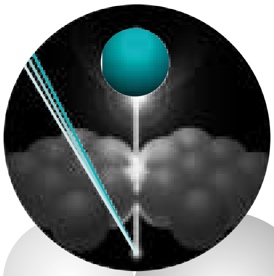
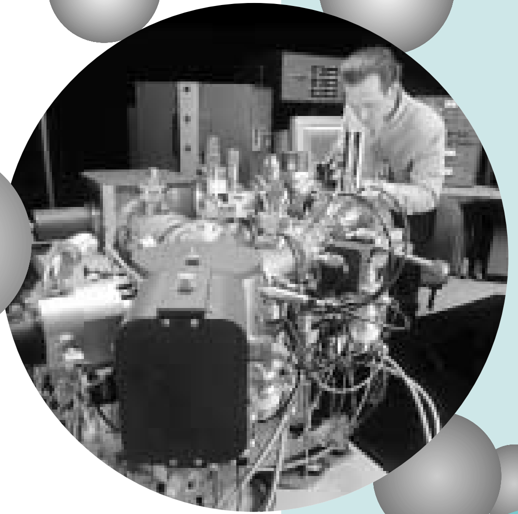


Surface Analysis



The Cameca IMS 5f dynamic SIMS is used to analyze the surface of a material or to determine the depth distribution of elements as the primary ion beam sputters through a material. It can identify materials from the lightest of elements up to those having 250 amu. (Jim Yost Photography/ PIX02009.)

We use surface analysis to determine the chemical, elemental, and molecular composition of material surfaces and interfaces. Depending on the technique used, the surface being probed may constitute just the top monolayer of atoms (which some hold to be the only true surface), or it may extend several microns beneath the top monolayer.

By bombarding a material with ions, electrons, X-rays, or photons in high vacuums, we map the elemental and chemical composition of specimens, study impurities and grain boundaries, gather bonding and chemical-state information, and perform depth profiles to determine doping and elemental distributions.

Our surface analysis team boasts more than half a century of experience in analyzing surfaces for materials that range from photovoltaics to microelectronics to polymers to biological specimens. For these analyses, we offer several sophisticated and complementary techniques, including *dynamic secondary ion mass spectrometry*, *static secondary ion mass spectrometry*, *Auger electron spectroscopy*, and *X-ray photoelectron spectroscopy.* To augment these techniques, we have recently added a new capability to our repertoire — *ion scattering spectroscopy* — which we use primarily for elemental analysis of the outermost surface layer of a material.

Dynamic Secondary Ion Mass Spectrometry (Dynamic SIMS)

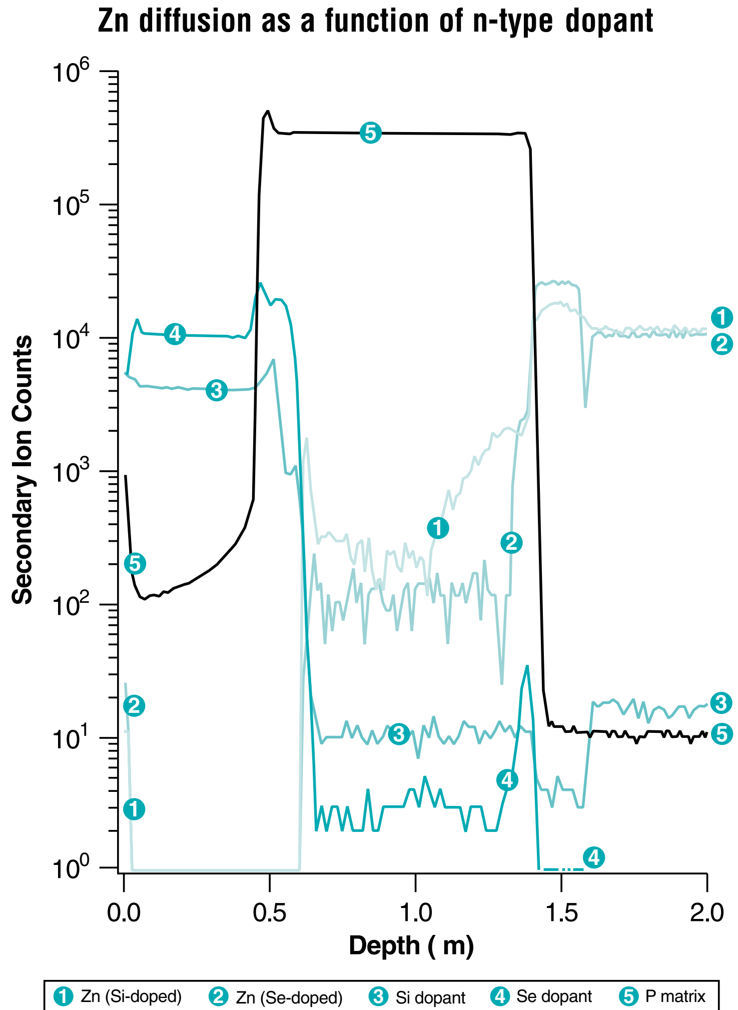
With dynamic SIMS, the surface of a sample is bombarded with a continuous, focused beam of primary ions. The impact of the ions sputters (ejects) atoms from the surface of the material, producing secondary ions in the process. The secondary ions are extracted into a mass spectrometer, which uses electrostatic and magnetic fields to separate the ions according to their mass-to-charge ratio. Ions of different mass-to-charge ratios are measured by changing the strength of the magnetic field. Dynamic SIMS determines the elemental composition and the trace levels of impurities and dopants in solid materials.

