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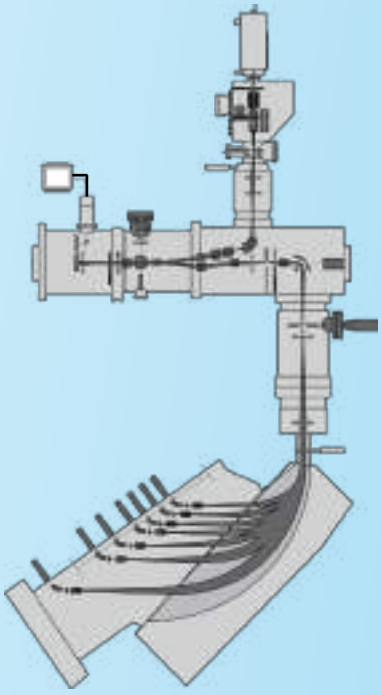


NanoSIMS 50L

Introduction to the instrumentation

Secondary Ion Mass Spectrometer
for trace element and isotope analysis
at sub-micron resolution

Design requirements for a Nanoscale SIMS microprobe

Fundamental limitation: SIMS sensitivity gets poorer as lateral resolution gets better

There are 1.25 E6 atoms in a silicon volume of 50 x 50 nm x 10 **nm** (one pixel). With an ionization of 0.005 and a transmission of 1, one can detect around 6250 atomic ions, or a detection limit around **0.1 at.%** level for one pixel. For comparison, using a CAMECA IMS SIMS with larger beam current will sputter 1.25 E12 atoms from 50 x 50 **µm** x 10 nm and be able to reach ar ppb level!

→ **COLLECTION & TRANSMISSION are CRUCIAL: no delayed extraction (low mass loss), no QUAD nor Ortho-TOF (low extraction), no collision cell or atm/vacuum interface (transmission).**

Ionization yield

Incorporation of Oxygen and Cesium in the first atomic layers is mandatory in order to enhance atomic secondary ion yield. Primary ion species like Ne, Ga, Xe, Au or Bi as used in FIB- or TOF-SIMS result in 100-1000x lower elemental signal. Flooding with Cs vapors would contaminate the instrument. Flooding with O_2 gas is not sufficient when using fast sputtering. Alternating implantation/analysis with two ion guns is possible at the cost of lost material during the reactive implantation, **unacceptable for very small feature analysis.**

Special case of biological SIMS

The yield of sec. molecular ions is very low, even using optimized projectiles (Ar-, CO_{2-} or water- massive cluster ions, C_{60} or Bi_3). Facing this fact the NanoSIMS approach for pushing the useful **lateral resolution** is then to intentionally **brake all molecules** of the surface by bombarding with energetic reactive ions (Cs+ and O-), detecting the ***more numerous resulting atomic ions*** and making use of isotope labeling by measuring isotopic ratios.

→ **The use of REACTIVE PRIMARY ION SPECIES (Oxygen and Cesium) is mandatory.**

Primary beam current density

In order to reduce contamination from the residual gas and detection noise one needs to sputter as fast as possible during the analysis. Hence not only the beam size but the reactive beam current density is crucial in defining the performance of a SIMS.

→ **Highest brightness reactive ion sources and shortest working distance are required to maximize the beam current density.**

Shadowing effects

The primary ion beam comes at inclined angle in all classical SIMS geometries. This induces primary shadowing of topographical samples and non symmetrical edge effect artefacts.

→ **A NORMAL geometry would be BENEFICIAL for both SHADOWING and ion COLLECTION.**

Mass Spectrometer Transmission

Due to the destructive nature of SIMS analyzing small features of a few tens nm in size requires detecting as many as possible of the very few emitted secondary ions, and as many targeted masses as possible in parallel. For optic aberration considerations the transmission of the ion micropobe mode is superior to the stigmatic microscope modefor sub-µm resolution.

→ **PARALLEL COLLECTION and Ion MICROPROBE mode are required.**

Mass Resolution and Lateral Resolution

High mass resolution is mandatory for small volume analysis (no second chance for another analysis elucidating possible mass interference !). Due to chromatic aberrations introduced by bunching the primary ion beam TOF-SIMS analysers with pulsed primary ion guns imply the choice between small sub-µm spot size OR high mass resolution. Delayed extraction implies a reduction of transmission for low masses and poorer mass scale accuracy.

→ **Need for High Mass Resolution *together with* High Spatial Resolution *and* High Transmission.**

Acquisition time and reproducibility

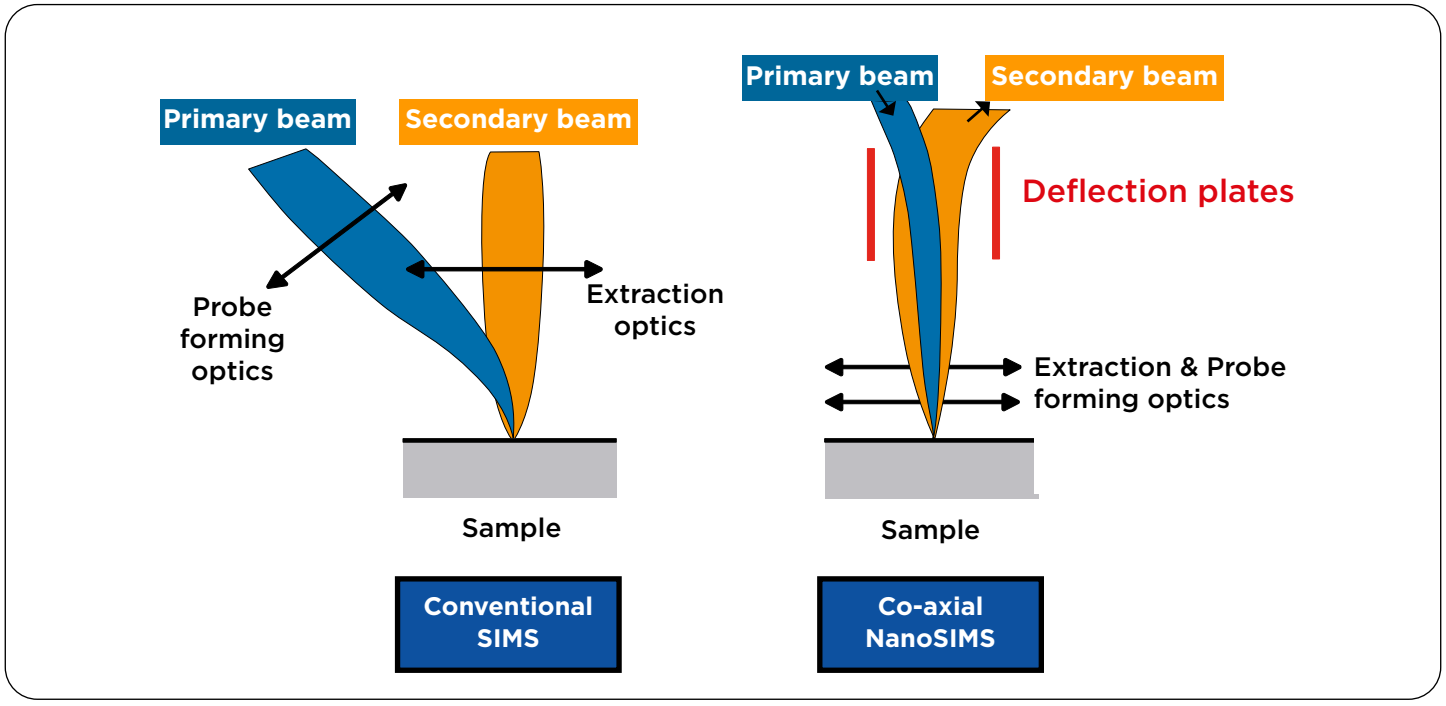
Precise isotopic data require good statistics and low noise (ex: 1 E-4 precision on a ratio requires statistically at least a few 1 E8 counts on the *minor* isotope). This requires DC sputtering mode to keep the acquisition time realistic and avoid side effects: drifts, contamination, noise, instabilities... Pulsed TOF-SIMS are limited by their low duty cycle and the detection principle (1 SI max per peak and PI pulse). Their reproducibility is limited to % level for many reasons (two beams, detector, data rate, pulsing, mass calib...). Magnetic sector analyzers working with DC beam and flat top peaks can reach tenth of permil isotopic *reproducibility* range.

