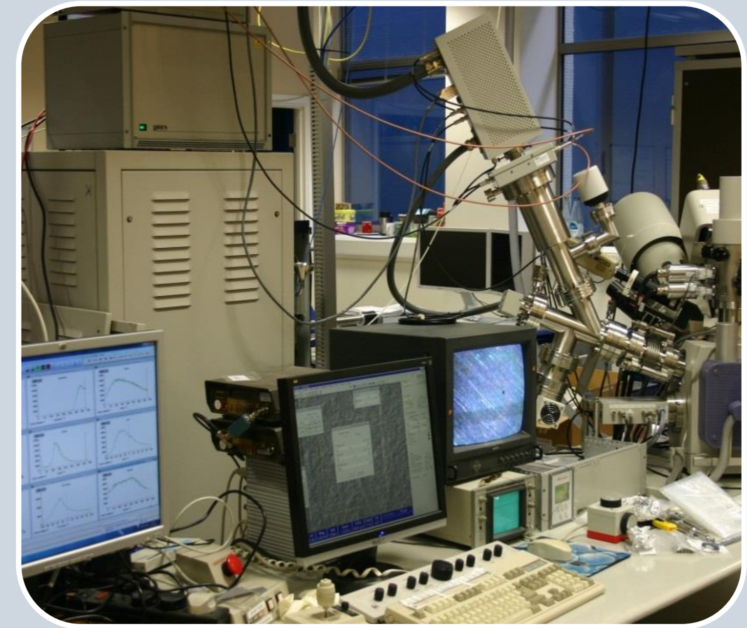
Material

- Oxide materials for high temperature Solid Oxide Fuel Cells are studied using the stable isotope oxygen-18 for surface reaction and diffusivity .
- Stress induced corrosion cracking in advanced metal alloys are also studies using oxygen-18 tracer techniques.

Objectives

- SIMS together with structural and chemical information from electron and x-ray analysis in two-beam scanning electron microscope
- Potential of simultaneous positive and negative secondary ion detection.





Zeiss Auriga SEM

♦FEG SEM electron gun column with electrostatic final lens.

♦Gallium ion beam column from Orsay-Physics (Cobra) with beam spot sizes that vary from 10nm .

♦Secondary electron detectors both in-lens and separate

♦Backscatter electron detectors in-lens

♦Positive or negative charged particle detector (SESI)

♦EDS

♦EBSD

Hiden EQS 1000 SIMS detector

♦Triple quadrupole electric mass filter for masses from 0.4 AMU to 300AMU

♦Electrostatic filter for ions at quadrupole entrance.

♦secondary charged ions detected individually by secondary electron multiplier.

♦separate vacuum pumping using a drypump and turbomolecular pump.

♦Software system for detector setup and control for spectra, depth profiles and images.

♦Residual gas analysis (RGA)