Applications

- Elemental analysis. Identifies all elements or isotopes present in a material, from hydrogen to uranium. Differentiates elements, isotopes, and compounds that have nominally identical atomic or molecular masses.
- Depth profiling. By sputtering into a material, dynamic SIMS determines the concentration of impurities or dopants as a function of depth. It is capable of resolving dopant and impurity levels whose concentration is as much as nine orders of magnitude less than the atomic composition of the material.
- Interface analysis. By sputter-removing material from the sample, dynamic SIMS determines the changes in composition or the diffusion of impurities from layer to layer.
- Microprobe image analysis. Uses raster scanning to produce microprobe images for detecting the lateral distribution of trace impurities. Image analysis in combination with depth profiling produces three-dimensional maps of elemental distributions.
- Direct image analysis. Uses the unique capabilities of the instrument to produce a direct secondary ion image — for rapid determination of the lateral distribution of trace elements.

Special Features

- Several primary ion sources, including argon, oxygen, and cesium. Cesium is especially useful for the analysis of lighter elements such as hydrogen, carbon, oxygen, and nitrogen. Oxygen is used to enhance sensitivity for boron and transition metals.
- Depth resolution <10 nm. Allows analysis of buried interfaces and determination of subtle differences in diffusion profiles.



Dynamic SIMS can be used for depth profiling. Here, depth profiles show how the diffusion of Zn in GaInP is a function of the n-type dopant. When Se is used as the n-type dopant, Zn does not redistribute during growth. When Si is used, significant Zn diffusion occurs. (Note: Dynamic SIMS output can be produced in full color, which allows the depth profiles to be more easily delineated for the user.)

MAJOR INSTRUMENTATION FOR SURFACE ANALYSIS

System	Analytical Technique	Typical Applications	Beam Source	Spot Size	Signal Detected
Cameca IMS 3f	Dynamic secondary ion mass spectrometry	Elemental and interface analysis, high-sensitivity profiling, mass scans	Primary ions (Cs, O, Ar)	10 to 200 µm	Secondary ions
Cameca IMS 5f	Dynamic secondary ion mass spectrometry	Elemental and interface analysis, high-sensitivity profiling, mass scans	Primary ions (Cs, O, Ar)	0.3 µm	Secondary ions
Cameca TOF SIMS IV	Static secondary ion mass spectrometry	Compositional and mass analysis, molecular and surface analysis	Primary ions (Ar, Ga, O)	<200 nm (Ga ion)	Secondary ions
Leybold LHS-10	Ion scattering spectroscopy	Elemental analysis of top surface monolayer, compositional depth profiles	Noble gas ions (He, Ne, Ar, Xe)	125 to 600 µm	Scattered ions
Physical Electronics PHI 670	Auger electron spectroscopy	Elemental surface analysis, multi-point analysis, small feature analysis	Focused beam of electrons	15 nm	Auger electrons
Physical Electronics PHI 5600	X-ray photoelectron spectroscopy	Chemical-state and elemental analysis, compositional depth profiles	X-rays	26 µm	Photoemitted electrons

- Mass resolution of M/ΔM ≥ 7000. Allows the separation and identification of isotopes, elements, and compounds that have nominally identical atomic or molecular masses.
- Sensitivities to 1 ppb (~10^{13} at/cm^3). Enables detection of trace levels of contaminants and dopants in materials. Quantitative with standard samples.
- Detection of hydrogen and deuterium. The only surface analysis technique capable of directly detecting hydrogen and deuterium in materials.

