



Welcome to the Materials Design Building and the Analytical
PicoProbe Electron Optical Beam Line Overview Site

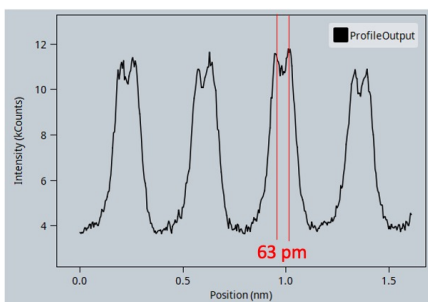
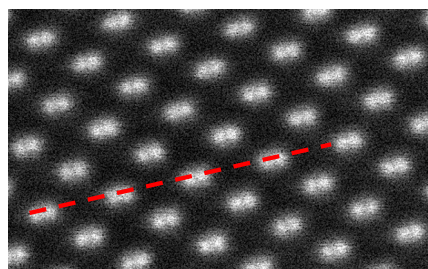
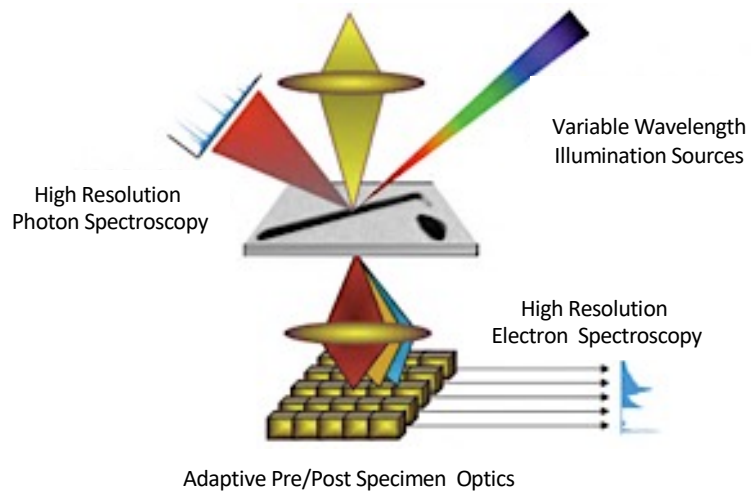
The Analytical PicoProbe Electron Optical Beam Line

The Analytical Picoprobe Electron Optical Beam Line is a unique instrument, resulting from a CRADA with ThermoFisher Scientific, who approached Nestor Zaluzec while at Argonne to collaborate, design, build and implement a prototype for the next generation of Analytical Resource for characterization at the highest sensitivity and spatial resolution.

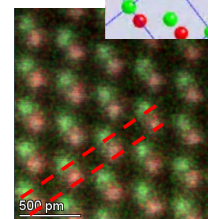
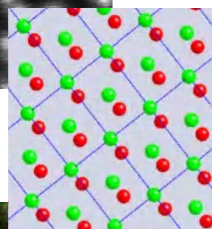
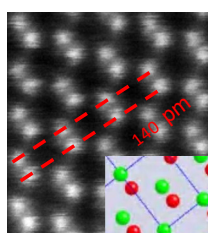
Picoprobe is an imaging, diffracton, and in-situ analytical instrument, with no equal, and is being used to address challenging problems in today's technologically important materials.

It's key features are shown on the next PPT. As of Sept 2024, a version of this instrument is now commercially available from ThermoFisher Scientific as the Spectra Ultra X Illiad System

The Analytical PicoProbe Electron Optical Beam Line



<211> GaN
Sub-Atomic Imaging
Resolution 55 pm



GaAs {110}
Sub-nm HyperSpectral
Imaging 140 pm

Key Features/Attributes

- E_0 : 30-300 kV Electron Source
- High Brightness Coherent Field Emission Gun
- Probe Corrector ($\delta x < 55$ pm)
- Ultra Monochromed EELS ($\delta E < 20$ meV)
- X_{PAD} 4.5 sR X-ray Spectrometer
- ZTwin Lens
- Next Generation Multi-Port Octagon
- HR/LowDrift Piezo Goniometer
- 4Kx4Kx16 bit CMOS Imaging Detector
- 4Kx4Kx16 bit Low Dose Electron Detector
- 128x128x30 bit Diffraction Camera
- CTEM/STEM/iDPC /4DSTEM Modes
- Analytical LBHV DTilt Holder
- Ambient ST XEDS Tomography Holder
- Ptychography/Tomography Holder
- Ambient ST XEDS Liquid Cell Holder
- Cryo Tomography/Spectroscopy ST Holder
- Heating ST Holder
- Inert Cryo Environmental Transfer Holder
- Dynamic Imaging ~ 0 -300 fps
- Multi-Modal Enabled
- Telepresence Enabled and Computationally Mediated



Prototype of the
ThermoFisher Scientific
Spectra Ultra X Illiad

ANL/ThermoFisher
CRADA #1300701

UofC NSF MRI #2117896

Image Resolution

CTEM < 100 pm / STEM ~ 55 pm

Temporal Resolution

0- 300+ fps (CTEM) ≤ 1 usec/pixel (STEM)

Spectroscopy

Worlds most sensitive AEM XEDS detector
UHR Electron Energy Loss Spectrometer + UltiMono

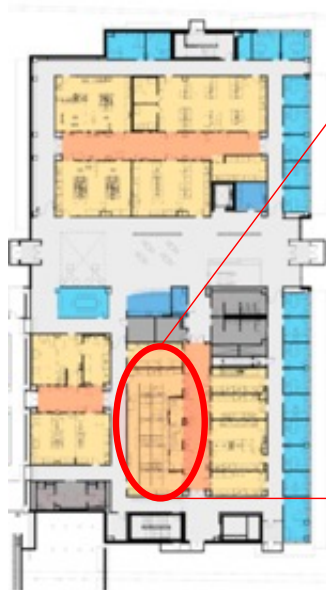
Environmental

Commercial and Custom (Ambient) in-Situ Holders
Cryo EM Imaging for Soft/Hard, Energy & Quantum Materials
Comprehensive Suite of In-situ Specimen Holders
including Inert Environment and Cryo transfer

