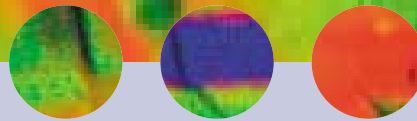


# SURFACE AND DEPTH PROFILING ANALYSIS AT CSMA

# CSMA



## INTRODUCTION

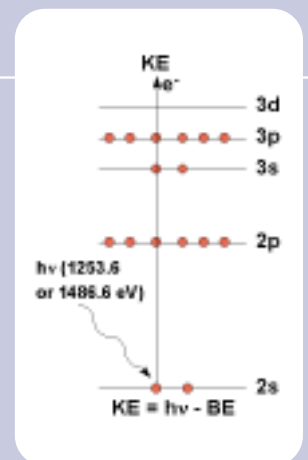
Understanding the **surface** and **interface** properties of modern materials is an essential requirement for a wide range of industries. Using the latest surface analysis techniques it is possible to measure the chemical and physical composition of the outermost atomic layers of any solid material, routinely. This region can control factors such as biocompatibility, lubrication, corrosion resistance, colour, finish, adhesion strength, odour and printability.

The most useful surface analysis techniques are based upon mass spectrometry and electron spectroscopy.

## SURFACE ANALYSIS TECHNIQUES

### X-RAY PHOTO-ELECTRON SPECTROSCOPY (XPS OR ESCA)

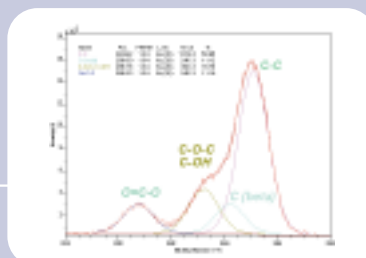
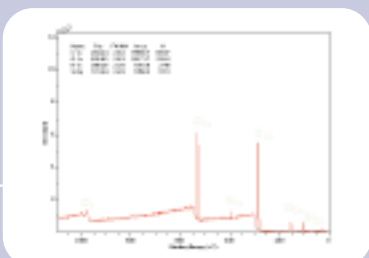
**XPS** uses an X-ray beam of energy  $h\nu$  to stimulate the emission of electrons from the sample. The electrons analysed do not have sufficient energy to escape from a depth of more than  $\sim 10\text{nm}$ , so the XPS technique is inherently surface sensitive. The kinetic energy (KE) of the emitted electrons is measured in an electron spectrometer and used to identify the elements present by calculating the electron binding energy (BE). The diagram shows the electron emission process.



*XPS photoelectron emission process.*

XPS provides :-

- Identification of all elements present in the surface (except H and He).
- Chemical state, oxidation state and functional group information. This is optimised through the use of a monochromatic x-ray source.
- Measurement of the thickness of surface layers and depth distributions in the 1nm - 10nm range using Angle Dependent XPS.
- Ready quantification of elements in the concentration range 100 at.% -  $\sim 0.1$  at.%, on conducting and insulating materials.



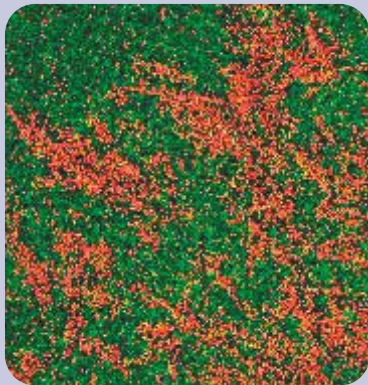
*XPS spectra of a polymer surface showing silicone contamination (left) and carbon functionality (right)*

## SECONDARY ION MASS SPECTROMETRY (SIMS)

**SIMS** involves the production of secondary ions by bombardment of the sample with a primary ion beam. The impact of the primary ion causes an atomic scale collision cascade within the surface layers of the sample and, at points remote from the point of impact, secondary ions are ejected from the surface. These ions are then analysed by mass spectrometry. CSMA uses both Time of Flight SIMS and Dynamic SIMS routinely for its problem solving applications

