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A Tour of the Hiden SIMS Workstation

– a cost effective materials analysis solution

Introduction

SIMS

Secondary ion mass spectrometry is the most sensitive of the generally available surface analysis techniques and finds application in a wide range of industrial and research settings.

- **semiconductors**
- **coatings technology**
- **fuel cell development**
- **photovoltaics**
- **metallurgy**
- **pharmaceuticals**
- **nuclear**

- **parts per billion sensitivity**
- **imaging chemical distributions**
- **depth profiling**
- **3D chemical mapping**
- **diffusion and dopant analysis**
- **isotopic analysis**

Why not try SIMS?

Despite the undoubted benefits of this extremely powerful technique, SIMS is often perceived as difficult and expensive, frequently regulating it to be a technique of last resort. This attitude has arisen because early SIMS instrumentation was highly complex and expensive both in terms of capital and running costs and also required PhD level staff to operate and interpret it. However, just as the complexity of early electron microscopes has given way to user-friendly semi-automated instruments, so the essence of SIMS analysis has become more refined. In this article we will take a tour around the Hiden SIMS Workstation, a complete, highly flexible general purpose SIMS tool, carefully designed for ease of operation and low cost of ownership – so SIMS can now be a frontline technique for everyone.

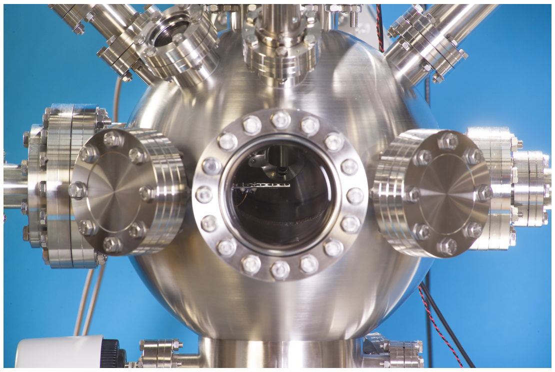
Hiden SIMS Workstation

Vacuum – *efficiency and flexibility*

The SIMS process relies on the ability of the primary ions to interact with the specimen and generate secondary ions that reach the detector. Operation in the ultra-high vacuum (UHV) regime prevents interactions with the residual gas.

The SIMS Workstation runs at UHV pressures (typically 1E-9 torr) and is based around an 18 port, spherical, turbomolecular pumped chamber. The ion guns and loadlock are each equipped with their own turbomolecular pumps and vacuum gauges, ensuring that the main chamber is at optimum cleanliness. Vacuum interlocks ensure safe operation and power supply protection.

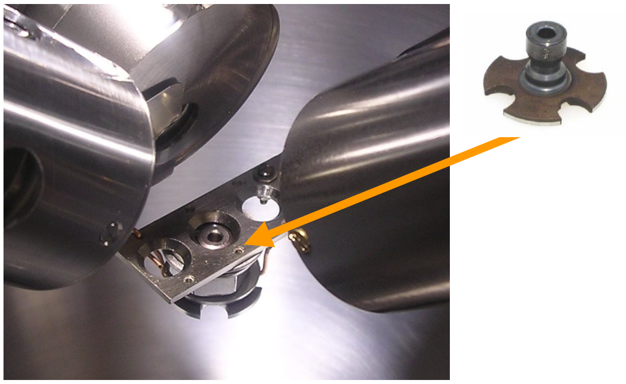
The entire system is fully bakeable and uses industry standard Conflat® type seals. There are a number of unused ports designed to permit the system to be easily reconfigured or for customers to add their own devices specific to their requirement, such as lasers, heating, cooling or mechanical attachments, X-ray and electron guns etc. The SIMS workstation is designed to be flexible and upgradeable, an instrument for tomorrow as much as today.



