PHI VersaProbe 4

Scanning XPS Microprobe


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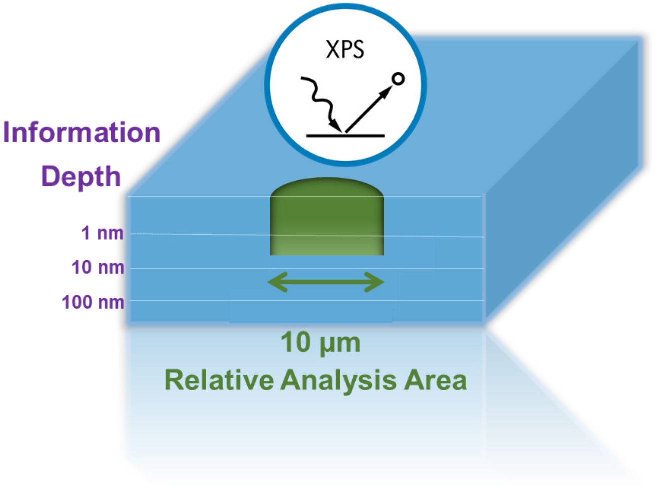
WHAT CAN XPS TELL YOU?

X-ray Photoelectron Spectroscopy (XPS) is the most widely used surface analysis technique because it can be applied to a broad range of materials and provides valuable quantitive elemental and chemical state information from the surface of the material being studied.

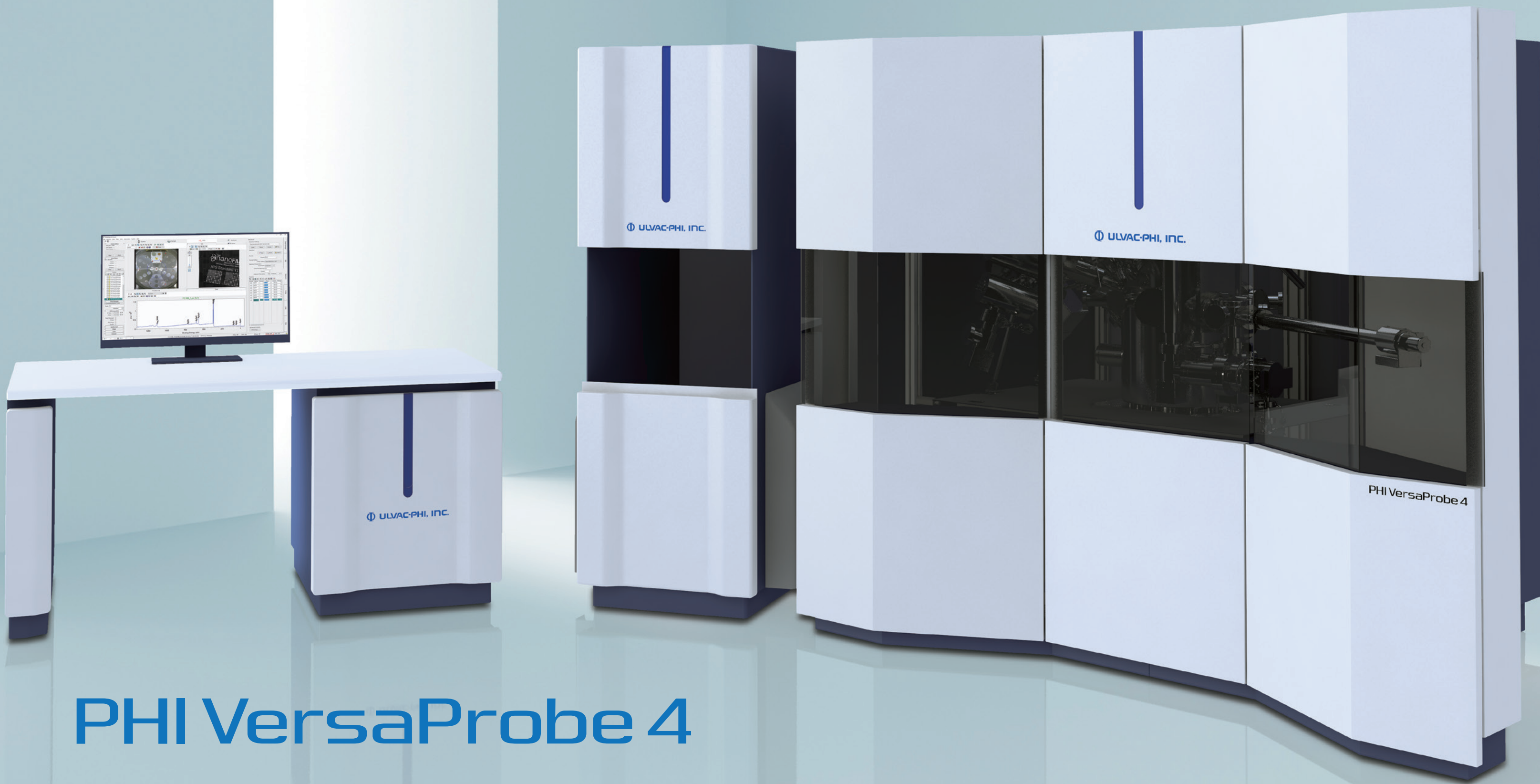
The information XPS provides about surface layers, thin film structures and interfaces is important for many industrial and research applications where composition plays a critical role in performance. It is widely used for polymers, metals, ceramics, catalysts, thin films, photovoltaics, batteries, wear coatings, nanomaterials, semiconductor devices, magnetic storage media, display technology, 2-D materials and biomedical devices.