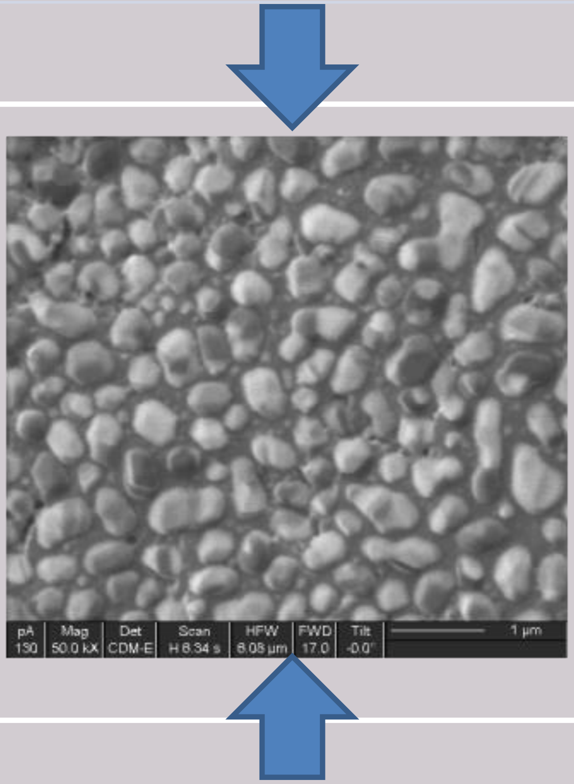
FEI FIB200 SIMS

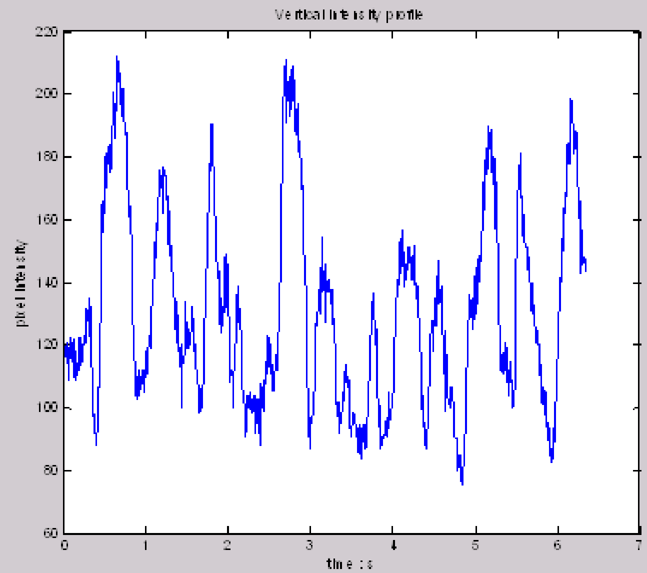
♦FIB200 workstation with single beam gallium ion gun used at energies to 30keV. Beam can be scanned with normal line/frame raster or within a pattern(s).

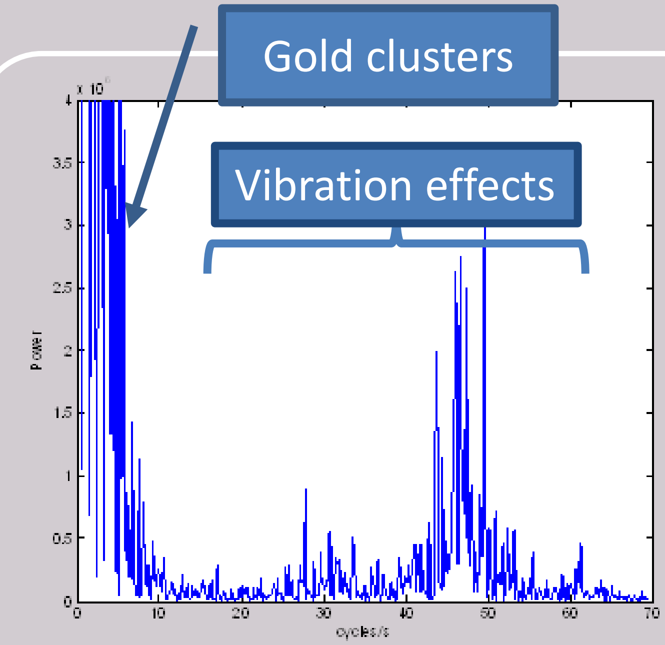
♦Gallium ion beam spot size varies from 10nm at 20pA to ~600nm at 20nA.

♦FEI designed quadrupole based SIMS detector with low field collection and without an electrostatic analyser in the secondary ion column .

♦SIMS measurements generate spectra, depth profiles or maps.







Vibration effects from image analysis

♦ Gold clusters on carbon imaged with Gallium beam at 30keV and 100pA aperture in both the FEI FIB200 and Zeiss Auriga SEM

♦ Line intensity profiles horizontally and vertical through the centre of the images were analysed for the power spectra.

♦ Drypump and turbopump damping and connections modified in response to power spectra