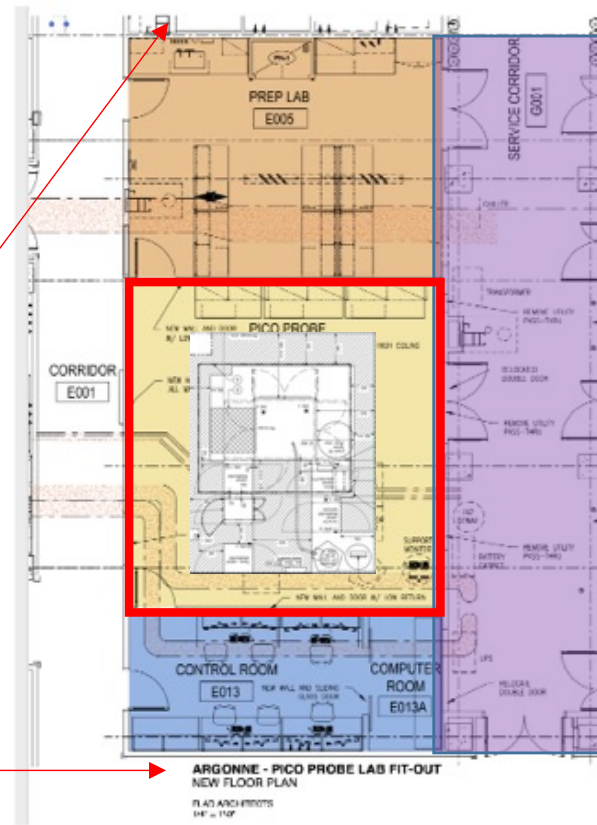
Future

Adaptive Holographic Optics (in Progress)
Higher Speed Electron Detectors

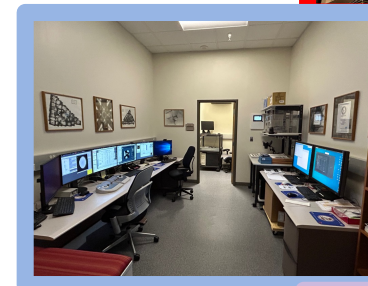
Materials Design Lab / Argonne The Custom Analytical PicoProbe Electron Metrological Suite



Plan View
Ground Floor



A custom built Metrology Suite
built by ANL to host PicoProbe
and facilitate operations



Environmental Controlled/Shielded
EMF (< 10 nT RMS AC)
Temp (< 1° C /day)
Vibrational /Acoustic Isolation

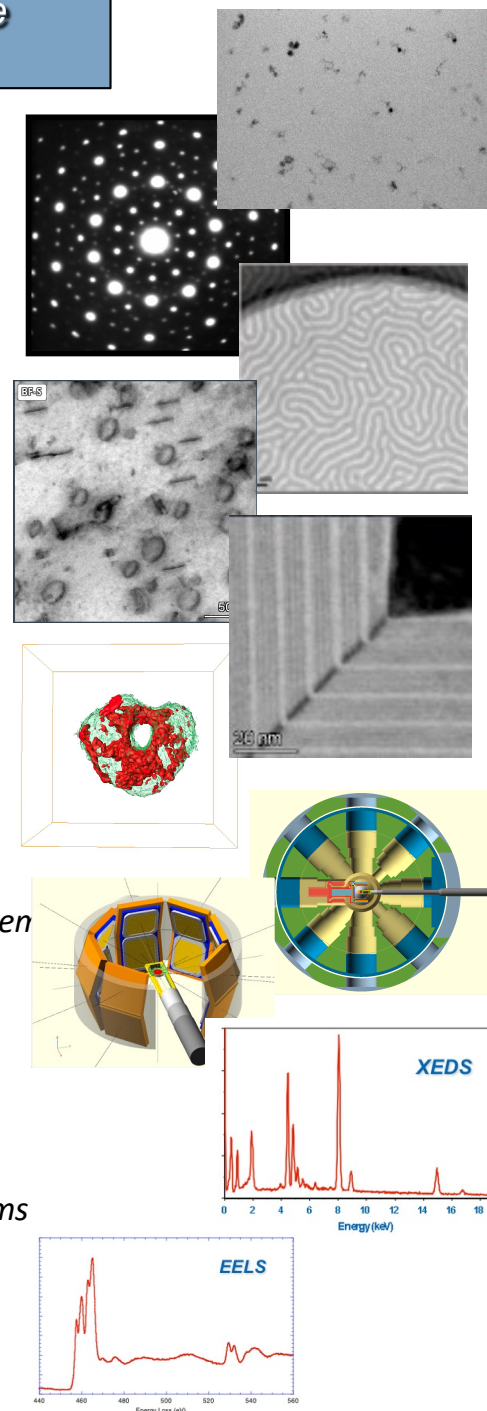
Current Soft / Hard Matter R&D at the PicoProbe

