VersaProbe III

Scanning XPS Microprobe

















VersaProbe III Scanning XPS Microprobe

X-ray photoelectron spectroscopy (XPS/ESCA) is the most widely used surface analysis technique and has many well established industrial and research applications. XPS provides quantitative elemental and chemical state information from surfaces and thin film structures. XPS is applied to a diverse range of materials applications including: polymers, metals, ceramics, catalysts, thin films, photovoltaics, batteries, wear coatings, nanomaterials, semiconductor devices, magnetic storage media, display technology, and biomedical devices.

The *VersaProbe* III is the latest generation of PHI's highly successful multi-technique XPS product line. It includes improved performance and additional technique options important for studying today's advanced materials. Physical Electronics (PHI) has been the leading supplier of surface analysis instrumentation (XPS, Auger, and TOF-SIMS) for over 50 years and PHI XPS instruments are the only XPS instruments that scan the X-ray beam, providing true SEM-like operation.

An array of excitation sources, ion guns, and sample treatment and transfer options are available to support your material characterization and problem solving requirements.

SEM-LIKE XPS MICROPROBE

100 µm 00 µm 50 µm 100 um 100 um 50 µm Multilayer Optical Coating Polymer Beads Corrosion 50 µm 50 µm 100 um 50 µm

Dental Implant

Tribology Wear Track

Surface Contamination

Similar to operating an SEM/EDS, X-ray induced secondary electron images (SXIs) are used on the PHI *VersaProbe* III for real-time location of features of interest and to select points/areas of analysis.

Unique Capabilities

PHI's scanning XPS microprobe instrument platform provides scanning X-ray induced secondary electron images (SXI) generated by scanning a focused sub-10 µm X-ray beam across the sample. Just like an SEM, SXI's can be used to navigate to areas of interest and to select areas for analysis in real time. SXI images provide 100% confidence in locating small features of interest and in avoiding areas with contamination and inhomogeneities for analysis. SXI images have a contrast mechanism that is dominated by photoelectron yield (composition), and therefore often reveal features that are not optically visible related to topography.



(Clockwise from top-left) SXI image of a patterned device structure; Spectra from three selected locations on the SXI obtained using a sub-10 µm X-ray beam; Si chemical maps; Si spectra extracted from regions on the Si map; elemental images obtained from the region selected from the SXI using sub-10 µm X-ray beam.









SEM-LIKE OPERATION

SEM-Like Workflow

A typical XPS analysis on the PHI VersaProbe III begins by collecting an SXI image that is quickly generated using a sub-10 μ m diameter raster scanned X-ray beam. Areas of interest for small or large spectral analysis or imaging are used to guide the next steps which may include: obtaining high energy resolution spectra for chemical state analysis, chemical state images, or compositional sputter depth profiles.

1 CRATER MULTI-POINT DEPTH PROFILE

Multi-Point Depth Profiling

A powerful capability enabled by PHI's unique scanning XPS microprobe technology is the ability to define analysis points on an SXI image and then obtain sputter depth profiles from multiple locations in a single sputter crater. For samples where sputtering area should be minimized, this is a powerful tool for analysis of neighboring features or on and off defect sites.



SXI of a patterned device structure showing analysis locations for a multi-point sputter depth profile.



Depth profile of the blue (silicide) point obtained using a sub-10 µm X-ray beam.



Depth profile of the green (oxy-nitride) point obtained using a sub-10 μm X-ray beam.



Depth profile of the red (oxide) point obtained using a sub-10 μm X-ray beam.



500 eV Ar⁺ sputter depth profile of a multi-layer coating on a glass substrate performed using Zalar rotation to enhance layer definition.

Thin Film Depth Profile Analysis

- Bend in ion column to stop neutrals
- Compucentric Zalar rotation
- Robust dual beam charge neutralization
- Micro-area depth profiling
- Single crater, multi-point depth profiling

THIN FILM DEPTH PROFILE ANALYSIS

Optimized Configuration

A focused X-ray beam, high sensitivity spectrometer, high performance floating column argon ion gun, turnkey dual beam charge neutralization, compucentric Zalar™ rotation, and advanced data reduction algorithms provide the highest performance XPS depth profiling capability available. The standard monatomic argon ion gun is capable of generating 5 eV to 5 keV Ar ion beams and is ideally suited for most inorganic depth profiling applications.

ARGON GAS CLUSTER ION BEAM (GCIB) OPTION

Organic Depth Profiling

It is well known that monatomic Ar ion guns used for inorganic thin film analysis typically cause severe chemical damage when sputtering most polymer and organic materials. PHI has led the way in developing and applying cluster source ion guns for the successful thin film analysis of polymer and organic materials. Our optional 20 kV Argon gas cluster ion beam (GCIB) and optional C_{60} ion gun have proven performance for depth profiling many polymer and organic films while minimizing the potential for chemical damage.





GCIB sputter depth profile of a graded OLED test structure showing the ability to preserve and observe the two organic species that make up the test structure. The montage plot of N 1s spectra, on the right, shows the spectra that were used to create chemical state plots for N with the linear least squares fitting algorithm in PHI data reduction software.



 20 kV C_{60} sputter depth profile of an inverted organic photovoltaic device that contains metal layers, organic layers, oxide layers and a mixed matrix layer with an organic and TiO, nanorods. Compucentric Zalar rotation was used to enhance layer definition.

C₆₀ CLUSTER SOURCE ION GUN OPTION

Mixed Matrixed Depth Profiling

With the introduction of cluster source ion guns for organic and polymer thin film depth profiling, interest has grown in applying these ion guns to inorganic structures that sustain chemical damage with monatomic Ar ion beam sputtering. Our experience has shown that some metalloids, oxides, and thin film structures that contain both organic and inorganic materials sustain less chemical damage and differential sputtering artifacts when depth profiled using a 20 kV C_{60} cluster source ion gun.

