Analytical Methods for Characterizing Battery Materials

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Sanjay Patel

Evans Analytical Group

Materials Characterization Microelectronics Test and Engineering Environmental Fate, Chemistry and Ecotoxicology CHEMIR

- Founded in 1978
- Leader in advanced surface analysis and materials characterization
- 700 employees
- Locations worldwide

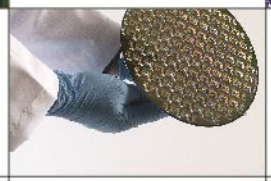
Analytical Techniques in Other Industries

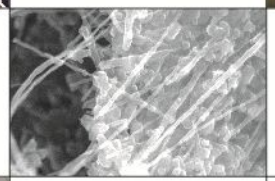
AgroChemical

Photovoltaic/ Solar

Coatings

Integrated Circuits





Nano Technology

Stents and other Medical Devices

Sputtering Targets

Electronic Materials

Ecotoxicology

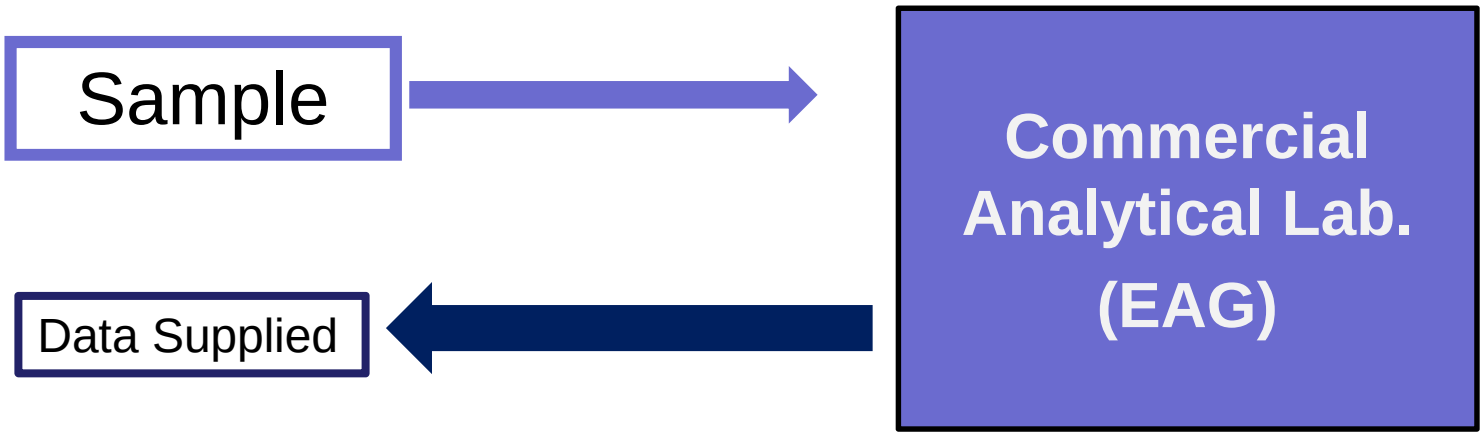
Turbines, Aircraft Engines/Aerospace

Drug Delivery Systems

Semiconductor Equipment

Metallurgy, DPA & RGA

Commercial Analytical Lab?



- Fully Confidential – no IP or patent related issues
- Fast turnaround – typically 3-5 days
- Access to best available techniques and procedures