Existing Programmatic Thrusts - via Advanced Electron Microscopy

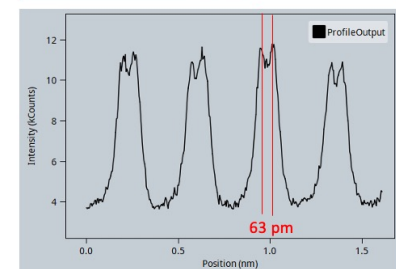
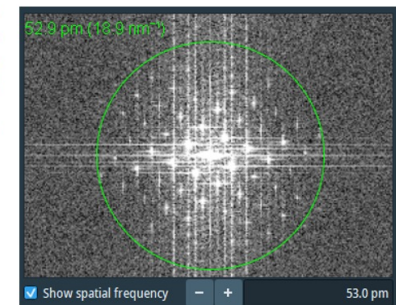
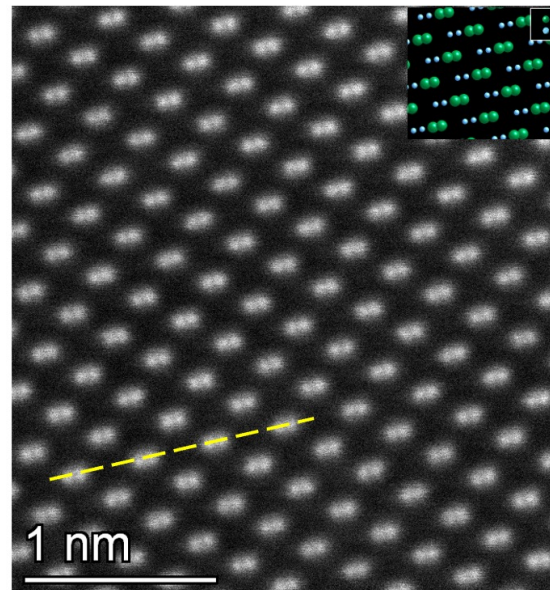
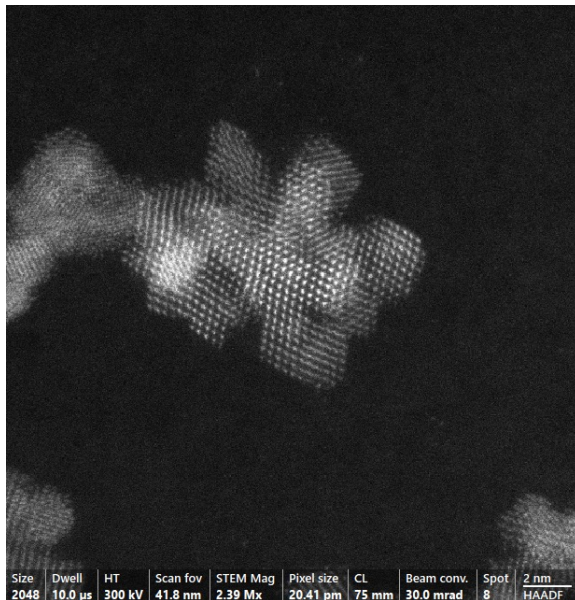
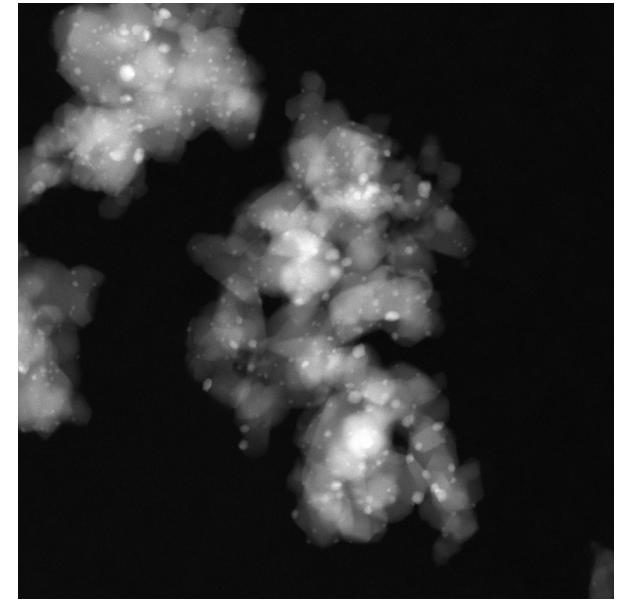
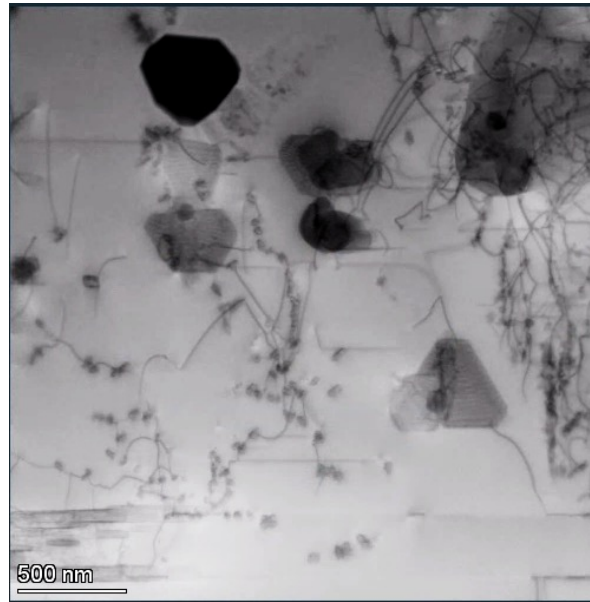
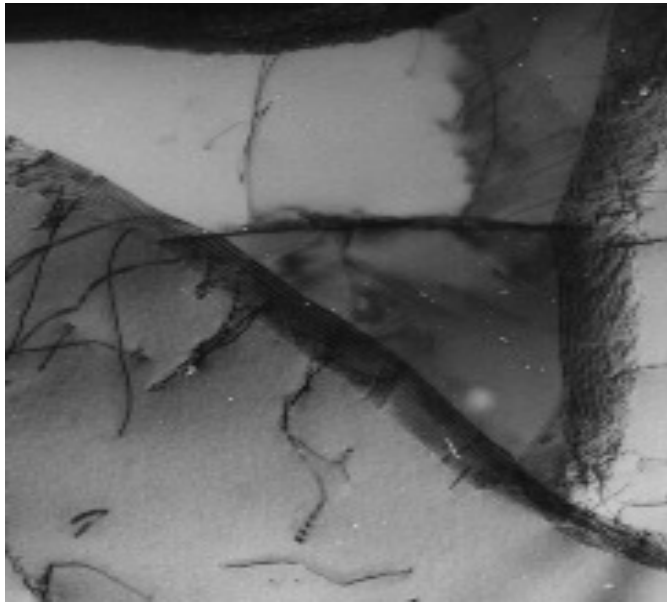
- *Materials Chemistry & Manufacturing Science and Engineering Initiatives*
 - ✓ *Energy Storage and Quantum Materials (ANL / ESRA - UofC / LESC)*
 - ✓ *Advanced Materials for Energy-Water Systems (EFRC / AMEWS)*
 - ✓ *Solar Energy Conversion in Photosynthesis and Photosynthetic Biomimetics*
 - ✓ *Inorganic/Organic Hybrid Materials*
 - ✓ *Irradiation Effects in Materials*
- *Soft/HardMatter Characterization, Instrumentation & Technology R&D*
 - ✓ *Instrumental/Experimental Developmental PicoProbe (ANL/TF CRADA)*
 - ✓ *Dynamic PicoProbe (NSF - MRI - UofChicago)*
 - ✓ *Analytical Metrology at High Spatial Resolution and Sensitivity*

Research Opportunities: Materials , Chemistry, Physics, Life Science, Engineering

- *Physical Sciences*
 - *Engineered Nanomaterials , Catalysts, Energy and Quantum Materials*
 - *Monolayers/2D Materials/Interfacial Species/ Defects/Plasmonics*
 - *Organic/Inorganic Heterostructures - Smart Materials for Advanced Functional system*
 - *Polymeric Systems: Self-assembly, templated growth,*
 - *Molecular, Colloid and Membrane Frameworks*
 - *Complex Fluids, Emulsions, Gels, Membranes*
 - *Amorphous materials*
- *Structural Biology/Life Sciences*
 - *Single particles, macromolecular complexes, inter/intra cellular processes, biosystems*
 - *Hybrid/Smart nanostructures for therapeutic medicine / Cancer*
 - *Intelligent coatings for encapsulation and separation*
 - *TM interactions in Bio-organic processes*



Traditional Electron Microscopy : Mesoscopic to Sub-Atomic Imaging

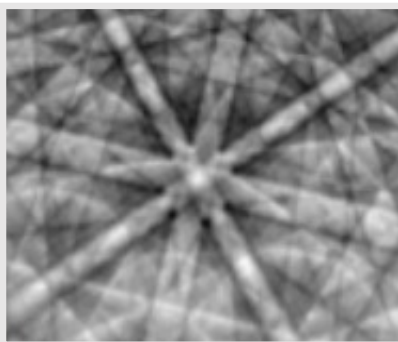
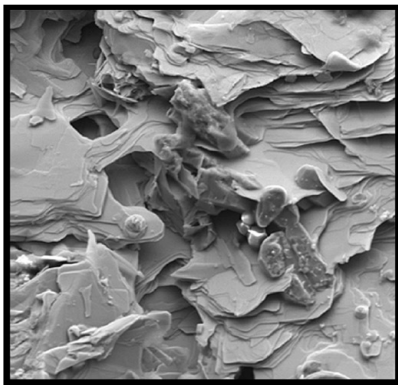


Most individuals associate Electron Column instruments/microscopes with imaging and PicoProbe covers the entire gambit from mesoscopic to subatomic

