

Multi-technique Scanning XPS/HAXPES

# PHI GENESIS



PHYSICAL  
ELECTRONICS  
A DIVISION OF ULVAC-PHI

# PHI GENESIS

## NO COMPROMISE!

Fully automated multi-technique XPS / HAXPES

### Features

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- ✓ Easy operation & multi-technique options
- ✓ Fully automated with sample parking
- ✓ High-performance large & micro area XPS analysis
- ✓ High speed & non destructive depth profiling
- ✓ Hard X-ray Cr K $\alpha$  source for HAXPES
- ✓ Comprehensive solution for batteries, semiconductors, organic devices, and other applications

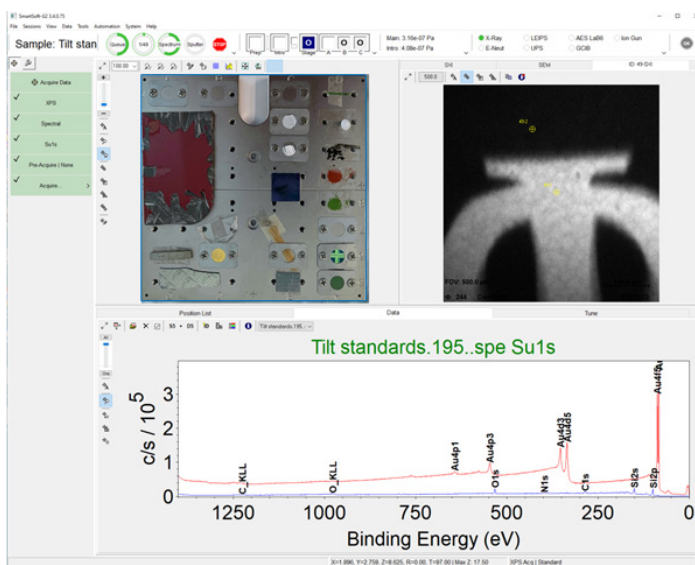




## Ease of Operation

PHI *Genesis* offers a new and improved user interface (UI) providing an intuitive and fully-automated operation of the high-performance instrument.

The interface allows users to access all necessary functions for setting up routine and advanced multi-technique acquisitions within a single screen, while still retaining essential features like navigation using intro photo and 100% accurate positioning from X-ray induced secondary electron images (SXIs).



## Intuitive User Interface

PHI *Genesis* features a simple, intuitive, and user-friendly interface that allows users to perform both basic operations and automated analyses using all available options.

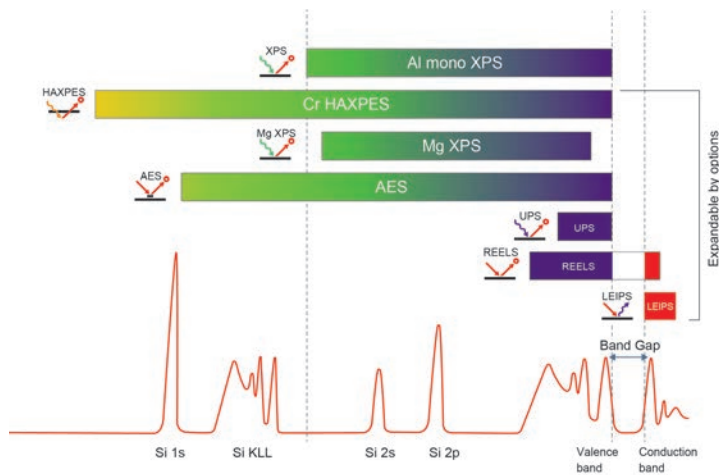
## Autotuning and Calibration

X-ray, electron, and ion gun sources can be calibrated and tuned automatically using the new autotuning feature.



## Multi-Technique Options

PHI *Genesis* facilitates same-area automated analysis using multiple techniques that can cover the full range of energy, from measuring the conduction band onset with Low Energy Inverse Photoemission Spectroscopy (LEIPS) to core-level excitation with Hard X-ray Photoelectron Spectroscopy (HAXPES). This unique ability to analyze the same area on a sample with all available multi-technique options provides unprecedented value that is not typically found in conventional X-ray Photoelectron Spectroscopy (XPS) instruments.

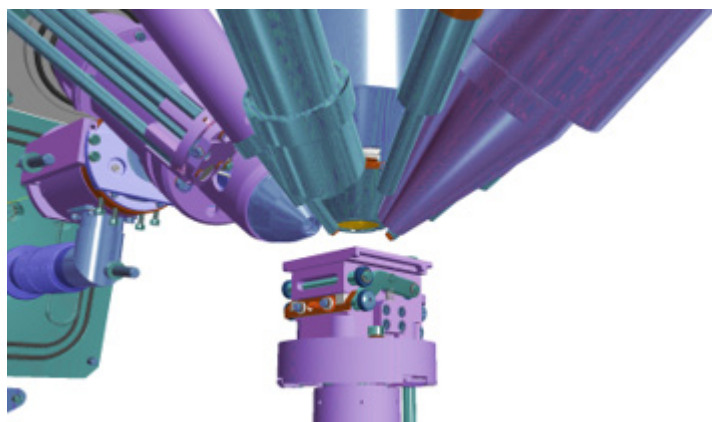


### No compromise solution:

PHI *Genesis* is a high-performance instrument with a wide range of options that can meet all surface analysis needs.