Analytical Considerations

Analytical consideration for R&D and manufacturing differ

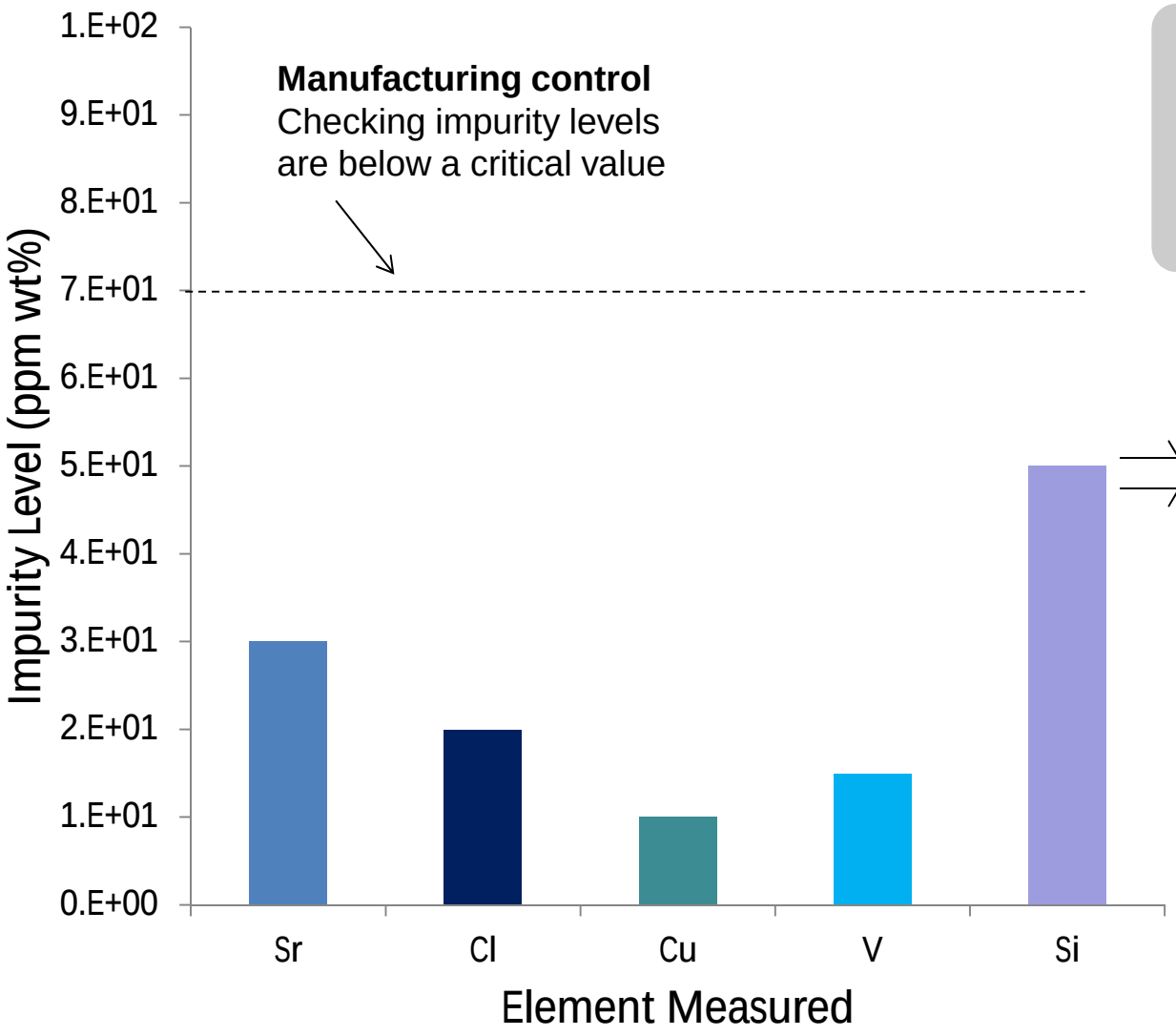
Research and Development

- Accuracy
- Lowest detection Limit
- Resolution
- Physical / Chemical Information