COMPLETE ELECTRONIC BAND STRUCTURE CHARACTERIZATION

Ultraviolet Photoelectron Spectroscopy (UPS) - Valence Band

Design of complex electronic material systems for display panels, flexible circuitry, and photovoltaics require knowledge of the basic properties of each component's band structure in order to achieve efficient charge transport.

The combination of ultraviolet photoelectron spectroscopy (UPS) and low energy inverse photoemission spectroscopy (LEIPS) provides a complete characterization of the valence and conduction bands, as well as useful parameters such as the band gap, ionization energy, work function, and electron affinity.



5 keV Ar₂₅₀₀⁺ GCIB depth profile of OLED multilayered film. *Ionization energy* is extracted from UPS spectra at each depth sputter cycle. UPS valence band spectra of copper phthalocynanine (CuPc), a hole transport material in organic light-emitting diodes (OLEDs). Biasing the sample (solid curve) allows one to calculate *ionization energy* or *work function*.

Samples provided by: Organic Optoelectronics Practical Development Center (i3-opera)

COMPLETE ELECTRONIC BAND STRUCTURE CHARACTERIZATION



Combined measurements from UPS and LEIPS

Electronic band structure for CuPc as determined by UPS and LEIPS. Band gap is calculated from combining *ionization energy* measurement from UPS and *electron affinity* measurement from LEIPS

Low Energy Inverse Photoemission (LEIPS) -Conduction Band States

LEIPS provides accurate values of electron affinity (EA) which is required for designing organic light-emitting diode, understanding band structure at metal-semiconductor and semiconductor heterojunctions and in studies of charge-transfer processes.

Low energy incident electrons (<5 eV) used in this technique are well-suited for analysis of organic materials with minimal damage.

The ionization energy can be obtained from the highest occupied molecular orbital (HOMO) of the UPS measurement. The electron affinity can be obtained from the lowest unoccupied molecular orbital (LUMO) of the LEIPS measurement. From the difference in those two values, the semiconductor band gap energy can be calculated.



AUGER ELECTRON SPECTROSCOPY (AES)

AES

When the features of interest are too small for XPS analysis, Auger Electron Spectroscopy is often used. The AES probe electron beam is up to 100 times smaller than the XPS X-ray beam, opening new possibilities for sample characterization at increased spatial resolution.

Within the VersaProbe III, the convergence of the optical, SXI, and SEM images allows for an intuitive approach to identifying regions of interest for analysis.

XPS and AES session tabs in the SmartSoft acquisition software are set up to operate seamlessly, allowing for in-situ analysis using both techniques at the same region of interest without moving the sample. Similar options for spectral analysis, depth profiling, line scans, and maps are available with both techniques.





(Clockwise from top-left) SEM image of a patterned electronic device; Multipoint AES analysis indicating regions containing Si, Al, and O; Chemical state information extracted from Points 1 and 2 based on Si peak position; Elemental maps with high spatial resolution.











REFLECTION ELECTRON ENERGY LOSS SPECTROSCOPY (REELS)



A peak rises at an energy 8.8 eV lower than the reflected incident electron, allowing the **band gap** of the SiO2 film to be measured



Loss spectrum provides relative concentration of hydrogen (loss peak at ~1.8 eV) which is not accessible by XPS and peak due to π -> π^* transition (loss at 6-8 eV) for polymers and 2-dimensional materials

REELS

REELS is a surface analysis technique in which a specimen is bombarded with an electron beam (≤ 1500 eV) and the energy distribution of the reflected electrons is measured. This energy distribution contains features corresponding to discrete losses of energy of the reflected electrons due to excited atomic states, valence band transitions and material bandgaps.

REELS capabilities:

- Electronic state and bonding state analysis on the surface
- Band gap measurement of semiconductors
- Compare the relative hydrogen content of materials
- Observe evidence for conjugation/aromaticity in materials
- Discrimination of sp²/sp³ bonds of carbon



VERSATILE TEST CHAMBER CONFIGURATION

Integrated Optional Accessories

The VersaProbe III test chamber is designed to accept multiple photon, electron, and ion sources that are focused on a common analysis point on the sample and are all controlled from the *SmartSoft* user interface.







CAPABILITIES

Standard Features

- Scanned, micro-focused, monochromatic X-ray beam
- X-ray beam induced secondary electron imaging (SXI)
- Dual beam charge neutralization
- 128 data channel detection
- Chemical state imaging
- Single Crater multi-point depth profiling
- Floating column monatomic Ar ion gun
- Compucentric Zalar[™] rotation
- Angle dependent XPS
- Five axis automated sample manipulator
- 25 mm and 60 mm diameter sample mount

Optional Accessories

- Low Energy Inverse Photoemission Spectroscopy (LEIPS)
- Reflection Electron Energy Loss Spectroscopy (REELS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- Electrochemical mount
- 20 kV C₆₀ ion gun
- Gas Cluster Ion Beam (GCIB)
- Scanning Auger Electron Spectroscopy (AES)
- Dual anode, achromatic X-ray source
- Hot /Cold intro & analysis chamber
- Custom sample preparation chambers
- Controlled environment transfer vessel



Physical Electronics - USA U

ULVAC-PHI Inc. - Japan

Phone:952-828-6100Email:sales@phi.comWeb:www.phi.com

Phone:81-467-85-4220Email:webmasterjp@phi.comWeb:www.ulvac-phi.co.jp