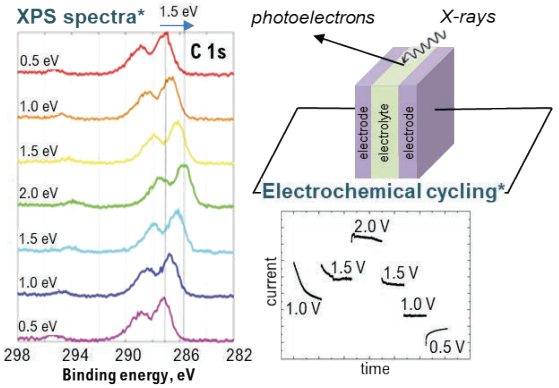
VersaProbe 4 Scanning XPS Microprobe: Unique Instrument for Battery Characterization

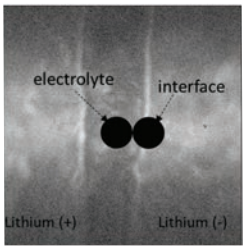
The *VersaProbe* 4 is a highly versatile, multi-technique instrument with PHI's patented, monochromatic, micro-focused, scanning X-ray source. The instrument offers a true SEM-like ease of operation with exceptional capabilities for micro and large area spectroscopy.



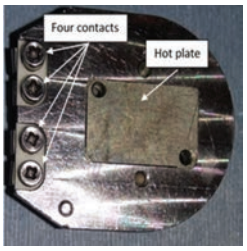
The fully integrated multi-technique platform of the *VersaProbe* 4 offers a complete suite of specialized solutions for in situ characterization of battery materials:

- Determine precise location of interfaces between battery components using X-ray induced secondary electron images (SXIs) for targeted small area analysis
- Electrochemical (biasing, polarization studies) experiments using 4-contact hot-cold stage for operando studios
- Inert gas transfer vessel to transfer samples from glove box or other analytical instruments
- Electronic band structure of organic and inorganic materials using UPS, LEIPS and REELS measurement from the same sample location

SXI visualization of electrodes and electrolyte

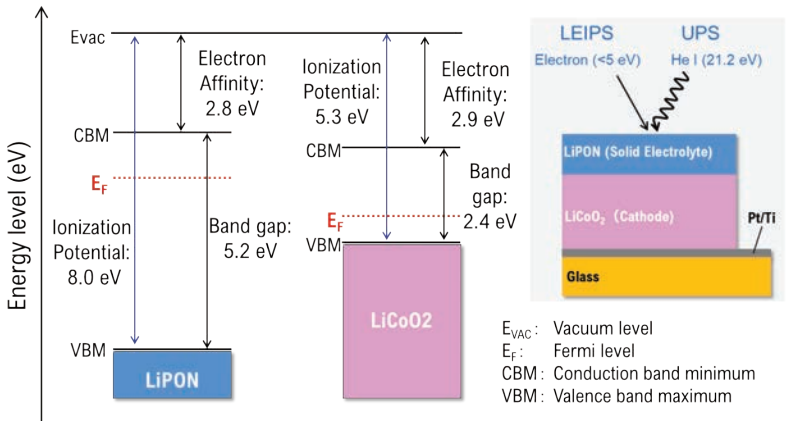


4-contact stage for polarization studies



Inert gas transfer vessel

Energy diagram determined by UPS/LEIPS



These features available on VP4 open new capabilities to investigate the components and interfaces of batteries at realistic operando conditions

**A.Benayad et.al. JPCA, 2021 https://dx.doi.org/10.1021/acs.jpca.0c09047*

What is XPS?

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 quantitative and chemical state information from the surface of the material being studied.

XPS is typically accomplished by exciting a sample's surface with mono-energetic Al Ka X-rays causing photoelectrons to be emitted from the sample surface. An electron energy analyzer is used to measure the energy of the emitted photoelectrons. From the binding energy and intensity of a photoelectron peak, the elemental identity, chemical state, and quantity of a detected element can be determined.

	XPS
Spatial Resolution	7 µm
Sampling Depth (nm)	1-10
Detection Limits	0.1 atom%
Information Content	Elemental (all except H and He) Short range Chemical
Organic Information	Yes
Quantification	Excellent
Depth Profiling	Yes

VersaProbe 4 Capabilities Enabling Battery Applications:

- Analytical Capabilities:
  - XPS (chemical state of surfaces/interfaces for all elements above He)
  - AES (high spatial resolution surface elemental analysis)
  - UPS/LEIPS/REELS (electronic band structure characterization)
- Sample handling:
  - Inert gas transfer vessel
- Sample processing:
  - In situ voltage cycling
  - In situ electrode deposition
  - Hot/Cold intro & analysis chamber
  - Sputter depth profiling using monatomic Ar, optional 20 kV C_{60} ion gun and/or Gas Cluster Ion Beam (GCIB)

VersaProbe 4 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 mounts

PHYSICAL ELECTRONICS
A DIVISION OF ULVAC-PHI

18725 Lake Drive East
Chanhassen, MN 55317, USA

www.phi.com
www.ulvac-phi.com

2500 Hagisono, Chigasaski
253-8522, Japan