**Multi-Technique Options**

XPS	UPS	HAXPES
LEIPS	REELS	AES
Ar ion	GCIB	C <sub>60</sub>



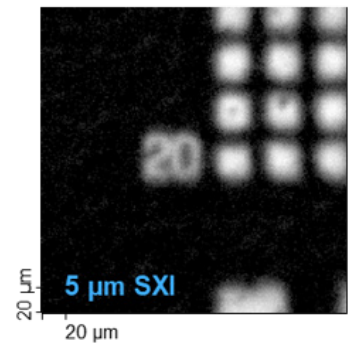


# Superior Micro Area Spectroscopy and Imaging $\leq 5 \mu\text{m}$ X-ray Spot Size

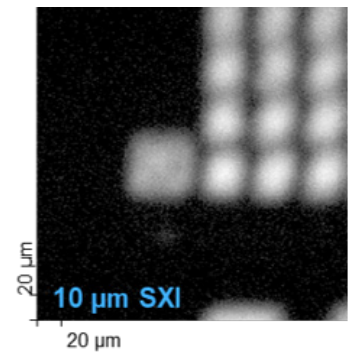
In PHI *Genesis*, a micro-focused scanning X-ray source is used for intuitive, SEM-like navigation using X-ray induced secondary electron images (SXIs). Multi-point small areas can be defined from SXI images with 100% certainty for all types of acquisitions including spectra, depth profiles, and maps.

A typical XPS analysis on the PHI *Genesis* begins by collecting an SXI image that is quickly generated using a sub- $5 \mu\text{m}$  diameter raster-scanned X-ray beam. Areas of interest for small or large area spectral analysis or imaging are selected from the SXI image 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.

## PHI GENESIS

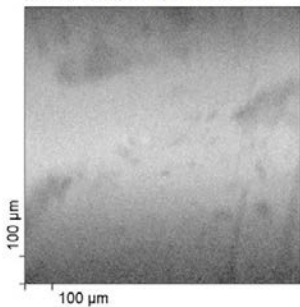


## PHI VersaProbe

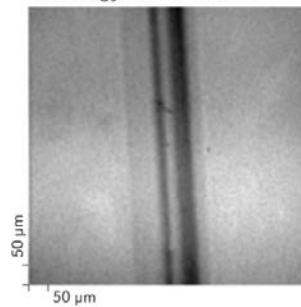


### X-ray Induced Secondary Electron Images (SXI)

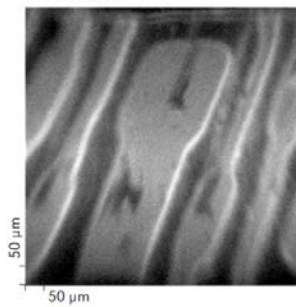
Surface Contamination



Tribology Wear Track



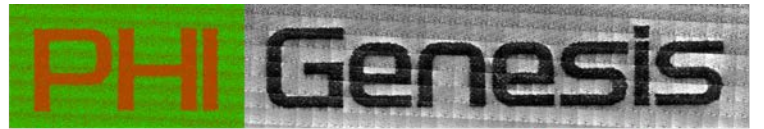
Corrosion



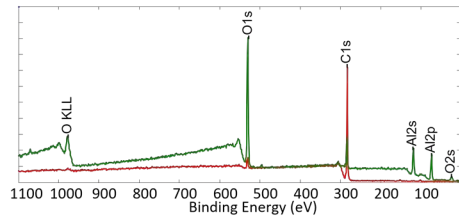


# SEM-Like XPS/HAXPES Microprobe

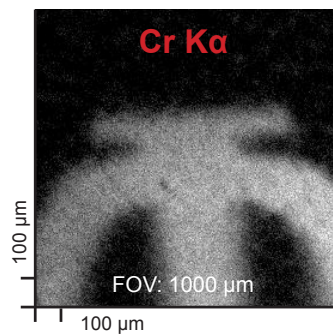
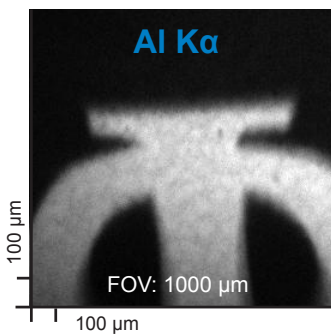
With the SmartMosaic feature, PHI Genesis allows users to image macroscopic areas by automatically stitching together multiple maps or SXIs into a single image called a mosaic, greatly expanding the analysis size while maintaining high resolution and focus. Stitched mosaic SXI images can be used to investigate the homogeneity of the sample across much larger areas than available in a single SXI image. Areas of interest can be selected from these stitched large area X-ray induced secondary electron images for spectroscopic analysis.