The NanoSIMS is unique in enabling *simultaneously* small spot of reactive species, high mass resolving power *with* near full transmission, *and* DC beam for high throughput and lowest background & noise.

Conventional and co-axial probe forming systems

Any design of a SIMS instrument must accommodate two conflicting needs :

- The objective lens of the primary ion column must be as close as possible to the sample in order to optimize its optical properties, leading to the highest beam density (the highest beam density (highest beam current in the smallest spot size).
- On the other hand, secondary ions are emitted in a half-space, with a large energy spectrum (~ 0-200eV). In order to collect the largest fraction of these ions, the extraction optics should also be placed as close as possible to the sample. As the extraction and objective optics have their own physical size, a compromise must be found leading to large sample/optics distances. The NanoSIMS design has escaped from this dilemma by switching to a new co-linear optics capable of simultaneously focusing the primary ions with high quality and collecting most of the secondary ions.



Advantages of co-axial configuration

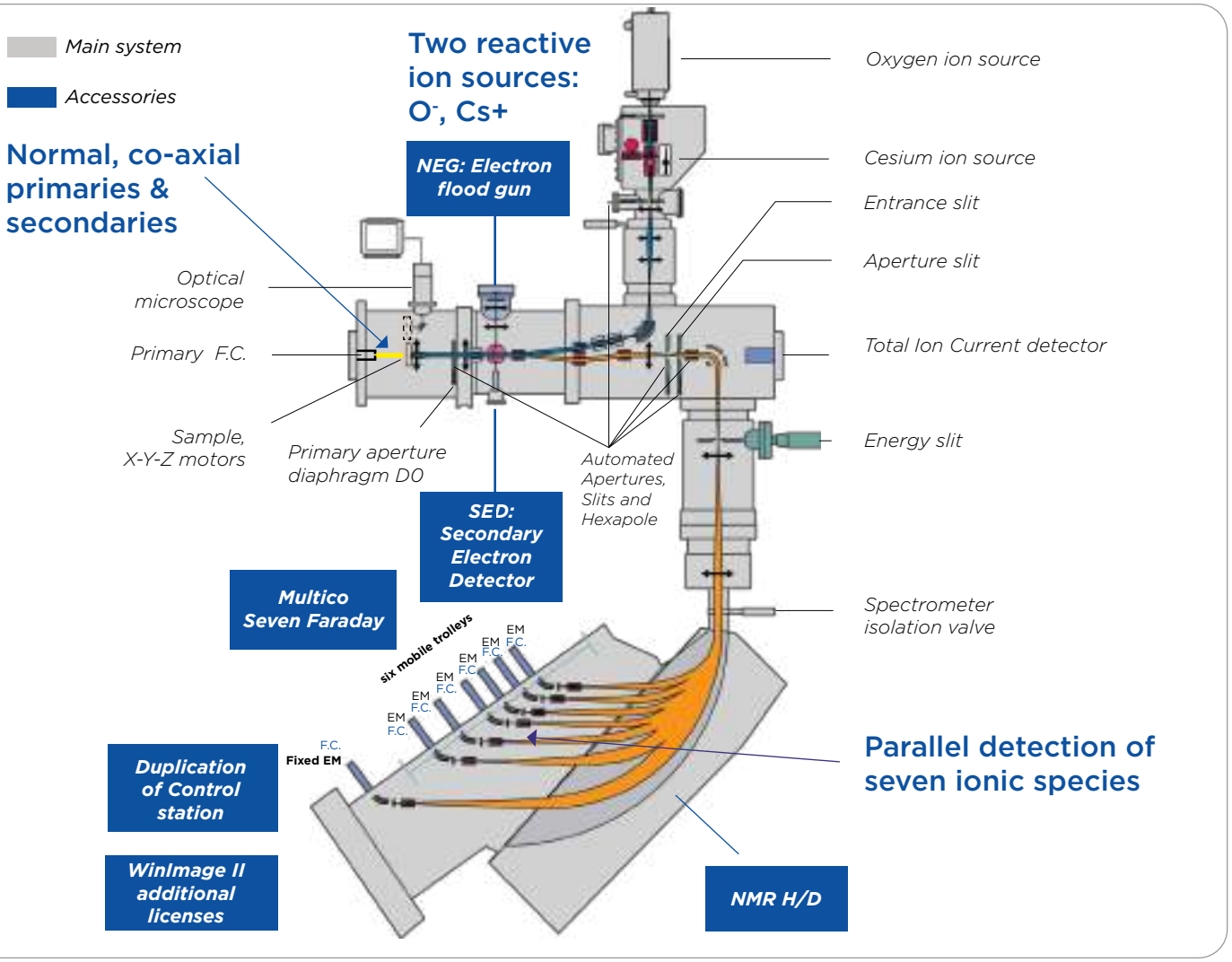
- Smaller spot size for a given beam current.
- Higher collection efficiency and dramatic reduction of the broadening of the secondary ion beam due to the initial angular and energy distribution. This will favor transmission of the analyzer at high mass resolution.
- Minimization of shadowing effects for non flat surfaces; access to hole or trench bottoms.
- Reduction of the beam and raster distortions as induced by the extraction field with an oblique incidence.

Constraints due to co-axial configuration

- Primary and secondary ions must be of opposite polarity and equal energy (Cs^+/ negative ions, O^-/ positive ions).
  1) This excludes MCs^+ technique for semi-quantitative SIMS analyses,
  2) It forces the use of O^- or O_2^- PI instead of O_2^+ primary ions for the electropositive elements. This is very beneficial for charge reduction.
  The negative aspectis is that oxygen ion sources have higher brightness for positive ions.
- Oxygen flooding can not be used (risks of arcing when increasing gas pressure).

The normal primary ion incidence with co-axial ion collection permits a maximized and uniform collection from samples with topography without shadowing.

NanoSIMS 50L synopsis & main accessories



NEG

Normal incidence Electron flood Gun for the analysis of strong electrical insulators with Cs^+ PI and negative SI if metal coating method is not sufficient or allowed.

SED

Secondary Electron Detector. Works only in negative secondary polarity with cesium primary ions. Can give nicely contrasted topographical images for illustration and sample visualization.

Multico Seven Faraday

The NS50L is equipped in standard with one electron multiplier (EM) in each trolley, plus one Faraday Cup (FC) on trolley #1. The M7FC option equips each trolley with one EM and one FC, together with a thermostated chamber housing the FC preamplifiers. FCs, generally used with 100s pA or a few nA beam current, are required for achieving low tenth permil reproducibility on isotope ratios from whole scanned area. They do not permit fast imaging as with EMs.