Modern Electron Metrology is a Collection of Analytical Tools

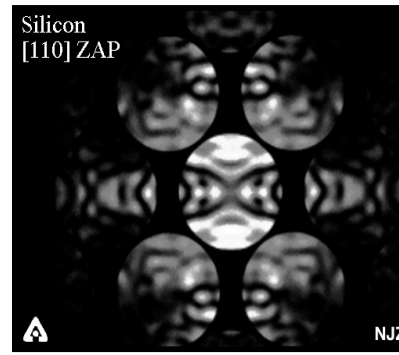
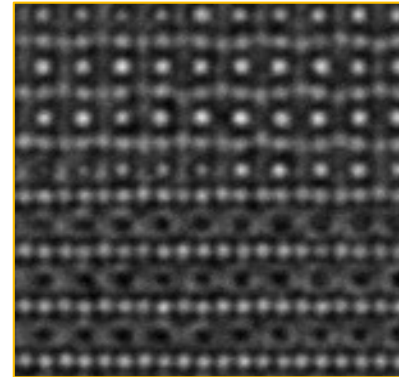
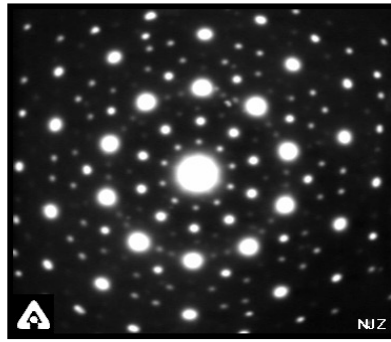
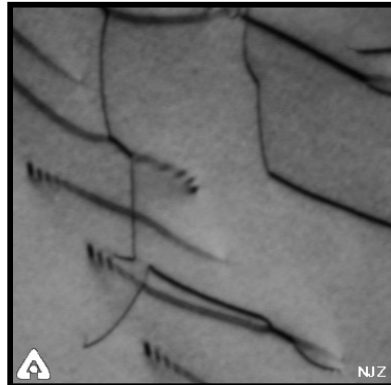
SEM

Scanning Electron Microscopy



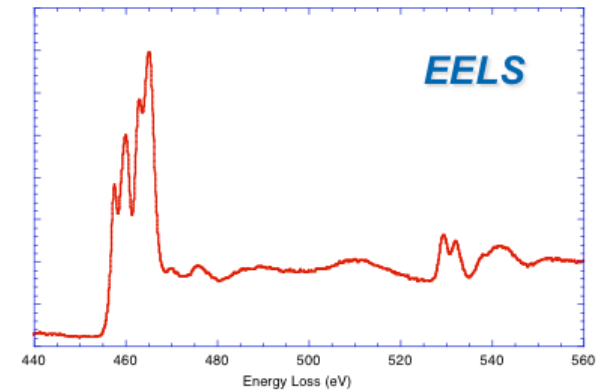
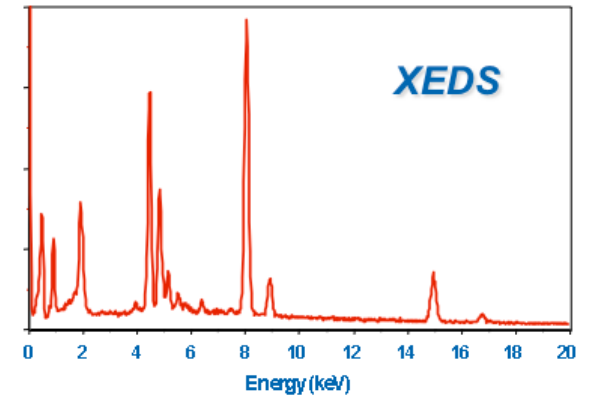
TEM - HREM

Transmission - Scanning Transmission -
High Resolution Electron Microscopy



AEM

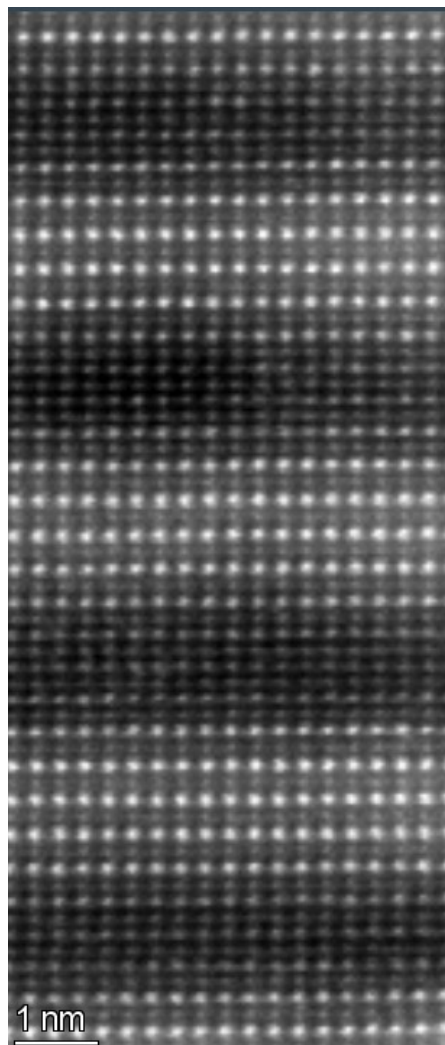
Analytical Electron Microscopy



Electron Metrology Today is more than just Imaging

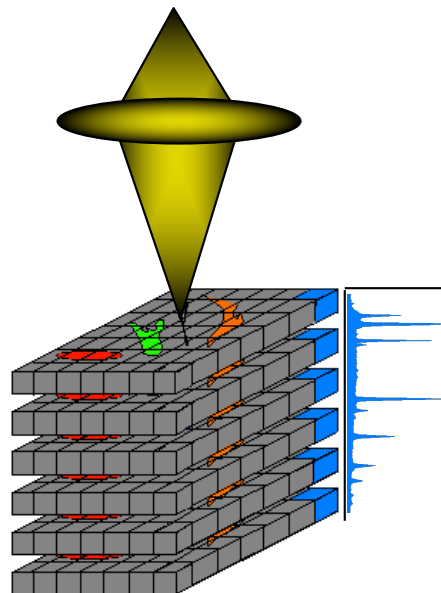
PicoProbe Also Enables Sub-Atomic Hyper Spectral Imaging