Static Secondary Ion Mass Spectrometry (Static SIMS)

Static SIMS uses a pulsed primary ion beam to sputter material from the top monolayer of a sample. Secondary ions are collected and focused into a reflectron time-of-flight (TOF) mass spectrometer, where they are separated according to mass. Mass separation is performed by measuring the length of time it takes secondary ions to reach the detector — the lighter the ion, the less time it takes to reach the detector. Highest mass resolution is achieved by using the shortest primary ion pulses — the shorter the pulse, the more precise the determination of the time it takes ions to reach the detector. The high transmission of the TOF analyzer (the static SIMS collects and analyzes between 20% and 60% of all secondary ions generated) allows the use of short pulses and low removal rates.

Applications

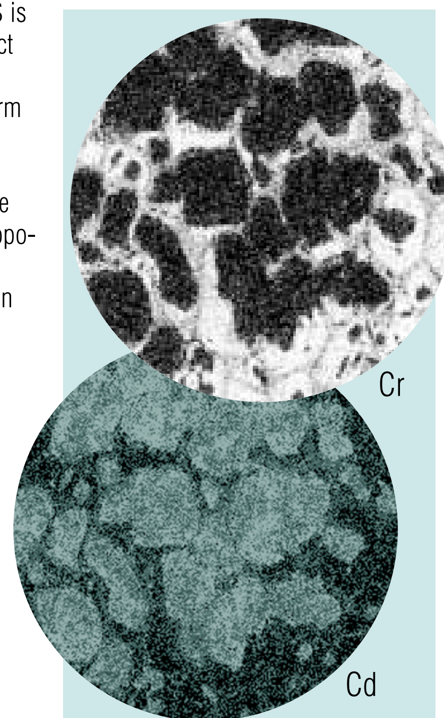
- Surface sensitive. Low doses of primary ions ensure that every secondary ion comes from an undisturbed region of the surface, allowing true surface analysis with SIMS sensitivities.
- Compositional analysis. Performs mass analysis to determine the elemental and/or molecular composition of the top monolayer of a material's surface. Can distinguish elements and molecules whose masses range from 1 amu to >10,000 amu. Can be quantitative with standards.
- Wide range of materials. Can be used for mass analysis of a wide range of organic and inorganic materials, including thin-films, solid-state materials, ceramics, polymers, biological samples, and catalyst particles.
- Imaging. Using a raster probe, static SIMS generates images to determine the lateral distribution of secondary ions, with a lateral resolution ≤ 0.2 µm.

Special Features

- Multiple ion sources. Two analytical sources — argon gas and gallium liquid metal — promote analytical flexibility. Also uses a low-energy argon or oxygen sputter gun for depth profiling with high depth resolution.
- Small spot size. The gallium ion source is capable of providing a spot size of less than 200 nm, enabling high lateral resolution.
- High vacuum of $<8 \times 10^{-10}$ torr. Promotes a clean specimen surface, with less recontamination, for a better analysis.
- A precisely pulsed acceleration potential and time-to-digital converter. Enables picosecond resolution of the flight time of secondary ions, and hence, allows a high mass resolution.
- Mass resolution of M/ΔM > 7000. Allows precise differentiation among isotopes, elements, and compounds that have similar atomic or molecular masses.
- Depth resolution. Using a low-energy sputtering beam, the static SIMS is capable of depth resolutions of <5 nm for shallow depth profiles.
- Lateral resolution. With the gallium source, the static SIMS is capable of 0.2 µm lateral resolution, while maintaining mass resolutions of >6000 FWHM (full width at half maximum).

Auger Electron Spectroscopy (AES) and Scanning Auger Microscopy (SAM)

With AES, a sample surface is bombarded with a focused beam of electrons, which generates Auger electrons that are collected and measured. Auger electrons have discrete kinetic energies that are characteristic of the emitting atoms, making AES particularly useful for identifying elemental composition. A key capability of AES is the ability to focus and deflect the electron beam, thereby allowing the analyst to perform scanning Auger microscopy, which is used to generate element-specific maps. These maps may be compared to topographic maps, such as those acquired by scanning electron microscopy (see *Analytical Microscopy* insert).



Spatially resolved AES images of Cr (upper image, dark areas) and Cd (lower image, light areas) in a CdTe solar cell with a Cr back contact. After high-temperature anneal, Cr is found in regions surrounding CdTe grains, indicating Cr diffusion from the back contact.

