Simultaneous detection of positive and negative secondary ions

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Motivation

- Simultaneous positive and negative SIMS for high spacial resolution surface imaging.
- Application to small feature dynamic SIMS analysis [1].

Instrumentation

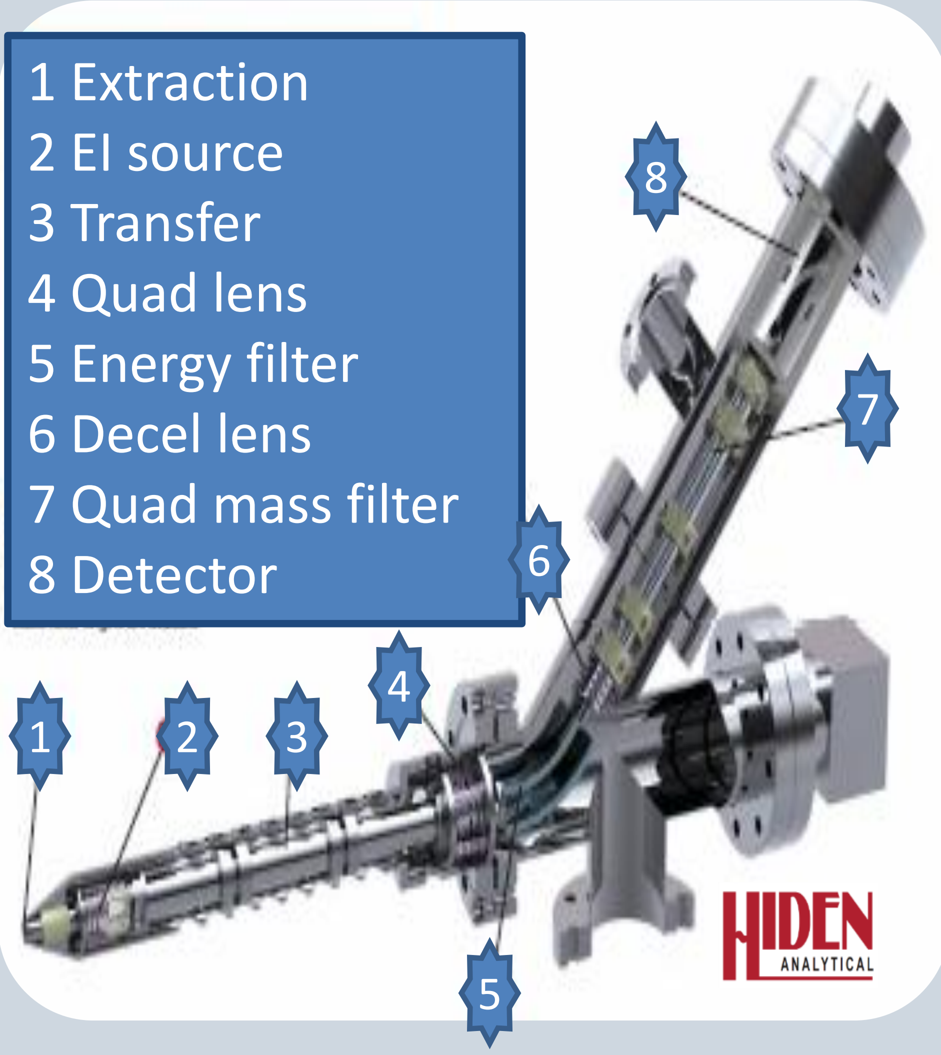
- A single beam gallium ion microscope, FEI FIB200 with a quadrupole-based SIMS detector
- The microscope has been fitted with a second SIMS detector, a Hiden EQS 1000, which is also quadrupole-based but is separately and differentially pumped [2].

Modelling

- The ray-tracing program , SIMION 3D [3], is used to model the instrument configuration.
- Secondary ions produced by sputtering a conducting oxide material are used to model secondary ion trajectories.