Manufacturing / Production Control

- Repeatability
- Cost
- Speed – turnaround time
- Sampling

Choosing the Analytical Method

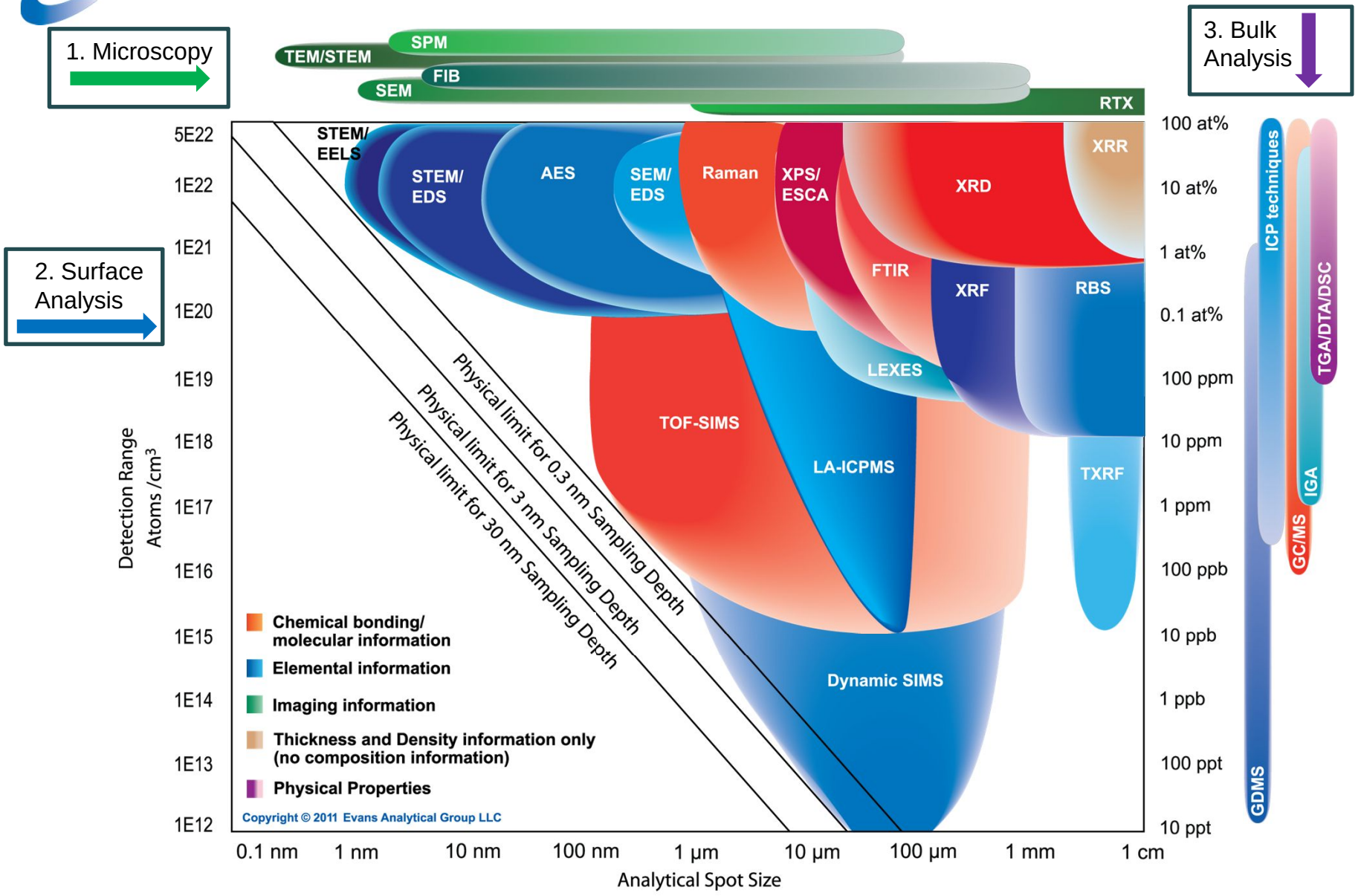


Manufacturing control
Checking impurity levels are below a critical value

Optimum Technique Selection - Costs Reduced

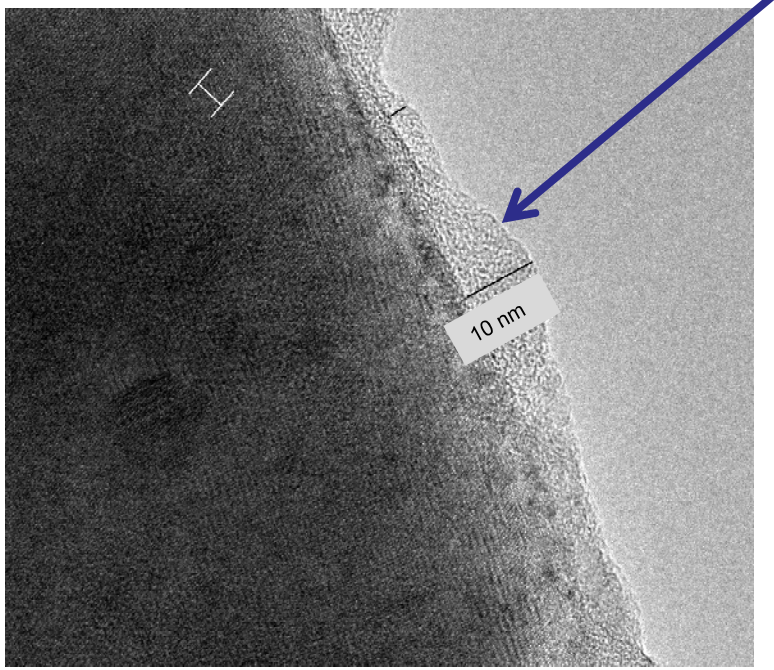
R&D
Measurement accuracy important

Bubble Chart for Analytical Techniques



Study of SEI

TEM, cycled cathode particle



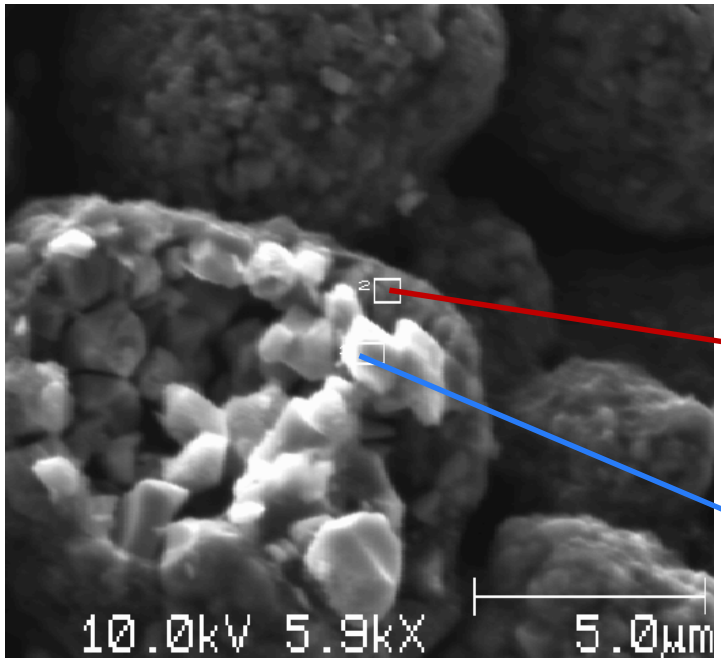
Analysis of SEI is essential for understanding electrochemical mechanism which results in loss of cell performance/cycle life.