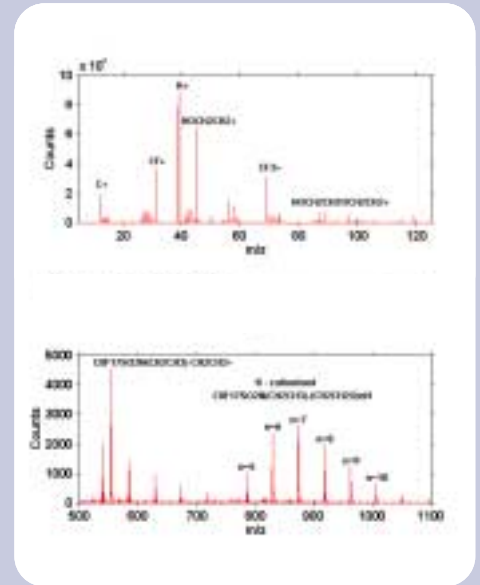
In **Time of Flight SIMS (ToFSIMS)**, a pulsed primary ion beam irradiates the surface of interest. The flight times and intensity of elemental and molecular fragment ions are measured in a time of flight mass spectrometer followed by conversion to a mass spectrum. The features and benefits of **ToFSIMS** are :-

- **Elemental and molecular information** from the uppermost surface layer of all solid materials including insulators.



*ToFSIMS image overlay showing the distribution of an organic crystalline deposit (red) on a filter paper*

- Detailed chemical information and **empirical formulae** through the use of reference library spectra and accurate mass analysis.
- Detection **sensitivity** in the ppm range for all elements.
- **Chemical mapping** of elements and molecular species with sub-micron resolution.
- Retrospective mass spectral and imaging analysis of complex surfaces using **Region of Interest** mode.



*ToFSIMS spectrum of the surface of a polymer mesh from a medical device identifying the molecular formula of a fluoro-surfactant treatment*

**Dynamic SIMS (DSIMS)** provides quantitative information on the elemental composition of the sample surface region from a few nm to several hundreds of microns in depth. The detection sensitivity of the technique is in the ppm - ppb range for all elements in the Periodic Table.

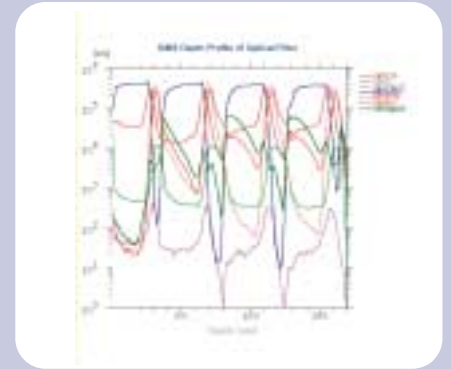
In Dynamic SIMS a primary ion beam of energy 0.5keV - 20keV is used to sputter-remove successive layers of the sample in a well-defined area ranging in size from typically 500 x 500µm to 10 x 10µm. The emitted positive and negative secondary ions are collected and mass analysed in a mass spectrometer (Cameca ims 4f). The secondary ion signals are representative of the sample composition and, with appropriate use of standard samples, can be used to produce quantitative information on the lateral and depth distribution of major elements, dopants and impurities in any solid material



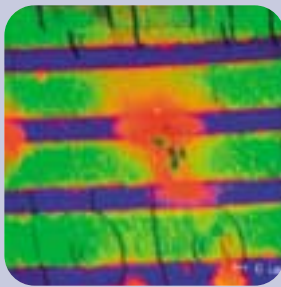


## THE FEATURES AND BENEFITS OF DSIMS ARE :-

- **Monitoring** of elemental composition on the surface with sensitivity in the ppm - ppb range - allows ready identification of process contaminants, impurities and surface treatments.
- **Depth profiling** from surface to bulk - provides characterisation of layer structures, buried features and interfaces.
- **Ultra-Shallow Depth Profiling** - specially developed for analysis of thin layer structures and shallow implants.



*SIMS depth profile of an optical filter device.*

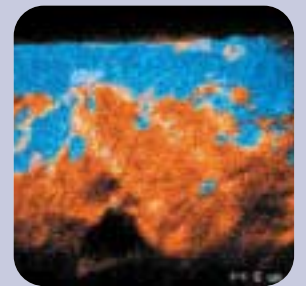


*SIMS image overlay showing corrosion features on a semiconductor device. (Al-blue; Si-green; TiO<sub>2</sub>-red)*

- **Chemical mapping** on feature sizes down to <math>0.5\mu\text{m}</math> - provides clear diagnosis of fault areas.
- **Line scan analysis** - gives precise registration of surface features and chemical boundaries.
- **Retrospective 3D analysis** provides chemical maps and area-selected depth profiles from complex materials.

- **Cross Sectional Analysis** - extended information from the surface features to bulk structure, particularly effective on complex, thick layer materials.