Analysis Chamber

Sample Handling

SIMS analysis was (and still is) heavily used by the semiconductor industry and this has always been noticeable in the sample stages provided for commercial instruments. However, most other customers do not have thin, perfectly planar, easily cut samples. In the real world it would be a great advantage to analyse entire components or industry specific test pieces. The sample stage and handling in the Workstation is designed around this philosophy. Firstly, the entire analytical system (ion guns and spectrometer) are above the plane of analysis, meaning that large and awkwardly shaped test pieces can be accommodated (Hiden can also design a customer specific chamber if required to take very much larger than normal specimens). The standard specimen holders are based on a flat plate with wire springs and these are easily modified to take a wide variety of samples, from small flat plates to standard embedded metallurgical samples. Entire small components, such as the fuel injector below, can be attached without modification. This is especially important where cutting may cause contamination or be very time consuming.



Fuel injection component mounted for analysis

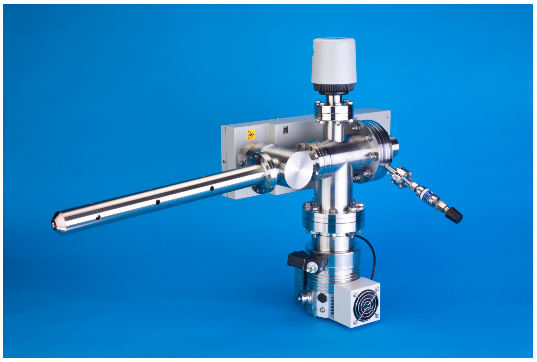
Samples are introduced to the main chamber via a turbomolecular pumped loadlock and UHV sealed magnetically coupled arm. This ensures that the main vacuum is not compromised and provides rapid transit from air to UHV. The standard holder provides for 5 or 10 small samples mounted behind 6mm diameter windows. Samples mount from the rear so that the front face is always correctly positioned for analysis.

Analysis Components

Having transferred the sample to the main chamber under UHV, SIMS analysis can now proceed. The standard complete Workstation is equipped with two ion guns, both introduced through 35 mm ports. The IG20 gas ion gun is used for bombardment with oxygen or inert gases, and the IG-5C caesium ion gun produces a focussed beam of caesium ions. At the heart of the whole system lies the MAXIM quadrupole SIMS/SNMS analyser that is responsible for the excellent sensitivity of the instrument.

IG-20 Gas Ion Gun

The IG20 is a highly versatile gun producing an intense focussed ion beam. Ions are produced by electron bombardment of gas admitted via a precision leak valve. For most SIMS analysis the gun is used with oxygen gas, providing the highest possible sensitivity for electropositive elements.



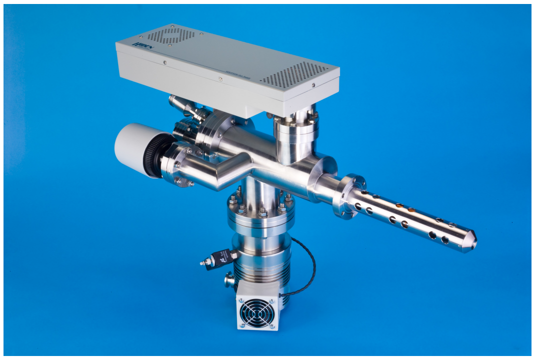
For sputtered neutral mass spectrometry, where enhancement of the ion yield is not required, the IG20 is run with argon. Gas consumption is low and a standard high pressure pure gas bottles will last many months, supplying gas to the leak valve via a regulator set at approx 0.5 bar.

The IG20 ion optical column comprises two three-element Einzel lenses, beam alignment stages, a bend to remove neutral particles and beam steering stages for scanning during depth profiling and imaging.

The beam steering electronics fit directly onto the ion gun feedthrough, removing the need for large multiway cables – a frequent source of unreliability on some systems.

IG-5C Caesium Ion Gun

The IG-5C opens up sensitive analysis of the electronegative elements as well as MCs^+ cluster detection (where M is the element of interest). Ions are generated by a miniature low power thermal contact ionisation source, manufactured by Hiden, which uses safe caesium salts. The source may be vented to air when still warm (a few minutes after operation ceases) and is user changeable in a few minutes (source lifetime is estimated to be 500 hours of operation).