References

1. '*Environmental fatigue crack nucleation in Ti-6Al-2Sn-4Zr-6Mo*'. Submitted for publication in Corrosion Science, Aug 2014
2. Presented at SIMS 19, Jeju, South Korea 2013 and accepted Aug 2014 for publication in SIA, DOI 10.1002/sia.5665.
3. Charged particle optics simulation software program, SIMION 3D is available from SIMION.COM



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)

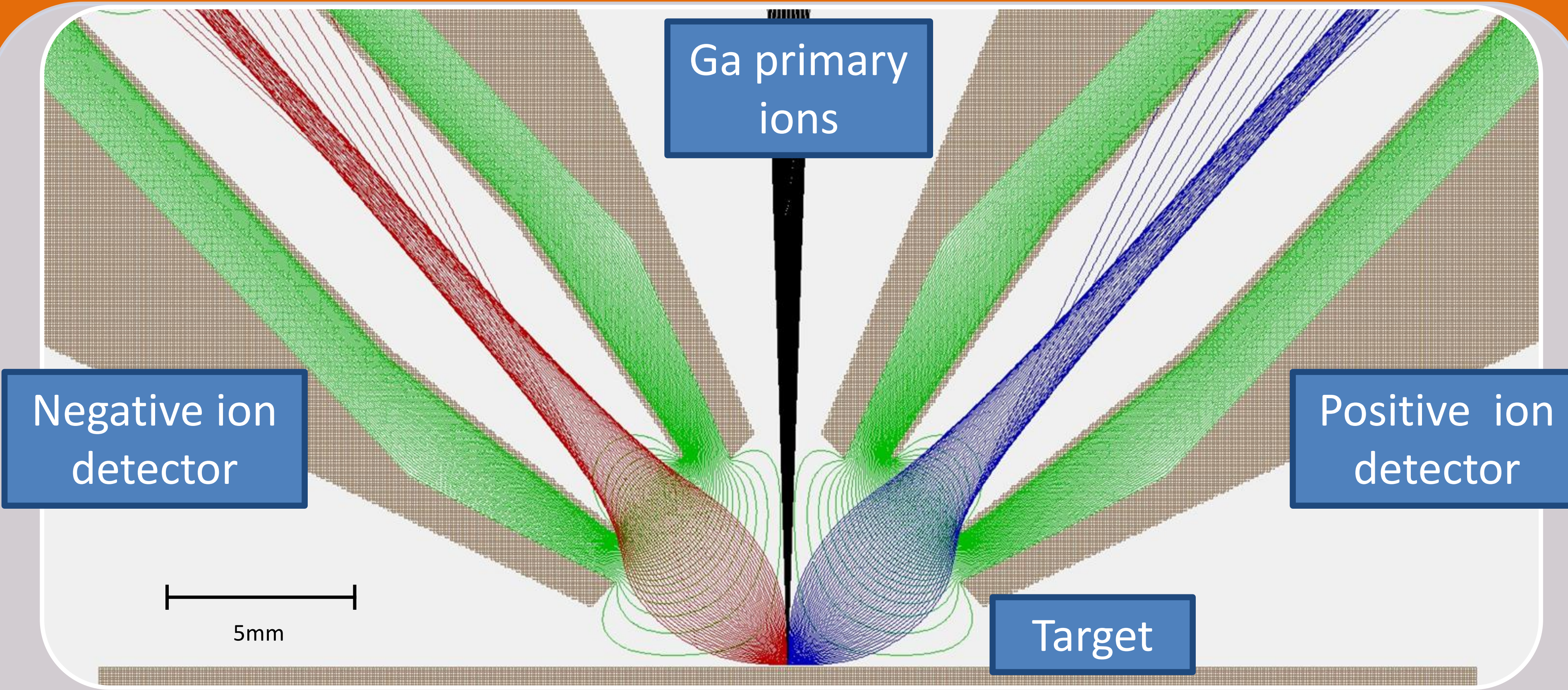
FEI FIB200 SIMS

♦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 but without an electrostatic analyser in the secondary ion column .

♦SIMS measurements generate spectra, depth profiles or maps.



Legend

Positive Ions —
Ga Primary Ions —
Negative Ions —
Equipotentials —

SPN Modelling in SIMION

♦ Configuration for SPN with two Hiden detectors is modelled for ray tracing of secondary ions sputtered by a normally incident gallium primary beam (black lines).