Physical Electronics (PHI) XPS instruments provide the ability to obtain spectra with a lateral spatial resolution as small as 10 µm for the Al Kα X-ray source.



Next Generation Multi-technique Scanning XPS Microprobe

High Sensitivity, Reliability and Versatility in a Single Instrument



PHI VersaProbe 4

PHI VersaProbe 4

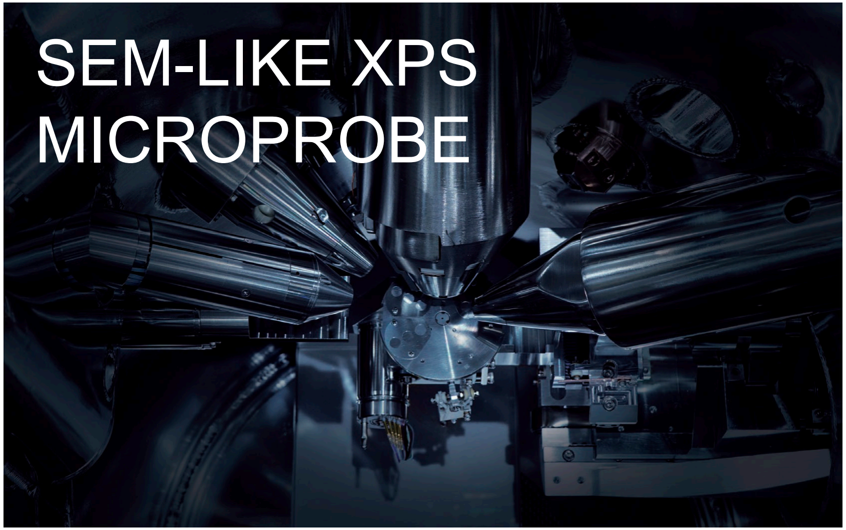
Scanning XPS Microprobe

The PHI *VersaProbe* 4 is the latest generation of PHI's highly successful multi-technique XPS product line with PHI's patented, monochromatic, micro-focused, scanning X-ray source. It includes improved performance, new large area imaging and mapping capabilities, and an environmentally friendly modern configuration with efficient power consumption, faster pumpdown and ergonomic design.

The fully integrated multi-technique platform of the PHI *VersaProbe* 4 offers an array of optional excitation sources, sputter ion sources, and sample treatment and transfer capabilities. These features are essential in studying today's advanced materials and in supporting your material characterization and problem-solving needs.

PHI has been the leading supplier of surface analysis instrumentation (XPS, AES, 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.

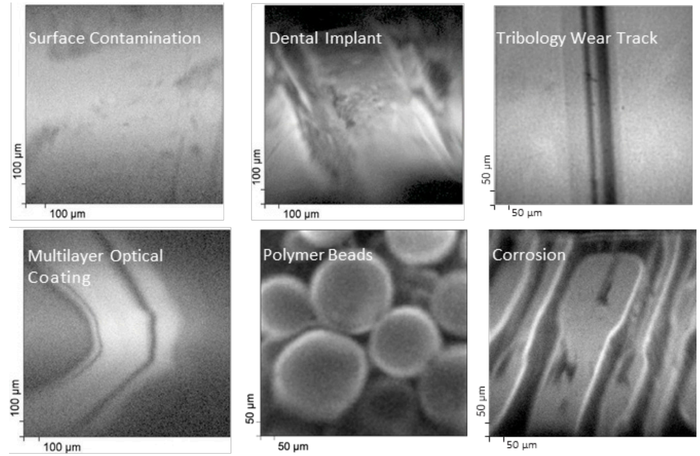
SEM-LIKE XPS MICROPROBE



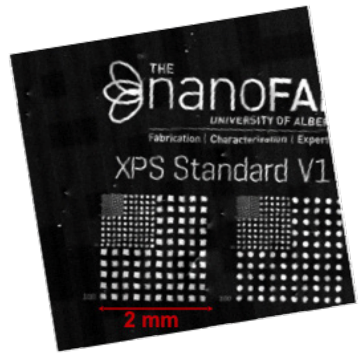
Unique Capabilities

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, SXIs 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.

