

EAG _aboratories



Industry Service Lab Accelerates the Development of Lithium-ion Batteries

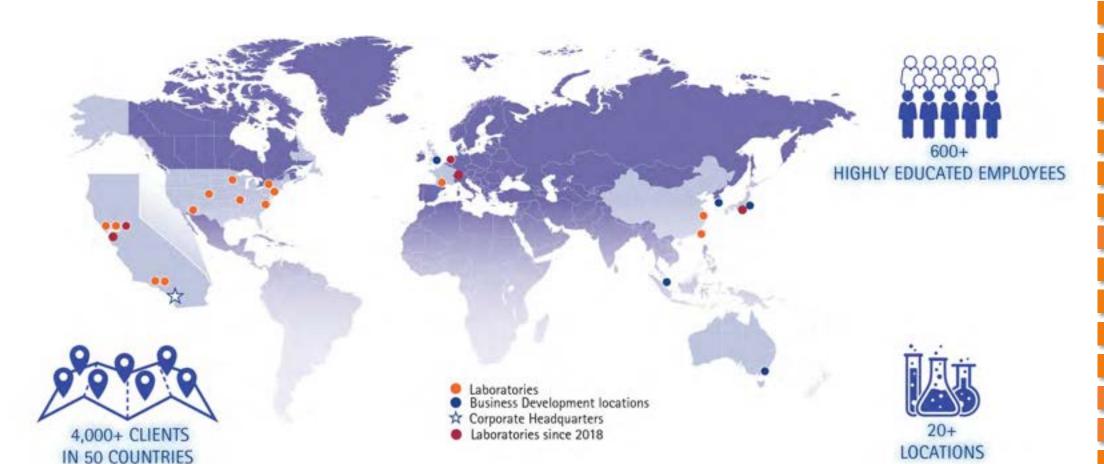
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Eurofins EAG

- Over 40 years experience in materials testing services
- Over 4,000 clients in 20+ facilities located in the United States, Europe and Asia
- ITAR registered and clients IP are secured

EAG Battery Lab

- A dedicated Battery Lab in **Milpitas, CA.**
- Total area ~2000 square feet with a dry room



All	Techniques	available at B	EAG
Most	are suitable ⁻	for battery a	nalysis
AFM	ETV-ICP-OES	MALDI	Tensiometry
ΑΡΤ	FTIR	Nano Indent	TG-DTA
Auger	GC	NMR	TG-EGA
BET & BJH	GC-MS/MS	NRA	TGA
Cryo-TEM	GDMS	ОР	TLC
DHEM	GPC	рН	ТМА
Dilatometry	HALT & MEOST	PIXE	TOF-SIMS
DPA	HFS	Pyro-GCMS	TXRF
DSC	HPLC	Raman	UHPLC
Dual Beam	IC	RBS	UV-Vis
DMA	ICP-MS	Refractromety	Viscosity
Dynamic SIMS	ICP-OES	RGA	XPS-ESCA
EBIC	IGA	Rheology	XRD
EBSD	LA-ICMS	RTX	XRF
EDS	LA-ICPMS	SAM	XRR
EELS	LCMS	SEM	
Ellipsometry	LC-MS-MS	SEM-CL	
Emissivity	LIBS	TEM-STEM	

- New services:
- Cell level and pack level cycling test
- Cell level cycling at different temperature (-40°C up to 100°C)
- EIS measurement: 10µHz to 1MHz
- Battery teardown inside Ar protected glove box
- Coin cell fabrication
- Defect, delamination of battery analysis by optical microscopy
- Elemental analysis by Laser-Induced Breakdown Spectroscopy (LIBS)
- Large cross-section and FIB/SEM tomography by Plasma FIB.
- Air-free transfer mechanism from glove box to other materials characterization instruments.