NMR H/D

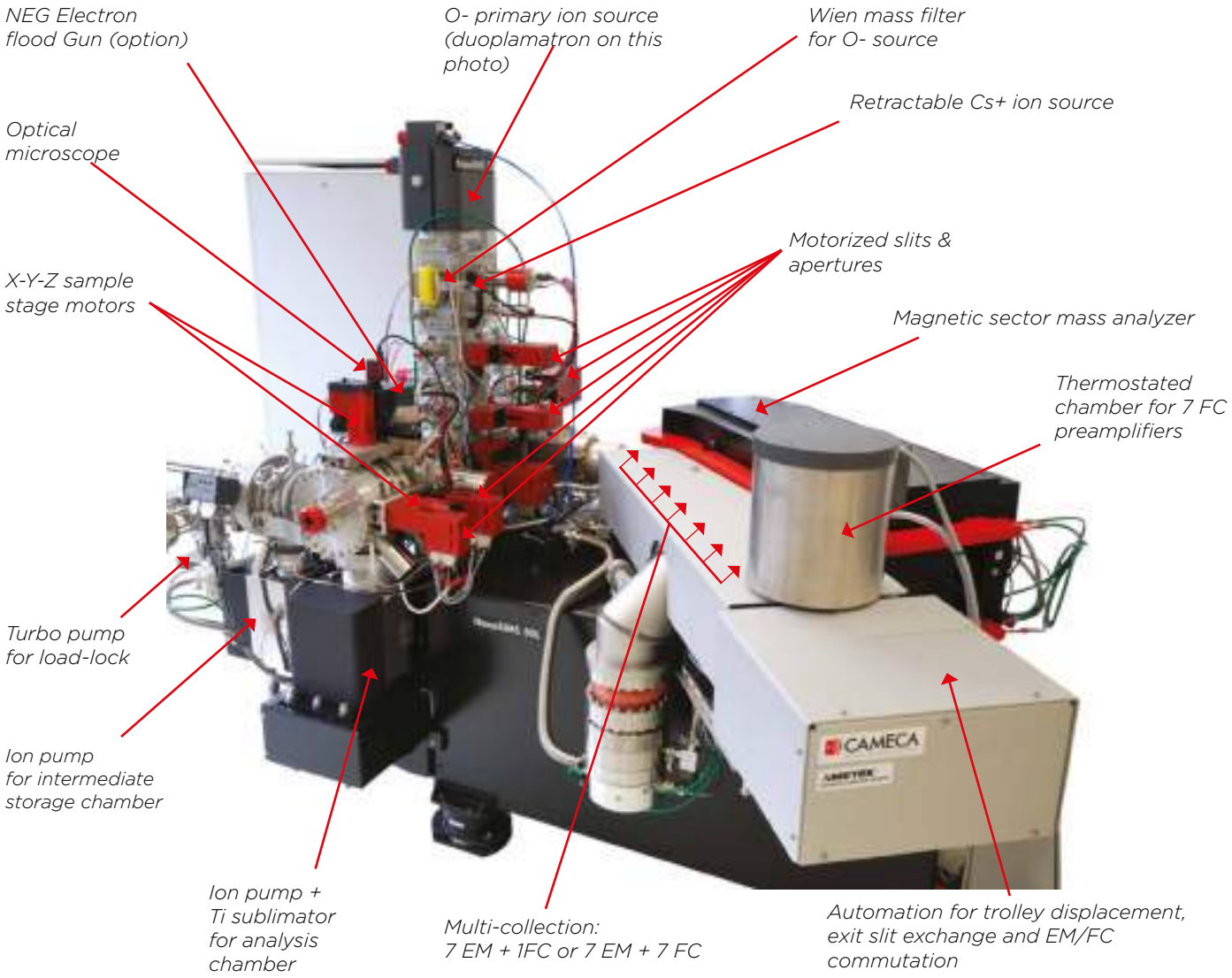
Additional NMR probe to ensure the best long term stability for hydrogen-deuterium measurements (the B field is too low for the standard NMR probe to improve stability on hydrogen compared to the standard Hall probe regulation).

Duplication of Control

Permits to control the NS from a neighbor control room (usually looking at the instrument through a window) keeping the instrument under best environmental stability and allowing best users comfort (no noise from rack ventilators and pumps).

The NanoSIMS can be fitted to specific applications. All accessories can be retrofitted on-site.

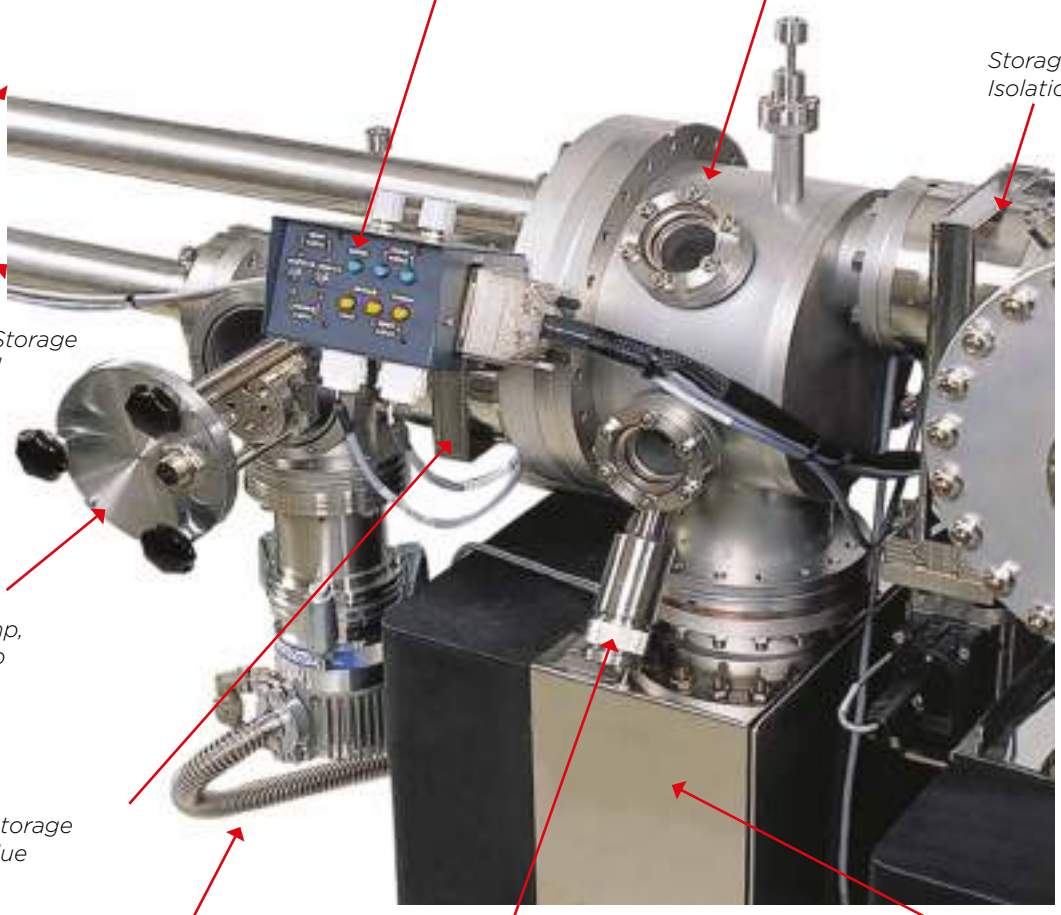
Key components



Key points of the NanoSIMS design:

- Co-axial primary and secondary ion beams
- Two switchable high brightness primary ion sources: Cs^+ and O^-/O_2^+
- In-situ optical microscope at separate position for sample observation and navigation
- Multicollection of seven selectable ionic species (EM/FC detectors) plus optional SEM detector (in negative secondary polarity) and total ion current detector (TIC)
- High overall mass analyzer transmission at high mass resolution
- UHV technology: dry primary pump, turbomolecular pumps, ion pumps and titanium sublimation, load-lock and intermediate storage chamber

Sample introduction



Key Points of the introduction

- Standard 2-inch load-lock with turbo pumping, dry nitrogen venting, heating lamp for sample degasing
- Manual magnetic transfer rod between the load-lock and the strorage chamber
- Intermediate storage chamber for eight 50mm diameter sample holders, with ion pumping
- Manual magnetic transfer rod between the storage and the analysis chambers
- Vacuum automation with independent processor
- UHV technology, integrated baking without dismounting

Overview and sample navigation

Complete instrument view with its two electronic racks and control desk. A third small electronic rack for the RF plasma O^- source is located behind the instrument. Primary pumps and fans are also located behind but can be deported in an adjacent service room where it is usual to locate water chiller, air compressor, gas bottles and UPS (uninterruptible power supply).