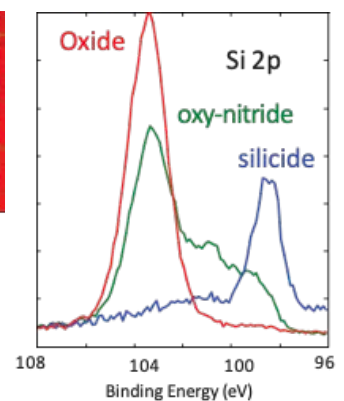
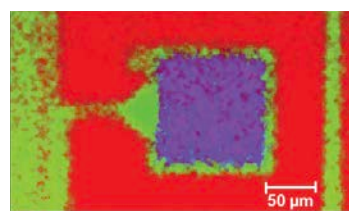
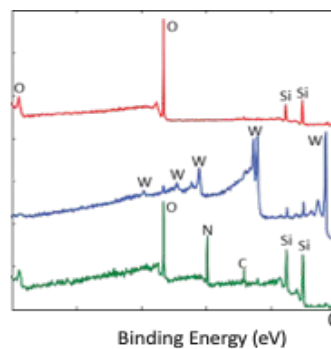
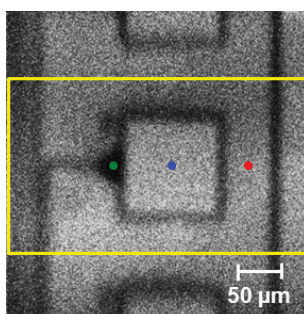
2 mm ~ 25 x 3 mm SXI mosaic image overlaid with ~ 5 x 3 mm C1s (red), Al 2p (green) chemical maps



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



All sources are aligned to the same point on the sample, enabling high spatial resolution imaging of both surface (<5 μm Al Kα) and subsurface (<14 μm Cr Kα) features at the same analysis point/area. With its micro-focused high energy Cr source, PHI Genesis is the only instrument on the market that enables high-resolution imaging of subsurface features, buried interfaces, and depth-resolved chemical/morphological changes.

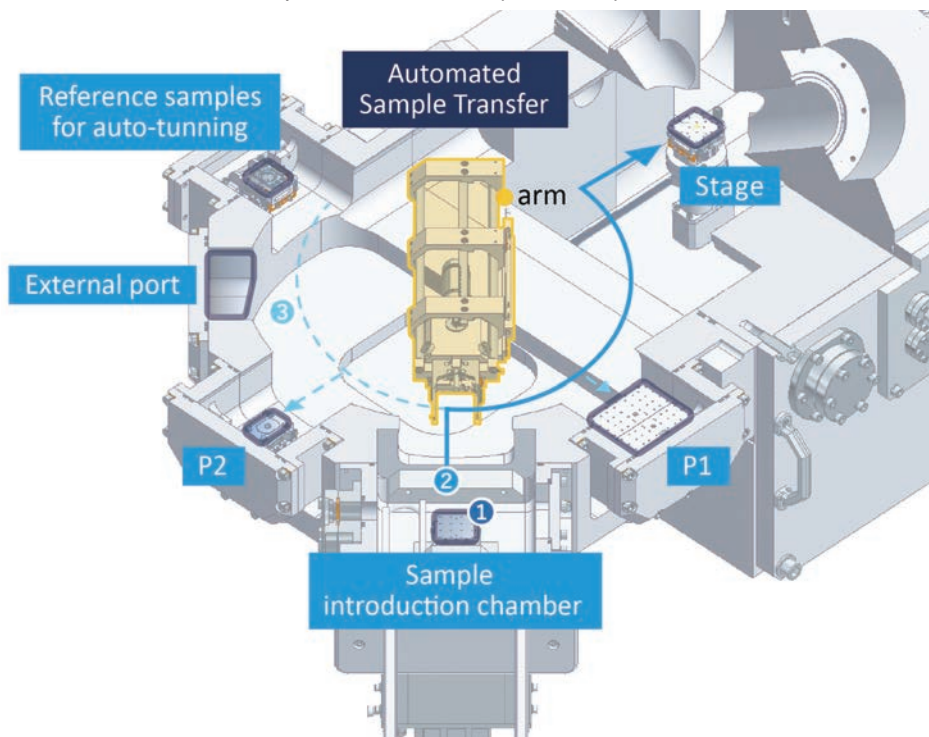


(Left to right) SXI image of a patterned device structure; Survey spectra from three selected locations on the SXI obtained using a sub-10 μm X-ray beam; Si chemical maps; Si 2p spectra extracted from defined regions on the Si map.

# High-Throughput Large Area Analysis

- Automated transfer of samples from intro to analysis chamber and parking positions
- Parking positions allow three sample holders plus *in-situ* holder with references to be kept inside the instrument for use without breaking vacuum
- 80 x 80 mm large sample holder is available for high throughput
- Can analyze a variety of sample types: powders, rough textures, insulators, large or oddly-shaped samples, and samples with varying heights on the same platen