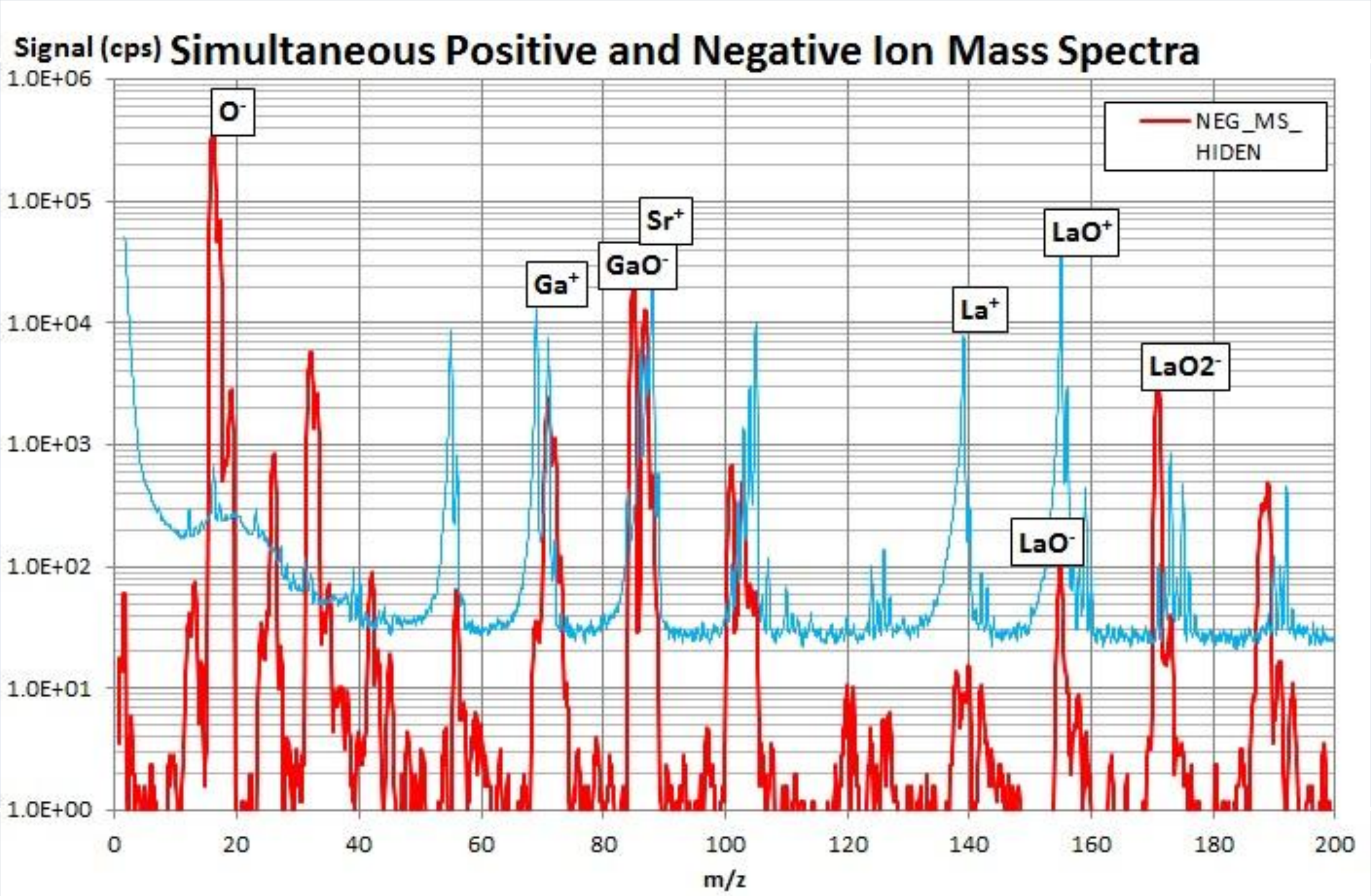
♦ SPN detection shown by trajectories of negative sputtered ions (red lines) and positive sputtered ions (blue lines) from the sample target into the SIMS detectors.

♦ The secondary ions have an angular distribution of ± 45 degrees from normal.

♦ The sample bias is 0V and the initial secondary ion energy is 5eV with 100V extraction potential (green lines) with equipotentials at 1V steps showing clearly the low extraction field strength

♦ Sample surface can be tilted and rotated to reflect the motion limits of the stage and to optimise the secondary ion collection efficiencies for both positive and negative ions.