Sample Navigation before analysis

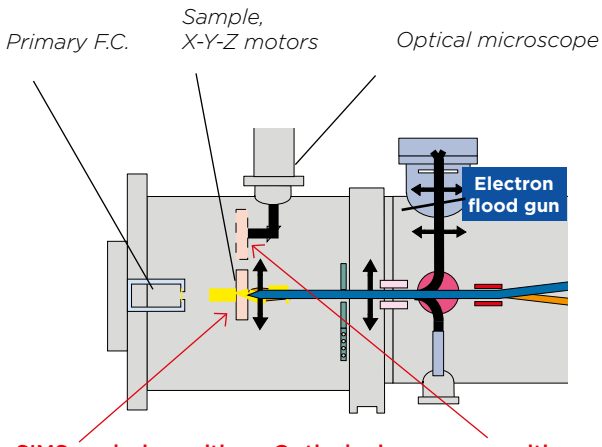
The usual way is navigating using the integrated **Optical Microscope**. Due to the short working distance of the immersion lens the optical microscope position is 40mm above the SIMS position. With a simple click of the mouse the sample is moved in seconds between the two positions. The field of view is 700µm and zoom inside the image is done numerically inside the 2052 x 2456 pixels of the digital color camera. With a white LED uniform illumination the lateral resolution is 1.5µm.

Point Logger

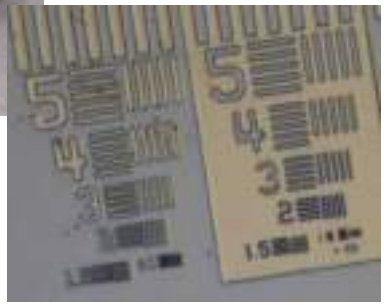
Another non-destructive navigation possibility is using the **Point Logger:**

- first an external image of the sample holder or sample (typically a SEM or an optical image from another instrument) is imported as a TIFF or JPEG,
- two reference points are calibrated in this image by moving these points in the analysis position.

It is then possible to drive the sample stage by clicking directly on the imported image.



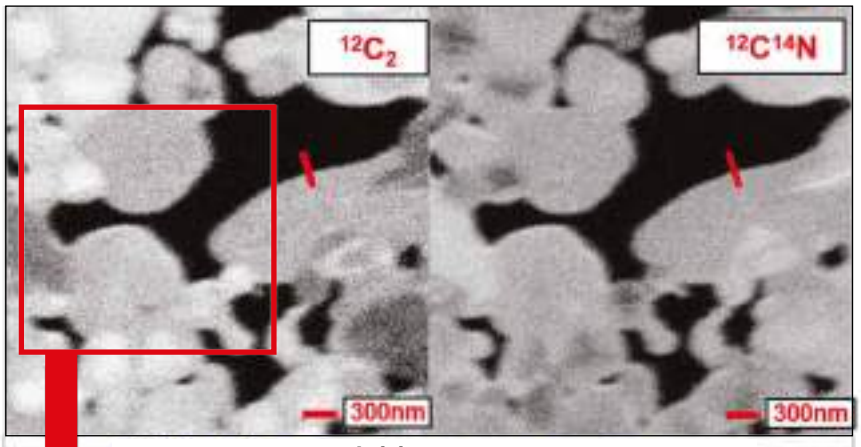
Zircon grains, 700 µm FOV



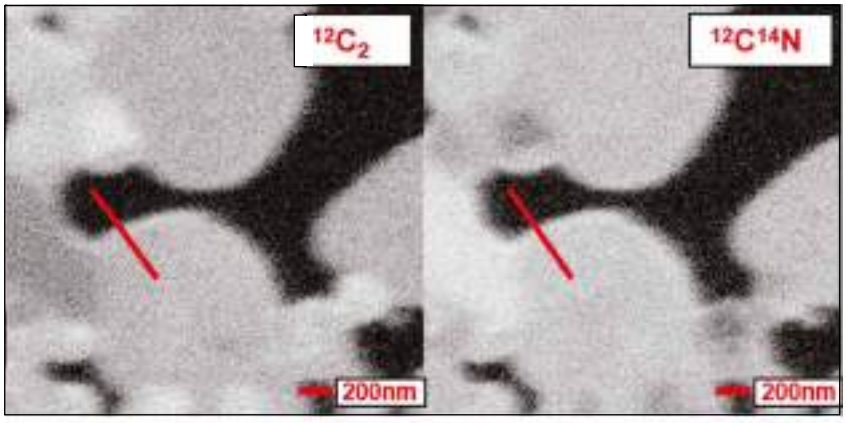
Digital zoom inside an image

Lateral resolution with cesium primary ions

The use of cesium primary ions is mandatory in SIMS for the analysis of electro-negative elements (H, C, O, N, F, Cl, P, Ge, Se, As, Br, Te, I, Au...). The enrichment of the top surface with cesium enhances the ionization yield (= sensitivity) by several orders of magnitude compared to non-reactive primaries (Ar, Ga, Au, Bi...). The NanoSIMS is equipped with the patented CAMECA Microbeam cesium ion source, guaranteeing the highest brightness available among commercial cesium ion sources. The source brightness (in mA/sr/cm^2) measures the ion current available within a given solid angle from a given source area. It is an invariant in optics: a perfect (= without optical aberration) primary ion column could at maximum re-obtain this brightness in the final spot size. The high brightness of the ion source, the short final objective working distance, its reduced aberration coefficients, and the normal incidence guarantee the best performance available from a SIMS microprobe for electronegative secondary ion microanalysis.



Field 4X4 µm



Field 2.5 x 2.5 µm

Best achieved lateral resolution: **25nm**

Two successive NanoSIMS images at increasing magnification on a TiCN sample giving sharp grain boundaries without artifact. A lateral resolution of 25nm is measured, determined by extracting the 16%-84% intensity line-scan from the image. The normal incidence/collection permits avoiding astigmatism and distortion introduced by an oblique incidence within an extraction field (resolution better in one direction than the other).

Conservative NS50L demonstrated specifications for 16keV Cs^+ primaries:
A) 50nm lateral resolution (16-84% criteria),
B) 2pA of Cs^+ in a spot of 100nm.

