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Laboratories

Time-Of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) Services

Time-of-Flight SIMS (TOF-SIMS) is a surface-sensitive technique, providing full elemental and molecular analysis with excellent detection limits

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is an analytical technique that uses a high-energy, primary ion beam to probe the surface of a material. The instrument is typically operated in the static mode for obtaining elemental and molecular-chemical information from both organics and inorganics. In this mode of operation, the sample integrity and chemistry are preserved by applying extremely low primary ion doses (less than 1×10^{12} ions/cm^2) during the entire experiment. This ensures that roughly less than 0.1% of the surface atoms or molecules are ever struck and damaged by the primary ion beam. TOF-SIMS is the most surface sensitive of the surface analytical techniques, with a depth of analysis of only approximately 1 nm. The technique has extremely good detection sensitivities, with detection limits for most elements in the parts-per-thousand to parts-per-million range.

TOF-SIMS works by rastering a pulsed beam of focused primary ions across the area of interest, resulting in the emission of secondary ions which are characteristic of the materials present in the top several monolayers of the sample. By accurately measuring the masses of the detected ions, they can be identified and related to the chemical species present on the sample surface. Use of a finely focused primary ion beam (typically, Gold or Bismuth cluster) makes it possible to record the lateral distribution of chemical species across a surface with micron to submicron spatial resolution. Because secondary ion yields vary dramatically from element to element and are highly dependent on the matrix from which they are sputtered, quantification with TOF-SIMS can be extremely difficult. Thus, in most cases, the technique is used more for qualitative purposes than for quantitative analyses. Relative comparison of a given species is possible between samples when the substrates are the same. Depth profiles can also be obtained if the technique is done in conjunction with ion sputtering (with either the same primary ion gun or using an additional Cs, O, or Ar cluster ion beam).

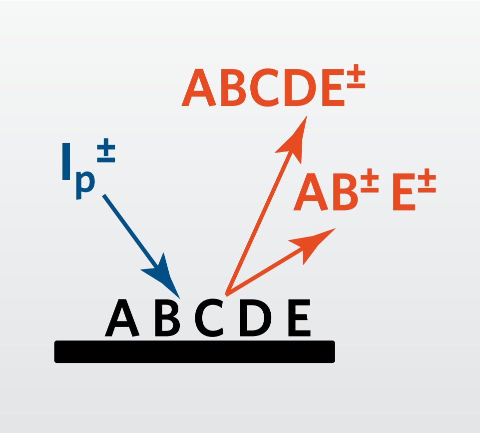


Figure 1: General schematic of how TOF-SIMS works

Strengths

- Surface sensitive; top few monolayers
- Detection limits in the ppm range
- Survey analysis
- Survey depth profiles
- Elemental and molecular information
- Can analyze insulators and conductors
- Sub-µm spatial resolution possible in imaging mode
- Major element composition possible, in some applications

Limitations

- Absolute quantitation is difficult without extensive calibration
- Can be too surface sensitive so careful sample handling/packaging is important
- Samples must be vacuum compatible
- Datasets can be complex

Common Applications

Its excellent surface sensitivity, ability to analyze organic materials and other insulators, excellent detection limits, and ability to provide elemental and molecular information makes TOF-SIMS an ideal technique for addressing the following types of applications:

- Surface characterization of organic and inorganic materials
- Imaging the lateral distribution of surface species
- Survey depth profiling
- Contaminant identification (down to the ppm range for elemental or molecular species)
- Quantitative analysis of surface metals on wafers
- Failure analysis, eg: adhesion, bond pads, coatings
- Evaluation of cleaning processes (QA/QC)
- Identification of stains, discolorations, and hazes
- Examining surfaces before and after processing to identify differences
- Comparison of samples processed or stored in different environments to determine surface changes

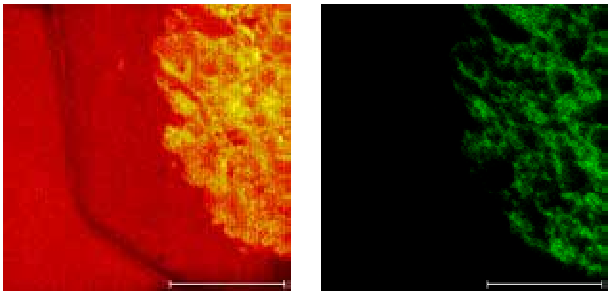
Industry Sectors and Technologies

- Medical devices
- Semiconductors
- Aerospace & defense
- Electronics
- Energy storage, batteries and solar/PV

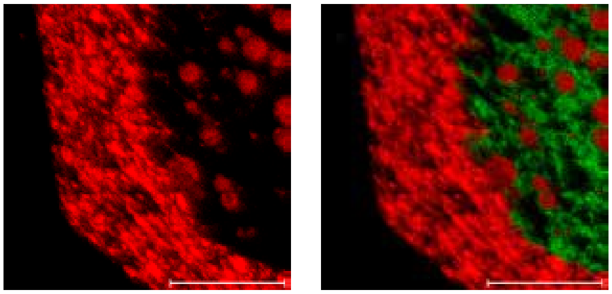
Case Study: Chemical Imaging of Drug Product

In this study the objective was to image ingredients in an over-the-counter (OTC) heartburn medicine. Lansoprazole is the API. The real power of TOF-SIMS imaging is shown in the upper right image which is the 2-dimensional image of lansoprazole molecules within the drug. An overlay of Mg and lansoprazole shows the complimentary distribution of each material. A similar map (not shown) for hydroxypropyl cellulose (HPC) confirms that it is located in the core of the pellet.