Fully automated multi-sample transfer platform



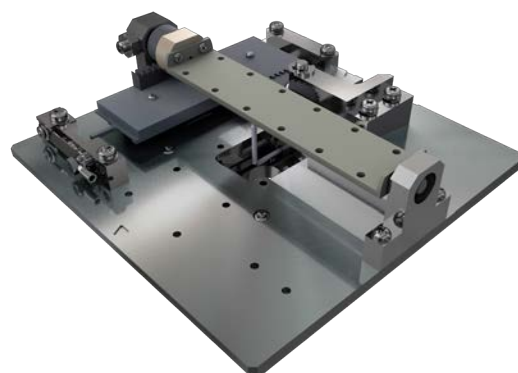
Standard holder 40 mm x 40 mm



Extended plate 80 mm x 80 mm



Dedicated tilt holder

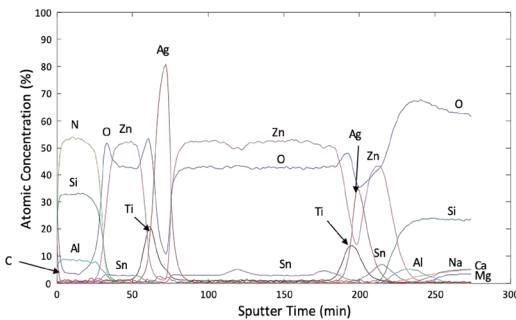




# Optimized Depth Profiling

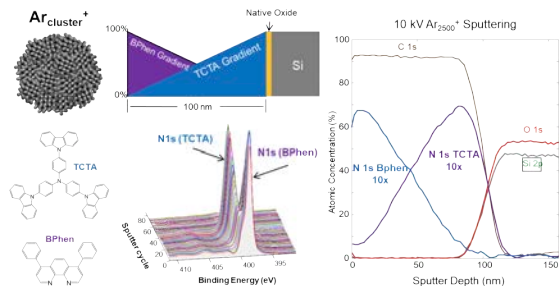
- The advanced software and precision 4-axis stage of PHI *Genesis* instruments enable precise Zalar compucentric rotation during sputtering, ensuring that the sputter crater is free from any shadowing defects or roughness.
- PHI's unique scanning XPS microprobe technology also enables data acquisition at multiple points within a single crater, a powerful tool for samples where sputtering area should be minimized or for more accurate analysis of heterogeneous samples.

## Ar<sup>+</sup> Sputtering



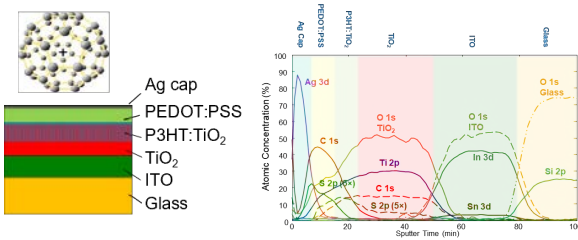
PHI *Genesis* is equipped with an Ar<sup>+</sup> ion gun for quick sample cleaning and removal of material. The advanced software allows for automated alternating sample sputtering with XPS and HAXPES data acquisition, making depth profiling a seamless, fast, and reliable process.

## Ar<sub>cluster</sub><sup>+</sup> Sputtering



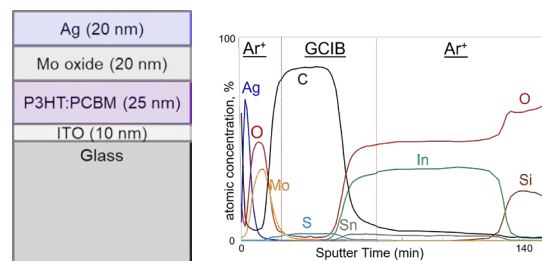
Ionized gas clusters offer a gentler approach to sputtering and cleaning fragile materials such as polymers without damaging the surface. PHI *Genesis* has an optional Dual Source Ion Gun that uses monatomic Ar<sup>+</sup> for rapid sputtering and size-selected clusters for delicate materials.

## C<sub>60</sub> Sputtering



For materials like oxides and metalloids that can't be easily removed by gentle sputtering with an argon cluster, PHI offers a C<sub>60</sub><sup>+</sup> gun that effectively removes them without causing damage from high impact energy. The C<sub>60</sub><sup>+</sup> gun also provides a consistent sputter rate across various materials, which makes it the best choice for probing complex layered interfaces in photovoltaics and semiconductor applications.

## Combining Options for Ultimate Performance



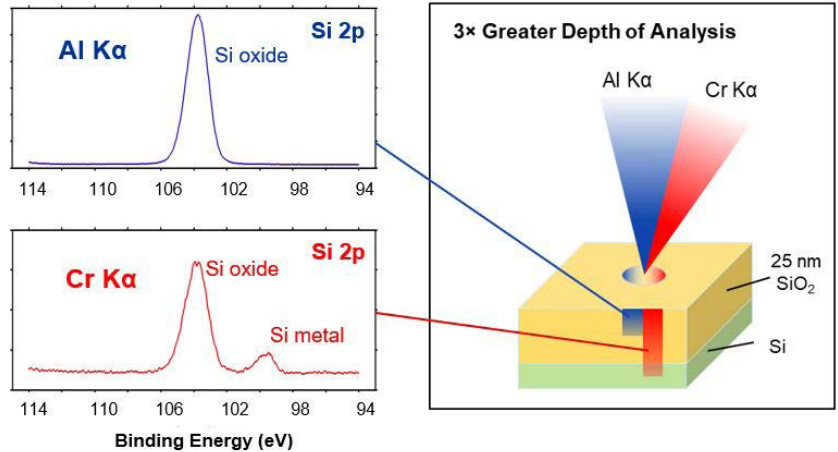
PHI *Genesis* instruments offer a unique advantage in that all analytical options are directed to the same point, allowing users to choose the best set of options to achieve their desired results. This includes the ability to use multiple ion guns during a single depth profile acquisition, as shown here for complex multilayered stack, which can optimize the speed of acquisition and minimize damage to the materials being analyzed.