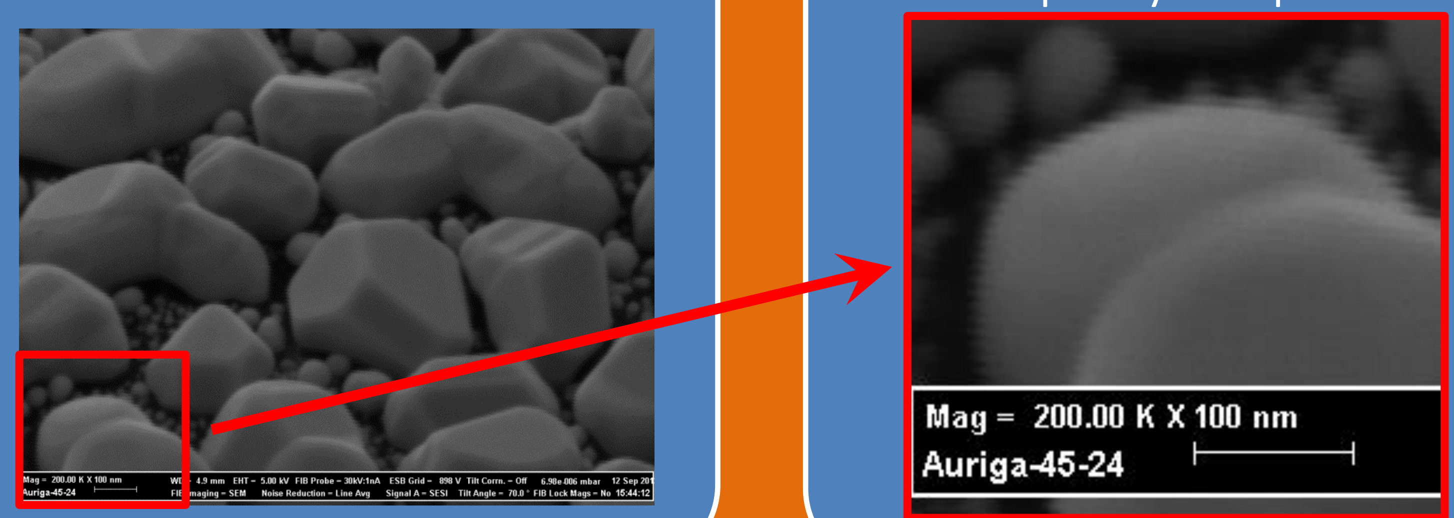
FEI FIB200

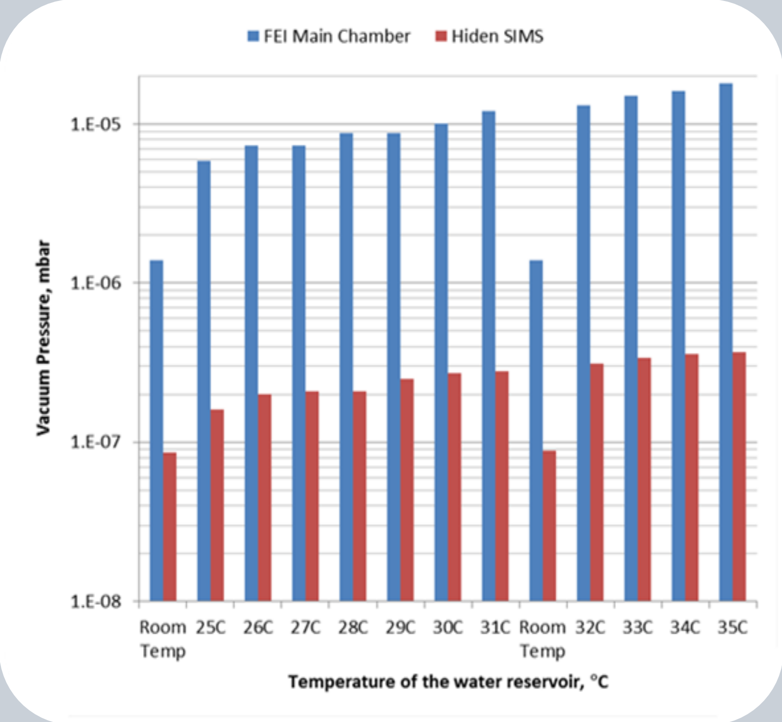
♦ Image degradation is not seen at any beam current after the modification to the Hiden SIMS probe pumping system .