♦ Sample Modelling SPN in SIMION allows insights into the way in which the ions are extracted and will allow a closer examination of the way in which the geometry has an influence on the efficiency of the ion collection.



Simultaneous detection of positive and negative secondary ions (SPN)

♦ Lanthanum strontium manganate ($La_{0.8}Sr_{0.2}MnO_3$) oxide target, positive and negative secondary ion spectra.

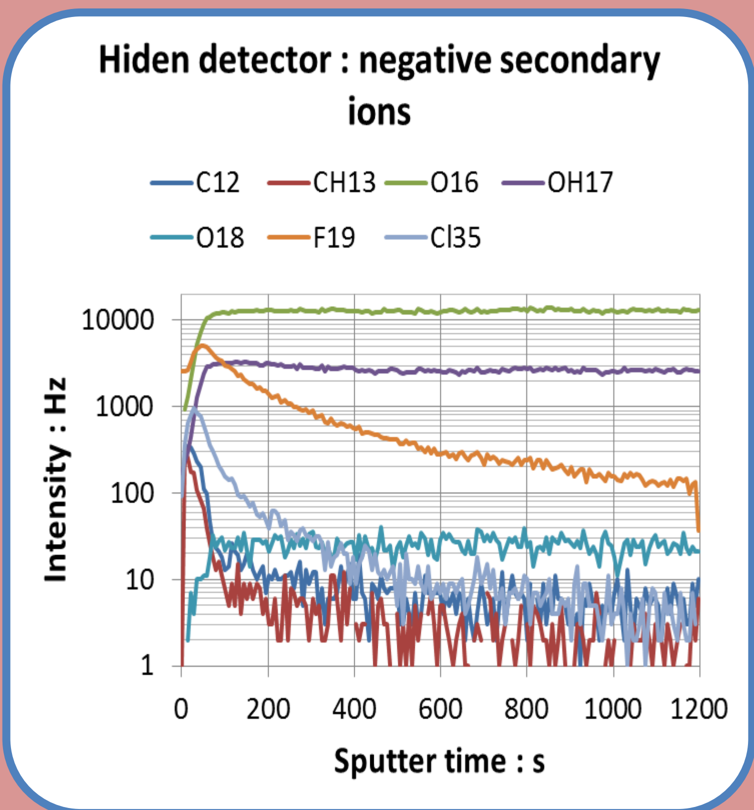
♦ Gallium FIB ion beam at 30keV and 20nA into a crater of 50 μm square on the oxide target.

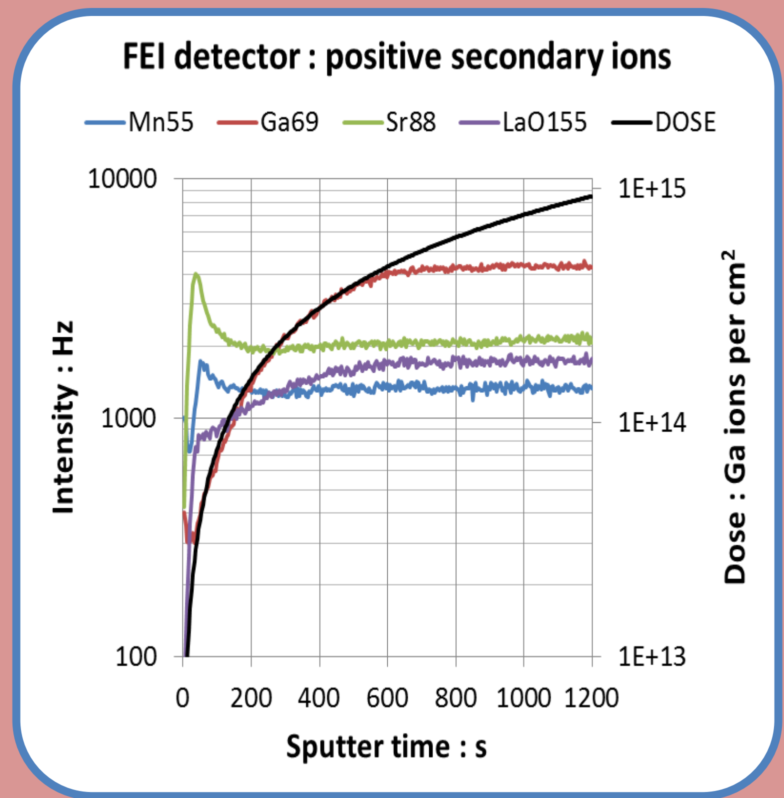
♦ Chamber pressure during sputtering was 7.3 x 10^{-7} mbar using a water leak into the chamber for positive ion enhancement.