Smart Mosaic allows a user to set up a mosaic acquisition of SXI images from large areas. Stitched mosaic SXI images can be used to investigate the homogeneity of the sample across much larger areas than available in a single SXI image.



Similar to operating an SEM/EDS, X-ray induced secondary electron images (SXIs) are used on the PHI *VersaProbe* 4 for real-time location of features of interest and to select points/areas of analysis.

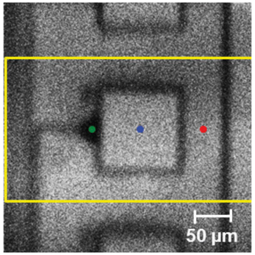


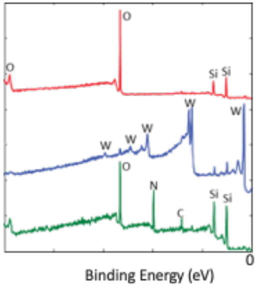
144 individual SXI images stitched together to produce a 5x5 mm secondary electron image that can be used for selecting areas for analysis.

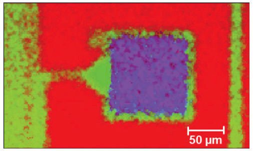
HIGH-SENSITIVITY SPECTRAL AND IMAGING ANALYSIS

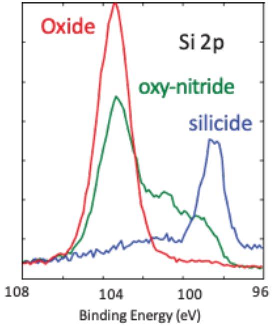
A typical XPS analysis on the PHI *VersaProbe* 4 begins by collecting an SXI image that is quickly generated using a sub-10 µm diameter raster scanned X-ray beam. Areas of interest for small or large spectral analysis or imaging are selected and used to guide the next steps which may include: obtaining high energy resolution spectra for chemical state analysis, chemical state images, or compositional sputter depth profiles.

Multispectral chemical maps can be acquired from areas of any size and stitched into a single large area chemical map. Overlays with a variety of color maps and transparency display options can be used to highlight areas that are spatially and chemically distinct.



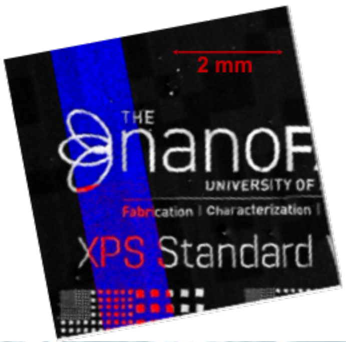




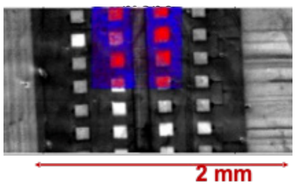


(Clockwise from top-left) SXI image of a patterned device structure; Spectra from three selected locations on the SXI obtained using a sub-10 µm X-ray beam; Si chemical maps; Si spectra extracted from regions on the Si map

SXI mosaic image overlaid with ~ 2.7 x 1.4 mm Ti 2p (red) and 5.5 x 1.4 mm Si 2p (blue) chemical maps



A 2.3 x 1.2 mm SXI mosaic image overlaid with ~ 0.7 x 0.7 mm W 4f (red) and Si 2p (blue) chemical maps