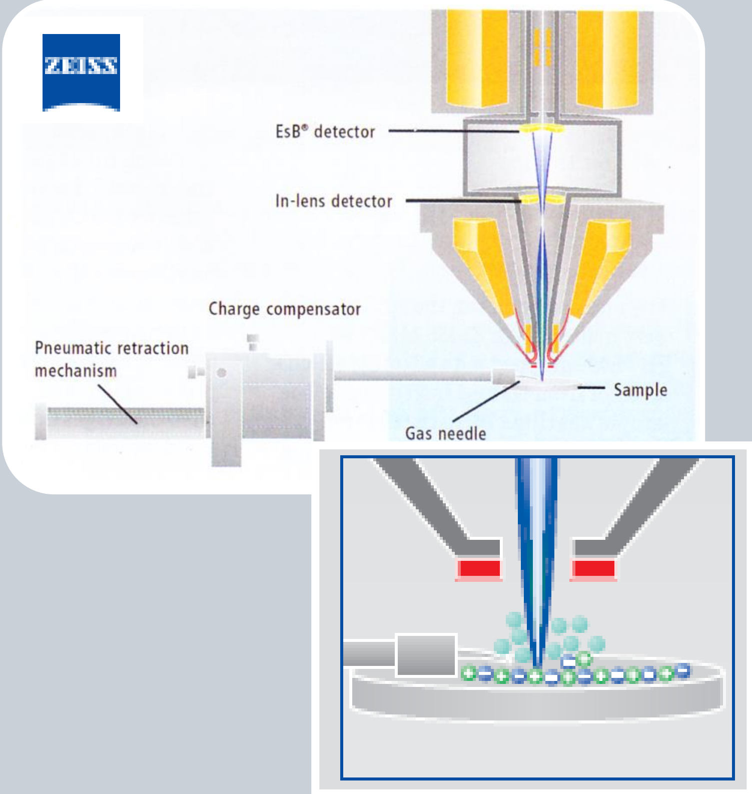
♦ Zeiss Auriga SEM-FIB

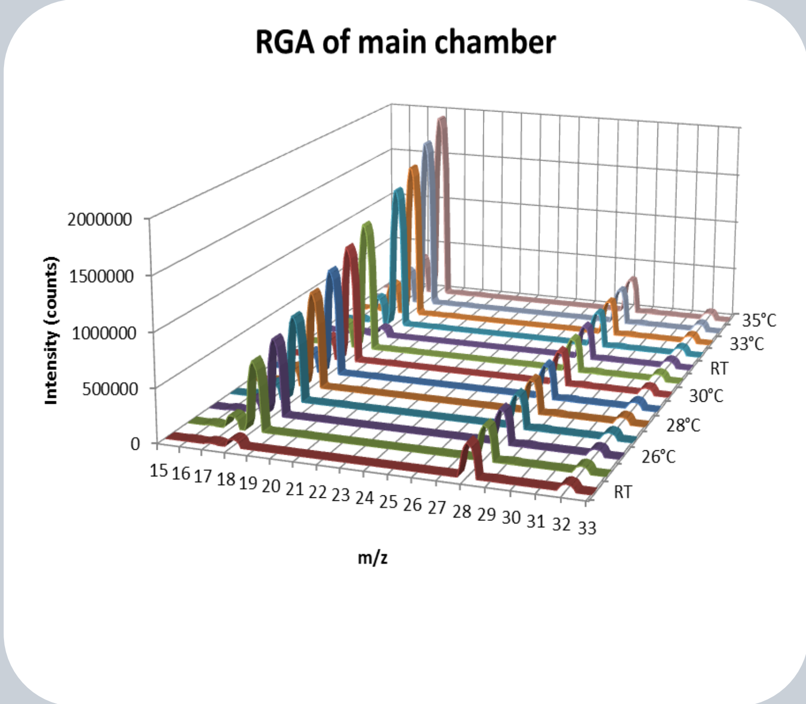
♦ Vibrational effects of the additional pumping system with the Hiden SIMS probe is seen in the SEM image of the gold cluster on carbon sample at 200kX magnification.

♦ Further damping for low frequency is required









Pressure differential : FEI FIB200

♦ FEI FIB200 instrument has a system for introducing water vapour only for positive ion yield M^+ enhancement.

♦ Chamber pressure is controlled by the reservoir temperature which contains hydrated magnesium sulphate (Epsom Salts).

♦ Graph shows that a differential pressure of ~2 orders of magnitude can be maintained.

♦ Hiden SIMS probe would have tripped off at the lowest reservoir temperature of 25°C.

Pressure differential : Zeiss Auriga SEM

♦ Zeiss Auriga SEM has a system for introducing oxygen gas as a jet directed at the sample surface for charge compensation. SE and BSE ionise local gas molecules to achieve a charge balance for normal imaging.