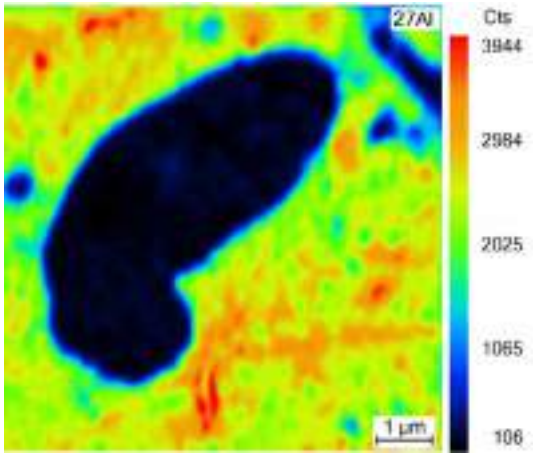
Lateral resolution with oxygen primary ions

The NanoSIMS was formerly equipped with the CAMECA duoplasmatron ion source used in O^- mode in order to benefit from the strong ionization enhancement of electropositive elements with oxygen implantation. Additionally, the use of primary negative ions offers the well-known advantage of much lower sample charging problems compared to positive primary ions (samples always tend to charge positively due to secondary electron emission).

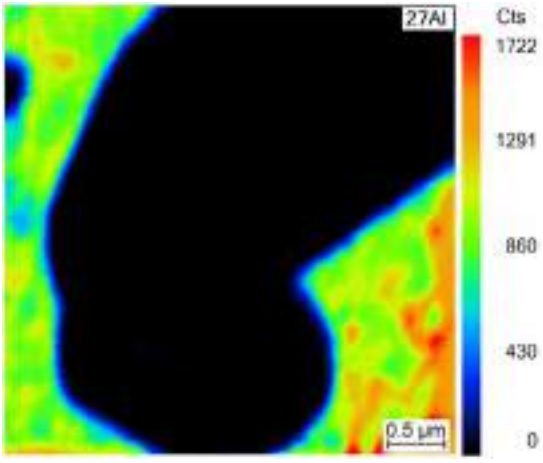
A **new RF-plasma O^- primary ion source** with higher brightness (in mA/cm2/sr) was adapted to replace the duoplasmatron. The improvements are:

- a much **longer lifetime** between cleaning (several months instead of weeks),
- a primary beam current **more stable over long term** (< 2% over 12 hours),
- the beam densities and lateral resolution of the NanoSIMS 50L are now **comparable with the cesium** performance.

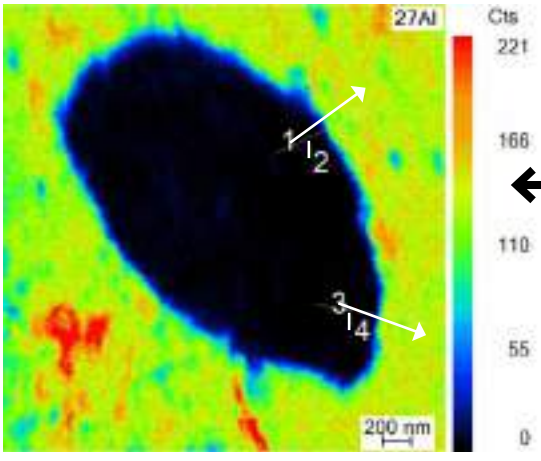
Three images below with beam current and 16-84% spot size, and one 16-84% line-scan illustrate this improvement of performance (images recorded during characterization phase, not contractual).



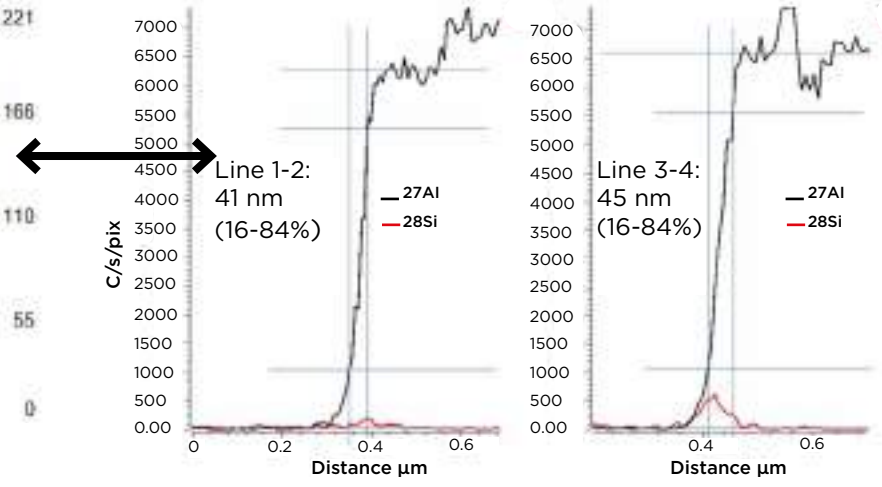
FOV 8 x 8 microns, 175 nm 9.2 pA



FOV 4 x 4 microns, 88 nm 1.65 pA



FOV 3 x 3 microns, ~ 43nm 0.2 pA



Two line-scans extracted from image on the left

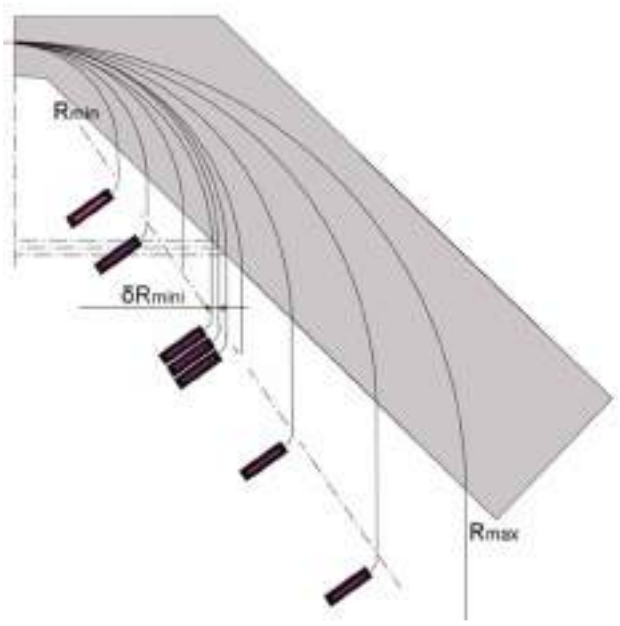
The NanoSIMS 50L now offers similar lateral resolution and beam density for Oxygen primaries compared to Cesium.

NanoSIMS 50L multicollection

The Multicollection analyzer of the NanoSIMS 50L can measure **Seven Masses in Parallel** with six trolleys moveable under vacuum and one trolley fixed at the highest radius (Rmax). Each trolley is equipped with scanning plates, selectable exit slits, cylindrical electrostatic sector and detector (EM or/and FC).
Scanning the voltage on the entrance plates permits recording a high mass resolution mass spectrum across a single mass unit, then selecting which peak to detect at this unit mass (ex: $^{28}Si_2$ or ^{56}Fe).
The adjustable combination of entrance and exit slits permits to control a) the mass resolving power and b) the peak shape (flat top peaks or "triangular" peaks).
The multicollection has two essential characteristics:

- **The Mass Range**, given by the factor **D = X 22** between minimum mass and maximum mass (ex: from 1 AMU to 22 AMU or from 10 AMU to 220 AMU depending on the selected B-field in the electromagnet.