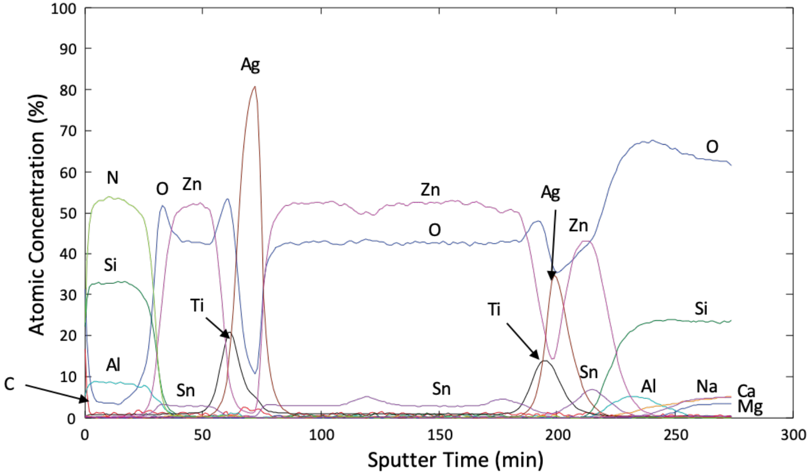
~ 25 x 2.5 mm SXI mosaic image overlaid with ~ 5 x 2.5 mm C1s (red), Al 2p (blue) chemical maps

OPTIMIZED THIN FILM DEPTH PROFILE ANALYSIS

Optimized Configuration

A focused X-ray beam, high sensitivity spectrometer, high performance floating column argon ion gun, automated dual beam charge neutralization, Zalar rotation, and advanced data reduction algorithms provides the highest performance XPS depth profiling capability available.

The standard monatomic argon ion gun can generate 5 eV to 5 keV Ar^+ ion beams and is ideally suited for most inorganic depth profiling applications.

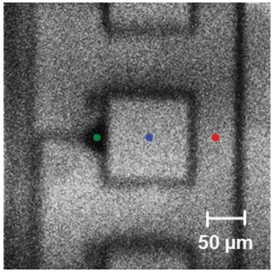


500 eV Ar^+ sputter depth profile of a multi-layer coating on a glass substrate performed using Zalar rotation to enhance layer definition.

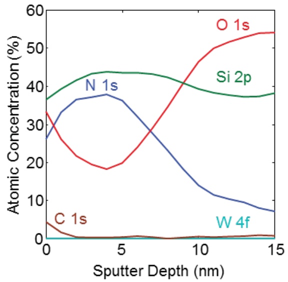
Thin Film Depth Profile Analysis

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

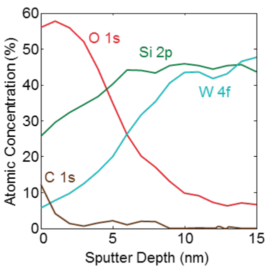
SINGLE CRATER MULTI-POINT DEPTH PROFILE



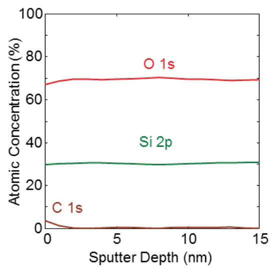
SXI of a patterned device structure showing analysis locations for a multi-point sputter depth profile.



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



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



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

Multi-Point Depth Profiling

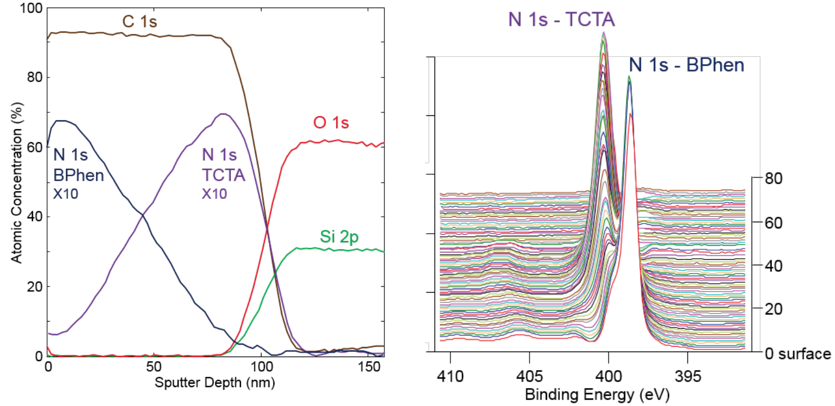
A powerful capability enabled by PHI's unique scanning XPS microprobe technology is the ability to define analysis points on an SXI image and then obtain sputter depth profiles from multiple locations in a single sputter crater. For samples where sputtering area should be minimized, this is a powerful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

The image registration option is coupled to PHI's unique SXI X-ray imaging capability to ensure XPS analysis is performed at the precise location of interest, even for very small features. Accurate positioning of very small features within a single sputter crater for analysis can be guaranteed.

ARGON GAS CLUSTER ION BEAM (GCIB) OPTION

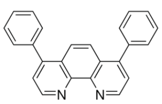
Organic Depth Profiling

It is well known that monatomic Ar ion guns commonly 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 profile plots for N with the linear least squares fitting algorithm in PHI data reduction software.

