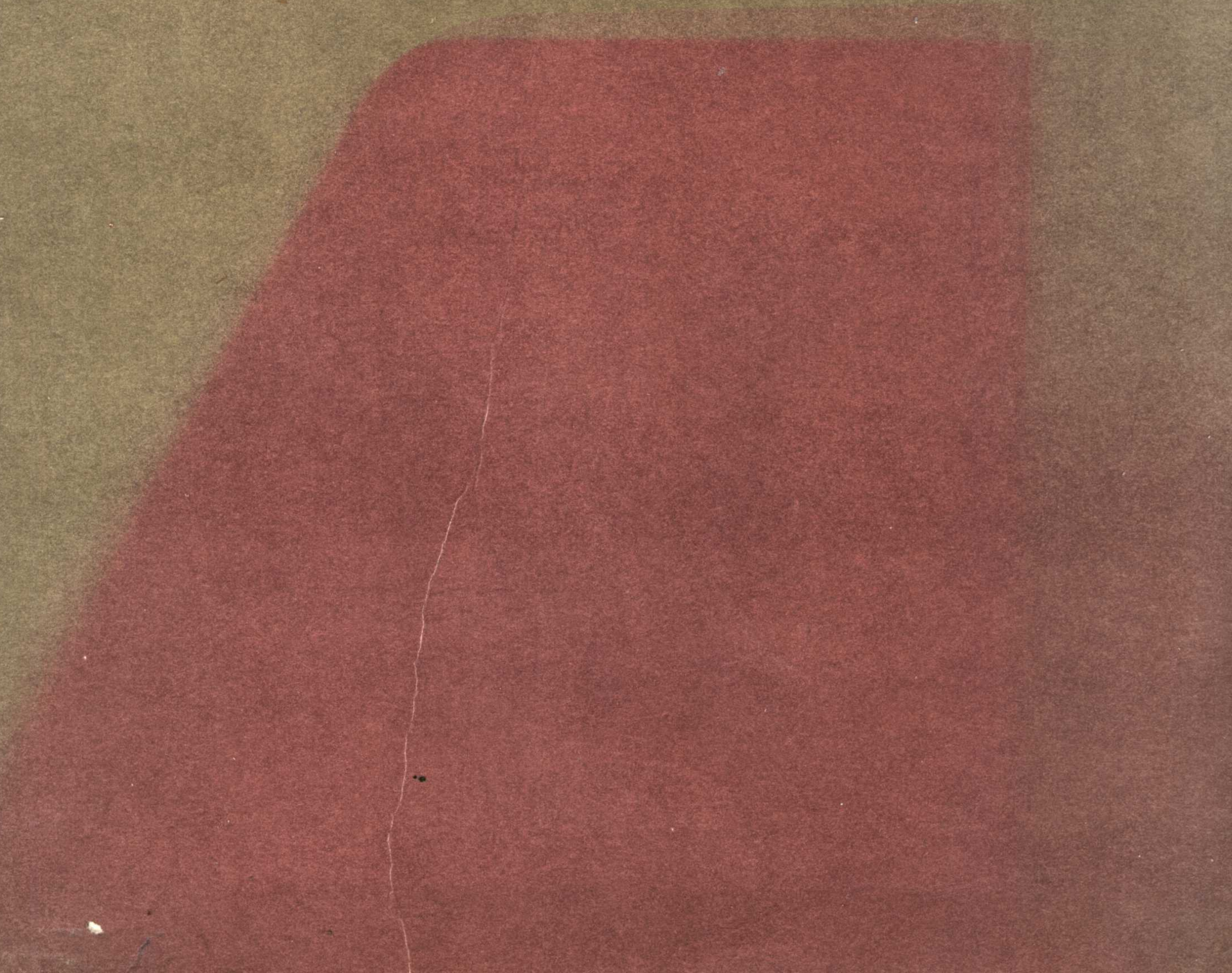
Spectroscopic Characterization Techniques for Semiconductor Technology II

Fred H. Pollak
Chairman/Editor



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SPECTROSCOPIC CHARACTERIZATION TECHNIQUES FOR SEMICONDUCTOR TECHNOLOGY II

Volume 524

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SPECTROSCOPIC CHARACTERIZATION TECHNIQUES
FOR SEMICONDUCTOR TECHNOLOGY II

Volume 524

INTRODUCTION

The semiconductor industry has gone through some very dramatic changes during the past several decades. The electronic (and optoelectronic) devices of the 1980s are a far cry from isolated large area junction transistors of thirty years ago. Many new developments have occurred, such as heterojunctions, quantum wells, and superlattices, which were undreamed of only a few years ago. These advances are the result of new technologies including microelectronic processing, structure fabrication, and interface preparation as well as thin film deposition methods such as VPE, MBE, MOCVD, etc. Thus there is an increasing demand for characterization techniques which can provide greater information with ever-increasing sensitivity, precision, and spatial resolution. Spectroscopic techniques offer powerful methods for probing relevant materials and device parameters.

