Probion Analysis

Semiconductor and solid material analysis expert

Analysis service for industry and research

We attend to your samples and analysis issues
We analyze your samples

With 20 years experience, Probion Analysis delivers an analysis service in the field of solid materials, especially semiconductors.

With an extensive range of analysis tools at our disposal (SIMS, XPS, CV profiler, TOF-SIMS, RBS, FIB-TEM), we take cognizance of your analysis issues and we suggest the most suitable solutions to provide the information you need: impurities presence and concentration, matrix composition, layers thicknesses, interface quality and interdiffusion, doping uniformity...

With a sound background regarding all sorts of semiconductor devices (Si-SiGe, III-V, II-VI, insulators, diamond ...), we also provide analysis solutions for other solid material: metallic coatings and alloys, glasses…

We know that a fast turnaround time is a major issue. So we propose short delivery times (5 days standard) or even 24h delivery arrangements for specific cases. After analysis delivery, we remain available for any further information on analysis results or possible analysis supplement.
### Solid materials analysis solutions

<table>
<thead>
<tr>
<th>Dynamic SIMS</th>
<th>Secondary Ion Mass Spectrometry</th>
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<tbody>
<tr>
<td>Provides profiles of atomic concentrations versus depth (dynamic SIMS). Elemental imaging and mass spectra capabilities. The best way for detecting very low concentrations (down to ppm or ppb depending on element and substrate), well suited for semiconductors analysis.</td>
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<tr>
<td>Depth range: from 10nm to 10µm. Depth resolution: down to 1nm.</td>
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<tr>
<td>Analyzed area: from 10µm to 500µm in diameter.</td>
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<tr>
<td>Equipment used by Probion: CAMECA 7F, CAMECA 4F (III-V), CAMECA 4F (Silicon)</td>
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<tr>
<td>Please, see our tutorial: <a href="http://www.probion.fr/tutorial.pdf">http://www.probion.fr/tutorial.pdf</a></td>
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<tr>
<th>XPS also known as:</th>
<th>X-ray Photoelectron Spectrometry</th>
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<tr>
<td>ESCA</td>
<td>Electron Spectrometry for Chemical Analysis</td>
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<tr>
<td>Provides matrix atomic composition or impurities concentrations and informations on chemical bonds.</td>
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<tr>
<td>No standard needed but poor detection limit (0.2at%).</td>
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<tr>
<td>Although dedicated to surface analysis (10nm), profiles versus depth may be obtained with a low depth resolution.</td>
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<tr>
<td>Equipment used by Probion: Micro XPS KRATOS, PHI Quantera SXM.</td>
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<tr>
<td>Please, see our tutorial: <a href="http://www.probion.fr/xpscourta.pdf">http://www.probion.fr/xpscourta.pdf</a></td>
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<tr>
<th>TOF-SIMS</th>
<th>Time Of Flight Secondary Ion Mass Spectrometry</th>
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<tr>
<td>The ideal tool for detecting organic molecules contaminations on surfaces. Extreme surface analysis: a few monolayers (static SIMS). Profiling also possible.</td>
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<tr>
<td>Equipment used by Probion: TOFSIMS 5 from IONTOF</td>
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<tr>
<th>TEM coupled with:</th>
<th>Transmission Electron Microscopy</th>
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<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
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<td>These two techniques are routinely coupled to observe the geometry and matrix composition of integrated circuits patterns (widths and thicknesses of transistors, metallic pads, insulating layers, alloys composition).</td>
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<tr>
<td>The FIB tool is used to cut an ultrathin (~0.1µm) vertical lamella in the 3D structure. This lamella is then analyzed by TEM.</td>
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<tr>
<td>Equipment used by Probion: FIB : FEI-200 FIB, TEM : Jeol 2000-FX-TEM</td>
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<tr>
<th>RBS</th>
<th>Rutherford Backscattering Spectroscopy</th>
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<tr>
<td>By using He(^{2+}) ions accelerated at 1-3MeV, this technique probes the sample over ~1µm depth. One analysis provides the energy spectra of backscattered ions. Different types of information can be deduced according to the sample structure.</td>
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<tr>
<td>Main use: matrix atomic composition of homogeneous substrates, ions implants doses measurements (over 1µm depths), thickness measurements (by modeling the He ions behaviour in the sample).</td>
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<tr>
<td>No standard needed.</td>
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<tr>
<td>Best sensitivity obtained for heavy atoms embedded in a matrix of light elements.</td>
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<tr>
<td>Equipment used by Probion: Van de Graaff accelerator 1-3MeV</td>
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<tr>
<th>CVP</th>
<th>Capacitive Voltage Profiling</th>
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<td>Ideal couterpart of dynamic SIMS analysis regarding dopants concentration measurement in semiconductors. This technique is specifically dedicated to the measurement of electrically active dopants, while SIMS measures the concentration of all dopant atoms (electrically active or not).</td>
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<tr>
<td>Provides n or p type active dopant concentration versus depth.</td>
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<tr>
<td>Equipment used by Probion: Polaron from Biorad</td>
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<th>VPD-ICPMS</th>
<th>Vapour Phase Decomposition Inductively Coupled Plasma Mass Spectrometry</th>
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<tr>
<td>Best technique for accurate measurement of surface contamination of silicon wafers</td>
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**Dynamic SIMS** (Secondary Ion Mass Spectrometry) – General outline