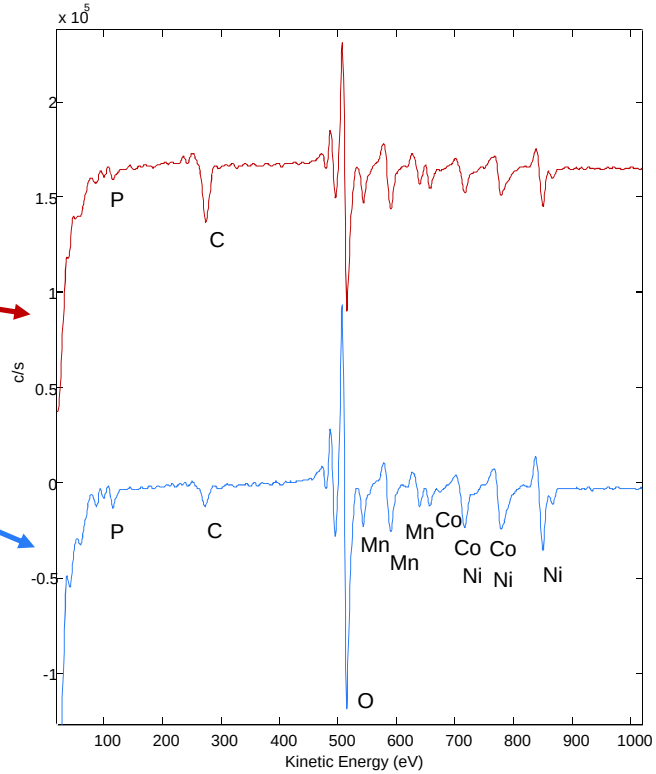
Characterization of SEI Requires:

- Small spot size to analyze individual particles
- Ability to measure elemental and molecular components
- Shallow information depth (few nms)
- Depth profiling ability