Within the last several years the SPIE has organized several conferences devoted to such characterization approaches including one on optical methods in San Jose in 1981 and another on more general spectroscopic techniques in Cambridge in 1983. Although quite a number of important methods were discussed at these meetings it was not possible to cover all relevant techniques. Also, in many of these areas the developments are occurring so rapidly that new approaches evolve almost every day. Therefore it was felt that another conference should be held in order to explore important methods not dealt within the previous meetings and to update others.

High energy beams of photons, electrons and ions are emerging as powerful tools for investigating bulk, surface, and interface properties with increasingly greater resolution and sensitivity. For example, electron microscopy, which can have spatial resolution down to the angstrom level, yields both structural and chemical information. The availability of tuneable synchrotron radiation is having a major impact on surface and interface studies as well as on X-ray topography. The oscillations in RHEED patterns during MBE growth are being utilized to gain valuable insights into the mechanisms of thin film fabrication. A number of other methods such as UPS, XPS, EXAFS, SEXAFS, PIXE, RBS, etc., were also reviewed.

The method of deep level transient spectroscopy together with electron paramagnetic resonance offers a powerful combination to characterize impurities and defects in both the bulk and at interfaces.

Optical techniques in the near ultraviolet to the far infrared are still some of the most powerful approaches for semiconductor characterization, including the properties of impurities. Many of the more mature methods, such as infrared transmission, modulation spectroscopy, Raman scattering, etc., are being employed in new ways to explore long-standing problems and also to gain important insights into new structures. New experimental methods are emerging, such as time-resolved spectroscopies down to the picosecond and femtosecond regimes, which will allow the dynamics of semiconductor systems to be investigated.

We hope that the readers of these proceedings (and of the proceedings of related SPIE conferences) will gain a better understanding and appreciation of the large number of methods available to characterize and diagnose parameters relevant to semiconductor technology. The use of the information that can be derived from these state-of-the-art techniques can be an extremely valuable tool in hastening progress in the development of semiconductor devices.

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High Energy Photons, Electrons, and Ion Beams

Chairman
E. R. Weber
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An Overview of Surface Analysis Techniques and Their Applications in the Semiconductor Industry
(New Developments in ESCA)

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Surface Science Laboratories, Inc., 1206 Charleston Road, Mountain View, California 94043

Abstract

Materials characterization, using surface analysis and depth profiling techniques, plays a vital role in all aspects of semiconductor technology from basic research to manufacturing. The techniques are:

SEM/EDX/WDX - Scanning Electron Microscopy/Energy Dispersive X-ray/Wavelength Dispersive X-ray
AES - Auger Electron Spectroscopy
ESCA - Electron Spectroscopy for Chemical Analysis
SIMS - Secondary Ion Mass Spectrometry
RBS - Rutherford Backscattering Spectrometry
LIMS - Laser Ionization Mass Spectrometry

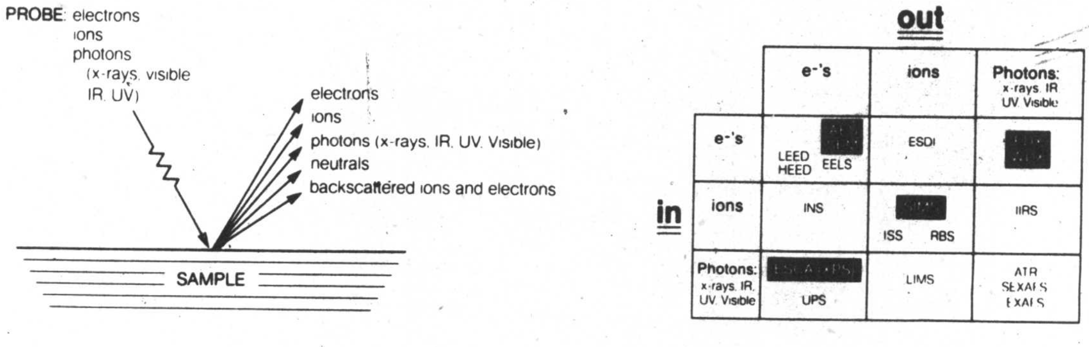
Chemical and elemental analysis of the first several atomic layers of a surface are performed. Detection limits range from less than a part per million to several atomic per cent, and areas of analysis can be as small as 5000 Angstroms in diameter. SEM capabilities provide topographical information at magnifications up to 200,000x.

An overview of these surface analysis techniques will be presented with an emphasis on the wide range of applications in the semiconductor industry. Recent advances in one technique in particular, ESCA, will be discussed.

Sorting out the alphabet soup

The use of acronyms is common in surface analysis and depth profiling (AES, ESCA, SIMS, RBS, LIMS, SEM, WDX, EDX, EBIC, BSE . . .); however, what seems to be a complex "alphabet soup" can be sorted out in a fairly simple manner, as illustrated in Figure 1. Table 1 explains the acronyms used in Figure 1. A sample surface, under vacuum, is probed with either an electron beam, an ion beam, or a flux of photons. Any of these probes cause several phenomena to occur and ions, electrons, and photons are emmitted or backscattered from the sample's surface. Which particles or photons are detected and what aspect of the emission phenomena is studied defines the technique being used and determines the information obtained from the investigation.