	Advanced Microscopy	Bulk Material Analysis	Chemical Analysis	Failure Analysis	Gas Analysis	Materials Characterization	Reliability Testing	Surface Analysis
Raw Materials								
Impurity survey analysis (electrode raw materials)		•			•	•		
Compositional analysis (metal/lithium content of electrode raw material)	•	•	•			•		
Phase identification (electrode raw materials)		•	•			•		
Manufacturing Process								
Elemental composition (electrode, electrolyte, separator)	•					•		•
Elemental analysis, mapping and depth profiling (electrode, separator)	•		•			•		•
Chemical state and composition (electrode, separator)	•		•			•		•
SEI characterization (electrode, separator)	•					•		•
Particle size, particle coating analysis and particle depth profiling (electrode, separator)	•					•		•
Identification and quantification of anions and cations (electrode, electrolyte)			•			•		
Levels of atmospheric species (electrode, electrolyte)			•		•			
Organic composition (electrode, separator)						•		•
Characterization of volatile organic species (electrode, electrolyte)			•		•	•		
Impurity detection (electrode, electrolyte, separator)		٠				•		
Organic compound degradation (electrode, electrolyte, separator)						•		•
Crystallinity phase (electrode, electrolyte)	•					•		
Thermal properties (electrode, electrolyte, separator)			•			•		
Application/Use								
Corrosion and contamination analysis	•			•		•		•
Delamination and void identification	•			•		•		
Battery Cycling							•	
Recycle								
Trace elemental analysis (recycled battery materials)		•			•			
Composition analysis (recycled battery materials)	•	•				•		

Optical Microscopy and Laser Induced-Breakdown Spectroscopy

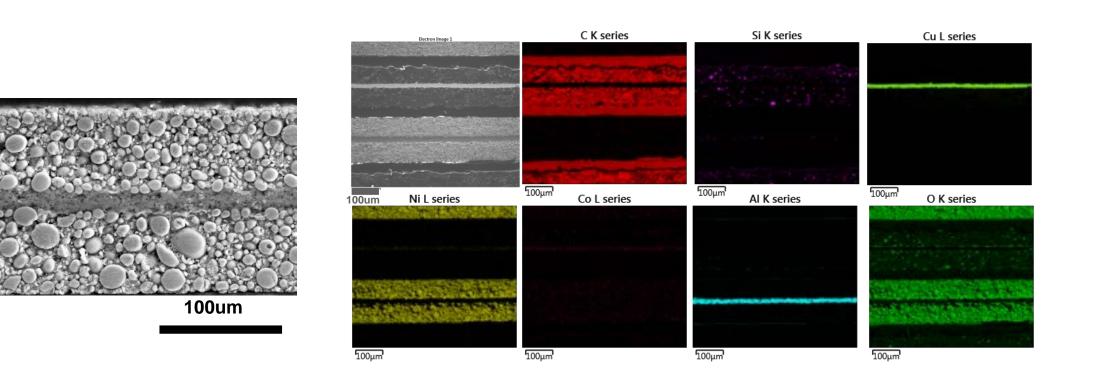
Digital Camera documents the big defect like delamination shown below.



• LIBS could provide elemental analysis instantly under Optical Microscope. Shown below is a cathode materials of LiFePO₄. Light element like lithium,

Scanning Electron Microscopy and Energy-Dispersive Spectroscopy

- High resolution SEM/EDS can provide more detailed information of the battery materials morphologies and elemental maps.
- Right maps are the 2D elemental distribution of a commercial battery cathode by SEM/EDS.