Small Area Elemental Analysis

Auger used for imaging LiNiCoMnO cathode particles ~1 micron size particles

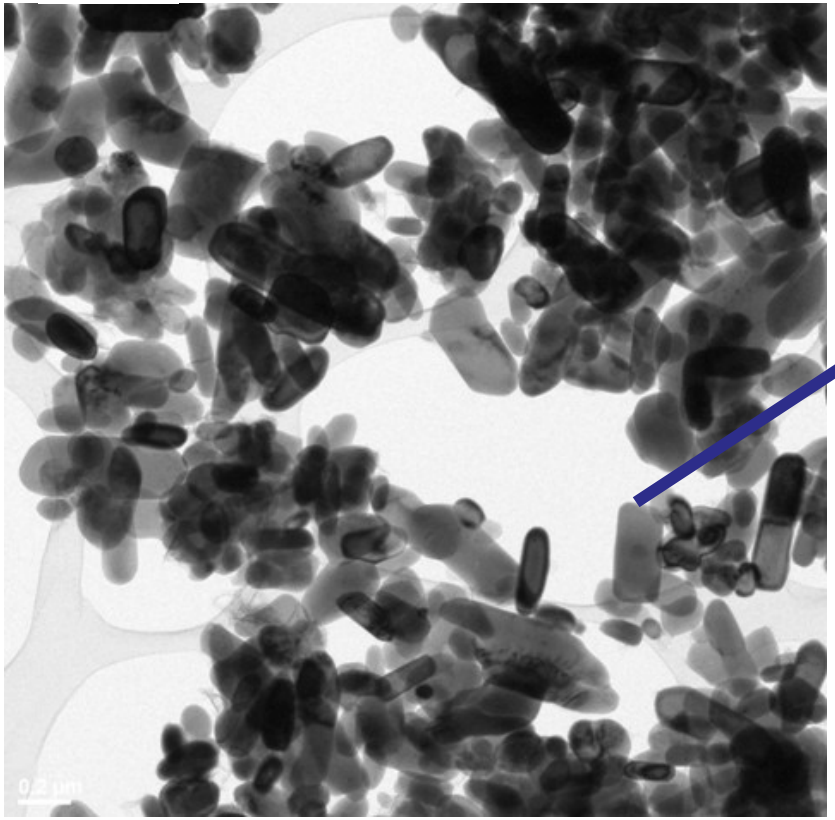


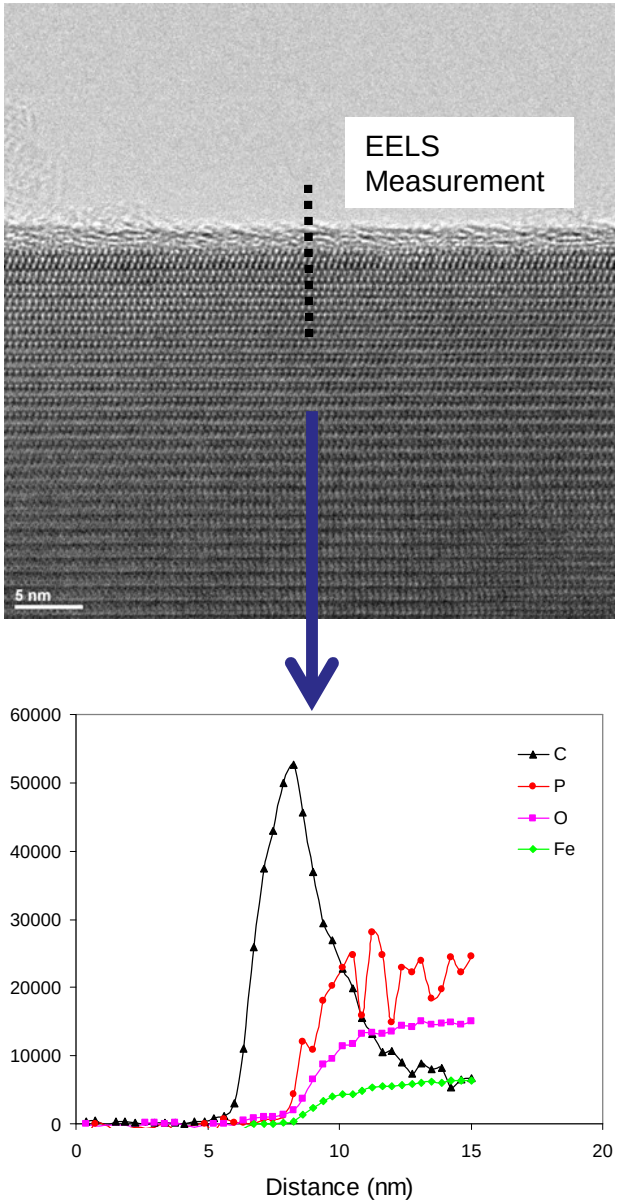


- Typical spot size ~ 50 nm
- Li difficult due to poor sensitivity
- Elemental analysis
- No molecular information