The most commonly used surface analysis and depth profiling techniques in an analytical laboratory are: ESCA (Electron Spectroscopy for Chemical Analysis), AES (Auger Electron Spectroscopy), and SIMS (Secondary Ion Mass Spectrometry). The use of RBS (Rutherford Backscattering Spectrometry), and LIMS (Laser Ionization Mass Spectrometry) has become much more common recently and their applications are steadily increasing. SEM/EDX/WDX (Scanning Electron Microscopy/Energy Dispersive X-Ray/Wavelength Dispersive X-Ray) are not strictly surface analysis tools but are used in conjunction with the other techniques in almost every analytical laboratory and grouped with the others in most discussions.



	e-'s	ions	Photons: x-rays, IR UV Visible
e-'s	AES SEM LEED HEED EELS	ESDI	EDX WDX
ions	INS	SIMS ISS RBS	IIRS
Photons: x-rays, IR, UV Visible	ESCA/XPS UPS	LIMS	ATR SEXAFS EXAFS

Figure 1. Probing a sample surface, under vacuum, with electrons, ions or photons results in several events, including the emission of ions, electrons and photons. Analyzing various aspects of these entities provides information about the sample.

Table 1. Defining the Alphabet Soup

ESCA - Electron Spectroscopy for Chemical Analysis
(XPS - X-ray Photoelectron Spectroscopy)
AES - Auger Electron Spectroscopy
SIMS - Secondary Ion Mass Spectrometry
RBS - Rutherford Backscattering Spectrometry
SEM/EDX/WDX - Scanning Electron Microscopy/Energy Dispersive X-ray/Wavelength Dispersive X-ray
LEED - Low Energy Electron Diffraction
HEED - High Energy Electron Diffraction
EELS - Electron Energy Loss Spectroscopy
INS - Ion Neutralization Spectroscopy
UPS - Ultraviolet Photoelectron Spectroscopy
ESDI - Electron Stimulated Desorbtion of Ions
ISS - Ion Scattering Spectrometry
LIMS - Laser Ionization Mass Spectrometry
IIRS - Ion Induced Radiation Spectrometry
ATR - Attenuated Total Reflectance
EXAFS - Extended X-ray Absorption Fine Structure
SEXAFS - Surface Extended X-ray Absorption Fine Structure

Reviewing the four most common techniques

ESCA. Electron Spectroscopy for Chemical Analysis (ESCA) employs low-energy X-rays (for example - the K-alpha line of aluminum at 1,487 eV) to excite the atoms of a sample. The X-ray photon energy is absorbed by an atom and is used to remove an inner shell electron. This electron is then emitted with a kinetic energy characteristic of the difference between the photon energy and the binding energy of the electron. The type of atom detected is determined by the energy of the emitted electron and the number of emitted electrons is proportional to the number of those atoms in the sample volume. These electrons are called photo-electrons. Although they are emitted from atoms fairly deep in the sample, only those in the top 1-10 monolayers are emitted without significant energy loss (from collisions) and only they are included in the ESCA peaks used for quantitative work.

Broad energy scans, called ESCA survey spectra, cover the entire range of detectable elements (ESCA does not detect hydrogen and its helium sensitivity is extremely low). Narrow energy scans, called high resolution spectra, show detail of a peak or peaks in the low resolution survey spectra. They yield more detailed information about the electron binding energies of the atoms involved and this relates to the chemical bonding of the atoms. Thus, high resolution spectra give chemical bonding information in addition to elemental composition. This chemical information is ESCA's primary "claim to fame".

Small-spot ESCA. Although ESCA has been extremely valuable as a surface analysis tool (especially because of the chemical information) it had been a slow, large area technique. Since 1981, several developments have been incorporated into ESCA instrumentation which have increased data acquisition speed, allowed areas as small as 150 microns in diameter to be analyzed and large samples to be introduced unbroken into the spectrometer (up to seven inch diameter disks).

The use of focused X-rays for truly small-spot ESCA was a major advance in instrument design. The focusing X-ray monochromator capable of producing a 150 micron diameter beam for ESCA was introduced in 1982 by Surface Science Laboratories in the X-Probe^{tm} family of ESCA systems and components. The innovations have revolutionized the technique, made routine depth profiling with ESCA possible for the first time and opened up many new areas of applications.

Rapid, routine depth profiling with ESCA. The ability to do routine rapid depth profiling using ESCA is one of the most important results of the development and introduction of focused X-rays for ESCA.

Until now, routine depth profiling and the ability to take a detailed look at a given depth or interface has been more conveniently done with Auger Electron Spectrometry. The previous impediment to depth profiling with ESCA was the large analysis area, typically several millimeters on a side. Ion etching of a large area was required and this is a slow and often nonuniform process. Also, ESCA data collection was slow. With small area analysis (approximately 150 microns in diameter) and high data rates (because of improved electron optics and parallel detection), these impediments have been eliminated.

An ESCA depth profiling application - analysis of a thin-film device. Devices made of multiple dielectric layers are being used increasingly in applications associated with many technologies.

