Evans Analytical Group

Battery Technology
materials characterization

Today's advanced batteries require a range of specialized analytical tools to better understand the electrochemical processes that occur during battery cycling. Evans Analytical Group (EAG) offers a wide-range of materials characterization services specifically for the battery industry to help with battery manufacturing, material R&D and failure analysis.

raw materials

During battery manufacture, an important factor affecting performance, and potentially safety, is the consistency in composition and impurity levels in the raw electrode materials. A number of different analytical techniques are available to address this. ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) is commonly used for analyzing the composition of electrodes, whereas ICP-MS (Inductively Coupled Plasma-Mass Spectrometry) is used for accurately determining lower level elemental impurities. GDMS (Glow Discharge Mass Spectrometry) allows full periodic table trace element analysis in a single measurement and is an ideal technique for monitoring the presence of unwanted impurities. IGA (Instrumental Gas Analysis) is the technique of choice when analyzing for gas forming elements such as H, C, N, O and S.

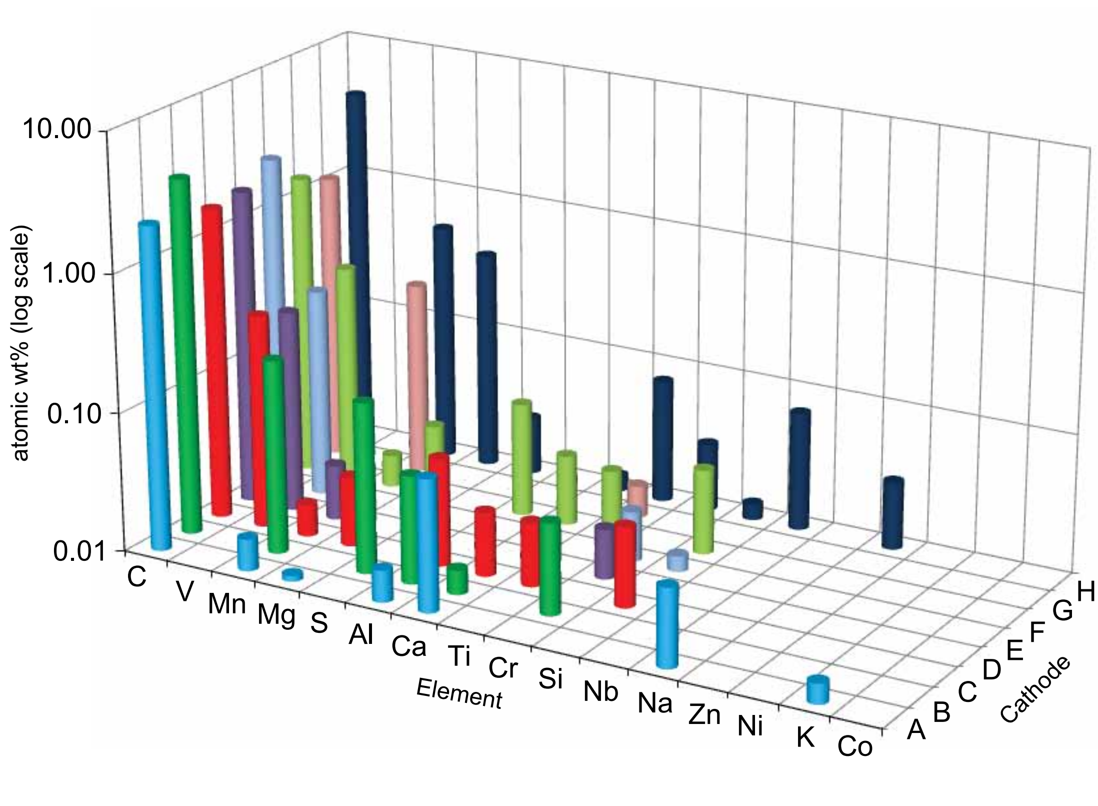
ICP-OES data from $LiNiCoAlO_2$ cathode samples from 3 different raw material suppliers showing variations in composition.

Elements	Supplier #1, wt%	Supplier #2, wt%	Supplier #3, wt%
Li	7.6	6.1	6.3
Al	1.2	1.3	1.5
Co	8.9	9.3	9.1
Ni	46.6	51.0	50.0

Atmospheric species measured using IGA from a $LiNiCoAlO_2$ cathode.