Elements Detected	Detection Limits	Mass Res.	Lateral Resolution	Depth Res.	Depth Profiling?	Imaging/ Mapping?	Organic Info.?
All elements and isotopes, H to U	To nearly 1 ppb (at/cm^3)	≥7000	1 µm	< 10 nm	Yes	Yes	—
All elements and isotopes, H to U	To 1 ppb (at/cm^3)	≥7000	0.3 µm	< 10 nm	Yes	Yes	—
Isotopes of organic and inorganic materials with amu of 1 to >10,000	1 ppm (at/cm^2)	>7000	≤0.2 µm	< 5 nm	Yes, limited	Yes	Yes
C to U	Sub-mono-layer	—	—	Surface monolayer	Yes	No	—
Li to U	0.5 at.%	—	15 nm	—	Yes	Yes	—
Li to U	0.5 at.%	—	26 µm	—	Yes, limited	Yes	Yes

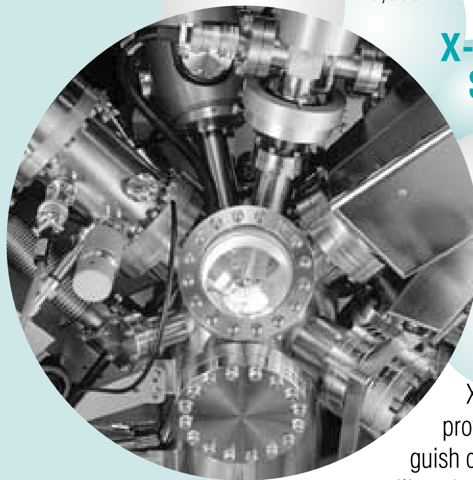
Applications

- Elemental surface analysis. Identifies and quantifies elemental compositions of solid surfaces, with a sensitivity to 0.5 at.% for lithium to uranium.
- Depth profiling. By sputtering a surface with argon ions, performs elemental analysis at various depths. Especially useful for bulk and volumetric analysis and for exploring impurities and diffusion present at junctions and grain boundaries.
- Line-scan analysis. By deflecting the electron beam in one direction, analyzes multiple points across a surface.
- Imaging. By employing raster scanning, performs SAM to produce compositional maps of a surface. Can also produce SEM micrographs with up to 20,000x magnification.
- Small-feature analysis. Because of small spot size and large magnification, can be used to explore small particles or intricate features, such as those found in microelectronics.

Special Features

- Field emission source. Produces a small spot size for electron beam with high current density.
- Small spot size. Spot size of several tens of nanometers results in lateral resolutions ≥ 15 nm.
- Nanoprobe. Allows raster scanning to produce compositional maps of small areas, with magnifications up to 20,000x.

X-Ray Photoelectron Spectroscopy (XPS)



The Physical Electronics PHI 5600 uses X-ray photoelectron spectroscopy to perform chemical-state and elemental analyses on a wide range of materials. (Jim Yost Photography/ PIX01437.)

The XPS technique involves the bombardment of a sample surface with X-rays and the measurement of the concomitant photoemitted electrons. The photoemitted electrons have discrete kinetic energies that are characteristic of the emitting atoms and their bonding states. XPS goes beyond elemental analysis to provide chemical information. It can distinguish chemical arrangements such as silicon-to-silicon bonds from silicon-to-oxygen bonds.

Applications

- Chemical-state analysis. Evaluates valence states, bonding environments, and the molecular composition of surface layers.
- Elemental analysis. Identifies elements from lithium to uranium, with detection levels down to 0.5 at.%.
- Imaging. Uses raster scanning to produce images with a spatial resolution of 26 µm.
- Depth profiling. By sputtering material from a surface, generates compositional depth profiles for materials up to 1 µm thick.
- Thin-films. Frequently used for the analysis of surfaces of thin-film materials.
- Polymers. Especially valuable for analyzing functional groups in polymers and other organic materials. Particularly useful in this regard when used as a complementary tool with static SIMS analysis.
- Catalysts. Evaluates the surface of catalysts to determine reactive species.
- Other materials. Valuable for chemical-state analysis of materials ranging from metals to insulators to semiconductors.

Special Features

- High vacuum of 10^{-10} torr. Reduces contamination for reliable analysis.
- High-energy resolution analyzer. Minimizes the wavespread of the photoelectrons as they enter the analyzer, allowing accurate determination of energies.
- Small spot size. The size of the analysis area can be tuned to as little as 26 µm. This enables the analysis of small areas and features.
- Aluminum and magnesium modes. X-ray beams of 1486.6 eV and 1253.6 eV impart precise energy to photoelectrons, allowing accurate determination of chemical and bonding information.
- Monochromated X-rays. Precisely restricts the wavelength to remove satellites (associated X-rays of different energies, that are less intense than the primary, and preferred, X-ray) from the AlK_{α} X-ray line. This ensures that the energy imparted to photoelectrons will be precise.
- Angle-resolved photoelectron spectroscopy (ARPES). With ARPES, photoelectron spectroscopy can be performed from a variety of angles, from normal to the surface to nearly parallel to it. This yields a nondestructive method for producing depth profiles of near-surface regions.

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