The bandpass, efficiency, and longevity of such devices are critically affected by submonolayer contaminants in or between dielectric layers. A depth profile of such a device is very difficult with Auger Electron Spectroscopy due to sample charging; however, it is staight-forward with ESCA because sample charging is minimal and can be neutralized.

Figure 2 shows an ESCA depth profile of a device with alternating layers of SiO_2 and ZrO_2. This profile was taken by alternately etching the sample with an argon ion beam, (for approximately one minute) and doing a rapid analysis for the set of elements present. The area analyzed was about 300 microns in diameter. The analysis time per element was approximately 10 seconds.

The atomic concentration profile, shown in Figure 2 was compiled by automatically measuring the area under each peak for the major elements present, quantifying the results and plotting the atomic % as a function of etch time. Figure 3 shows a perspective compilation from smoothed 10 second accumulations of oxygen (1s) spectra, illustrating the chemical information obtainable. The oxygen peak is presented as a function of time during the profile which corresponds to depth in the sample. The depth etched was approximately 0.4 microns. The peak energy shift from one layer to another, gives information about the different chemical environments of the atoms in the different layers. The foreground peaks correspond to the greatest depth.

In this case inner layer contamination was not found, but a significant amount of carbon (~30 atom %) was present on the outermost surface.

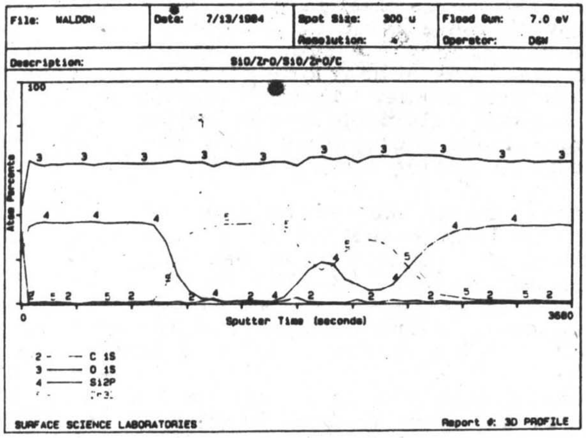


Figure 2. An ESCA depth profile of a multilayer device. The profiling rate was 50 Å/min.

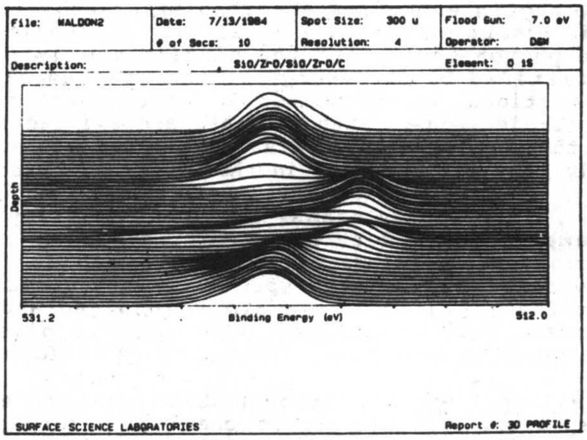


Figure 3. A perspective view of all the oxygen (1s) spectra used in the profile in figure 2. This plot provides a qualitative view of variations in chemistry as a function of depth.

AES. Auger Electron Spectroscopy involves the ionization of inner-shell electrons by an energetic (up to 10 keV) electron beam. An outer electron can then fill the vacancy created by the loss of the inner-shell electron, imparting its transition energy to another electron which is then ejected from the atom with energy in excess of the energy required to break its bond. The energy of the ejected "Auger" electron is thus characteristic of the type of atom involved. There are several such characteristic AES transitions (and therefore peaks) for most elements.

By raster scanning across a surface (with synchronized scanning of a display), and setting the detection system on a particular elemental peak, a picture of the surface distribution of that element is generated. Ion etching is used in conjunction with Auger analysis to produce concentration depth profiles of elements within a sample.

The "claim to fame" of AES is its extremely small area of analysis (one square micron or less) and rapid depth profiling. The analyst can determine elemental composition and distribution in three dimensions.

SIMS. Secondary Ion Mass Spectrometry (SIMS) employs a beam of ions (usually argon, cesium, or oxygen) at energies ranging typically from 500 eV to 5 keV to sputter material from the surface of a sample. Ions produced in this process are called secondary ions. These secondary ions are analyzed with a mass spectrometer, which distinguishes among elements and their isotopes by measuring the mass-to-charge ratio of the detected ions. SIMS is thus able to detect both hydrogen and deuterium, which ESCA and Auger analysis cannot do.

SIMS' "claim to fame" is its sensitivity, which can be as good as a fraction of a part per billion. It is especially sensitive to elements with low ionization potential (ie. Na, K) as well as elements with favorable electron affinities (ie. Group V and Group VI elements). However, spectral interferences, and wide variations in secondary ion yields cause significant variations in detection limits for routine trace analysis. Detection limits range from less than a part-per-billion to parts-per-thousand in routine analysis.

SIMS is used routinely in the semiconductor industry to investigate dopant and impurity depth profiles at very low levels after ion implantation or diffusion and the changes which occur in those distributions after subsequent processing. Like AES, the X,Y distribution of an element on the surface is obtained using beam rastering techniques. SIMS is also a three dimensional microanalysis technique.