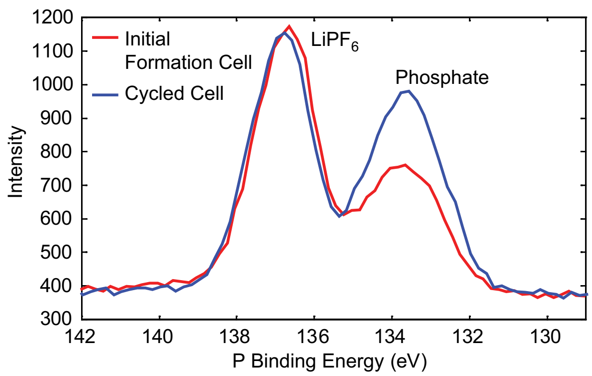
Elements	Composition wt %
O	30.9
C	0.3
N	0.2
S	<0.001

This plot shows GDMS data acquired from eight $LiFePO_4$ cathode samples from a range of suppliers. Only selected elements are shown here for clarity. The presence of a number of different unwanted impurities was repeatable over multiple samples from a given batch and provided valuable information to the battery manufacturer. One supplier's batch had unusually high amounts of impurities, in particular Mn and Mg, and the batteries manufactured from this batch resulted in unacceptable cycle life.



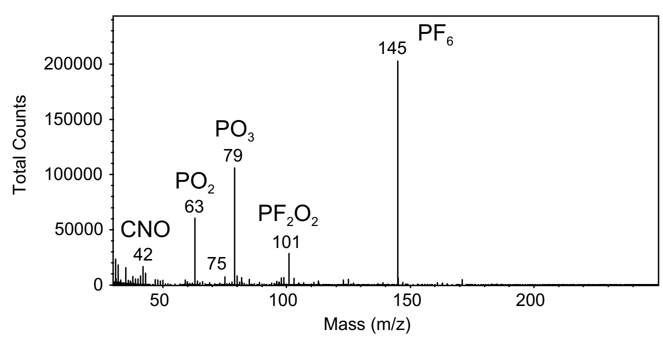
surface chemistry & composition

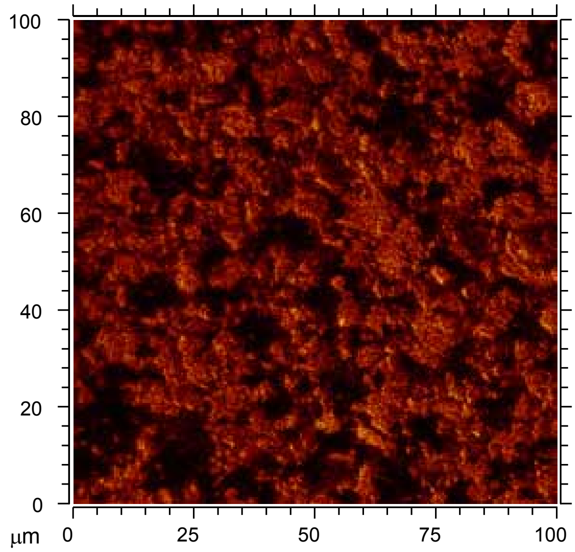
With increasing demands for higher battery performance and improved safety, a better understanding of the factors affecting performance, cycle life and possible failure mechanisms is essential. The chemical state of the battery components such as the cathode, anode, separator, electrolyte, contact layers and additives, at various stages of cycling, provides vital information about the electrochemical processes that occur during battery use. EAG offers elemental and molecular analysis using a number of different analytical tools to address this need. Moisture sensitive materials are extracted from cells in a moisture controlled environment and can be transferred to instruments for analysis without exposure to air or moisture.



XPS (X-Ray Photoelectron Spectroscopy) is a commonly used technique for investigating the surface chemistry of electrodes. Here the phosphorus chemistry on a graphitic anode before and after battery cycling is compared. After cycling there is a clear increase in phosphate bonding relative to $LiPF_6$.