HAADF Image



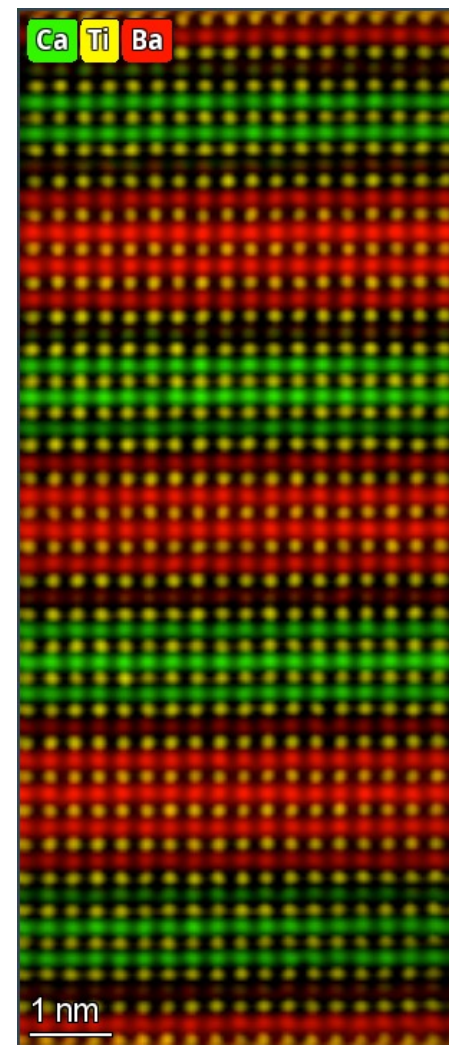
*Imaging does not always answer metrological questions
Spectroscopic Analysis is needed in today's
complex materials*

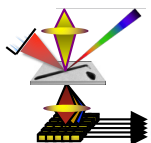
Artificial Perovskite Superlattice
 $\text{CaTiO}_3/\text{BaTiO}_3$



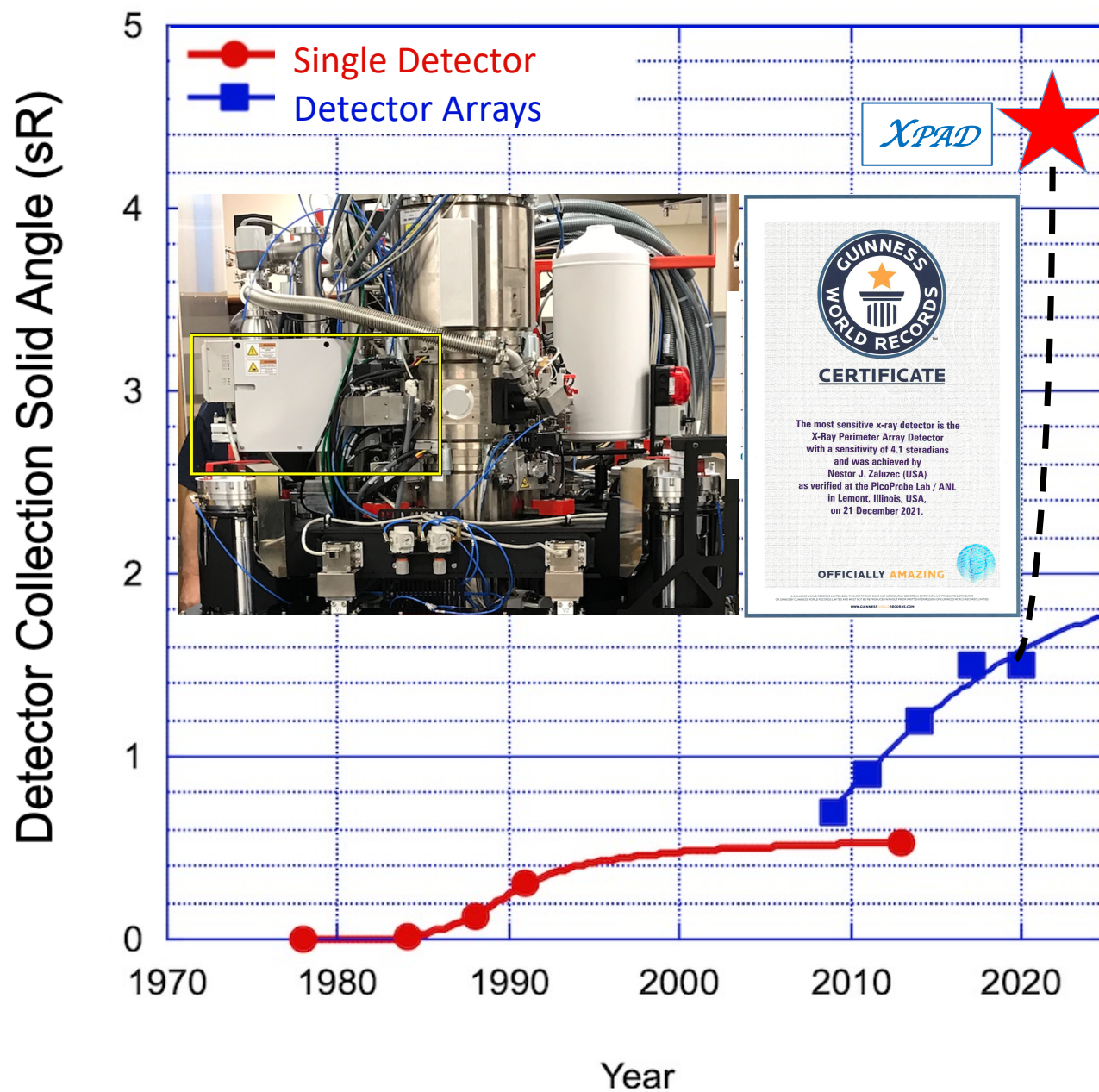
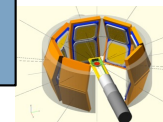
HyperSpectral
Imaging
using the XPAD