The **Cameca IMS 4F** at CSMA is the industry-standard instrument for DSIMS analysis. CSMA scientists have developed a unique range of methodologies to optimise problem-solving from the whole range of technologically - important materials including metals, ceramics, polymers, fibres, printed circuit boards and paper products,



*SIMS image overlay of cross-section of hard wood showing penetration of lacquer treatment (C<sub>1</sub>H<sub>5</sub>-blue) into wood (CN-brown).*

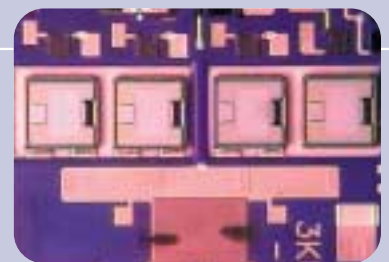


## SUPPLEMENTARY TECHNIQUES

In many cases a range of supplementary techniques may be used to help solve the problem. For example a visualisation of the sample area by optical microscopy or scanning electron microscopy is often the initial step in the analytical process.

### OPTICAL MICROSCOPY

CSMA has a range of optical microscopes which provide visualisation of areas from several cm down to  $\sim 10\mu\text{m}$  in size. These are used for precise area location and provide a digitally recorded image of the sample before or after surface analysis investigation.



*Optical micrographs of part of an automobile engine management control pcb (Field of view 2cm and 200 $\mu\text{m}$ )*

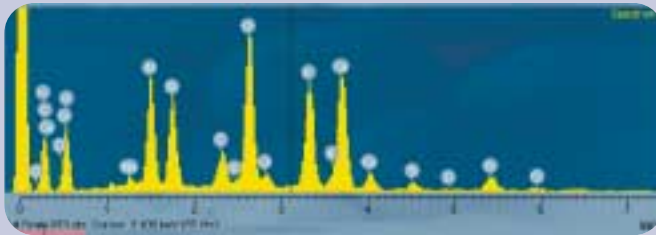
## SCANNING ELECTRON MICROSCOPY (SEM)

SEM provides detailed micro-structural and compositional information on sample areas from ~1mm to <math>10\mu\text{m}</math>. The features and benefits of SEM analysis include :-

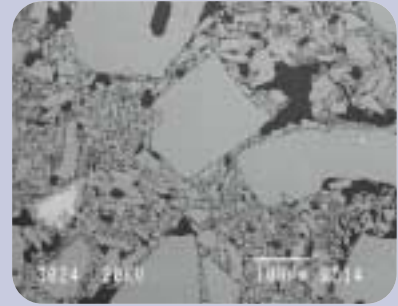
**Topographic information** from the sample surface using Secondary Electron mode

**Elemental contrast** visualised using Backscatter Electron mode

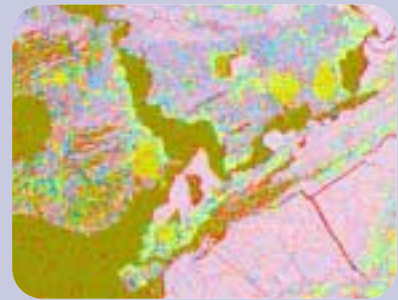
**Energy Dispersive Spectroscopy (EDS)** - for quantitative compositional information from Na - U with sensitivity down to ~0.1 at. % and a sampling depth of 0.1 $\mu\text{m}$  - 2 $\mu\text{m}$



*EDS spectrum from a KCl - attacked refractory sample*



*Backscatter electron image of silicon carbide grains in a refractory sample.*



*Distribution of components in refractory sample [potassium chloride (pink), alumina (yellow) and calcium (green)].*

## STYLUS PROFILOMETRY

In Stylus Profilometry the sample surface is scanned linearly on regions of interest aligned using an optical microscope system. The technique provides measurement of :-

- Average surface roughness
- Step heights
- Calibration of crater depths following depth profile analysis

**In addition to the above techniques, CSMA also provides the following on a routine basis :**

Fourier Transform Infra Red Spectroscopy (FT-IR)	Atomic Force Microscopy
Raman Spectroscopy	Contact Angle
Laser Scanning Optical Microscopy (LSOM)	Differential Scanning Calorimetry (DSC)
GC-MS	Thermo-Gravimetric Analysis (TGA)
Ion Chromatography	Dynamic Vapour Sorption
ICP-MS	Surface Area / BET

CSMA provides a complete surface analysis service to industry to accommodate every level of demand:

- rapid turnaround analysis (24 hours)
- problem solving and failure analysis
- litigation and expert witnesses
- training courses
- reverse engineering and competitor analysis
- materials and product development
- patent registration / infringement

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