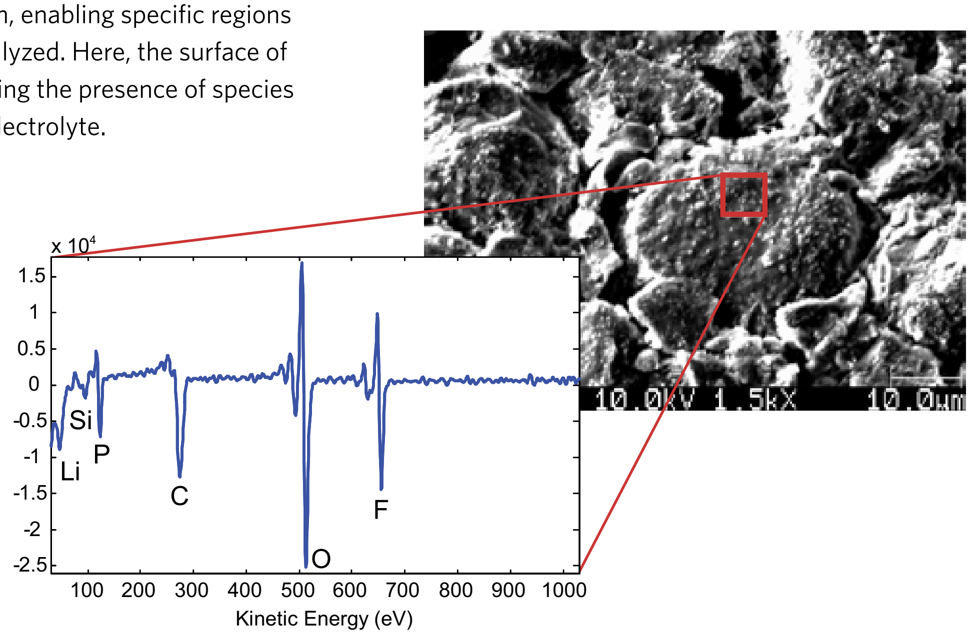
TOF-SIMS (Time-Of-Flight Secondary Ion Mass Spectrometry) has the unique ability of analyzing both organic and inorganic components and allows a full investigation of decomposition products; the presence of impurities; or any other surface changes during cycling. This spectrum was acquired from a cycled battery with a $LiCoO_2$ cathode using $LiPF_6$ electrolyte. A number of molecular species of interest are identified on the surface of the cathode that did not originate there.



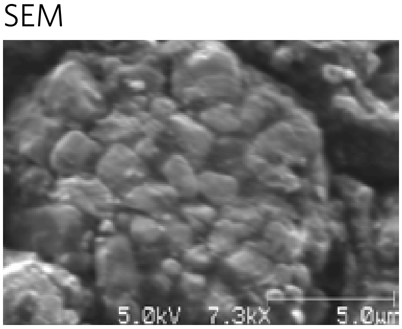


This is a secondary ion image recorded from the surface of a lithium titanate ($Li_4Ti_5O_{12}$) anode. By imaging different species on the sample, TOF-SIMS allows a better understanding of the lateral chemical distribution of species of interest at various stages of cycling. Sputtering with an ion beam also allows species of interest to be depth profiled, which is particularly useful in studying the chemical composition of SEI layers as a function of depth.

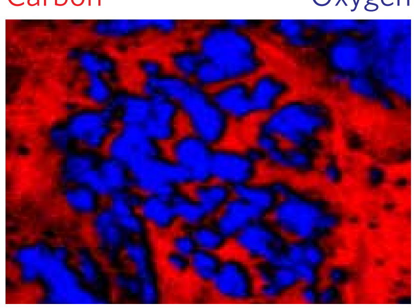
Auger Electron Spectroscopy (AES) allows elemental analysis using an electron beam focused down to ~10-20 nm, enabling specific regions of interest on electrode particles to be analyzed. Here, the surface of a cycled graphitic anode is analyzed showing the presence of species characteristic of both the anode and the electrolyte.



SEM



Carbon Oxygen

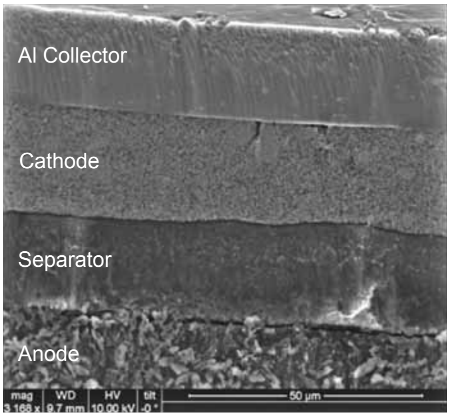


Using the elemental mapping capability of Auger, a cycled $LiNiMnCoO_2$ cathode is analyzed showing a high carbon signal at the extreme surface, from electrolyte residue surrounding the cathode particles.