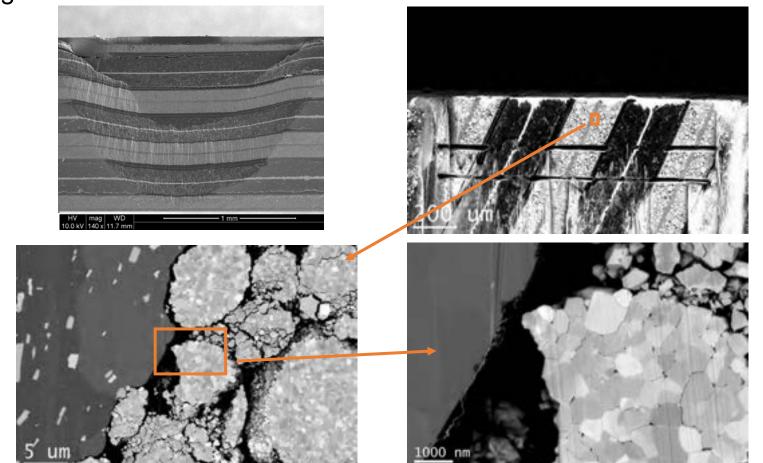
Ion Polishing and Plasma Focused Ion Beam

In order to study the active materials within the whole electrode, large cross sections are necessary. Now mm size cross section can be obtained within EAG battery lab by two methods:

- Broad beam ion polishing
- Plasma FIB

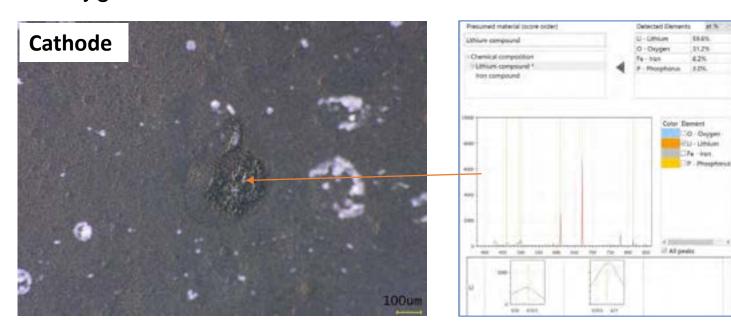
Both methods can maintain battery electrode structure integrity without damage.







carbon, oxygen could be detected with um resolution.

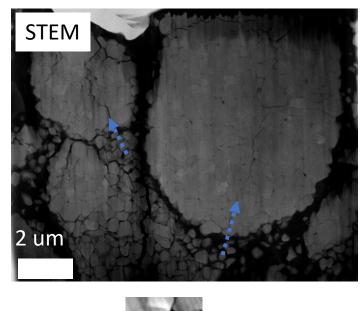


Transmission Electron Microscopy

• TEM has higher resolution than OM and SEM.

Surface

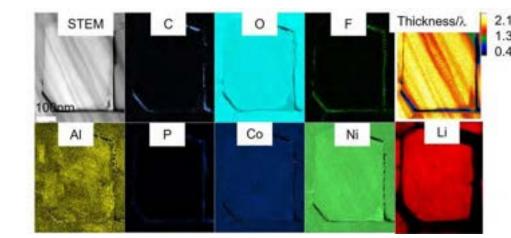
- Surface structure degradion could be investigated with atomic resolution AC-STEM.
- Lithium ion could also be observed with ABF-STEM image directly.



		\mathbf{c}
	300-	
	200-	
	150- 100-	
ABF-S	TEM ADF-STEM	

TEM and EDS/EELS

STEM/EDS/EELS provides 2D elemental maps

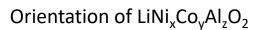


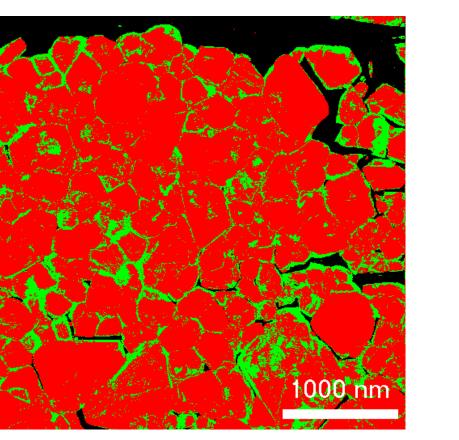
EELS helps understanding the chemical state of transition metal. As shown below, Mn changed into lower oxidization state at the surface, indicating the degradation.