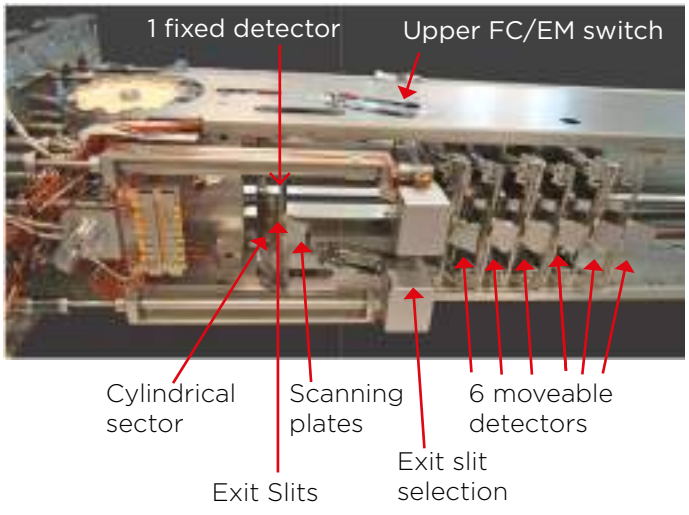
- **The Mass interval between detectors:** minimum mass separation between two adjacent small detectors at the highest radius: dM = M_{max}/58 (one AMU interval between neighbor detectors is possible up to mass 58 AMU). Thus for example masses 56, 57 and 58 AMU can be recorded simultaneously but only 100, 102 and 104 AMU or 145, 148 and 151 AMU. If one wants to record 145, 146, 147, 148, 149, 150 AMU:
  - one solution is to work in monocollection (one detector) and switch the B-field for each mass.
  - It is possible to mix modes: ex record 145, 148 on two detectors, switch B-field and record 146, 149, switch B-field and record 147, 150 AMU, etc.
  - It is also possible to use trolley jumps instead of (or together with) B-field jumps.
  Each mass set-up contains the trolley position, B-field, deflector voltage and all necessary parameters. Hence it is also possible to perform electrostatic peak jumping: alternate between $^{12}C^{15}N$ and $^{13}C^{14}N$ or between $^{12}C^{14}N$ and $^{12}C_2{}^2D$, on one detector using deflection plates before the exit slit.



Multicollection characteristics:

Rmax = 680mm, Rmin = 145mm. D= $(Rmax/Rmin)^2$
Inside the magnetic field, the radius R of ion trajectory is proportional to the square root of the mass M of the ion:R_M = a * sqrt(M) or R_1/R_2 = sqrt(M_1/M_2).
δR_{min} (mini physical R-interval between EMs) = 5.8mm.
Hence:
RM = 680/ sqrt (M_{max}) * sqrt (M), so δM_{min} (mini mass interval between 2 adjacent det.) = sqrt (M_{max}) * sqrt (M) * δr_{min}./ 340.
or: δM_{min} ~ sqrt (M_{max} * M) * 0.017

Multicollection overview:



7 masses in parallel with single mass unit separation up to 58 AMU,
7 FC option with individual FC / EM switch under vacuum,
Mono-, Multicollection and hybrid modes (electrostatic, magnetic and trolley jumps)

Analyzer transmission versus mass resolution

The NanoSIMS is a SIMS analyzer with high transmission at high mass resolving power (MRP = M/dM). To a first approximation, the NS50L MRP is inversely proportional to the magnification of the spectrometer and to the entrance slit width; As the magnification is itself proportional to the radius, the MRP is theoretically constant along the focal plane. The theoretical MRP is then degraded by angular and chromatic aberrations of various orders. The conception work, initially done by Prof. G. Slodzian at the UPS, Orsay, France, was to maximize the collection and the transport of (the few) secondary ions of various energies and directions and to shape the secondary beam into a small beam waist, while minimizing aperture (angular) and chromatic aberrations of the analyzer. Finally the detector efficiency is the last parameter affecting the overall useful yield (UY: nb. of detected ions per sputtered atoms). A characteristics of the NanoSIMS is to always work in high mass resolution: by design there is a beam waist at entrance slit location even when removing all apertures. In addition, the analyzer transmission is maintained very high when increasing Mass Resolution, result of:

- a very strong, normal electrostatic extraction field allowing a very early secondary ion focusing,
- a limited field of view (but large collection angle) enlarged with a dynamic emittance matching system,
- a careful transport and rectangular shaping of the secondary beam resulting in the use of small slit sizes compared to the magnet size, reducing aberrations,
- the correction of the second order mass spectrometer optical aberrations,
- miniaturized electron multiplier detectors with discrete anodes. Working at 8kV ion energy impact on the first dynode and with low noise preamplifiers, they permit high quantum efficiency and, coupled with small exit slits, very low background noise.

MRP	T (%)
3500	100
5910	68
6120	65
6770	56
7120	51
7390	45
7885	39.9
9470	29
9615	24.5

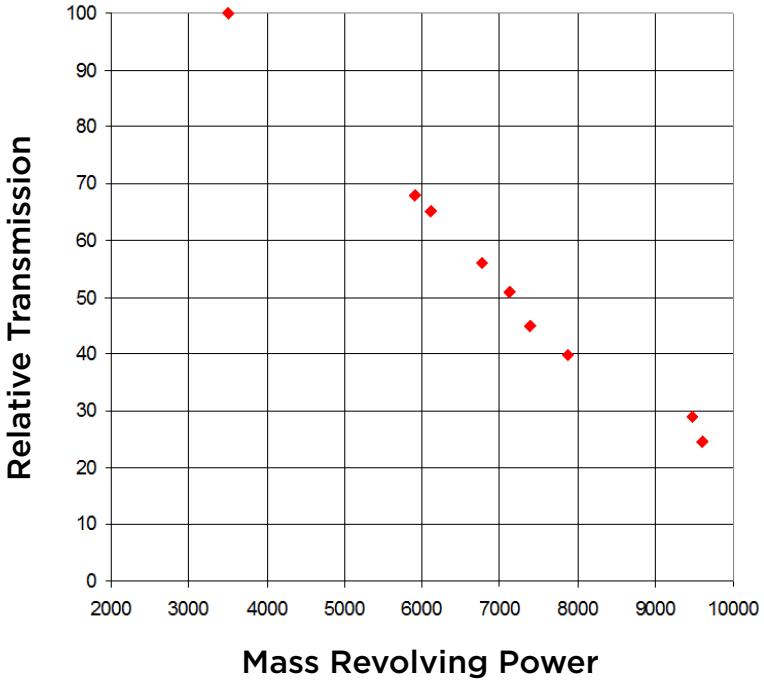
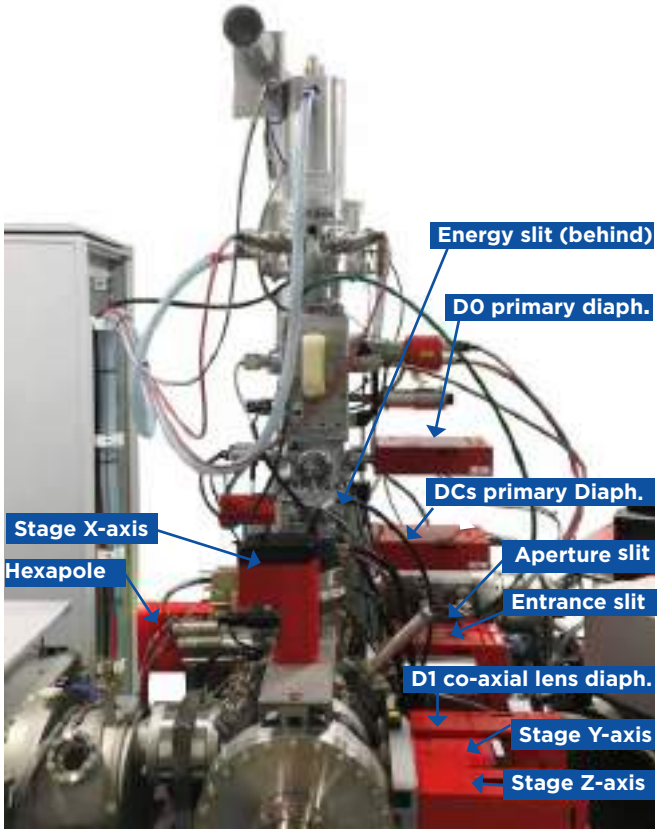


Table and corresponding plot: relative transmission as a function of mass resolving power. Without any slit, mass resolution is 3500 and transmission is taken as 100%. Other transmissions are referred to this one. Mass resolution is taken as M/dM = R/4 * L10-90, where R is trajectory radius and L10-90 is line width corresponding to 80 % of intensity.