2-dimensional chemical imaging of finished drug products can be invaluable for understanding the location and distribution of organic and inorganic constituents. This information is important for formulation scientists. It can also be critical in intellectual property cases where patent law frequently dictates the location and function of materials.



Total Ion *lansoprazole*



Mg *Overlay*

Figure 2: TOF-SIMS ion images of cross-sectioned drug bead

Case Study: Root Cause Determination of Adhesion Failure in Medical Device Packaging

A medical device sterile package experienced adhesive failure at a polyethylene-ethylene acrylic acid (co-polymer) heat seal to polyethylene. If undetected, these types of failures can lead to loss of sterility and result in serious harm to patients. Adhesion failures are frequently chemical in nature and can be caused by very low levels of surface contaminants.

Ethylene acrylic acid (EAA) was added at 3% to improve the adhesion and lower the crystallinity of the polyethylene (PE). Good and bad heat seal surfaces were examined. Both surfaces contained ions indicative of PE (C_2H_3, C_3H_5, C_4H_7, etc.) and EAA (CH_3O, C_2H_5O, etc.). However, the Bad heat seal also contained intense peaks characteristic of a hydroxyhydrocinnamate compound. Such compounds are common antioxidants under the Irganox® brand. The elevated levels of the antioxidant on the heat seal surface were concluded as the root cause of the adhesion failure.

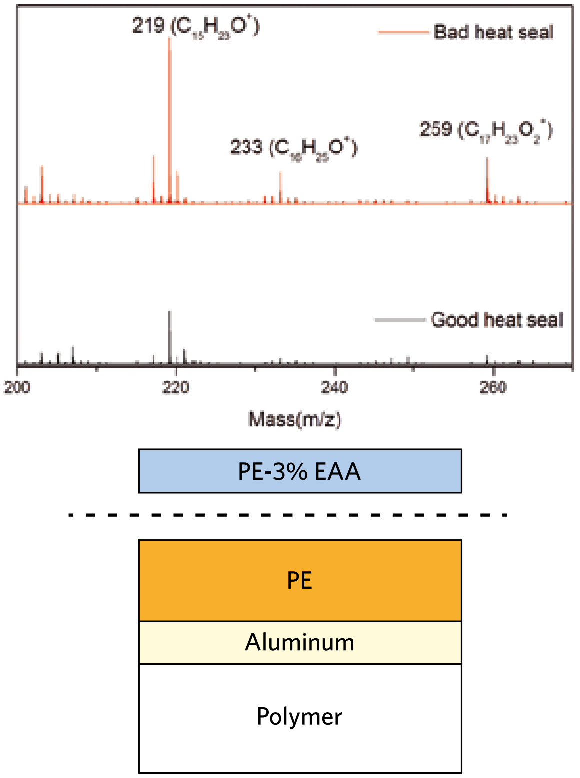


Figure 3: TOF-SIMS spectra and schematic of the adhesive failure in medical device packaging

Complementary Techniques

Other surface analysis tools with similar depths of analysis or applications include X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES) and Fourier Transform Infrared Spectroscopy (FTIR). XPS provides quantitative concentrations and chemical bonding information that is not normally obtained directly using TOF-SIMS. AES can provide better spatial resolution images for elemental species, but with poorer sensitivity. FTIR can provide complementary organic information and has a greater information depth and access to commercial library spectra. This may make FTIR a better choice for identification of macroscopic amounts of material, where extreme surface information may not be of prime interest.

TOF-SIMS at EAG

EAG has several TOF-SIMS instruments located at our Sunnyvale, CA and Chanhassen, MN labs. Some of these instruments contain special capabilities such as large sample stages or sample cooling for the analysis of semi-volatile materials in vacuum. Our TOF-SIMS experience is unsurpassed with many of our TOF-SIMS scientists having more than 10 years of practical TOF-SIMS experience across a range of industries and applications. Historically, the California lab of EAG (Charles Evans & Associates) was directly involved in the early commercialization of TOF-SIMS instruments giving EAG unmatched practical analytical experience.

At Eurofins EAG, our technical experts have many years of experience working with a wide range of materials. With our breadth of techniques and expertise, we work closely with our clients to tailor the analytical plan to their needs. Contact us today to learn how we can help.