# BENEFITS OF A HARD X-RAY SOURCE

## 3x Depth of Analysis

The Cr X-ray source has a photon energy of 5414.8 eV and provides for depths of analysis roughly 3 times those obtained using an Al X-ray source. This allows for:

- Analysis of buried layers and interfaces deeper than traditional XPS;
- Decreased effect of chemical state damage induced by ion sputtering;
- Access to additional transitions of higher binding energy for complementary chemical information and interpretation;
- Reduced effect of surface contamination
- XPS and HAXPES analysis from identical sample positions.



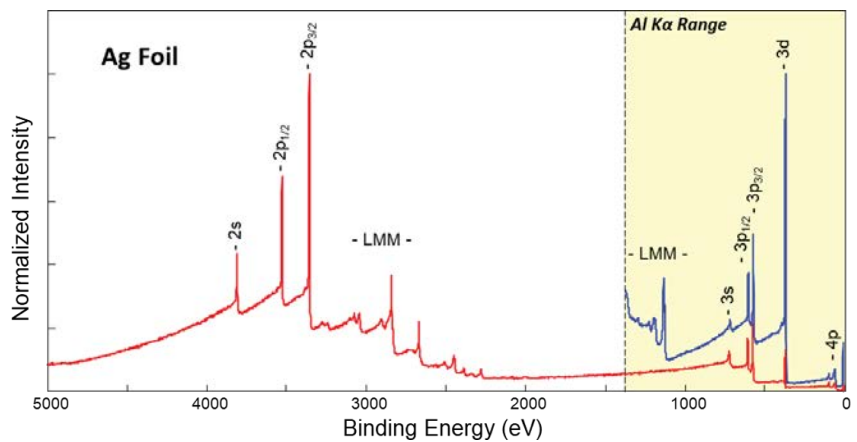
Si 2p high-resolution spectra obtained using Al and Cr sources from a 25 nm SiO<sub>2</sub>/Si sample. Signal from Si metal is detected in the spectrum obtained using the Cr source due to the larger information depth with respect to the Al source.

## Additional Transitions

Using the Cr source allows access to 3x the energy range that is available using an Al X-ray source, unlocking transitions from different sample depths.

Chemical information from both surface (XPS) and subsurface (HAXPES) depths can be obtained for depth-resolved composition without ion beam sputtering.

Both X-ray sources are aligned to the same focal point of the electron energy analyzer, enabling same-area analysis using either XPS or HAXPES, thus providing chemical information from small or large areas at different sample depths.



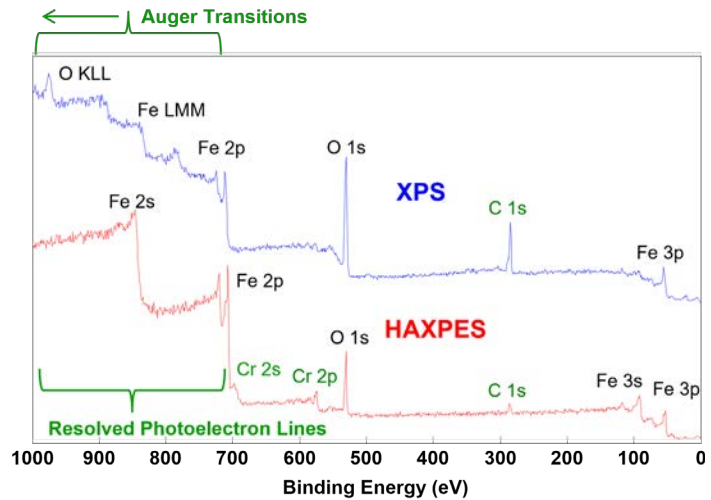
Overlaid survey spectra of Ag foil collected with Al and Cr X-rays. Due to the higher photon energy of Cr X-rays, higher binding energy transitions become available.



# Elimination of Auger Peak Interferences and Contamination Effects

Collecting spectra from the same sample using both Al K $\alpha$  and Cr K $\alpha$  X-ray sources demonstrate the ability to shift Auger transitions and clearly resolve overlapped photoelectron lines.

HAXPES is much less surface-sensitive than XPS, reducing the need to clean adventitious carbon contamination prior to analysis.

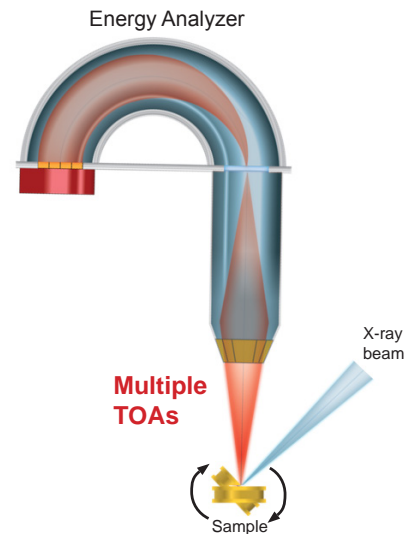


In the survey spectrum obtained from a stainless steel sample with the Al X-ray (blue trace), the Fe and O Auger transitions overlap with photoelectron lines. Using the Cr X-ray source (red trace) the Auger peaks are shifted and the photoelectron lines can be resolved.