The NanoSIMS 50L multicollection spectrometer design permits benchmark transmission at high Mass Resolving Power, in DC mode for high throughput analysis and low detection limits.

Automation and control interface



All moveable apertures, slits and hexapole are computer controlled

The lab manager would typically tune the instrument for a given application and store the setting in the computer. Other users can then re-load the setting, fast check, navigate, select the analysis areas and start the acquisition or launch chained acquisitions on saved sample positions.

The benefits are an easier operation (important especially for multi-user operation), a better reproducibility for high precision isotopic ratios (sub-permil level) and a higher throughput (faster tuning checks or pre-sputter at high current followed by analysis at high resolution in chain mode).

Automatic secondary alignment software routines can be performed before acquisition or added in the acquisition chaining. For high precision isotopic ratios, automatic EM high voltage tuning can be also added to compensate for potential aging of the electron multipliers at high count rate.

The operations that are still manual are only the sample transfer, the Cs/O ion source switch and the oxygen leak valve adjustment for the RF-plasma ion source.

The local control of the instrument is performed through the combined use of a dedicated control keyboard to access directly key parameters and assign some to a three-wheel keypad, and a PC with two large screens, working under Microsoft Windows 10.

The near-full automation of the NanoSIMS 50L has improved reproducibility, throughput, remote support and facilitated operation especially for multiple users.

Remote control through the internet & Control duplication accessory

Below is a view of the two screens used for controlling the instrument. The left screen displays the control of the multicollection. The right screen displays here the vacuum control, sample navigator and tuning presets. This would be the standard PC interface used together with the local dedicated keyboard and 3-wheel pad.

LEFT PC screen

RIGHT PC screen

Virtual Keyboard

In addition, a virtual (digital) dedicated keyboard is available as shown at the bottom of right screen. The 3-wheel pad function is replaced by PC mouse rollpad (3 speeds). Numerical entries of parameter values are also possible**.**

The NanoSIMS can be controlled **locally** using the real dedicated keyboard or **remotely through the internet** using this virtual dedicated keyboard. In this latter case a single screen PC equipped with Team Viewer software is used and the switch between the two screens is done with a tab. This is the configuration used by CAMECA service engineers to localize problem or help on a tuning from our factory.

Duplication of Control (accessory)

For best environmental stability for the instrument and less noise for the operator it is possible to control the instrument from another neighbor room, generally viewing the instrument through a window.

The PC screens, keyboard and mouse are duplicated with some complementary hardware (max: 15m). The dedicated CAMECA control keyboard can be connected in the control room for normal operation or on the instrument's desk mainly during some maintenance. It does not change the recommendation to deport water chiller, UPS, primary pumps, air & nitrogen bottles or compressor in an (air-conditioned !) neighbor service room.

NanoSIMS 50L controlled from a separate room (IGGCAS Beijing, China).

Remote control enables better stability, easier use from office or home, sharing between long-distance multiple users and faster service support and diagnostic.

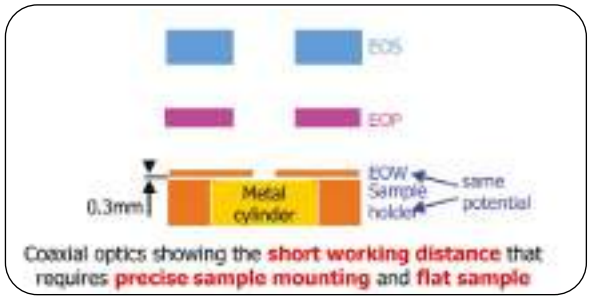
Sample mounting: principle

Dimensions: The critical point for sample mounting comes from the short distance (300µm) between the immersion lens electrode and the top surface of the sample holder. The sample is pressed from the rear against a circular lip of 100µm thickness and 700µm width. The sample surface is thus at 400µm from the immersion lens. The Z movement of the sample stage can be used to adjust the sample/extraction distance at 400µm +/- 50µm but the holder surface can not be raised by more than 300µm without contacting the immersion lens ! In addition the sample surface must be flat in order to define a uniform electrical field, crucial for the good working of the co-axial lens.

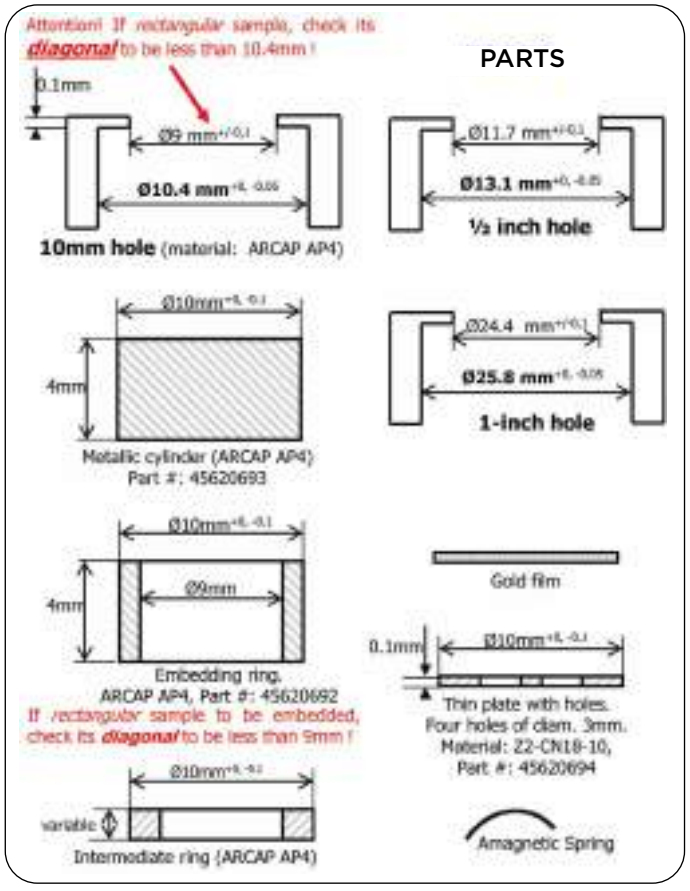
Cleanliness: the sample must not degas in order to avoid risks of arcing. Typical working condition is in the low E-9/ high E-11 mbar range. Samples should always be handled with clean gloves, tools and aluminum foil; avoid paper or wooven tissues. The sample must adhere strongly to its support (double sticky tape or insulating particles pressed into gold foil, etc.) in order to avoid it jumping into the immersion lens due to the strong electrostatic field and eventual charging ! This might lead to venting of the NS and dismounting of the immersion lens for cleaning !