The ion gun is of a two lens design with two sets of independent alignment stages. The upper stage is positioned immediately after the source to ensure that the emerging beam is optimised as soon as possible onto the optical axis. The beam passes through a defining aperture that is easily serviceable by the end user, either for replacement or to modify the beam characteristics. The lower alignment stage incorporates a double bend to remove neutrals and approximates two small-angle electrostatic sectors. The ions are finally focussed by a low spherical aberration lens and scanned by a group of four short plates. The thermal management of the IG-5C is provided automatically via the PC controlled ion gun interface unit.

Ion Gun Control


Hiden Ion Gun Controller

Filament 1 ON: 2.1 A
Emission: 50.0 mA
E-Energy: 70.0 V
Ion-Energy: 5000 V
Condenser: 2455 V
Objective: 2564 V
Upper-X: 53.5 %
Upper-Y: -2.4 %
Deflection-X: 0.0 %
Deflection-Y: 0.0 %
Span-X: 14.2 %
Span-Y: 5.9 %
Value: 2455.0
Settings: IG20 500nA 100µm spot
Instrument state: Normal: Running
Monitor: Total
Detector FSD: 100uA

This new generation ion gun controller is microprocessor based and is accessed via a very intuitive PC interface designed by an instrument user.

Using the controller it is possible to store and recall all ion gun settings. This makes life for the analyst very easy as a click of the mouse can switch between settings for

- high current depth profiling
- low current fine focus imaging,
- low energy high depth resolution profiling
- large area sputter cleaning
- other customer specific tasks

Saving settings during tuning also permits experimentation without loosing your best set-up!

Diagnostic modes are instantly available with beam parameters (current and beam shape) measured on an electron suppressed faraday collector and displayed live on the PC.

PC Ion gun control interface

A useful feature is the automatic switching of the target bias between modes which ensures that the correct bias is applied when running analyses or diagnostics.

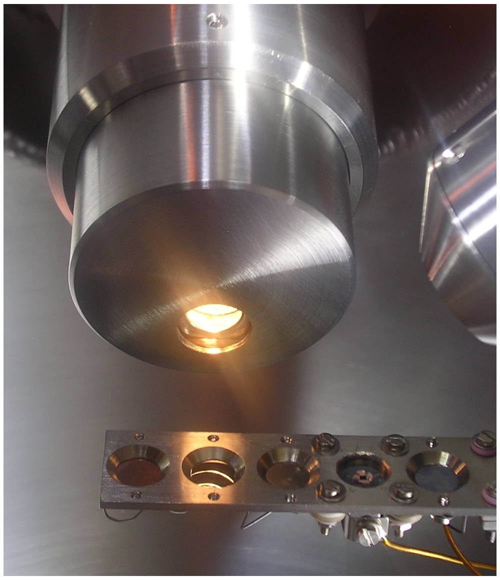
MAXIM Spectrometer

The MAXIM SIMS/SNMS spectrometer is mounted on the back of the chamber in an off axis position and has been specifically designed to give sensitive, reproducible, analyses. Ions are collected from the sample by a shaped extraction field and energy filtered using a parallel plate system, with the energy resolution matched to that of the quadrupole analyser. The 9 mm triple filter has molybdenum rods for thermal stability and the pulse counting detector has a 4 keV post acceleration potential to increase efficiency at high mass. The spectrometer is available in mass ranges of 300u, 510u and 1000u for different applications.

Immediately behind the extractor is a high efficiency electron impact ion source which can be used for either sputtered neutral mass spectrometry (SNMS) or residual gas analysis.