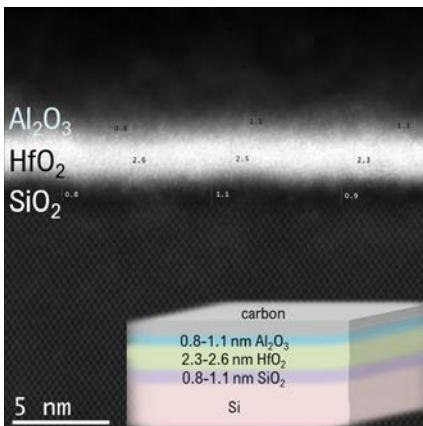
# Non-Destructive Depth Profiling

Using a higher energy, hard X-ray source probes deeper into a material than conventional XPS, allowing to analyze the unperturbed composition and chemical state of buried interfaces and compare surface to bulk-like features.

PHI *Genesis* can perform depth profiling of a sample surface easily without risking damage caused by sputtering. This is achieved by taking spectra at different take-off angles (TOAs), combined with PHI's advanced and automated structure determination analysis software called *StrataPHI*. PHI *Genesis* precisely and automatically controls the sample angle, location, and X-ray beam to ensure that the signal is generated from exact area of interest, even when a small X-ray spot size is used.

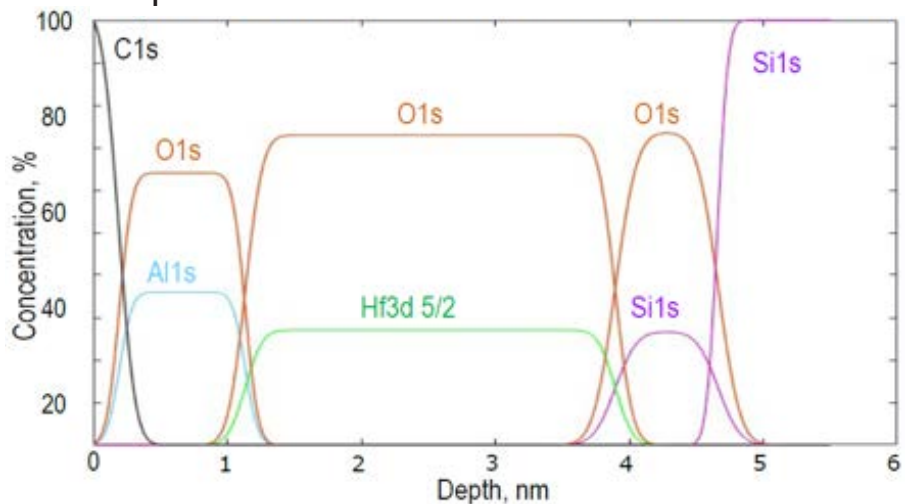


## Multilayered Device



Layers visualized via TEM

## Sample Structure Determined from AR-HAXPES





## Applications

High-throughput quantitative chemical structure-to-property optimization is essential for research and development of advanced functional materials used in all-solid-state batteries, semiconductors, photovoltaics, catalysts, and many other applications. These complex multi-component materials require a comprehensive analysis of their chemical structure to achieve optimal properties.

The PHI *Genesis* scanning X-ray spectrometer offers unsurpassed high performance with a high degree of flexibility and automation to meet the requirements for all types of scientific applications.

Application areas include:

- Semiconductors, batteries, organic devices, catalysts, quantum dots, nanoparticles, bio- and life-science materials, polymers, ceramics, metals, and many additional solid materials and devices.

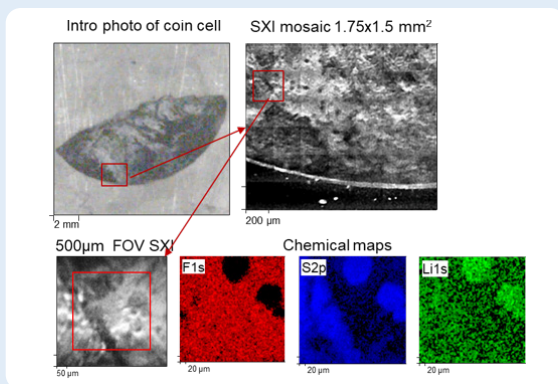
# Wide Variety of PHI Genesis Options for Research Needs

## Batteries

XPS

Transfer Vessel

### High-resolution XPS imaging of tested battery cell

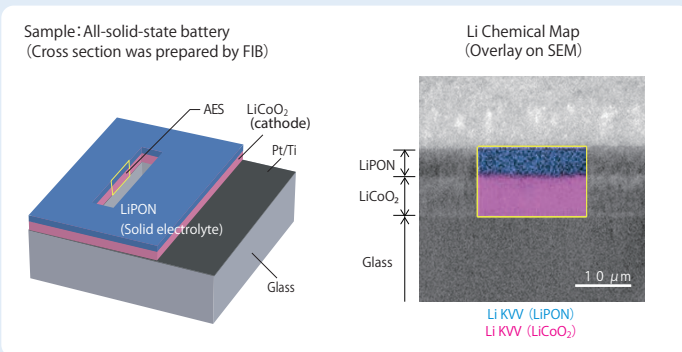


- Sub-5 µm X-ray beam generates X-ray induced secondary electron images (SXIs), providing 100% confidence in locating small features.
- Stitched SXI mosaic can be used for selecting areas for small area spectroscopic and imaging analysis.
- Multispectral chemical maps visualize distribution of individual components of SEI layer. Maps show distribution of PDVF binder and Li sulfide formed during cycling.