♦ Two separate mass spectra results are shown in the chart with simultaneous SIMS detection whilst the single crater was sputtered into the target.

♦ Hiden SIMS detector for negative ion spectrum, FEI FIB200 SIMS detector for positive secondary ions.

♦ Hiden SIMS detector for negative ion spectrum shows a background count-rate that is uniformly zero over the full mass range compared to the FEI SIMS detector.

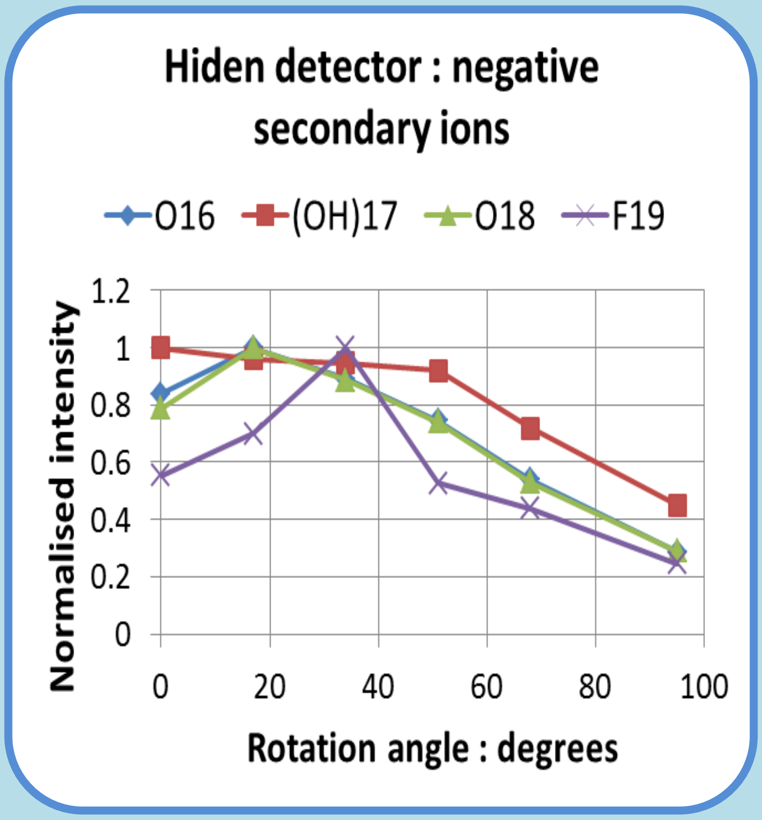


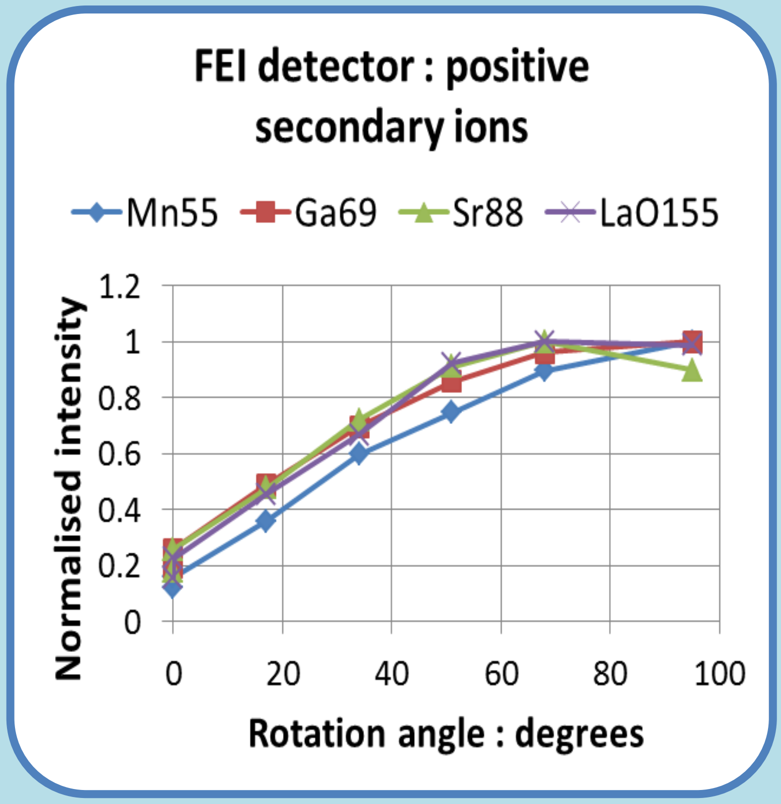


Pre-equilibrium sputtering region for Lanthanum strontium manganate (LSM) oxide target

- Gallium ion sputtering at 7keV, 1.29nA of the conducting polycrystalline oxide $La_{0.8}Sr_{0.2}MnO_3$. SRIM estimates the ion range as 5.3nm, straggle 2.6nm.
- Gallium positive secondary ion yield achieves a steady state yield after 600s of sputtering at a dose of 4 x 10^{14} gallium ions per cm^2. The molecular ion $^{155}(LaO)^+$ is the same.