Entrance to the MAXIM

SNMS – *crossing the boundary of quantification*



MAXIM running SNMS analysis

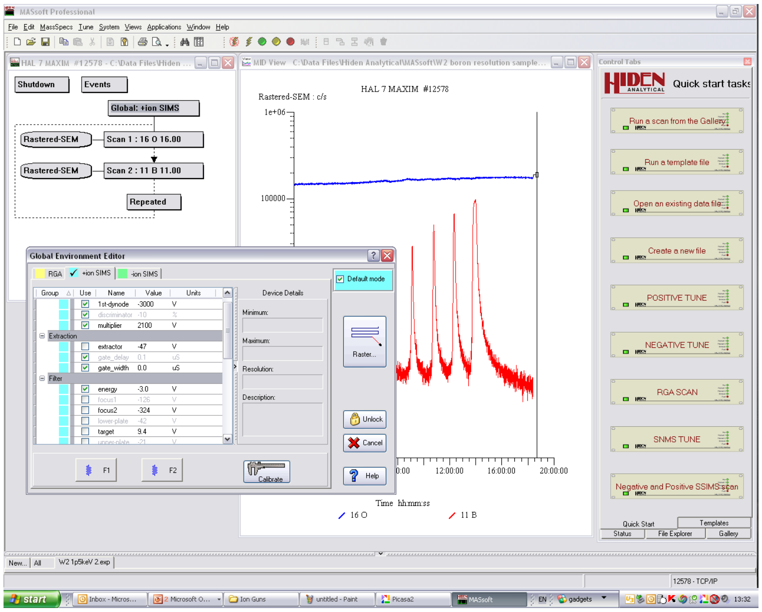
SIMS analysis is highly sensitive and ideal for the analysis of dopants, diffusion studies and contamination. Quantification schemes for SIMS generally assume that the impurity being measured is dilute (less than a few percent). Above this *dilute limit* the probability of ionisation becomes dependent upon the impurity concentration itself, as well as the chemistry of the matrix. Sputtered neutral mass spectrometry (SNMS) neatly overcomes this limitation.

Firstly, most ions from the specimen (the SIMS signal) are rejected by suitable target and deflection potentials. Next, the neutral particles drifting from the sample are ionised in the electron impact source; they are separated from residual gas ions, also forming, by their kinetic energy which is significantly higher. As ionisation occurs away from the sample surface the ionisation probability is always the same, thus the SNMS signal is easy to quantify.

Instrument Control – taking out the guesswork

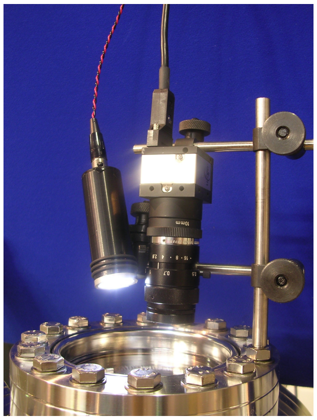
The instrument is controlled using the Hiden MASsoft software suite. This provides automatic efficient and reproducible optimisation of the secondary ion column and spectrometer – taking out the guesswork. If required all of the parameters may also be manually tuned. MASsoft provides a very powerful set of spectrometer, rastering, gating and data handling controls, with a simple clear process flow tree. A facility is provided so that the amount of control may be limited, with a few options for the inexperienced user right up to full control for those wishing to experiment.

Usefully, commonly undertaken tasks (like column tuning and spectrum collection) may be set as simple user programmable *quickstart* buttons, enabling an analysis to be initiated by a single click of the mouse.



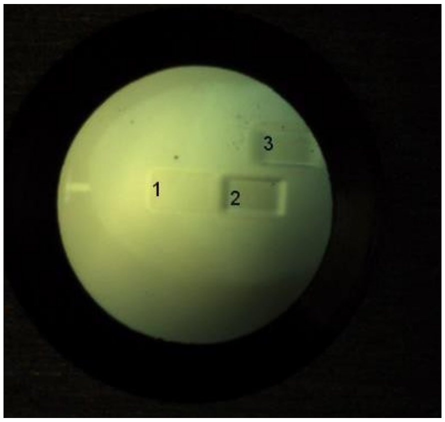
Group	Use	Name	Value	Units
	☑	1st-dynode	-3000	V
	☑	discriminator	-10	%
	☑	multiplier	2100	V
Extraction				
	☐	extractor	-47	V
	☑	gate_delay	0.1	uS
	☑	gate_width	0.0	uS
Filter				
	☑	energy	-3.0	V
	☐	focus1	-126	V
	☐	focus2	-324	V
	☐	lower-plate	-42	V
	☐	target	9.4	V
	☐	upper-plate	-21	V