BPhen



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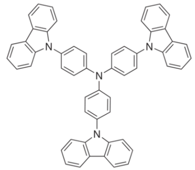
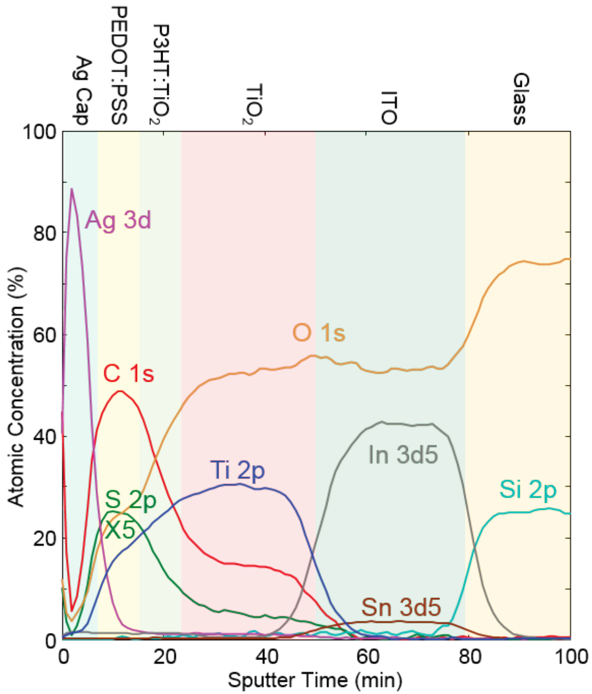


C_{60} GAS CLUSTER ION BEAM OPTION



20 keV 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_2 nanorods. Zalar rotation was used to enhance layer definition.

Mixed Material Depth Profiling

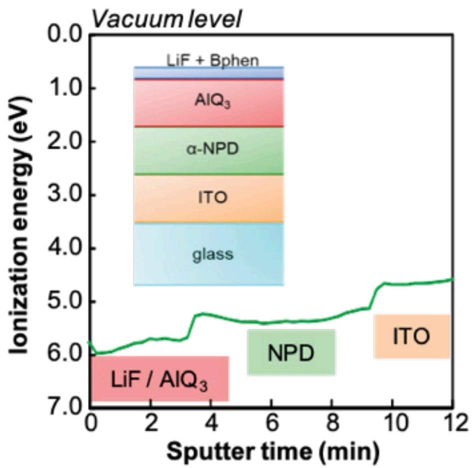
With the introduction of cluster source ion guns for organic and polymer thin film depth profiling, interest has grown in applying these ion guns to inorganic structures that sustain chemical damage with monatomic Ar ion beam sputtering. Our experience has shown that some metalloids, oxides, and thin 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

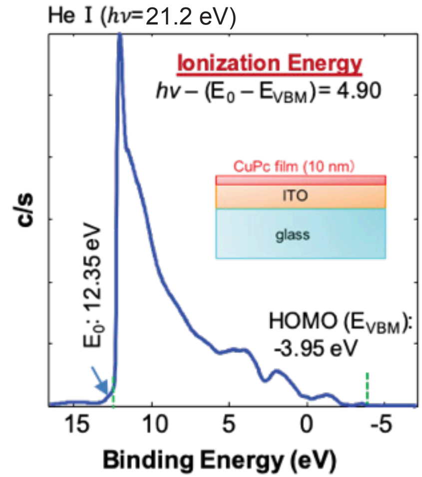
Ultraviolet Photoelectron Spectroscopy (UPS) - Valence Band

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.



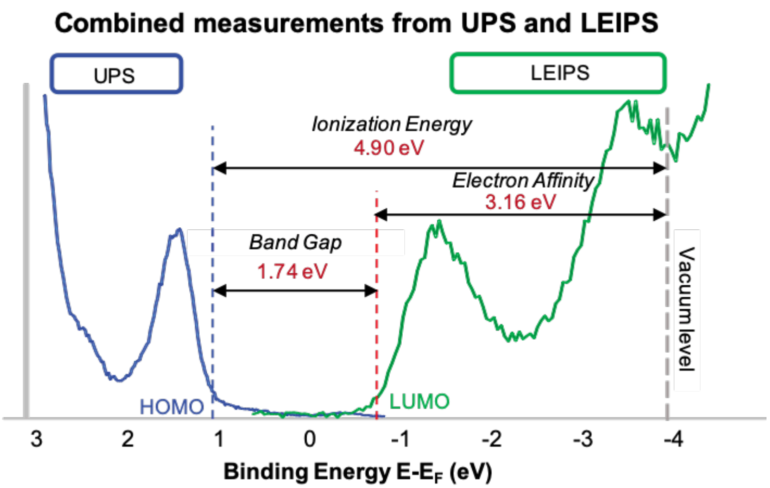
5 keV Ar_{850}^{+} GCIB depth profile of OLED multilayered film. *Ionization energy* is extracted from UPS spectra at each depth sputter cycle.