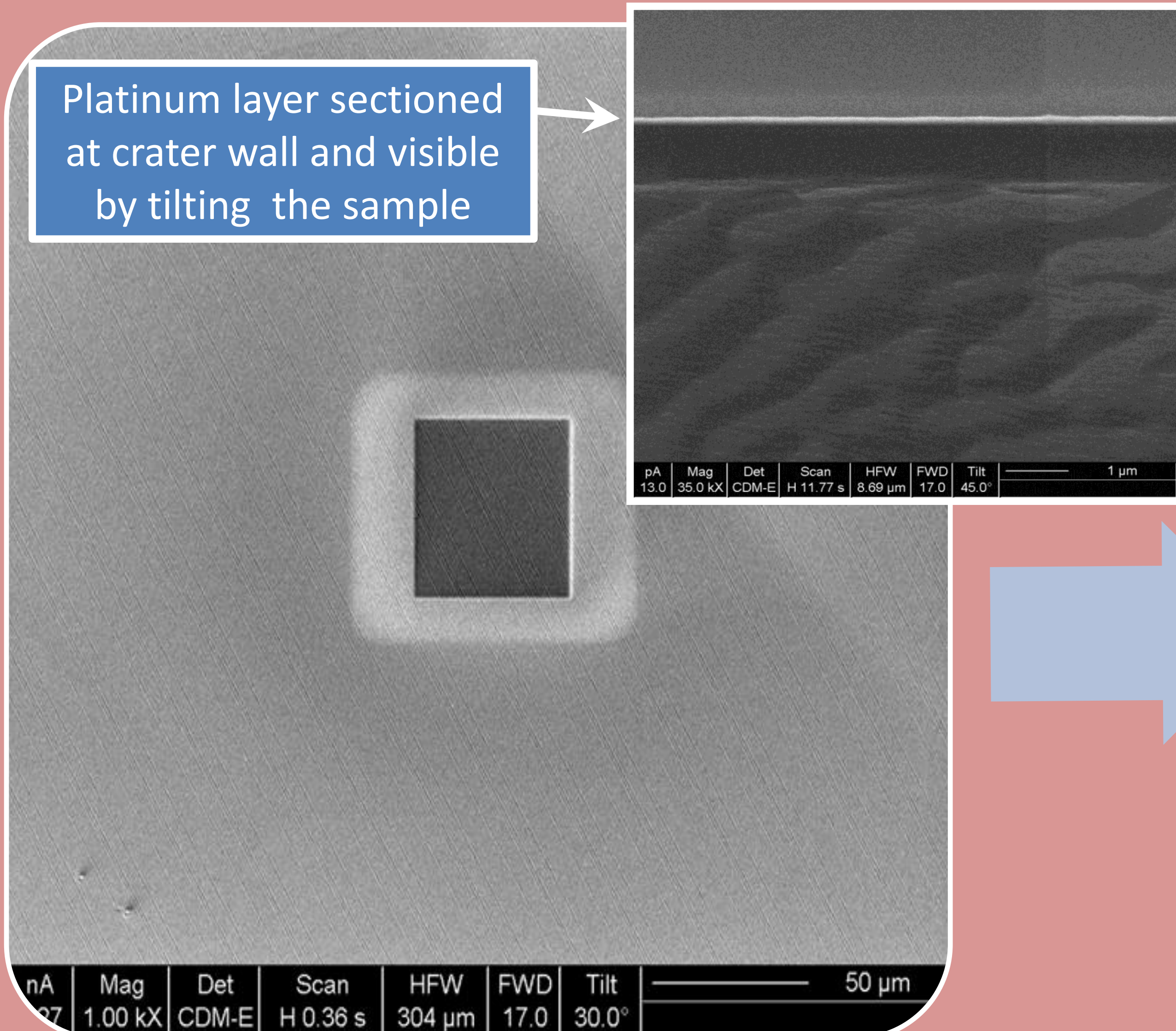
♦ Chamber pressure is controlled by flowrate and gas source pressure.