Sample viewing



Viewing system

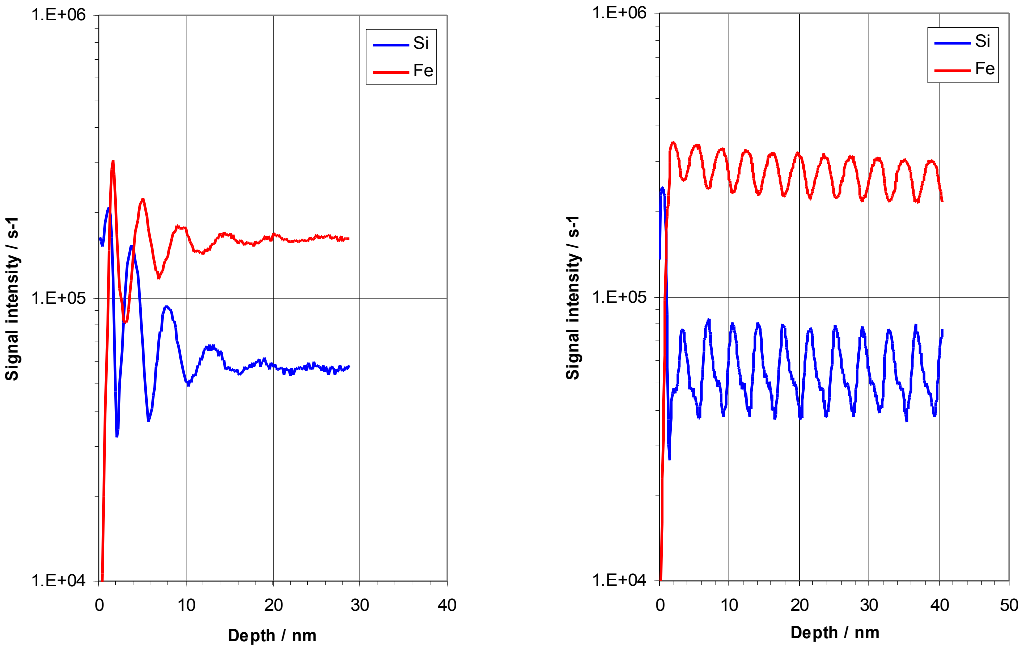
It is often overlooked but a clear view of the sample before and during analysis makes the analyst’s job significantly easier. It allows areas to be accurately and confidently targeted. In some cases colour changes during analysis of thin films provide added information about the erosion rate and consistency. The workstation is equipped with a normal incidence colour CCD camera and LED cold light source, giving a clear view of the sample surface.



Sample view of analysis craters.

Oxygen flooding

When making high depth resolution depth profiles, especially at low energy (below 2 keV) with oxygen, the surface is known to roughen and degrade the very resolution being sought. However, if the surface is made to fully oxidise during analysis the depth resolution is maintained. This can be achieved by either mounting the gun at normal incidence (very possible on the SIMS Workstation) or by flooding the sample area with oxygen. In the Workstation, a fine capillary brings oxygen gas to exactly where it is required, bringing about a drastic improvement in resolution.



2 keV O_2, 100nA 45° without Oxygen Flood

2 keV O_2, 100nA 45° with Oxygen Flood (4E-6 torr)

Effect of oxygen flood on analysis of 3.6nm Fe/Si neutron mirror

Conclusion

The Hiden SIMS Workstation has been developed with strong interaction with those used to performing SIMS analyses for both contract service work and research in many fields of analysis. The philosophy of design has been simple, to produce a proper SIMS analysis instrument which combines high sensitivity, flexibility and ease of operation, but also to make it cost effective both in terms of capital and ongoing costs.

The Hiden SIMS Workstation has achieved these aims, being the only mid-priced instrument to incorporate both oxygen and caesium ion guns – a necessity for general analysis. It provides a true UHV environment, flexible loadlock and sample handling system. The embedded SNMS facility ensures that the tool covers the full analytical concentration range, from ppm through to 100% bulk and the data system means that valuable results are easily stored and exported to other applications. It is easy enough to configure for production line analysis, yet will provide the dedicated research professional with a tool limited only by imagination.

SIMS is now a frontline technique for everyone.