Two techniques recently becoming popular

RBS. Rutherford Backscattering Spectrometry (RBS) uses high energy helium ions, typically 1 to 3 meV to perform non-destructive depth profiles in the outer 0.5 to 3.0 microns of the solid material. This technique is less commonly found in laboratories because it requires a helium ion accelerator. Only recently has RBS become more widely used because of the commercial availability of complete systems.

Samples are bombarded with the energetic helium ions and collisions occur with the outer surface and sub-surface atoms. The backscattered helium ion experiences an energy loss during backscattering which is characteristic of the atom with which it collided (the Rutherford Process) and an energy loss characteristic of the distance into the sample at which the collision occurred. Thus both elemental analysis and distribution with depth are determined simultaneously. Backscattered ions are detected using surface barrier detectors and multichannel analyzers. The accessible depth is dependent on the density of the material, but is generally in the range of 0.5 - 3 microns.

The "claim to fame" of RBS is its ability to provide quantitative information on the composition and thicknesses of multicomponent thin film structures without the use of standards. Also RBS is used to study crystallographic perfection after ion implantation and thermal processing.

Semiconductor applications include the stoichiometry of noble and refractory metal silicides and the composition of silicon and copper doped aluminum. Its use in the investigation of materials systems involving thin films is increasing.

LIMS. Laser Ionization Mass Spectrometry (LIMS) was first introduced about five years ago. A microfocused laser beam is used to probe a surface in one of two modes: Laser desorption (LD), which uses low laser-power density and laser ionization (LI) which uses higher power density. In both modes the ions produced are analyzed using a mass spectrometer of the Time-of-Flight (TOF) design.

Laser desorption produces ions from only species adsorbed on the surface and the spectra consist mostly of ions of the alkalies and molecular ions characteristic of organic contaminants. In laser ionization the ions are produced during vaporization of the sample surface and mostly atomic or fragmented molecular ions are found in the spectra. The ability to distinguish between adsorbed materials and materials incorporated in the sample matrix is a valuable application of LIMS.

The utility of the laser microprobe lies in its ability to obtain a mass spectrum of the contents of a volume of solid material 1-2 microns in diameter and either one to several monolayers thick in the case of laser desorption, or 1000-3000 Angstroms thick in the case of laser ionization. A rapid survey analysis of a localized volume can be obtained in the time required to locate the desired area (minutes) and the time required to acquire a TOF mass spectrum (60 microseconds).

Figure 4 illustrates and summarizes the probe and detected event aspects of these five techniques.

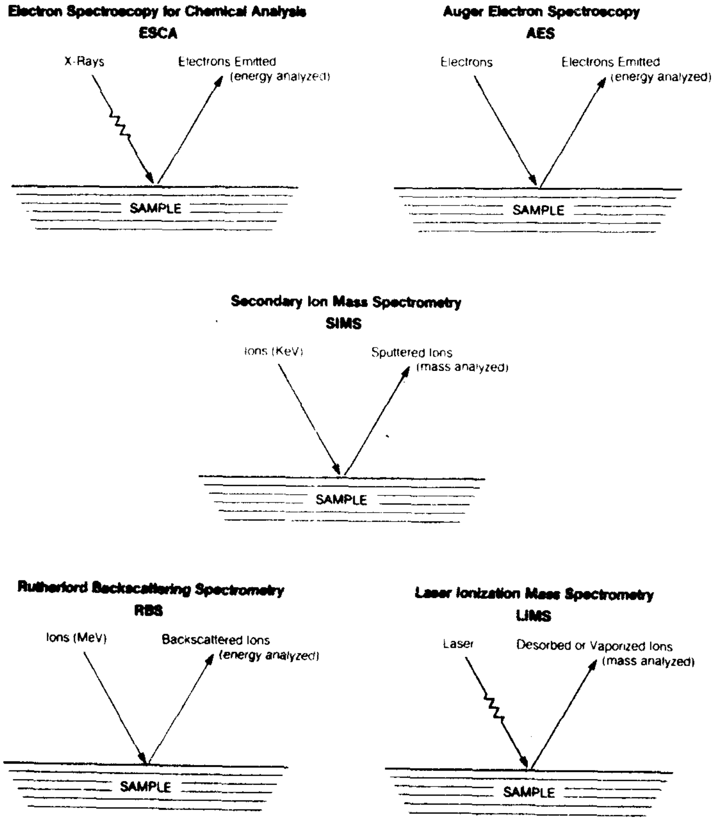


Figure 4. The probe and detected event are illustrated for the five most commonly used elemental analysis and depth profiling techniques.

Scanning Electron Microscopy and its related techniques

SEM/EDX/WDX. In the scanning electron microscope (SEM), a beam of electrons from 1 to 40 keV is directed at the surface of a sample in a raster pattern. The low energy secondary electrons that are emitted from the surface are detected and the detected current used to intensity modulate the Z axis of a cathode ray tube. An image is formed on the display by scanning in synchronism with the electron beam. If the sample is not electrically conductive it is usually necessary to coat it with carbon, gold, or some other metal to provide a conductive surface.