HyperSpectral Image

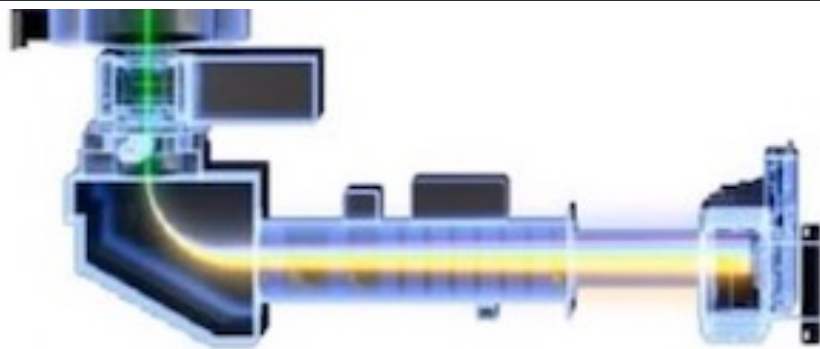
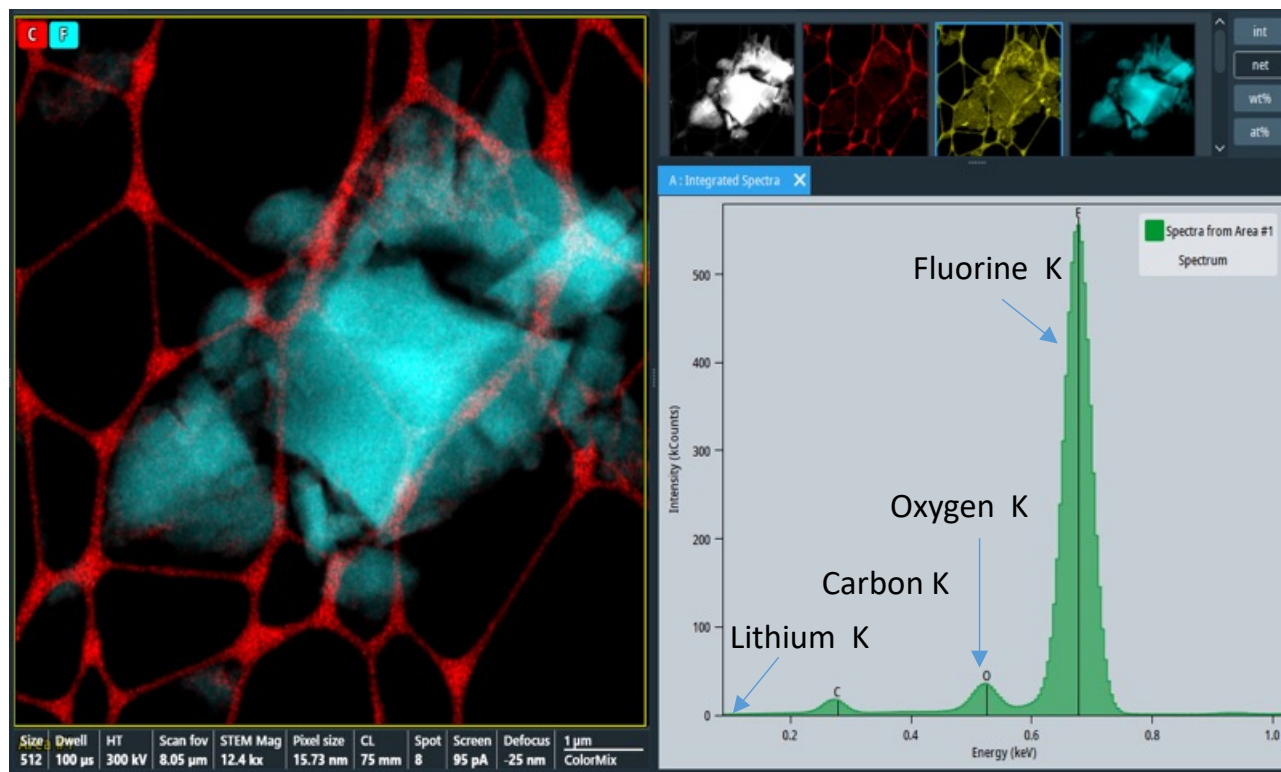




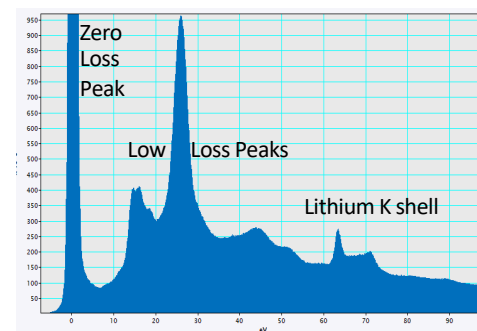
The XPAD is key to PicoProbers Sub-Atomic Hyper Spectral Imaging



