Combined SIMS and SNMS
- a method for quantification in complex matrices

G.A. Cooke, C. Robertson, S. Davies, C.L. Greenwood, D.L. Seymour, S. Doughton and T.H. Russell

Plasma and Surface Analysis Division, Hiden Analytical Ltd, 420 Europa Boulevard, Warrington, WA5 7UN, UK Tel: +44 (0)1925 445 225

INTRODUCTION

Secondary Ion Mass Spectrometry (SIMS) and Sputtered Neutral Mass Spectrometry (SNMS) are both surface analysis techniques in which the specimen is bombarded by an ion beam and sputtered particles are detected by a mass spectrometer. The critical difference between the techniques is that the SIMS ions are generated directly by the sputtering process, whereas those detected in SNMS are produced by ionisation of neutral particles some time after leaving the surface. The separation of the sputtering and ionisation events in SNMS results in a near-constant ionisation probability for each element, irrespective of the material from which it originated. This is in contrast to the well known matrix effect of SIMS [1] where the ionisation probability during sputtering can vary by orders of magnitude, depending on the surface chemistry.

This also means that the SIMS quantification requirement for matrix matched reference materials is absent from SNMS.

The techniques are quantified in different ways and are complimentary in the concentrations that they monitor, with SNMS covering 100% to 0.1% atomic concentration, and SIMS typically providing quantification in the range 3% to below 10^{-6}%.

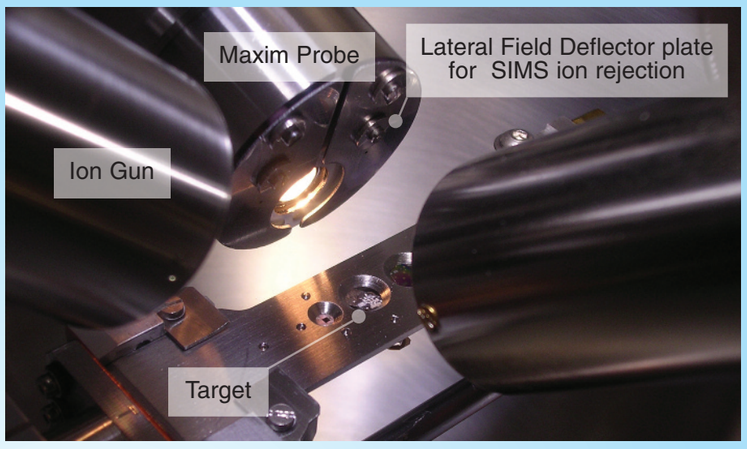
The aim of this work is to demonstrate that SIMS and SNMS data may be recorded from *within the same depth profile* in a quasi-simultaneous way, such that the SNMS may be used to quantify the matrix composition at each point which can then be use to provide accurate quantification of the impurity, by application of the SIMS RSF relevant to the alloy.

Hiden SIMS Workstation

- designed around high sensitivity quadrupole SIMS analyser (Hiden MAXIM)
- fitted with an electron impact ionisation cel
- efficient rejection of SIMS ions during SNMS analysis by means of lateral electric field
- rejection of residual gas ions generated in the cell by ion energy analysis
- automatic tuning of secondary ion column
- rapid switching between detection modes
- positive and negative ion SIMS
- sputtered neutral mass spectrometry
- residual gas analysis

SNMS MODE

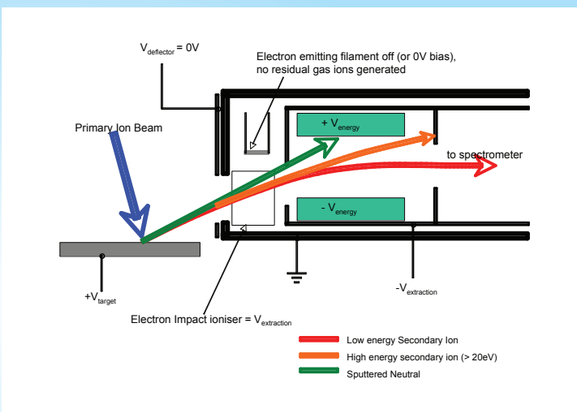
An electron impact ionisation cell is located very close to the front of the MAXIM probe in order to subtend the largest solid angle from the sampling point for the collection of neutral particles. Typically an electron energy of 40 eV is used in order to reduce the generation of ESD signals, for some elements 20 eV is required. However, there is still significant ionisation of the residual gas even at UHV pressures. The two signals, sputtered neutrals from the sample and those generated by the residual gas may easily be separated by the significant difference in their energy distributions, with most of the sputtered neutral having energies in excess of 5 – 10 eV and those of the gas below 1eV. This is accomplished by biasing the electron impact cage and use of the parallel plate filter. SIMS ions are rejected by biasing the target and application of a lateral field at the entrance to the probe, forcing them away from the spectrometer axis.

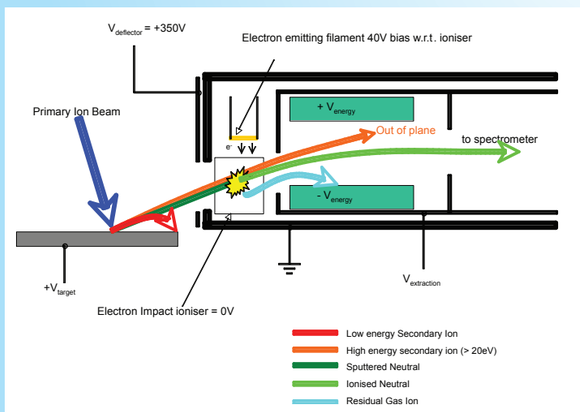


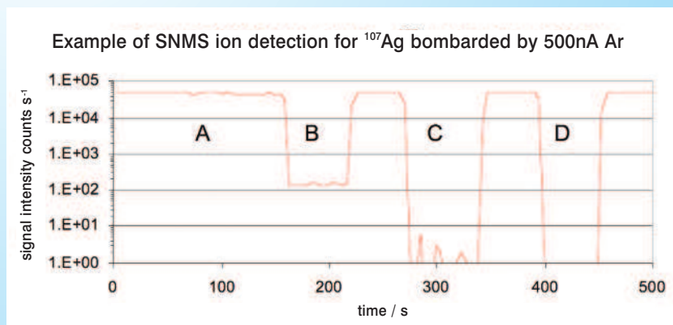
SNMS analysis with election impact ioniser active