Resin embedding: dehydration and selection of a UHV-compatible embedding materials is mandatory; minimizing its volume is good. For geology, embedding can be done inside a metallic cylinder, followed by polishing and metal coating. For biology, microtomed sections (100-500µm thick typically) can be laid flat on a conductive surface (ex: silicon wafer) and let dry. Some resins used in the NanoSIMS: Korapox 439 epoxy, LR White, EpoCure, EpoxiCure, Varian Torr Seal Low Vapor Pressure Resin. Also used: Wood metal (In-Bi alloy melting at 78°C).

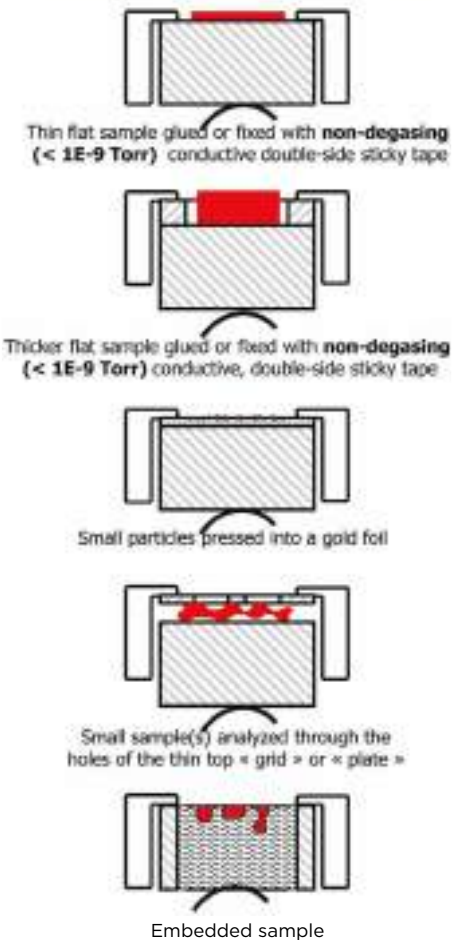
Sample mounting: Below is a schematic of a sample holder with 10mm holes (different holders and hole sizes are available, see the product description), with parts giving an idea of some possible mountings (schematics not at scale).



Coaxial optics showing the **short working distance** that requires **precise sample mounting** and **flat sample**

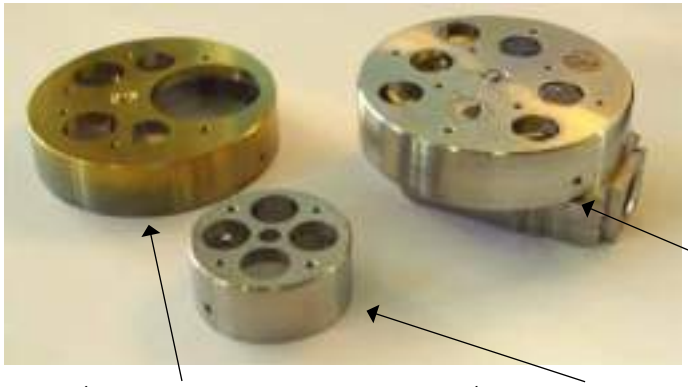


SOME SAMPLE MOUNTINGS



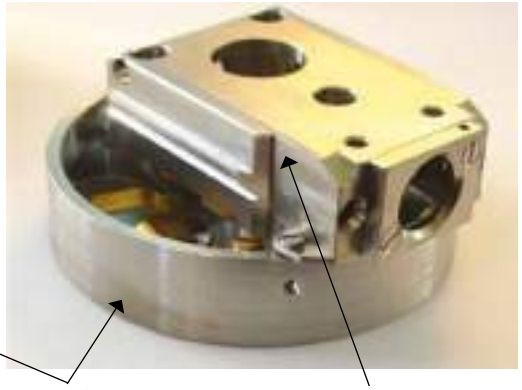
Sample mounting: parts

Various sample holders can be mounted (screwed) on a transfer shuttle. It is possible to simultaneously load two shuttles on the sample stage : two 1-inch, or one 1-inch and one 2-inch sample holders. The second 1-inch sample holder, generally used for standards, can be brought in SIMS position but not in the optical microscope position.



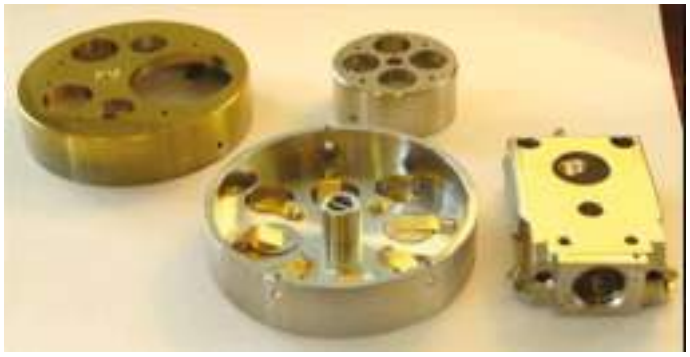
50mm/2-inch diam. «WU-MPI» sample holder with one 1-inch, two half-inch and two 10mm holes. Ref #: 45620643

25mm/ 1-inch diameter «Standard» sample holder with four 10mm holes. Ref #: 45620641



50mm/ 2-inch diameter «Biology» sample holderwith eight 10mm holes. Ref #: 45620642

Shuttle Ref #: 45621551

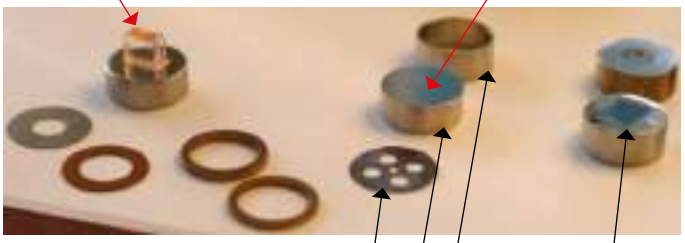


Reverse view of the « Biology» sample holder, unscrewed from its shuttle. One can see the springs pushing the sample cylinders in their hole against their lips.

"Harvard" holder with 16 holes and image of microtomed sections of resin-embedded tissues deposited on a 5x5mm silicon square.

Ex. of wrong mounting!
Must be remounted with front reference.

Thin samples (ex: biological sections) must be deposited on the **POLISHED side** *of the metallic cylinder !*

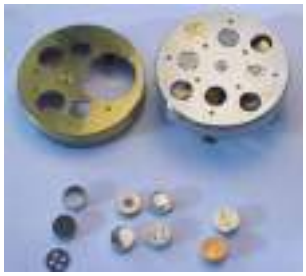


Embedded sample

10mm diam. embedding ring, Ref #: 45620692

10mm metallic cylinder, Ref #: 45620693

4-hole thin plate Ref #: 45620694

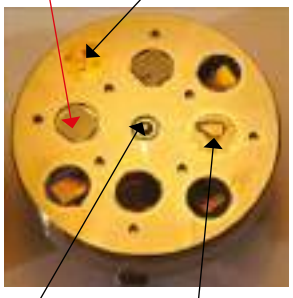


Set of 5 round silicon wafers: 2x10mm, 2x1/2-inch and 1x1-inch diameter. Ref #: 45639765

10mm diam. sub-holder for 3 TEM grids. Ref #: 45639345. Correct use: round carbon-film grids on hollow 3mm cylinder, half-grids on plain 3mm cylinder.

Biological thin cross-section deposited on **7.3 x 7.3mm** *silicon square (***diagonal***=10.3mm).*

Sample under 4-hole thin plate



Embedded sample

Anti-vibration finger contacting the front electrode of the objective lens.

NanoSIMS 50L



Introduction to the Instrumentation

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