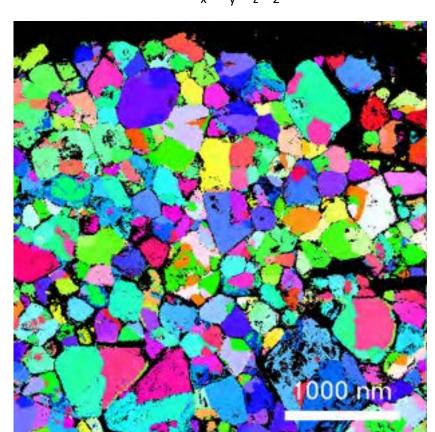
Precession Electron Diffraction

• TEM/PED provides phase distribution and grain orientation map at nm scale. As shown below, NiO was detected around cathode grains where near the edge or crack. Cathode grain has a homogeneous distribution based on their orientation maps.

Phase Map LiNi_xCo_vAl_zO₂ NiO







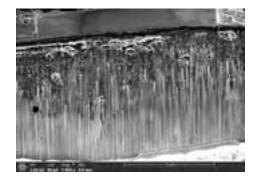


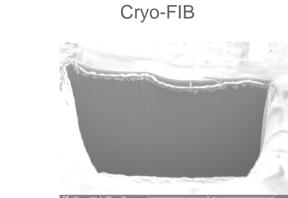
Cryo FIB and Cryo TEM/EELS

• RT FIB-creates significant artifact while Cryo FIB mitigates beam damage.

Room Temperature-FIB

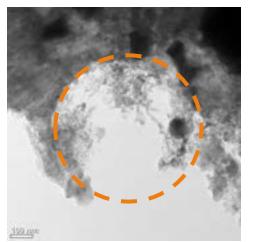
Core

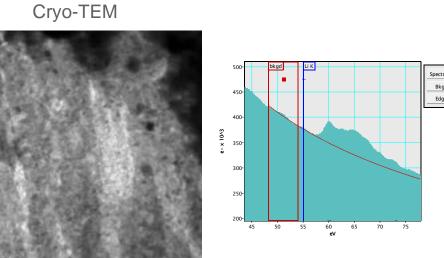




RT TEM on Li-containing materials is challenging due to beam damage. While structure was preserved at cryo temperature which makes elemental analysis possible by EELS.

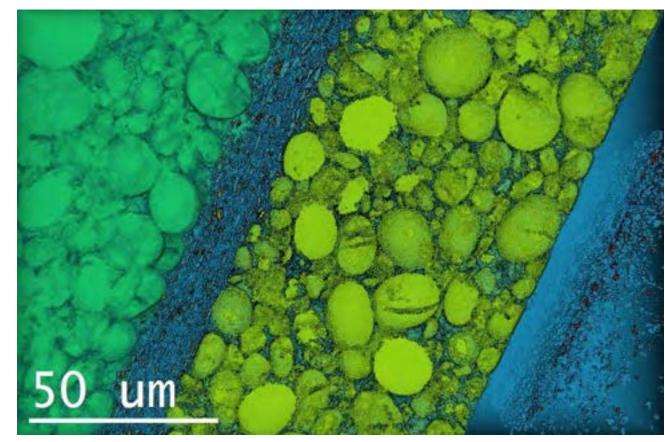
Room Temperature-TEM





FIB/SEM Tomography

- 3D Tomography based on FIB/SEM directly visualize the morphologies, pore structure, grain distribution. While Ga based FIB can only provide a small field of view, the new developed **PFIB** makes it possible to study materials with a larger FOV (shown below 150umx100umx100um data set).
- Elemental tomography is also possible with the aid of EDS while collecting FIB/SEM.



Atom Probe Tomography

• APT tomography reveals nm scale 3D structure and composition analysis includes light elements and trace elements (down to tens of ppm level) for cathodes like NCA, NCM, LFP and LCO as well as silicon-based anodes and oxide based solid state electrolytes. Shown below is a representative map for certain elements in the battery cathode.