Atomic concentration profiles versus depth

**Physical aspects:**
1. Primary ions emission (O$_2^+$ or Cs$^+$).
2. Sample sputtering by the focused and scanned primary beam.
3. Secondary ions emission from the sample surface.
4. Secondary ions mass filtration by the spectrometer.
5. Detection of filtered secondary ions.

**Characteristics:**
- Depth resolution: up to 1nm/decade (no roughness)
- Crater depth: 10nm-10µm
- Detection limit: 1ppm-1ppb
- Sample size: 200µm (chips) - 50mm (wafers) (cut out if too large)
- Optimal sample size: 7mmx7mm
- Crater size: 10µmx10µm to 500µmx500µm

**Applications:**
- Profiles of atomic concentrations versus depth
- Impurities detection (ppm to ppb detection limits)
- Quantification of dopants and impurities concentrations in most semiconductors
- Quantification of matrix composition in many semiconductor and insulator alloys
- Visualizing multilayer stack structures, diffusion profiles ... (quantification of depths)
- Elemental imaging (2D cartography of one element distribution on sample surface)

**Equipment used by Probion:**
- CAMECA 7F (latest generation instrument)
- CAMECA 4F (III-V)
- CAMECA 4F (Silicon)
Dynamic SIMS - Examples

High depth resolution: quantum wells resolving

Low detection limit: Sulphur in InP: 11 ppb

Alloys composition measurement: $x$ in Al$_x$Ga$_{1-x}$As versus depth
Dynamic SIMS - Examples

Mass spectrum characterization of a layer contamination:
Comparison with an uncontaminated reference sample

Arsenic (75amu) is found in sample A

Ionic imaging of a contaminant on a GaN sample
**XPS (X-ray Photoelectron Spectrometry) – General outline**

Matrix atomic composition and chemical bonds analysis

**Physical aspects:**
1. X-ray beam emission (Al K\(\alpha\) or Mg K\(\alpha\), \(\approx 1200-1500\)eV).
2. The deep level electrons of sample atoms are excited.
3. Some reach the surface and escape (photo-electrons emission).
4. Photo-electrons energy is filtered by the electrostatic analyzer.
5. Filtered electrons are detected.

As a result, one obtains a spectrum of binding energies of samples electrons, each peak corresponding to a particular deep electronic level such as O\(1s\), C\(1s\), Sn\(3d\), Pb\(4f\) ... The peaks are identified to the deep electronic levels they come from.

Their integration enables to calculate the matrix composition.

**Example:**
Pb 56at\%, Sn 36at\%, O 11at\%

**Characteristics:**
- Analyzed depth (single analysis): \(<10\)nm
- In combination with ionic sputtering: \(\sim 300\)nm (see next example)
- Detection limit: 0.2at\%
- Analyzed area: \(\sim 200\)µm (diameter)

**Applications:**
- Quantification of matrix composition without standard, close to surface (\(<10\)nm)
- Impurities detection (0.2at\% detection limits)
- Profiles of composition versus depth (10-500nm)
- Information on chemical bonds

**Equipment used by Probion:**
- Micro XPS Kratos
- PHI Quantera SXM
The binding energy of deep level electrons is slightly influenced by the chemical environment. Below, a silicon emission peak is deconvoluted into three components, each one corresponding to different chemical bonds.