### AES lithium chemical map of solid-state battery cross-section

AES

Transfer Vessel



- Li-based materials, especially solid-state, are sensitive to electron beam irradiation.
- The high-sensitivity analyzer available on the PHI Genesis enables fast acquisition of AES chemical maps at low beam current (300 pA).

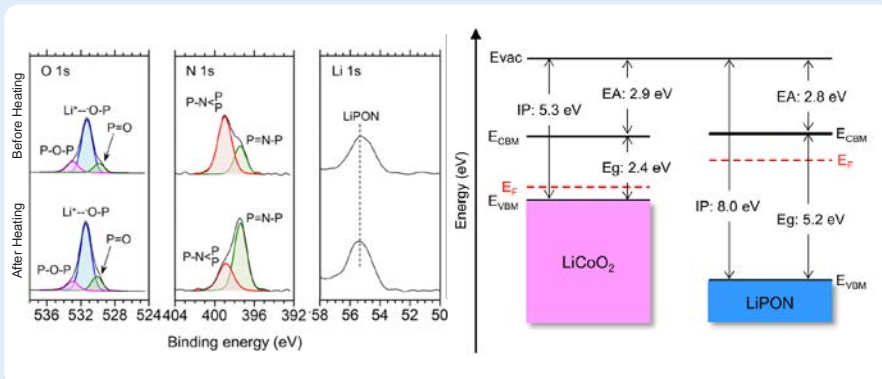
### Heating stage

### Chemical and electronic spectroscopic analysis of solid-state battery

XPS

UPS

LEIPS



Heating revealed temperature-induced interaction between battery components that impacted chemical and electronic properties.

- Change in chemical state suggests intermixing between the layers in the battery.
- Decrease in band gap for LIPON due to diffusion and interaction with the LiCoO<sub>2</sub> Layer.

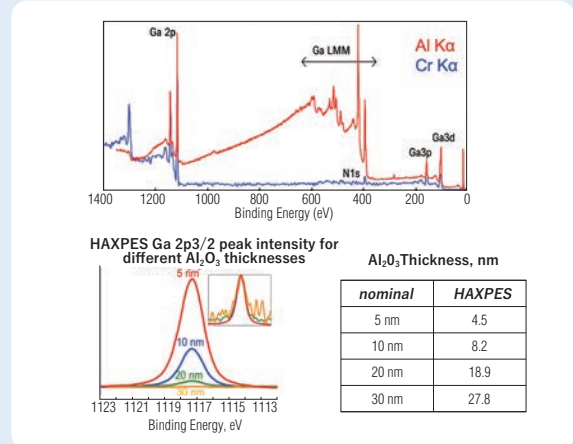
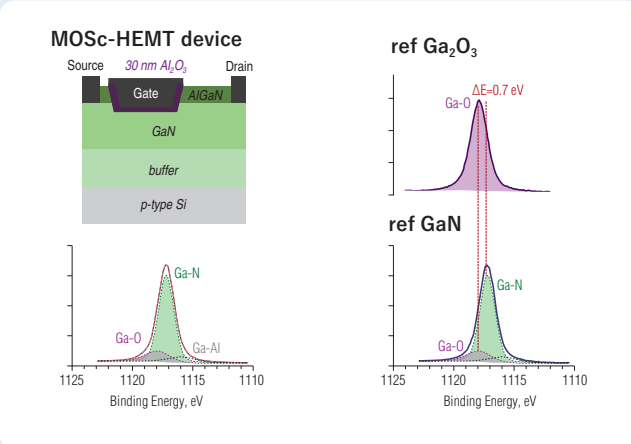
# Semiconductors

HAXPES

StrataPHI

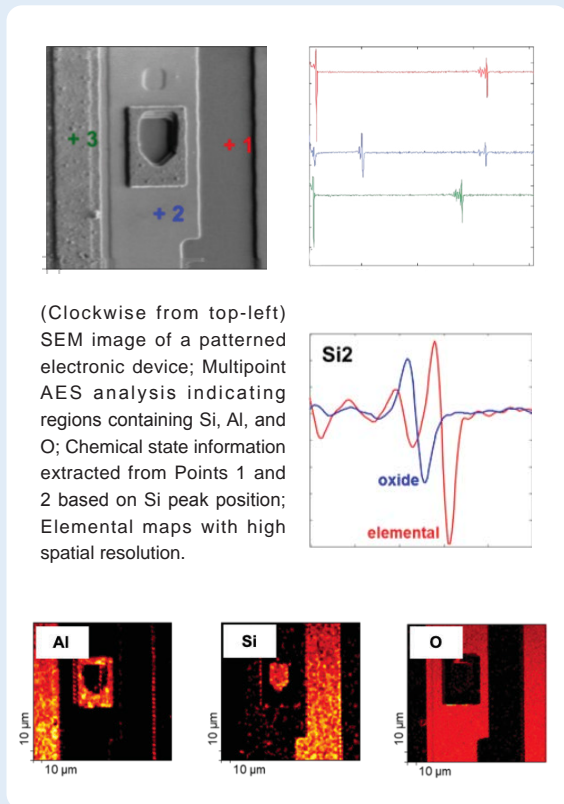
Semiconductor devices generally consist of complex thin films containing many elements, and their development often requires non-destructive analysis of chemical states at interfaces.