- The atomic ions $^{55}Mn^+$ and $^{88}Sr^+$ achieve steady state sputtering after 200s of sputtering, dose 1.6 x 10^{14} gallium ions per cm^2.

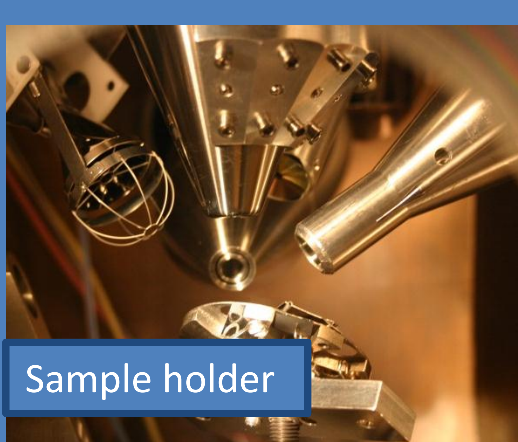
- Negative atomic oxygen ions $^{16}O^-$ and $^{18}O^-$ achieve steady state sputtering after 80s of sputtering, dose 6.4 x 10^{13} gallium primary ions per cm^2.
- The natural abundance for oxygen-18 is 0.00204. The value measured is (0.002±0.0004) at each point in the profile after 80s of sputtering. The PIDD for each point in the profile is 5.6 x 10^{12} gallium primary ions per cm^2.
- The oxide surface is covered with a thin hydrocarbon contamination layer.
- SBE for Sr is 1.7eV, for Mn is 3.0eV and for La is 4.42eV from SRIM.
- Sputter yield for La 0.73, Sr 0.47 and Mn 1.43