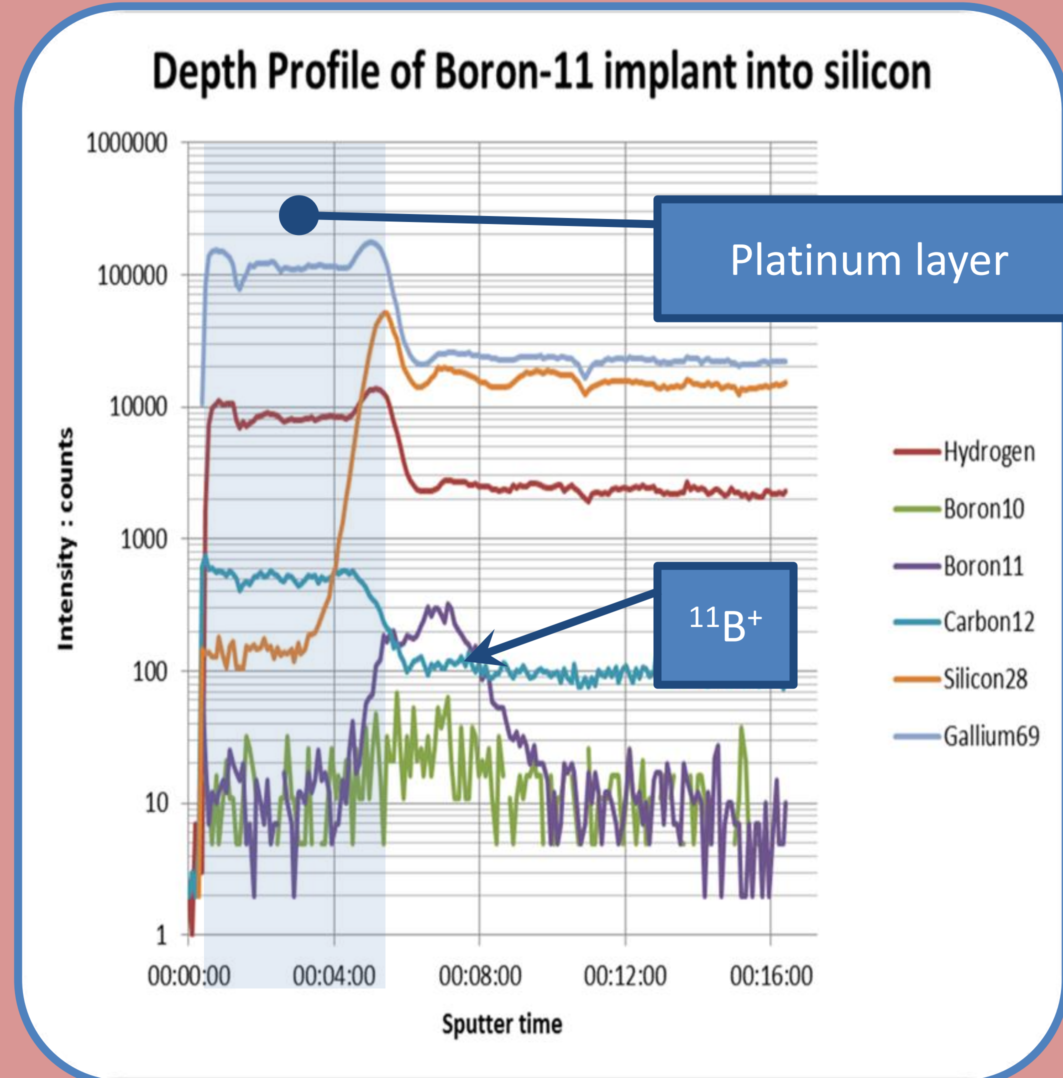
♦ Approximately two orders of magnitude pressure differential is maintained.

♦ Yield enhancement ratio for M^+ with oxygen coverage compared to a clean surface.

♦ A. Benninghoven 1976

Z	M	R
24	Chromium	1000
38	Strontium	800
42	Molydenum	615
25	Manganese	500
74	Tungsten	389
22	Titanium	308
23	Vanadium	300
73	Tantalum	286
26	Iron	233
56	Barium	150
13	Aluminium	100
12	Magnesium	90
41	Niobium	83
28	Nickel	75
14	Silicon	69
29	Copper	23
32	Gemanium	5