HAXPES plays a critical role in acquiring information from buried interfaces, such as GaN under gate oxide film, that cannot be obtained through other techniques.



- The high-energy Cr K $\alpha$  X-ray source probes beneath the thick oxide layer without sputtering and avoids critical overlap between N 1s and Ga LMM Auger line, which is an issue when using Al K $\alpha$  X-ray source.
- Ga is still detected with the presence of a 30 nm surface oxide layer.
- The thickness of alumina layers can be accurately estimated using *StrataPHI* software for thin-film structure optimization.

AES



When the features of interest are too small for XPS analysis, AES is often used.

- The electron beam in AES is up to 100 times smaller than the XPS X-ray beam, opening up new possibilities for sample characterization with increased spatial resolution.
- The convergence of the optical, SXI, and SEM images allows 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-beam charge neutralization allows for AES analysis of some insulating samples.

# Organic Devices

UPS

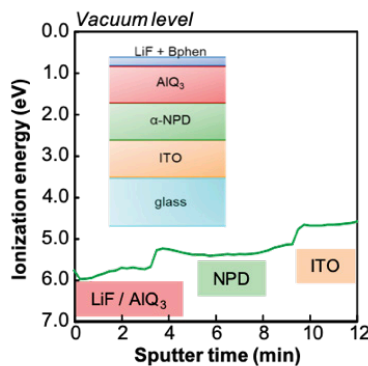
LEIPS

GCIB

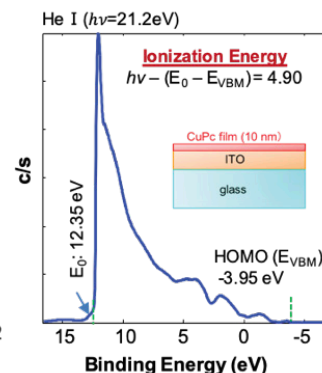
Efficient charge transport is essential for the design of complex electronic material systems, such as display panels, flexible circuitry, and photovoltaics.

This requires a comprehensive understanding of the electronic band structure.

Low energy inverse photoemission spectroscopy (LEIPS) provides electron affinity, which is required for designing organic light emitting diodes (OLEDs), understanding band structure at metal-semiconductor and semiconductor heterojunctions, and in studies of charge-transfer processes.

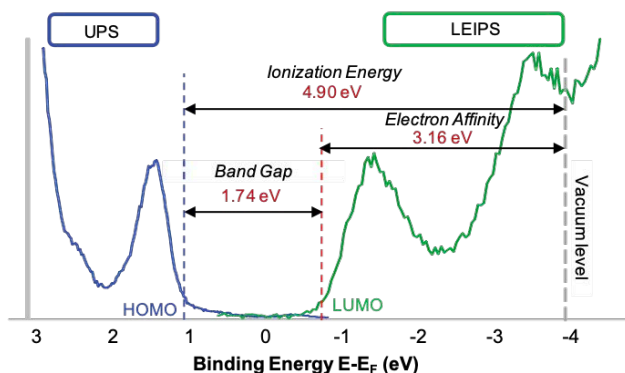


5 keV Ar<sup>+</sup> GCIB depth profile of OLED multilayered film. Ionization energy is extracted from UPS spectra at each depth sputter cycle.



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

## Combined measurements from UPS and LEIPS



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.

- 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.
- This provides a complete understanding of the electronic structure of materials.

UPS

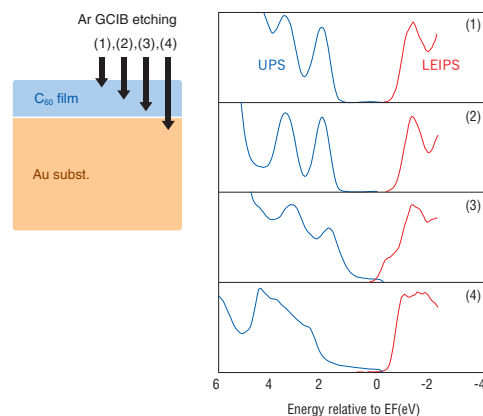
LEIPS

GCIB

Energy diagram of organic layers as a function of depth can be determined by combination of UPS/LEIPS and Ar-GCIB depth profiling.

The evolution of the energy band diagram using UPS/LEIPS and Ar-GCIB at multiple locations in a C<sub>60</sub> and Au sample:

- (1) C<sub>60</sub> film surface
- (2) C<sub>60</sub> film after surface cleaning.
- (3) C<sub>60</sub> film /Au interface
- (4) Au surface



## Optional Accessories

- HAXPES
- UPS
- LEIPS
- AES
- REELS
- Dual anode X-ray sources (Mg/Mg, Mg/Al, Mg/Zr)
- Dual source ion gun for Ar-Monatomic/Ar-GCIB
- Ar-GCIB cluster size measurement tool
- 20 kV C<sub>60</sub> cluster ion gun
- Narrow acceptance angle aperture
- Sample heating and cooling
- 4-contact voltage application
- Transfer vessel
- Dedicated intro pumping
- High magnification sample observation microscope (live view)
- Sample positioning system (SPS)



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