The SEM extends the magnification range possible with an optical microscope and provides a considerably better depth of field (sample thickness that is in sharp focus). SEM's with magnifications up to 200,000x and greater are available.

Electrons striking a surface also produce X-ray emission. In energy dispersive X-ray analysis (EDX), the characteristic energy of the X-rays that are emitted is detected and analyzed, giving composition information for elements above sodium in the periodic chart. (Windowless detection allows all elements heavier than boron to be detected.)

When EDX is used with a scanning electron microscope, the combined techniques can provide information concerning both surface morphology and the composition of the outer layers of the sample; however, they are not strictly surface elemental analysis techniques. The SEM detects signals from the surface to provide images and the EDX detects X-rays from about the top 1-3 microns of the sample to provide the elemental information.

Adding wavelength dispersive X-ray (WDX) analysis to an SEM/EDX system increases sensitivity in general (to ppm levels) and reduces problems of peak overlap. WDX also extends the elemental analysis range to include B, C, and O. Application examples for WDX include the investigation of oxides, nitrides and dopants in bulk electronic materials and thin films, solving trace level contamination problems, and the identification of alloys.

In addition to the detection of low energy secondary electrons, several other analysis modes and presentation modes are available on some SEM/EDX/WDX systems. A brief summary of these and some of their applications follows:

Absorbed current and electron beam induced current (EBIC). Image contrast can come from differences in the amount of absorbed current. Electrical paths are highlighted and defects such as shorts and sub-surface leakage paths are visible in the micrograph. EBIC is a modification of the absorbed current mode in which excess carriers are produced in a junction by the high energy primary electrons. The mobile carriers are detected as current which is used to modulate the X axis and therefore produce a micrograph which highlights junction areas.

Backscattered electron detection (BSE). With BSE, the picture contrast is induced by differences in atomic number. For example, in a complex thin film structure cross-section, the image contrast shows compositional differences which can then be identified by EDX or WDX. Multilayered structures such as diodes can be studied in cross-section and layer thicknesses investigated with much better contrast than using the secondary electron mode. (However, the ultimate resolution possible is somewhat less than in the standard secondary detection mode.)

Split screen imaging. This presentation mode allows dual magnification pictures, and direct comparisons using different analysis modes: morphology vs. electrical activity, morphology vs. atomic number, or one half of the screen for an overview and the other for a high magnification picture of a feature of interest. An example is shown in figure 5.

Dual element mapping. Taking advantage of the two element display, this feature provides comparison of element locations using side-by-side element maps. An example is shown in figure 6.

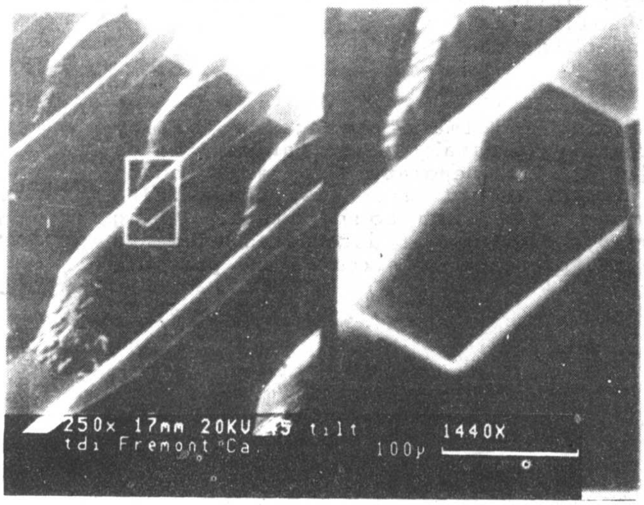


Figure 5. Dual magnification picture of a switching device.

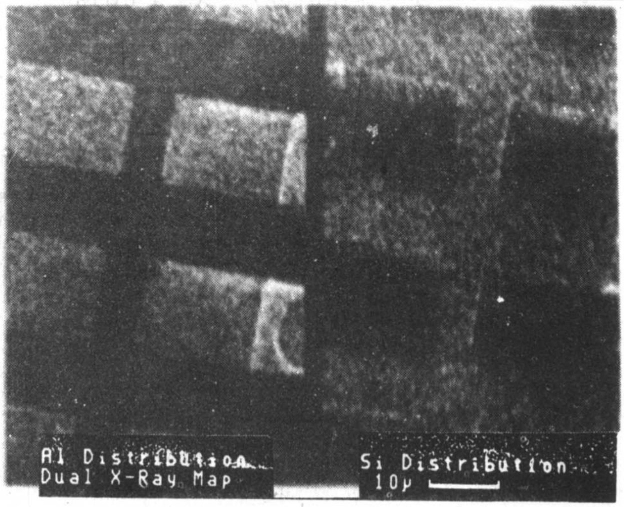


Figure 6. Dual X-Ray map of aluminum and silicon on a device surface.

Stereo pair presentation. Using split screen imaging, images are taken at a 6° angle difference to provide three dimensional information (using a stereo viewer).

X-ray maps and line profiles of light elements. Single or dual element X-Ray distribution maps and line scans can be displayed (an example is shown in figure 7) and also simultaneously mixed with any electron image as a background reference. If dual element mapping is selected the split screen function is automatically selected.