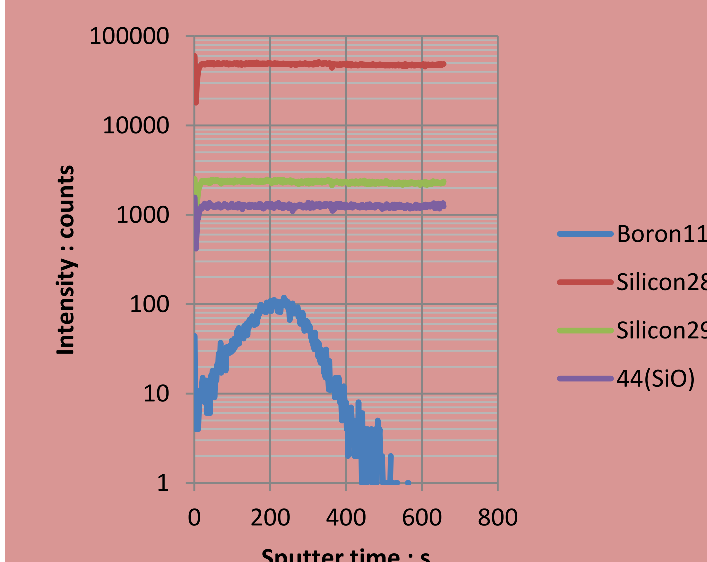
Depth profile of Boron in Silicon

- Small area, 70um x 70 um coated in-situ with ~200nm of a platinum organic.
- Ga^{+} FIB sputtering (30keV, 3nA) into area of 50um x 50 um centered within the platinum.
- Inset image shows the crater wall and crater base for depth estimation.
- Water vapour in main chamber, pressure 8.8×10^{-6} mbar.
- Platinum remains at edge throughout the depth profiling despite gallium beam skirt.

^{11}B implant, dose 1.6×10^{15} per cm^2 at 40keV into silicon

- Peak concentration ~0.25%at
- SRIM estimates ^{11}B peak at 144nm depth below surface which is distinguishable on the Log-Lin plot.
- Oxygen enhancement for both silicon and boron are below 100.
- TofSIMS profile, Oxygen sputtering at 1keV. 25keV Bi pulses.

TofSIMS measurement of Depth Profile



Sarah Fearn, IonTOF ToFSIMS5

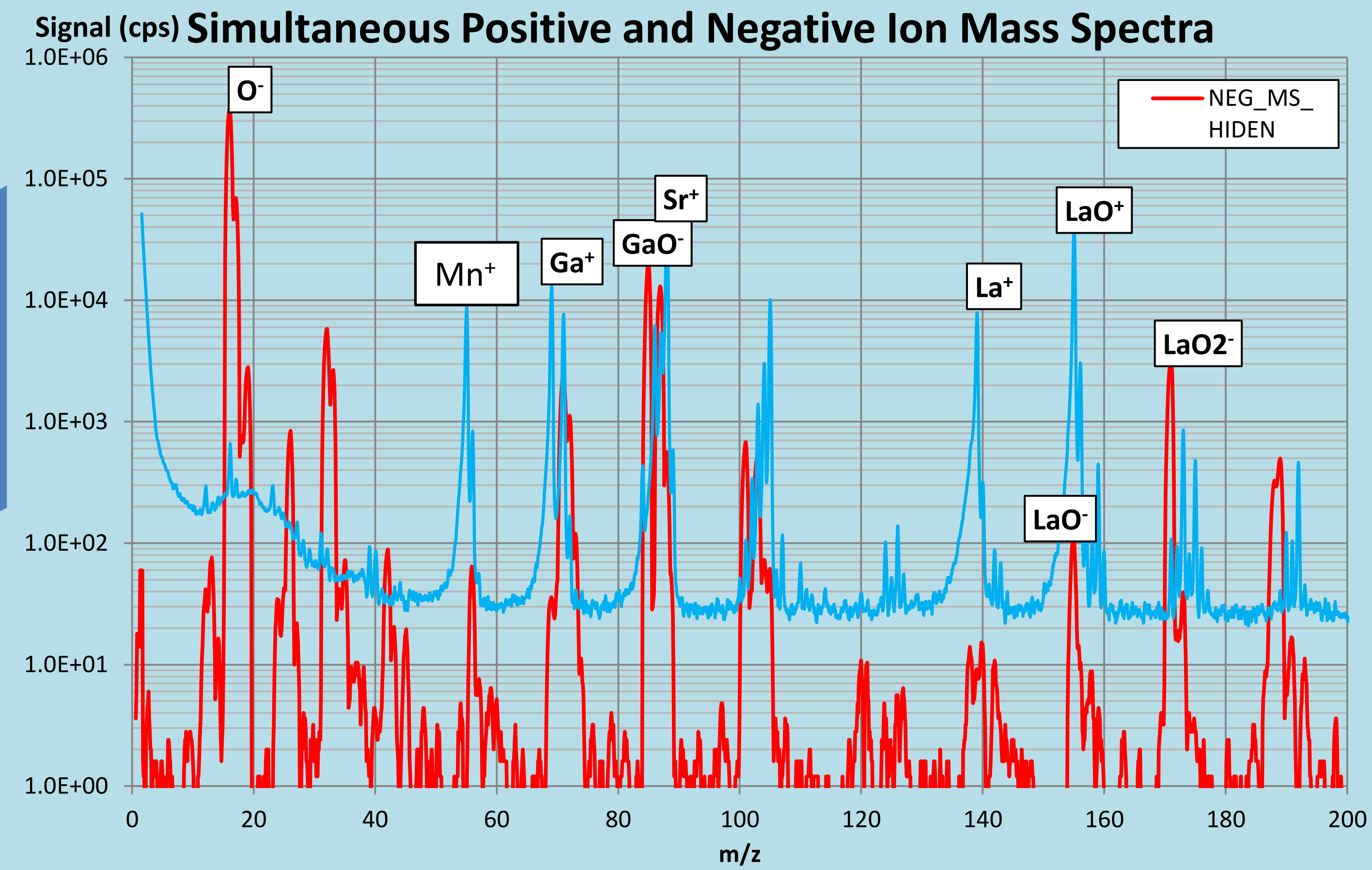
Simultaneous detection of positive and negative secondary ions in the single beam FEI FIB200 SIMS instrument