SIMS MODE

For the analysis of secondary ions (using positive ions as an example - polarities are reversed for negative ions) an extraction potential, typically of the order of -20 to -60V is applied to the internal structure of the probe, including the ionisation cage. This generates an extraction field protruding from the entrance aperture which captures ions from the target. A positive target potential assists in this process. Ions are collected from an off-axis position and the parallel steering plates sited immediately behind the cage act as an energy filter directing ions of 5 -10eV ejection energy onto the axis of the quadrupole mass spectrometer. The filament is not required for SIMS.







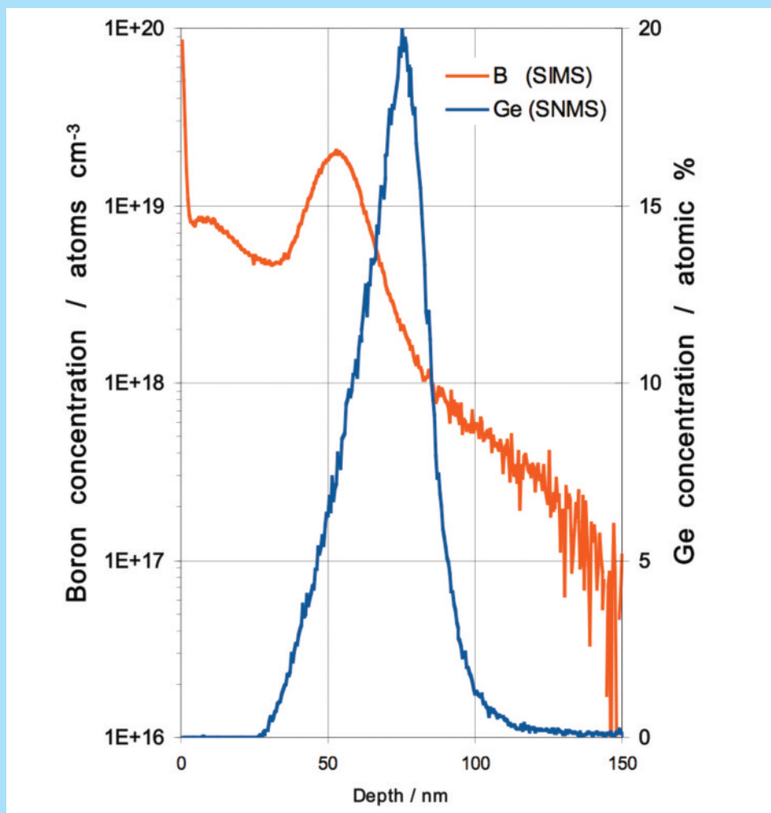
A - SNMS signal from ^{107}Ag sample
B – filament off <0.3% of the detected signal is due to secondary ions
C – Ion gun off background signal from residual gas
D – Filament and Ion gun off, no background signals.

SIMS

- SIMS signals vary non-linearly with the matrix composition (matrix effect), often over orders of magnitude.
- accurate quantification is possible when the impurity (dopant) is dilute within a uniform matrix (<3%).
- Quantification requires matrix matched reference material to provide a relative sensitivity factor (RSF).
- Using SIMS to accurately determine the matrix (alloy) composition is not possible in the majority of materials.
- Detection limit can be low ppb.

SNMS

- Ionisation is separated from sputtering – minimal matrix effects
- RSF is constant so specialist reference materials are not required, easily available alloys will suffice.
- SNMS signals vary linearly with composition and quantification methods are essentially simple
- Detection range is typically 100% to 0.1%
- Interference from molecular species is dramatically lower than with SIMS.



SIMS and SNMS data collected during the same analysis

Combining SIMS and SNMS in the same analysis

The Hiden MASsoft spectrometer control software provides an extremely flexible flowchart style control of all of the relevant voltages permitting SNMS and SIMS conditions to be swapped in approx 500ms and making it realistic to collect both sets of data within the same depth profile analysis. The sample used in this example has a graded SiGe layer of approx 20% Ge peak concentration and includes boron doping. Analysis was undertaken using 5 keV O_2^+ primary ions bombarding at 45° in order to provide a good sensitivity for the boron layer.

Conclusions

- Combined SNMS and SIMS detection in the same analysis has been demonstrated.
- SNMS can determine the local matrix composition so that correct SIMS RSFs can be applied (this can even be done on a point-by-point basis using a mathematical fit to the change in RSF with composition).
- SNMS can provide quantification above the dilute limit where the matrix effect prevents accurate application of SIMS.
- Using a sample with composition across the detection boundary of the two techniques permits the SIMS RSF to be determined quickly and easily without manufacture of a special reference material.

References

1. **SIMS: A Practical Handbook for Depth Profiling and Bulk Impurity Analysis**, R.G Wilson, F.A.Stevie and C.W.Magee, John Wiley and Sons NY 1989

For Gas Analysis, Plasma Characterisation, Catalysis and Surface Science

Tel: +44 (0)1925 445 225 Fax:+44 (0)1925 416 518 E-Mail: info@hiden.co.uk www.HidenAnalytical.com