Example:
- C-Si-O3: 61%
- Si-O2: 15%
- C2-Si-O2: 24%
TOF-SIMS (Time Of Flight Secondary Ion Mass Spectrometry)

Extreme surface mass spectra for detection of organic compounds (amines, amides, resins, silicone molecules, fluoride ...)

Physical aspects and characteristics

TOF-SIMS is a static SIMS analysis technique: unlike dynamic SIMS, only the first monolayer is analyzed.

Thanks to a soft sample sputtering, intact organic molecules deposited on sample surface can be ionized and detected.

One analysis provides a mass spectrum in the range 1-10000amu: chemical compounds can then be identified thanks to their spectral signatures.

Applications:

Detection of organic contaminations on surfaces
Imaging of molecules distribution (spatial resolution: <1µm)

Equipment used by Probion:

TOF-SIMS 5 from IONTOF
**FIB-TEM** (Transmission Electron Microscopy+Focused Ion Beam)

Vertical structure imaging at nanometer scale

**Part 1: FIB preparation**

An ultrathin lamella (~100nm) is cut out in the vertical structure of the sample and deposited on a sample-holder

**Part 2: TEM observation**

The lamella is observed by TEM (various imaging modes available)
RBS (Rutherford Backscattering Spectroscopy)

Matrix atomic composition and thickness measurements

Physical aspects and characteristics

High energy He\(^{2+}\) ions (1-3MeV) are sent on the sample, a fraction of them is backscattered and detected.

One analysis provides the energy spectrum of backscattered He ions.

The sample characteristics are probed over 1\(\mu\)m depth.

No standard needed

Applications:

Quantification of:
- substrate composition (at/cm\(^3\))
- implantation dose (at/cm\(^2\))
- layer thickness (<1\(\mu\)m)

By modeling the He ions behavior in the sample structure, one can calculate a simulated energy spectrum. By fitting the model to the experimental result, one or several characteristics from the sample structure can be determined.

Equipment used by Probion:

Van de Graaff accelerator

Example:

Spectrum from a TiN layer on a silicon substrate.

RBS analysis gives a better sensitivity for heavy elements embedded in matrix made of light elements. Here, Titanium is the heaviest element.
CVP (Capacitive Voltage Profiling)

Measurement of electrically active dopants in semiconductors

**Physical aspects:**
The electrical contact between electrolyte and doped semiconductor creates a Schottky junction.
By varying the voltages, capacitive measurements are made and carrier’s concentration is deduced.
The electrolyte has two roles: chemical etching of sample (depth information) and electrical contact for capacitive measurements.

**Applications:**
Ideal counterpart of dynamic SIMS analysis regarding dopants concentration measurement in semiconductors. While dynamic SIMS measures the concentration of active and inactive dopant atoms, CVP detects only the active ones.
Provides profiles of n or p type active dopant concentration versus depth.

**Equipment used by Probion:**
Polaron from Biorad

**Example:**
Boron shallow implantation in silicon before annealing.
The figure shows a comparison between a dynamic SIMS measurement and a CVP one.
The two superimposed curves reveal that an important fraction of boron atoms are not electrically active close to the surface (0-6nm).
On the CVP curve, the data density is higher for higher doping.
VPD-ICPMS (Vapour Phase Decomposition Inductively Coupled Plasma Mass Spectrometry)

Measurement of surface contamination of silicon wafers

Physical aspects:
The silicon wafer is exposed to HF vapor, which reacts with the surface native or thermal oxide (vapor phase decomposition). The matrix of silicon dioxide is removed by formation of SiF (gas form). A liquid droplet is then pipetted onto the wafer surface and moved over the entire surface area of the wafer for VPD residue collection. The droplet is transferred automatically into a sample vial for elemental analysis by ICP-MS.

Applications:
VPD-ICPMS analyses surface contamination by a wide range of elements of the periodic table from Li to U at one time, with unrivalled detection limits.

Equipment used by Probion:
VPD : Expert™ from IAS
ICP-MS : NexIon™ from Perkin-Elmer
Contact us

We study your analysis issues and advise you on the best technical options.
Please, contact us for further information.

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