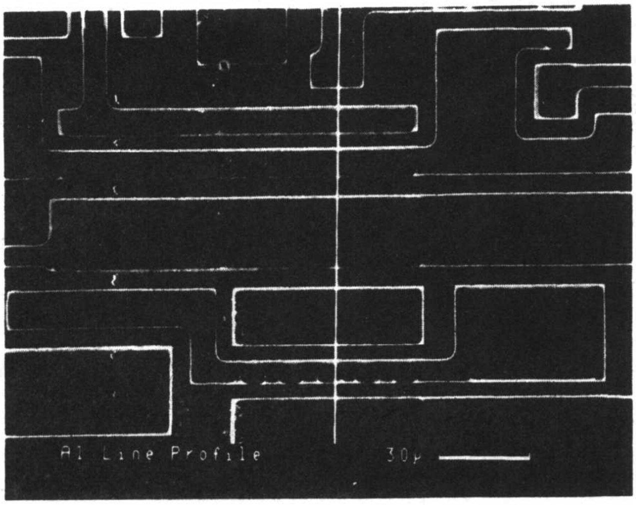


Figure 7. An aluminum line profile of an integrated circuit. The solid line, adjacent to the spectrum indicates the area profiled.

Digital X-ray mapping. Digital X-ray mapping and image storage capability enhance the quality of presentation of microanalysis data. In the CamScan Series 4 system at Surface Science Laboratories, up to four separate X-Ray maps can be acquired simultaneously and stored at the normal recording speeds, these being superimposed onto a TV rate visual image before photographing. Using multielement mapping one can determine the distribution of associated elements and compare different element maps to show the presence of mixtures and possible compounds.

Applications

There are many applications for all of these techniques. Figures 8 through 11 illustrate just a few of these.

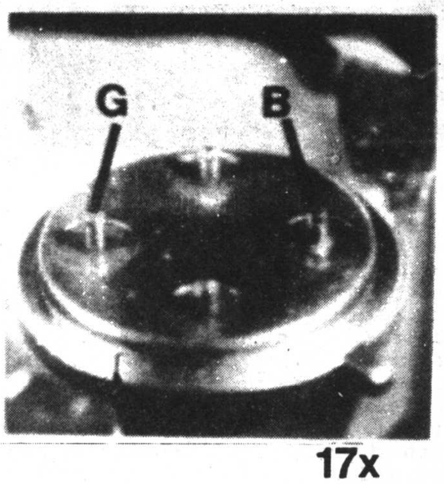
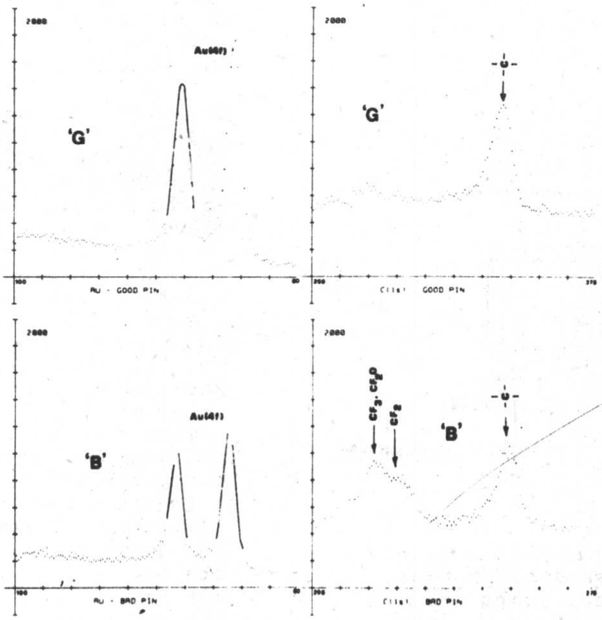


Figure 8. The photograph shows a gold-plated header used to package semiconductor devices. This header is one of a series tested to determine the causes of bond failures. Each of the four insulated pins is .018" in diameter, and is used to connect to the device via small wire bonds. Conventional ESCA analysis would not give useful data, due to the large analysis area.

Examination of both the good pin ("G") and the bad one ("B") with the X-Probe^{tm} revealed the presence of $-CF_3$ and $-CF_2-$ groups on the bad pin, indicating fluorocarbon contamination.

The small spot size (~150 microns) of the X-Probe^{tm} thus enabled us to obtain very precise chemical information about a very small sample area, and solve an otherwise difficult problem.



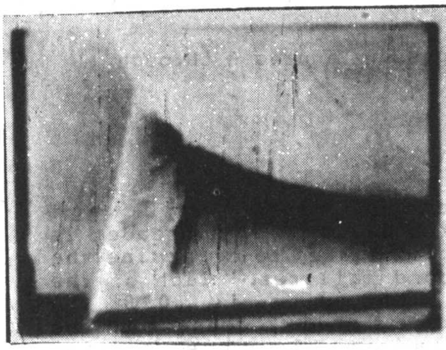


Figure 9. Bonding problems are a recurring cause of product rejection in the semiconductor industry. Surface chemical analysis using a combination of SEM, AES, and ESCA is a powerful tool for solving problems. Examination of failed bonds using an SEM will often detect corrosion and in many cases, Auger analysis shows that halogen contamination at extremely low levels from etching or an acid cleaning step was the culprit. In all cases the bonds had passed pre-bonding die inspection.