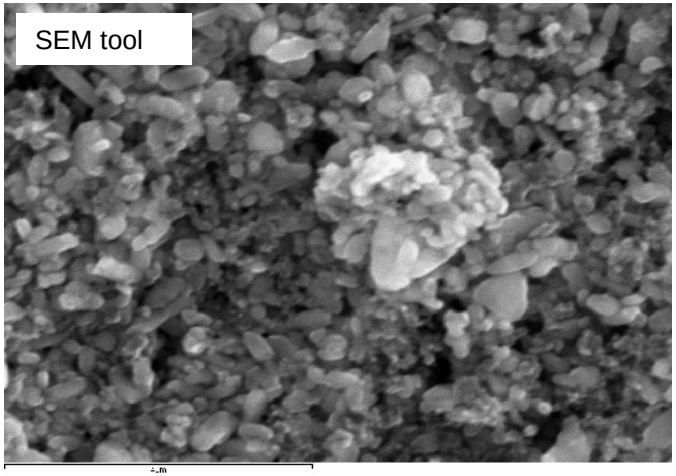
TEM / EELS

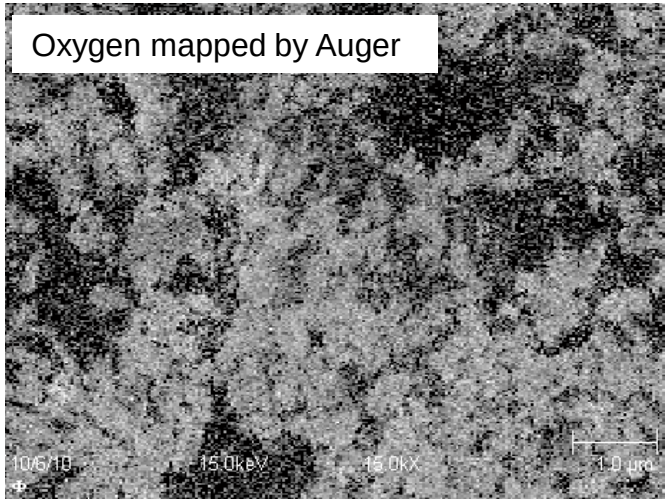
TEM

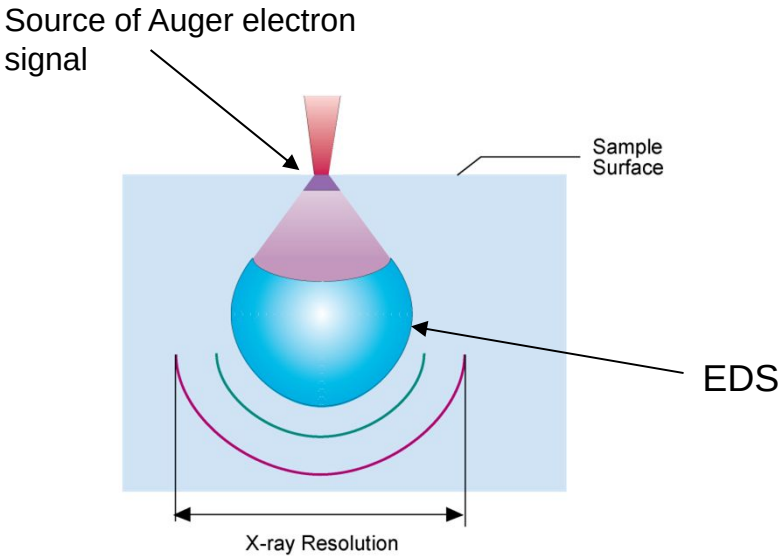


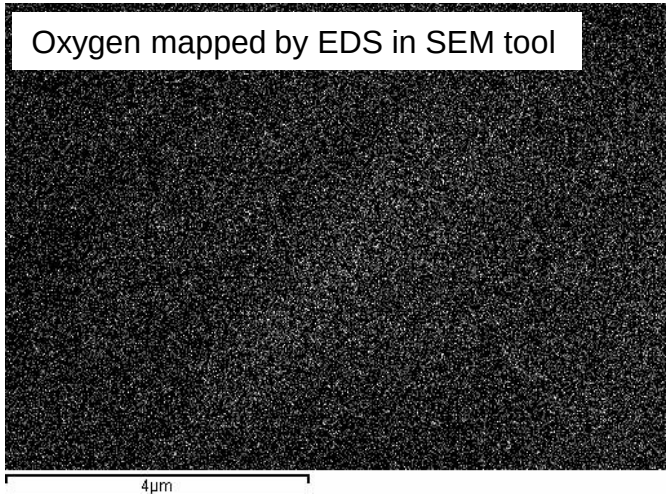


SEM / EDS







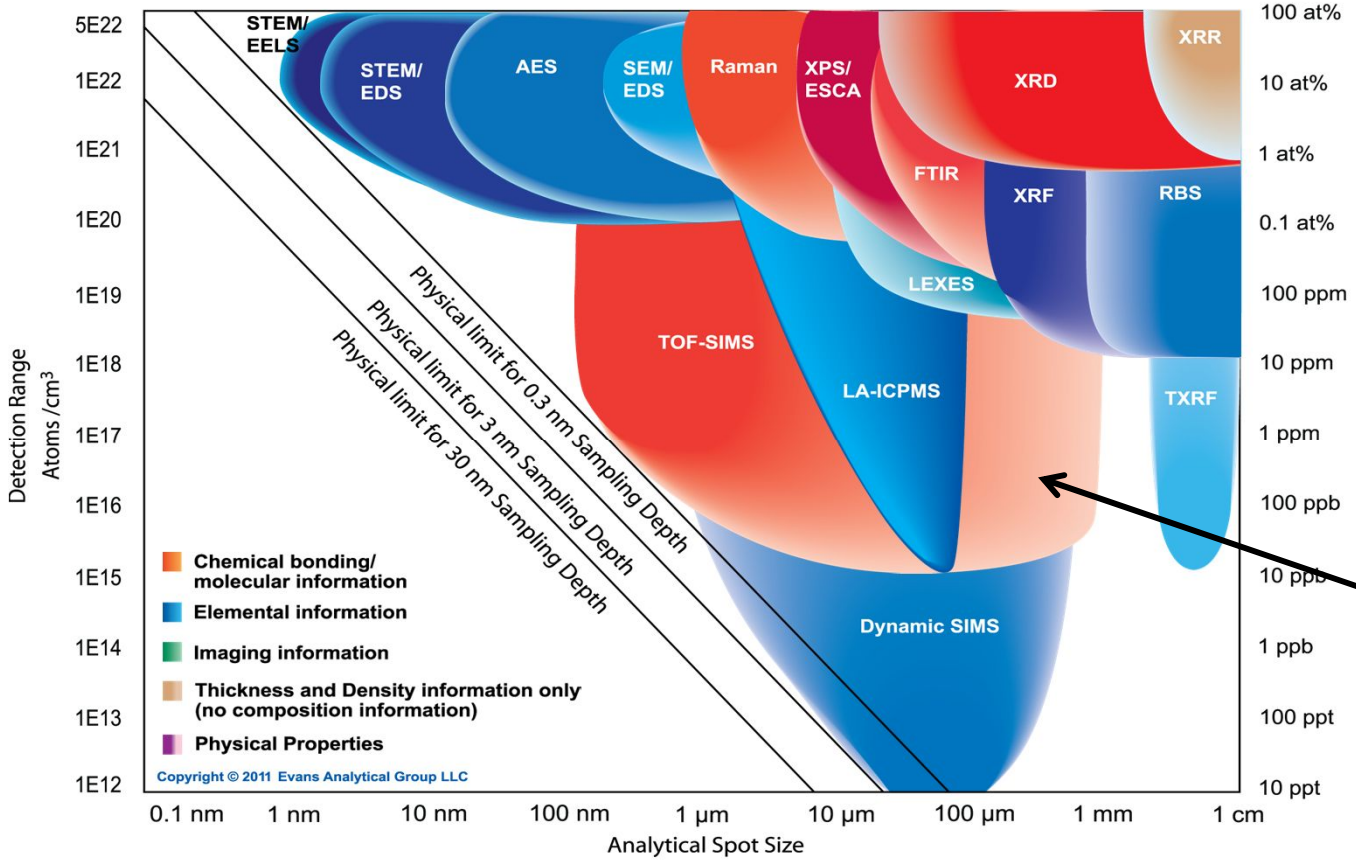


Bubble Chart for Analytical Techniques