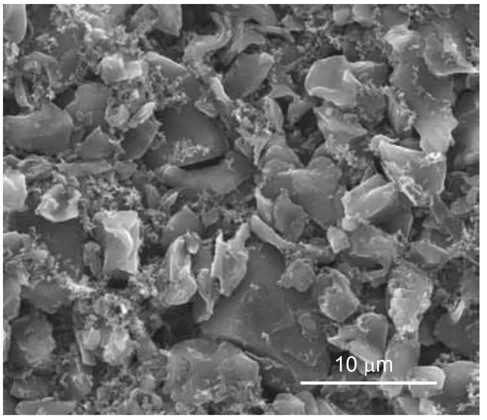
morphology & uniformity

Electron microscopy techniques such as SEM (Scanning Electron Microscopy) and TEM (Transmission Electron Microscopy) are essential techniques to investigate morphology, particle size, particle coatings, mixing efficiency and defects. These techniques often employ elemental mapping capabilities such as EELS (Electron Energy Loss Spectroscopy) and EDS (Energy Dispersive X-Ray Spectroscopy) which provide valuable information about elemental composition and location/distribution.

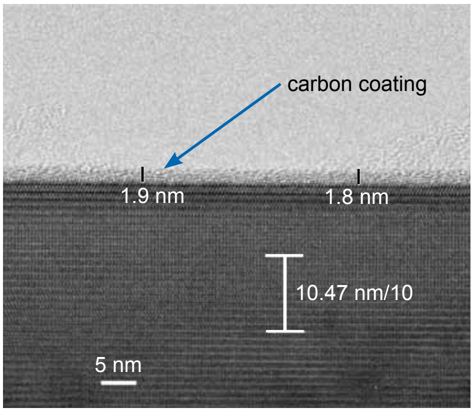
This is an example of an SEM image of a cycled lithium polymer battery prepared by ion milling. Film thickness variation and imperfections at the interfaces can be easily inspected. Each layer can be further inspected at greater magnification.



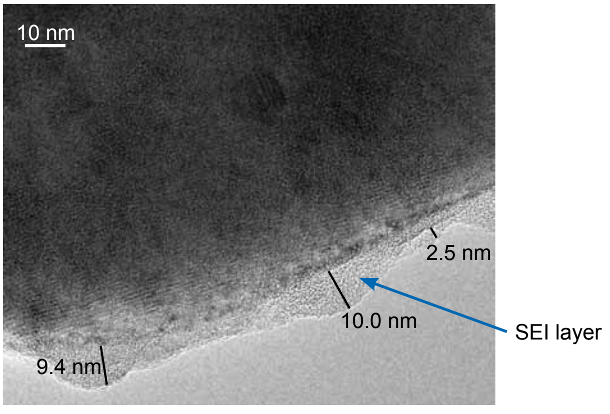
This is an SEM image of a freshly prepared carbon based anode film. Variation in particle size and mixing efficiency can easily be investigated. Software can be used for further image processing to aid in this task.



This TEM image of a freshly prepared cathode shows an amorphous carbon coating on a $LiFePO_4$ particle. The thickness can be accurately measured, allowing thickness variation to be monitored. The elemental composition of the coating can be confirmed by EELS.



This is a TEM image of a cycled $LiFePO_4$ particle showing the formation of an SEI (solid-electrolyte interface) layer on the surface. Determining the composition of SEI layers via EELS or EDS can provide important insights into possible SEI growth mechanisms.



composition

impurity surveys

phase identification

thermal stability

elemental analysis

chemical analysis

electron microscopy

Battery Technology
materials characterization

Technique	Typical Battery Applications
ICP-OES	Electrode composition
SEM	Morphology, mixing and film uniformity, particle size
GDMS	Raw material quality control
Raman / FTIR	Impurity detection, carbon phase
TEM	Particle size, particle coating analysis, crystallinity phase
EELS / EDS	Elemental analysis/mapping
XPS	Chemical state, composition
TOF-SIMS	Organic composition, SEI characterization
Auger	Elemental mapping, particle depth profiling
TGA / DTA / DSC	Thermal properties
SIMS	Elemental depth profiling of metals
GC-MS	Characterization of volatile organic species
XRD	Crystallographic phase
IGA	Levels of atmospheric species
ICP-MS	Electrode impurities
RTX	Alignment of internal components
XRF	Elemental composition

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