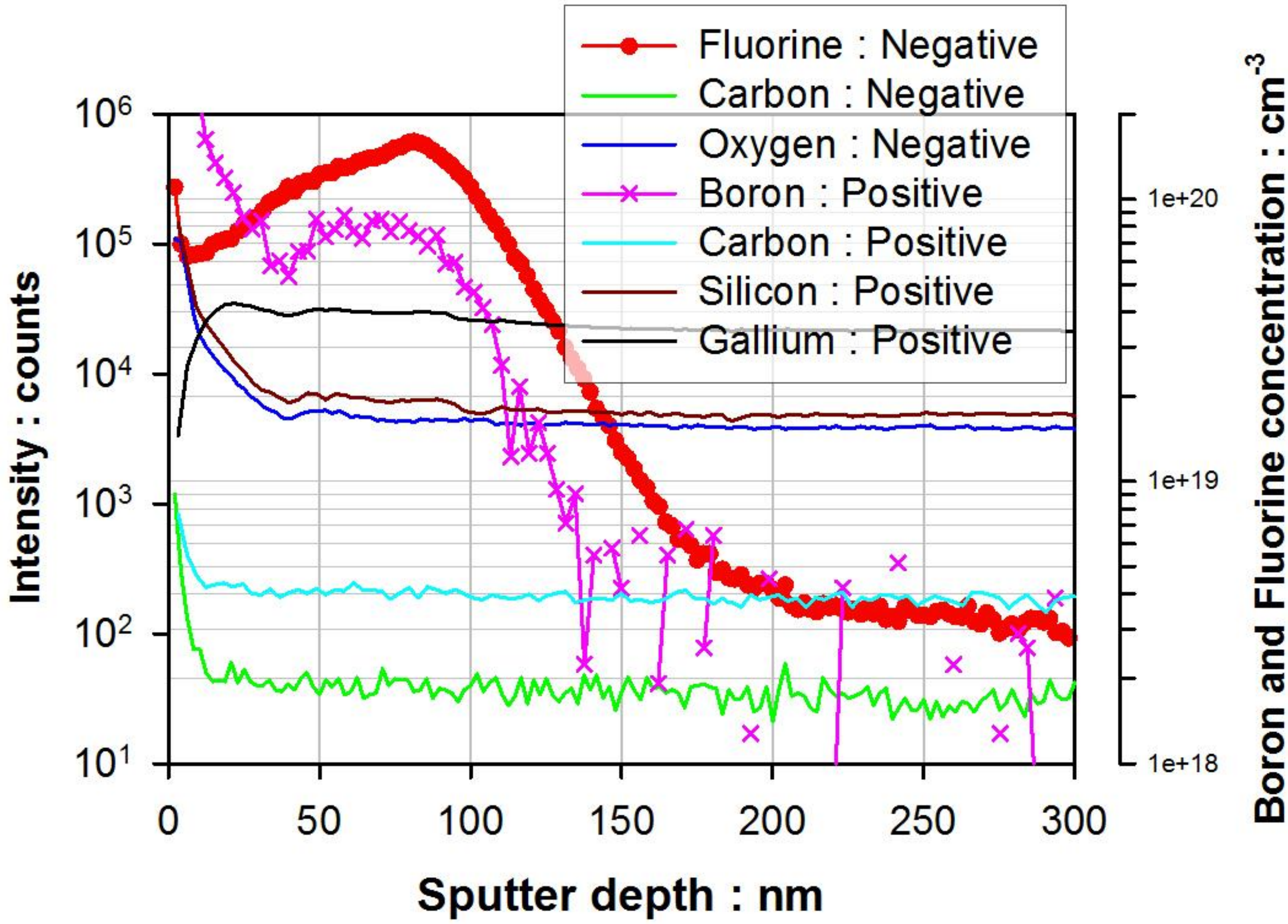


Optimisation of sample orientation between detectors

- Sample was rotated with a fixed tilt between the two detectors
- Sample holder shown with a tilt of 30° at angle of 31° away from Hiden detector.



- LSM oxide sample sputtered with gallium primary ions at 30keV and 3.28nA.
- Graphs show normalized yields of several atomic and molecular matrix secondary ions as sample holder is rotated away from facing the Hiden SIMS detector at 0° rotation angle.
- Optimization of the performance for SPN detection involved a slight loss of signal of between 80 and 90% compared to single detector operation.



BF_2 ions at 70keV implanted into silicon

♦ SPN SIMS analysis shows both boron and fluorine peaks.

♦ Sputter depth profile with FIB gallium ion beam at 30keV with a range of 27nm in silicon. Surface carbon and oxygen contamination layers are mixed into the substrate.

♦ Boron and fluorine concentration based on the estimates of peak concentration from SRIM. No attempt made to estimate boron enhancement in the surface oxide or the sputter-rate change.

♦ Two separate mass spectra results are shown in the chart with simultaneous SIMS detection whilst the single crater was sputtered into the target.

Conclusions

- Simultaneous positive and negative SIMS has been achieved using two quadrupole-based SIMS detectors.
- With the SIMS detector configuration fixed, optimization of the sample position involves very little reduction in collected secondary ion intensity.
- The added SIMS analytical facility to a single beam microscope has demonstrated its potential in a very short time since its installation in late July 2013.