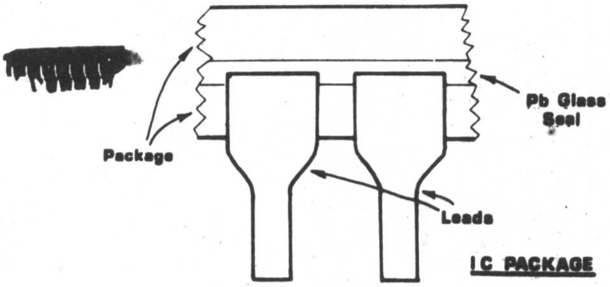


Figure 11. Electrical leakage across the lead glass seal in this IC package was traced, using ESCA, to excess lead deposited in the glass.

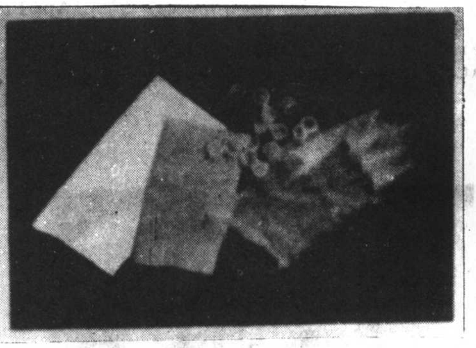


Figure 10. In today's semiconductor technology, processes which produce SLI and VLSI circuitry require extraordinary procedures for contamination control. Contaminants transferred from any source can cause extremely serious problems if they reach the surface of the wafer, either by direct contact or through contact with objects which eventually touch the wafer.

SSL's Contamination Transfer Test for gloves, finger cots, and industrial wipes involves touching the cot or glove (while on the hand) to the surface of a piece of clean aluminum foil. In the case of industrial wipes, the aluminum test piece is wiped or rubbed with the object to be tested.

The aluminum foil used in the test must be carefully pre-characterized so that the test results indicate only the contaminants transferred to the foil by touching it with the glove, cot or wipe.

Typical contaminants found after touching the aluminum foil with finger cots include: Sodium, Chlorine, Sulfur, Potassium, Magnesium, Silicon, Carbon, Phosphorous, and Copper. A given batch of finger cots usually show a marked consistency in the types and amounts of contaminants detected but large differences occur between lots or batches. A few lots are found to be exceptionally clean, and none show traces of all the contaminants listed. Other potential sources of contamination transfer are protective plastics or solder resist and coating masks.

Summarizing the technique parameters and the applications.

Table 2 summarizes the parameters of interest for ESCA, AES, SIMS, SEM/EDX/WDX, RBS and LIMS.

Analytical Parameter	**AES**	**ESCA**	**SIMS**	**SEM/ EDX/WDX**	**RBS**	**LIMS**
Probe Particle	electrons	X-rays	ions	electrons	ions	photons (laser)
Detected Entity	electrons	electrons	ions	electrons/X-rays	ions	ions
Detectable Elements	All > helium	All but hydrogen	All	All > beryllium	All > helium	All
Routine Small Area Analysis (diameter)	subμm	** 150-300 μm's	subμm to several μm's	1-3 μm's (EDX/WDX) 0.01μm(SEM)	~ 1mm	~ 1μm
Surface Sensitivity	~ 10-40Å	~40Å	~ 3-10Å	> 10,000Å (EDX/WDX)	Depth Resolution is 25Å--200Å	LD-adsorbed material LI~1000Å
Routine Detection Limits* (atom%)	0.3-1	0.1-1	10^{7} - 10^{1}	1 (EDX) 10^{4} (WDX)	0.01-10	10^{4} - 10^{2}
Routine Survey Analysis Time (data acquisition)	5 min.	5 min.	5 min.	2 min. (EDX) 30 min. (WDX)	15 min.	< seconds

* Detection limits are expressed as a range because different elements have different sensitivities. Minimum figures are difficult to achieve in routine work.

**Many older ESCA systems are limited to areas several millimeters on a side

Table 2. Typical Parameters for the Common Surface Analysis Techniques

A partial list of examples of applications for surface analysis and depth profiling techniques in the semiconductor industry is presented below.

General applications/problem solving in semiconductor technology and processing

Detecting Impurities.
Analyzing for contaminant distribution.
Identifying residues - from rinsing, polishing, etching, plating
    cleaning, gas line contamination, ...
Determining the sources of haze, discoloration, spots, & streaks - from
    roughness, oxides, residues, pump oil, thickness variation,
    particulates, deposits, impurities, ...
Identifying Particulates.
Monitoring solvents and other liquids for potential residue.
Locating surface segregation problems.
Investigating plating problems, i.e. poor grain integrity,
    solderability, delamination.
Tracing the source of high resistivity.
Tracing the source of electrical leakage from contaminants.