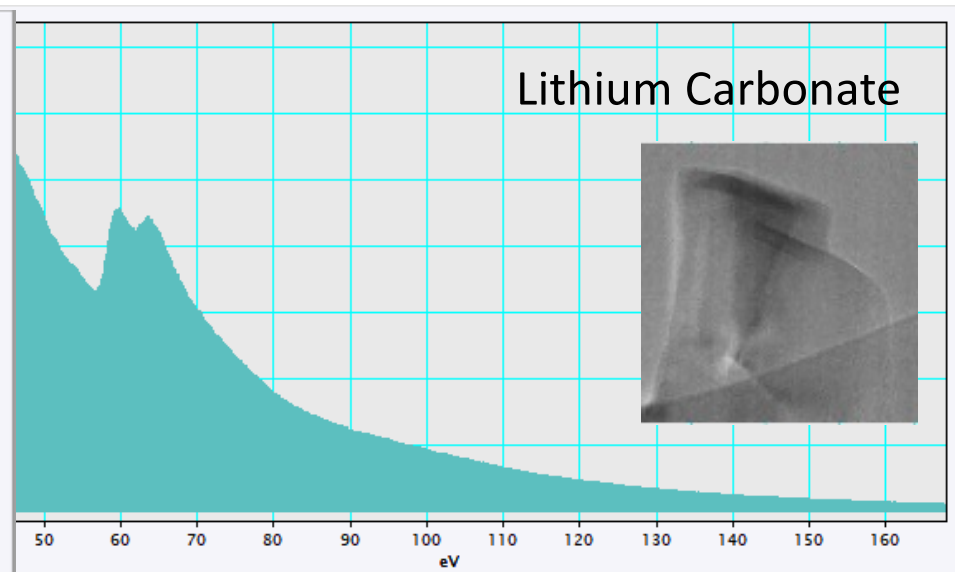
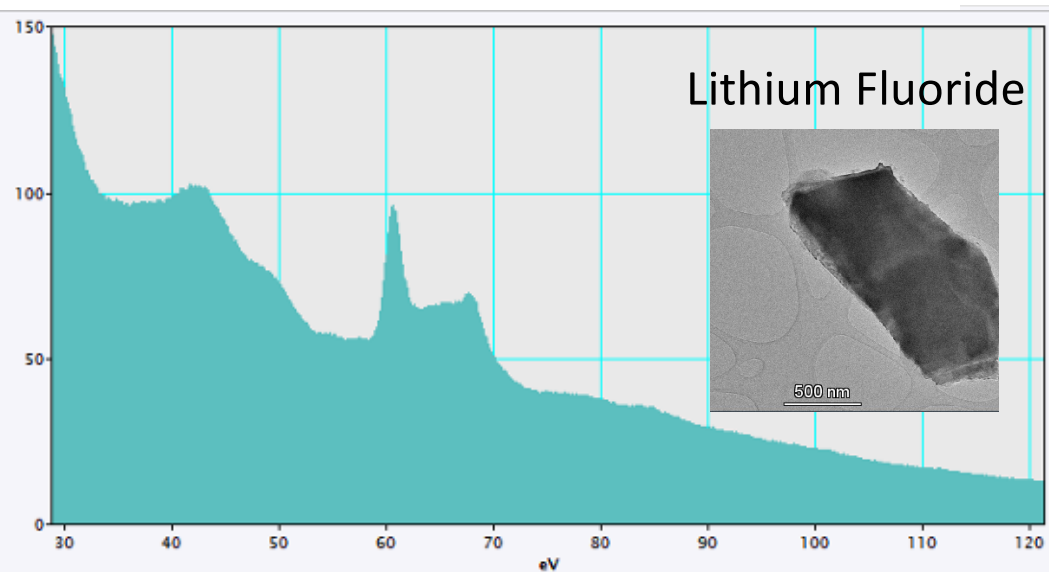
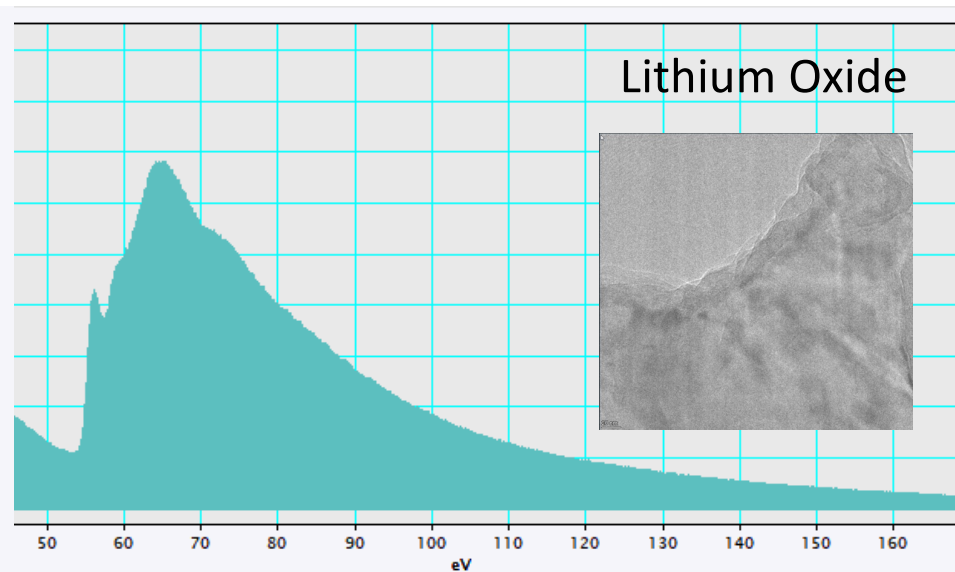
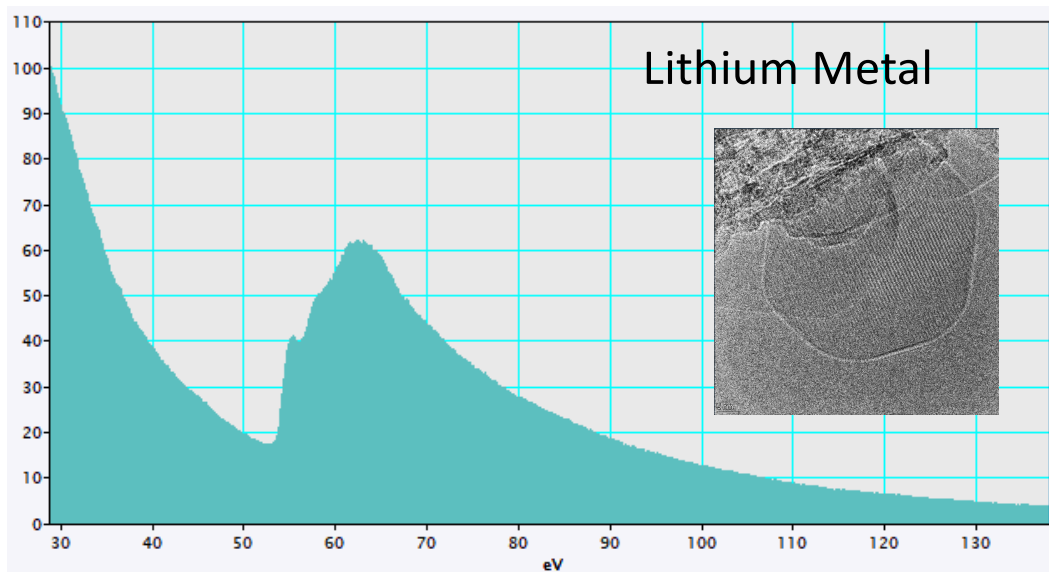
Hyperspectral Imaging of Light Elements Compounds
Requires Operation at the Highest Sensitivity
Electron Spectroscopy (EELS) Complements the Limitations of X-ray
Spectroscopy in this Regime



Electron Energy Loss Spectrometry



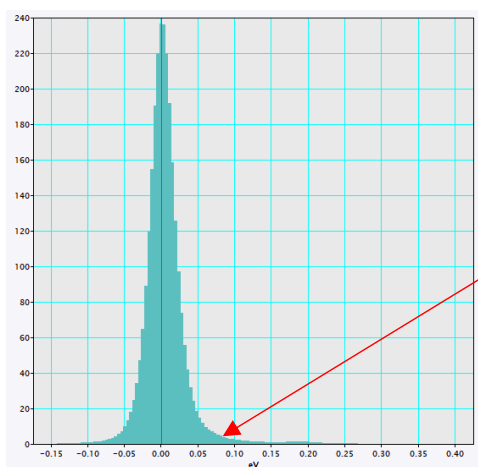
EELS Lithium Core Loss (K Shell) Spectroscopy



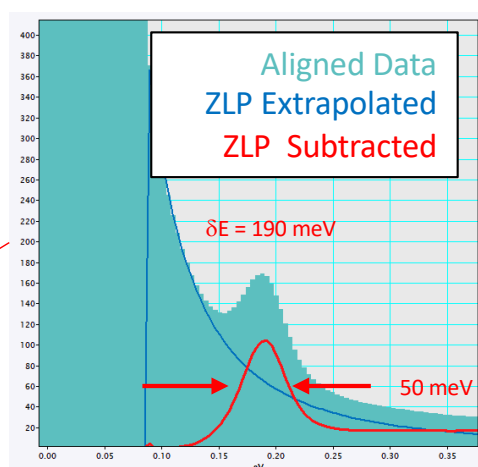
High Energy Resolution Lithium K Shell and Near Edge Structure via EELS

$E_0=300$ kV, $\theta_E=3.4$ mR

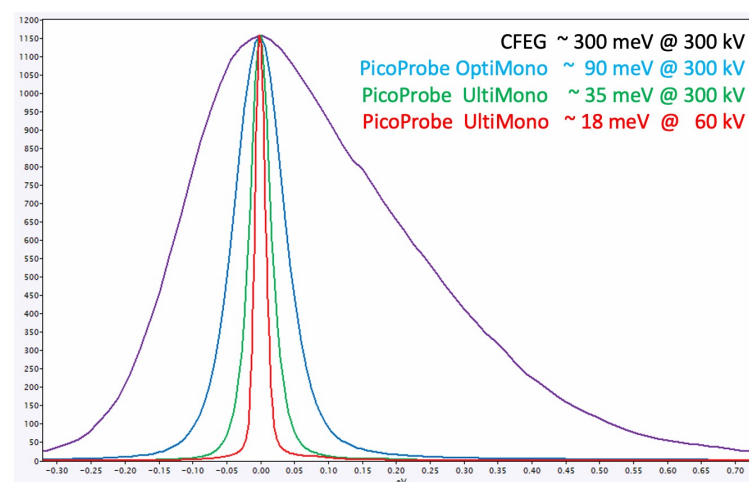
Ultra High Energy Resolution Vibrational Spectroscopy of Lithium Compounds Facilitates SEI Identification