UPS valence band spectra of copper phthalocynanine (CuPc), a hole transport material in organic light-emitting diodes (OLEDs). Biasing the sample allows one to calculate ionization energy or work function.

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

COMPLETE ELECTRONIC BAND STRUCTURE CHARACTERIZATION



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

Low Energy Inverse Photoemission Spectroscopy (LEIPS) - Conduction Band Characterization

LEIPS provides accurate values of electron affinity which is required for designing organic light-emitting diodes, 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. The semiconductor band gap energy can be calculated from the difference in energy between those two.

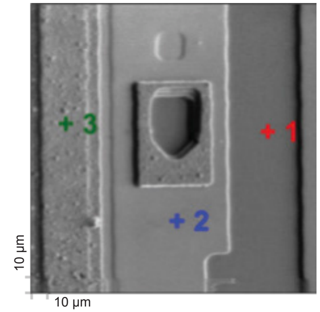
AUGER ELECTRON SPECTROSCOPY (AES)

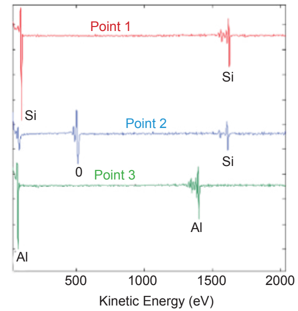
When the features of interest are too small for XPS analysis, Auger Electron Spectroscopy (AES) is often used. The AES probe electron beam is up to 100 times smaller than the XPS X-ray beam, opening up new possibilities for sample characterization at increased spatial resolution.

With the *VersaProbe* 4, the convergence of the optical, SXI, and SEM images allows for an intuitive approach to identifying regions of interest for analysis.

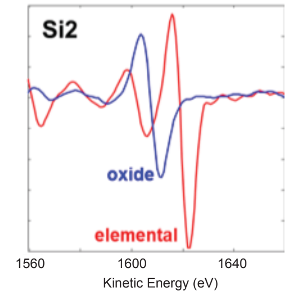
XPS and AES session tabs in the *SmartSoft* acquisition software are set up to operate seamlessly, allowing for in-situ analysis using both techniques at the same region of interest without moving the sample. Similar options for spectral analysis, depth profiling, line scans, and maps are available with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

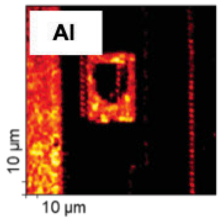
Dual charge neutralization allows for AES analysis of insulating samples.

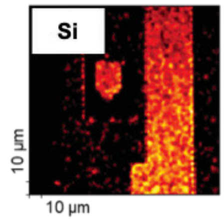


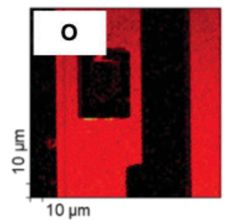


(Clockwise from top-left) SEM image of a patterned electronic device; Multipoint AES analysis indicating regions containing Si, Al, and O; Chemical state information extracted from Points 1 and 2 based on Si peak position; Elemental maps with high spatial resolution.

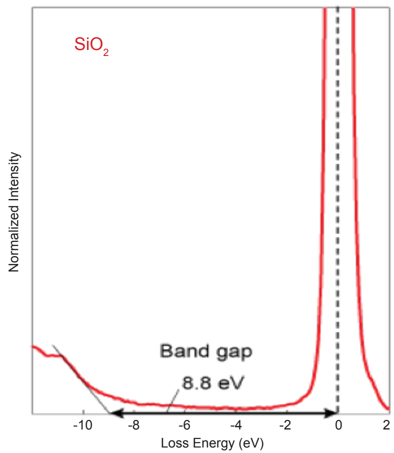




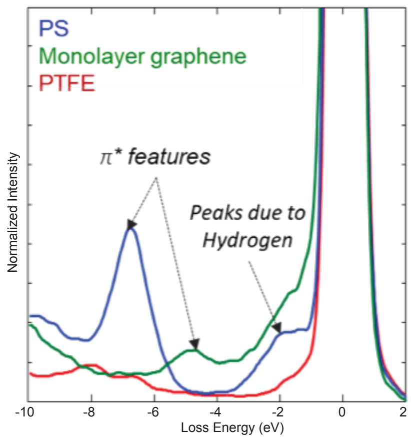




REFLECTION ELECTRON ENERGY LOSS SPECTROSCOPY (REELS)



The band gap is measured by taking the difference in energy between the reflected incident electron and energy of the peak arising near 8.8 ev.