Auger – elemental analysis of small areas

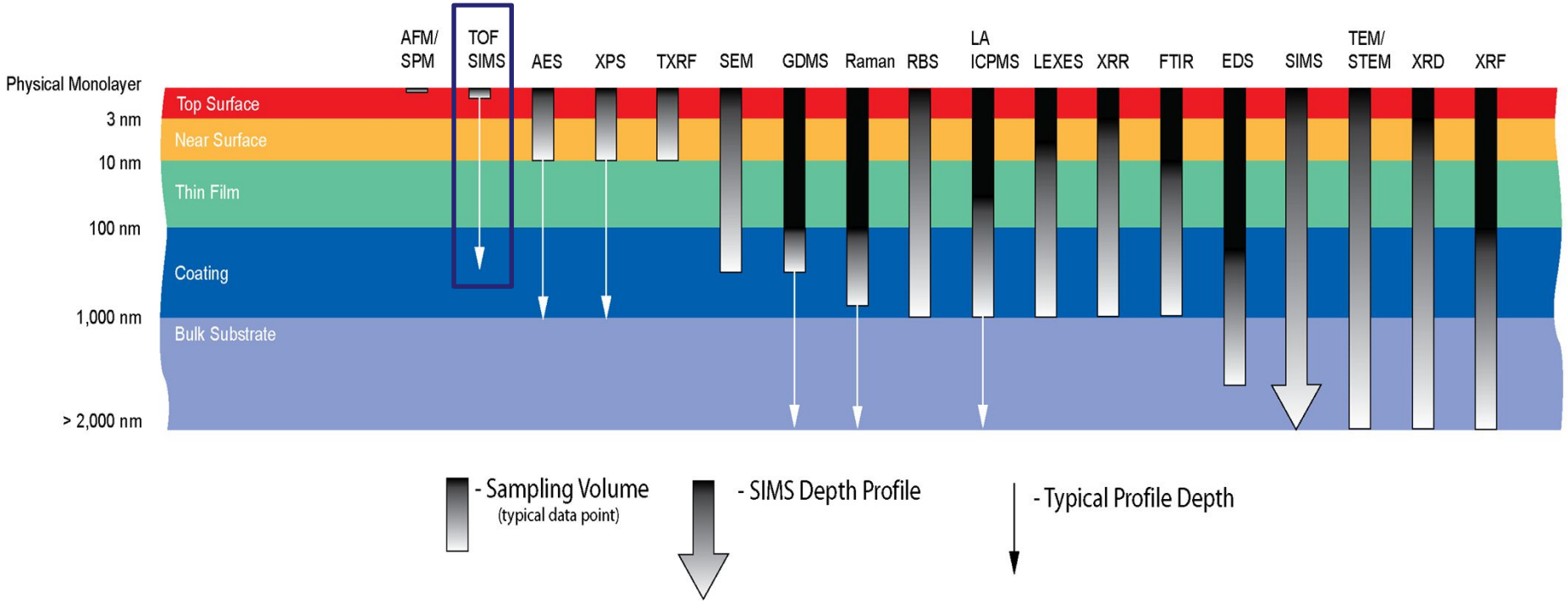
EELS/EDS – elemental analysis during microscopy

XPS – Chemical/molecular information

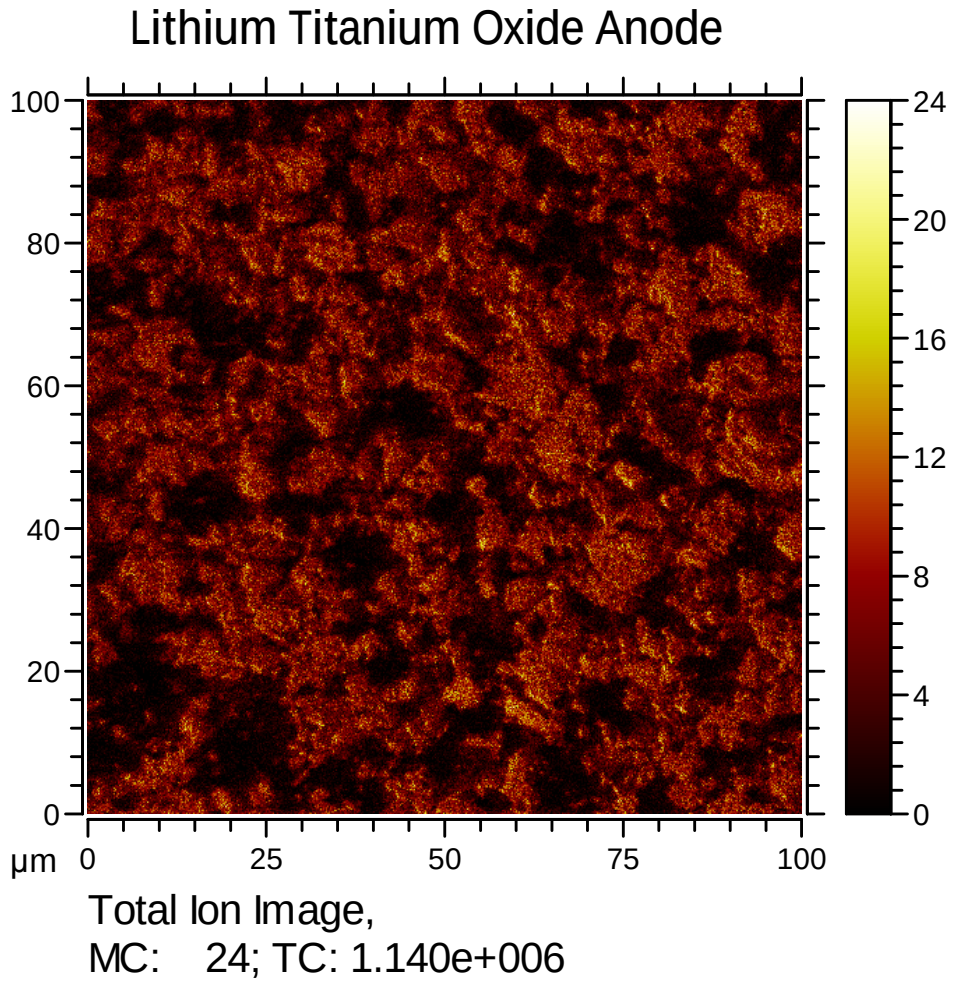


TOF-SIMS
Small area analysis
Molecular information
Elemental information
Depth profiling capability