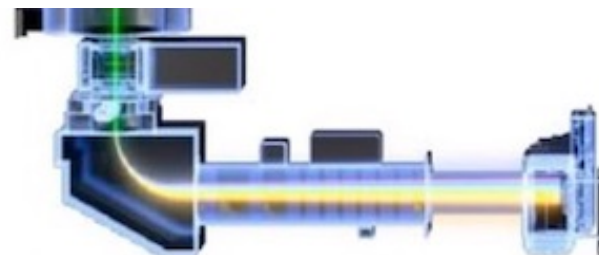
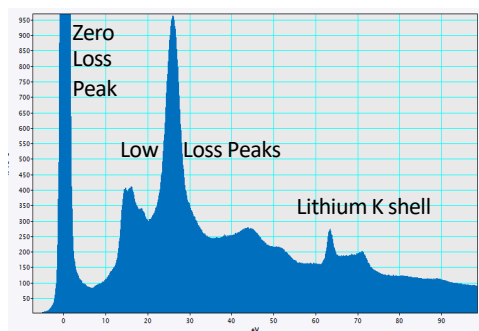
Lithium Carbonate
 $E_{FWHM} < 30 \text{ meV}$ Required



Spatially Resolved Vibrational
information in "Tail" of the
Elastic Zero Loss Peak

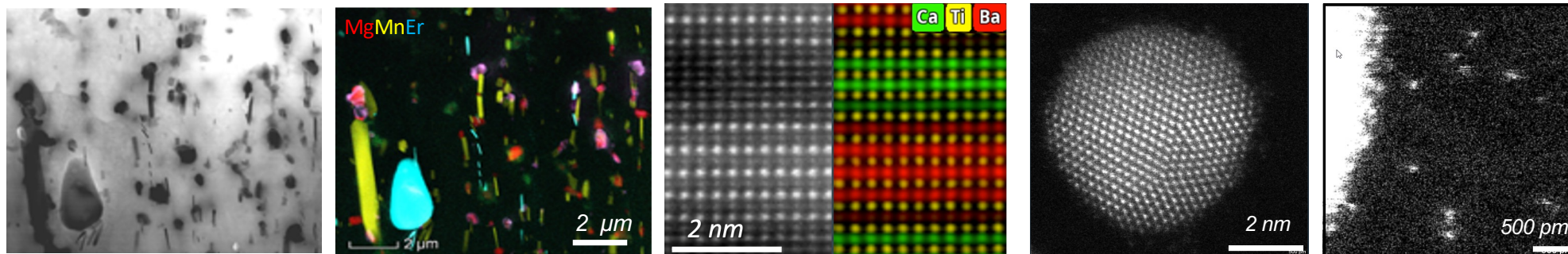


PicoProbe UltraMonochromator
Facilitates Vibrational Spectroscopy

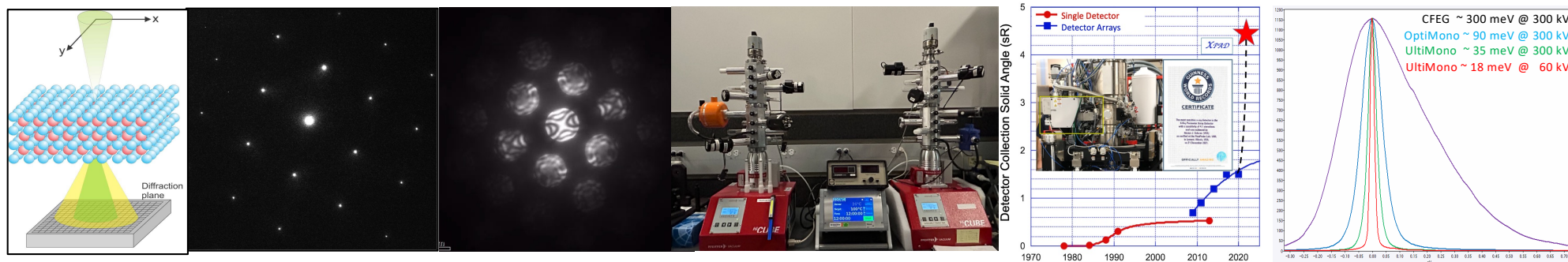


HR Electron Energy Loss Spectrometry

Dynamic In-situ, Multi-Modal, Multi-Dimensional, Correlative, High Spatial Resolution Imaging, Spectroscopic and Diffraction Metrological Characterization



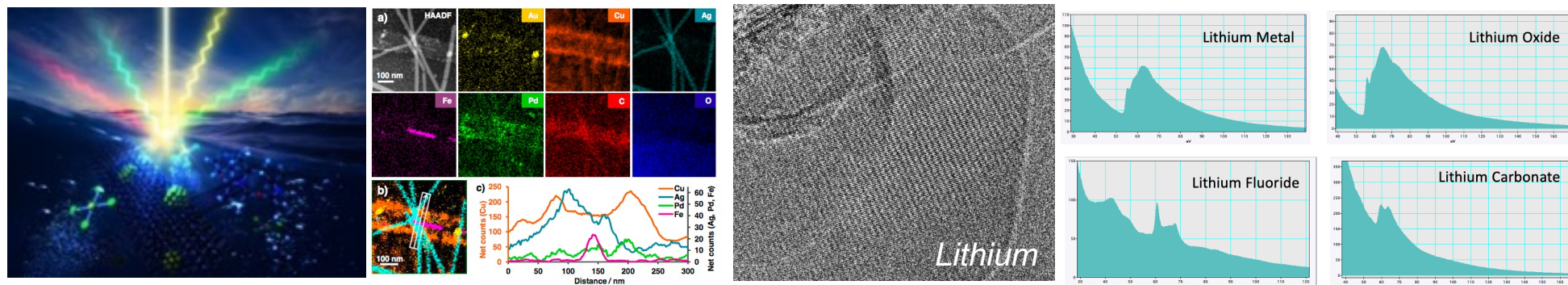
Materials Characterization and Engineering from the Mesoscopic -> Sub-nanosopic -> Single Atom imaging



Conventional / Convergent Beam and Position Resolved Diffraction

In-Situ Experimental Resources (LN₂-> 600°C)

Leading-Edge Spectroscopy



In-situ NP Evolution Vacuum/Cryogenic/Gaseous/Liquid Media

HyperSpectral Imaging and Metrology of Advanced Energy & Quantum Materials

Picoprobe is an imaging, diffraction, and in-situ analytical instrument, with no equal, and is being used to address challenging problems in today's technologically important materials.

If you can't detect it, then you can't measure it.

*Thanks !
Questions ?*