Loss spectrum provides relative concentration of hydrogen (loss peaks at ~1.8 eV) which is not accessible by XPS as well as peaks due to $\pi \rightarrow \pi^*$ transition (loss at 4-6 eV) for polymers and 2-dimensional materials.

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.

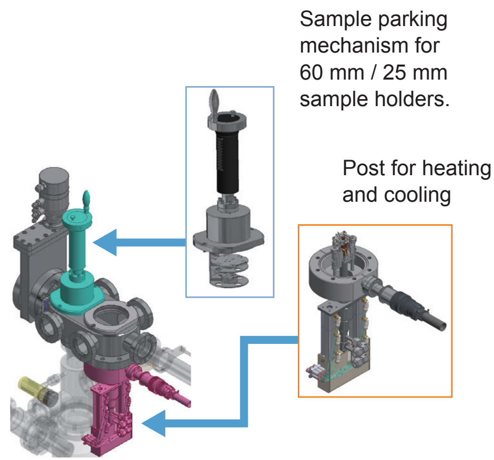
REELS capabilities:

- Electronic state and bonding state analysis on the surface
- Band gap measurement of semiconductors and insulators
- Compare the relative hydrogen content of materials
- Observe evidence for conjugation/aromaticity in materials
- Discrimination between sp^2/sp^3 bonds of carbon

WIDE SELECTION OF SAMPLE HANDLING SOLUTIONS

- Heating and Cooling Stage
- Electrochemical Heating and Cooling Stage
- Large Sample Introduction Chamber
- Sample Preparation Chamber
- Transfer Vessel

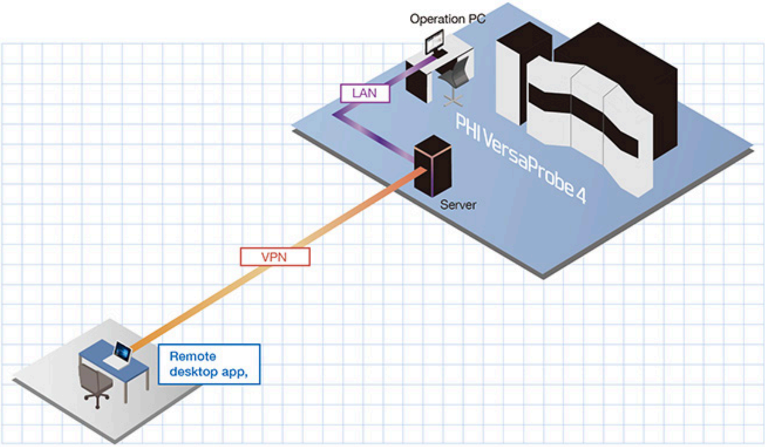
A sample introduction chamber compatible with all PHI XPS sample holders for applications such as a transfer vessel, sample parking mechanism, heating and cooling mechanism, etc.



The transfer vessel allows samples to be introduced to the instrument without exposure to atmosphere. This is particularly useful for samples that should not be exposed to air or water, such as battery samples. The sample holder in the transfer vessel is compatible with other PHI instruments, such as our *nanoTOF* 3, *Quantes* and *710* AES, and allows for a combination of multiple surface analysis methods.

Transfer vessels for sample holders. 25mm holders can be used in the 25 mm transfer vessel while all sample holders can be loaded into the 60 mm transfer vessel.

AUTOMATION AND REMOTE ACCESS



The PHI *VersaProbe* 4 allows access to the instrument via the customer's local area network or the internet.