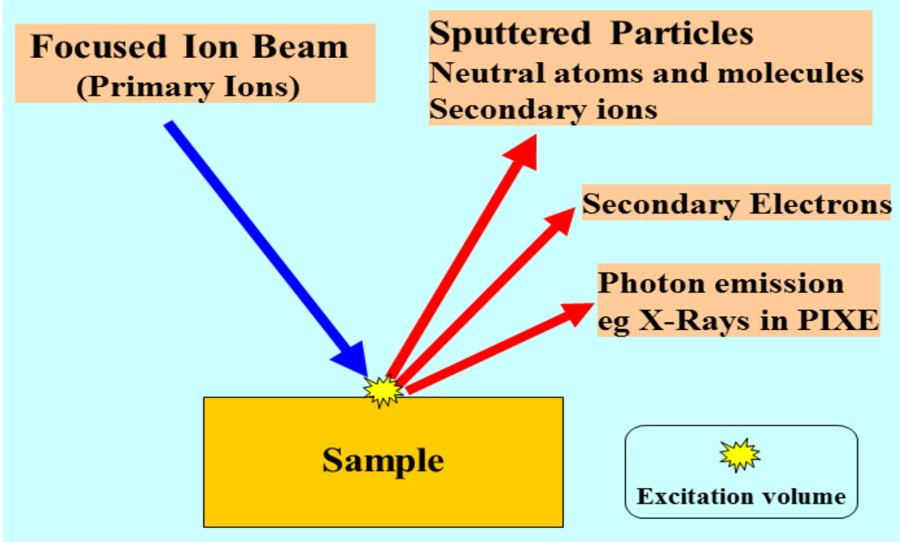
Lanthanum strontium Manganate target

Positive SIMS_ FEI SIMS detector

Negative SIMS_HIDEN SIMS detector

- Gallium FIB ion beam at 30keV and 20nA into a crater of 50 microns square on the surface of the target.
- Chamber pressure during sputtering was 7.3 x 10^{-7} mbar using a water vapour leak into the chamber for positive ion enhancement.
- Two separate mass spectra results are shown in the chart with simultaneous SIMS detection whilst the single crater was sputtered into the target.





Particle emission for low energy ion impacts

♦ Positive and negative secondary ion detection using both the Hiden SIMS probe and the Zeiss SESI detector.

♦ Secondary electron detection using inlens secondary electron detectors within the Zeiss electron column and the Zeiss SESI detector.

♦ X-Ray emission detector using standard Oxford Instruments SDD EDS/EDX detector.

Discussion

- Feature selection is often best achieved by exploiting a range of well established and very familiar analytical techniques that are standard on SEM instruments.
- Correct sample mounting and orientation in different instruments from different manufacturers can be very difficult. Finding micron-sized features on transfer between instruments can be very time consuming.
- Recent developments in low energy electron beam columns now allow for both electron and ion beams to have approximately the same excitation volume on the sample surface by correct selection of electron energy and ion energy. This leads to imaged data at nanometer resolution that is directly comparable. SIMS compositional information can be matched to the topographical and structural information from secondary electron and EBSD imaging.

Conclusions

- The added SIMS analytical facility to both the two beam and single beam microscopes has demonstrated its potential in a very short time since its installation in late July 2013.
- SIMS facilities alongside the more mainstream analytical techniques available on most two beam microscopes ensures an increased access and awareness of the technique.
- Potential for simultaneous positive and negative SIMS has been achieved.

Acknowledgements

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