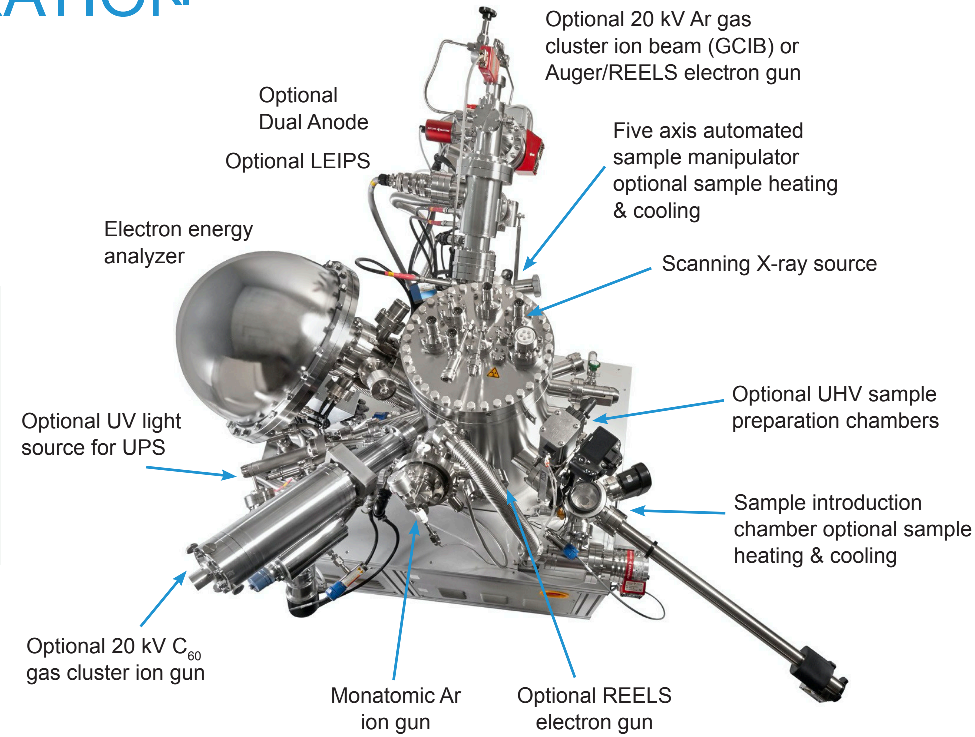
Once a sample has been introduced into the analysis chamber, it is possible to navigate the sample using intro photos and SXI, create automated queue of acquisition tasks, set measurement conditions, check the measurement status, and analyze the data remotely.

The instrument can also be remotely diagnosed by our expert staff.

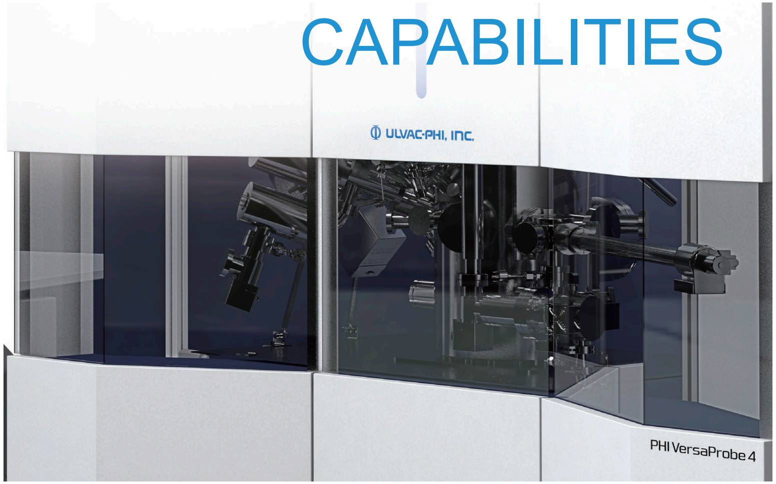
VERSATILE ANALYSIS CHAMBER CONFIGURATION

Integrated Optional Accesories

The *VersaProbe* 4 analysis 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)
- Chemical state imaging
- Large area imaging and mapping
- Dual beam charge neutralization
- 32 physical channel, 128 data channel detection
- Reliable automation and remote operation
- Single crater multi-point depth profiling
- Floating column monatomic Ar ion gun
- Zalar rotation
- Angle-dependent XPS
- Five axis automated sample manipulator
- 25 mm and 60 mm diameter sample holder
- Environmentally friendly modern configuration

Optional Accesories

- Low Energy Inverse Photoemission Spectroscopy (LEIPS)
- Reflection Electron Energy Loss Spectroscopy (REELS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- Advanced *Strata*PHI software for thin film structure analysis
- Electrochemical sample holder
- 20 kV C_{60} ion gun
- Argon Gas Cluster Ion Beam (GCIB)
- GCIB Cluster size measurement tool
- Scanning Auger Electron Spectroscopy (AES) with dual beam charge neutralization
- Dual anode, achromatic X-ray source
- Hot/Cold stage in introduction and analysis chamber
- Custom sample preparation chambers
- Controlled environment transfer vessel

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