Depth of Analysis / Information Depth



TOF-SIMS Imaging Capability



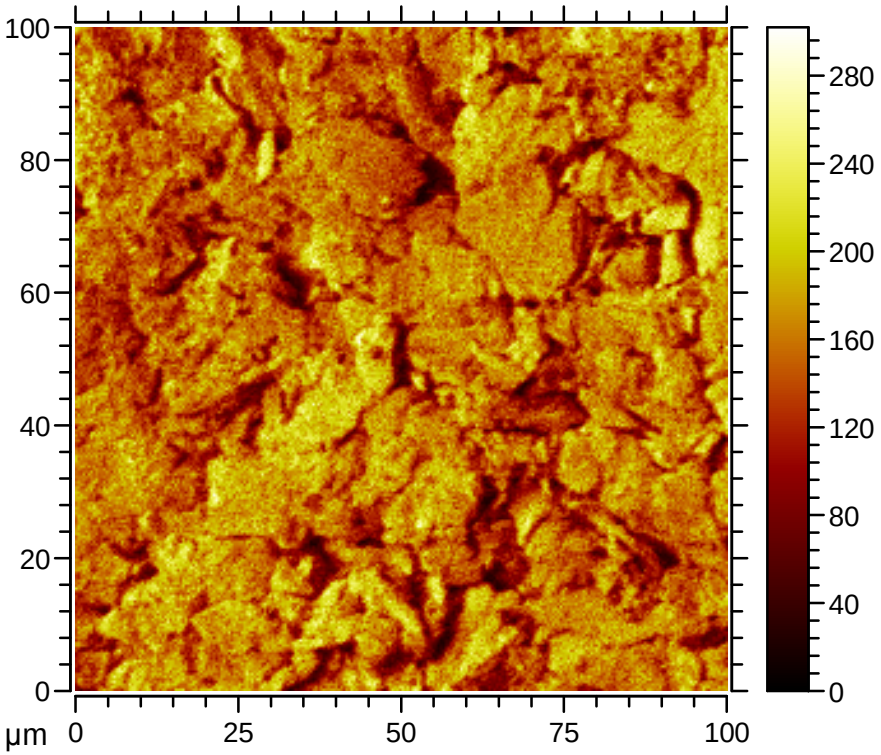
1. Ion Image – can mass select

2. Images recorded as a function of depth

3. Entire mass spectrum recorded per pixel – allowing any mass to be imaged or depth profile generated at any region

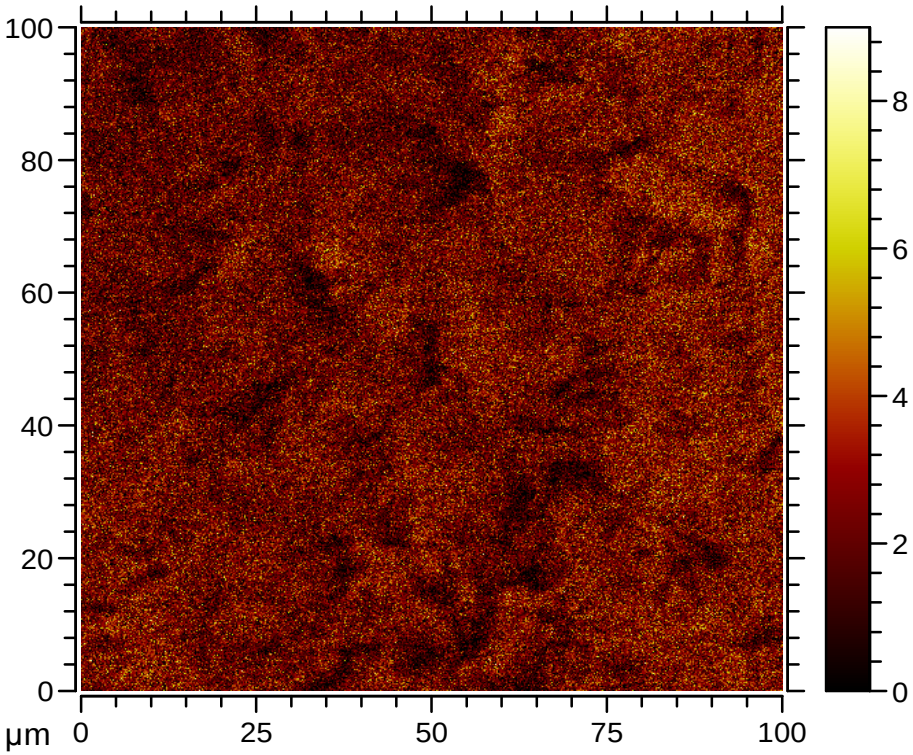
TOF-SIMS Mass Selection

Secondary Ion Image
Total Counts



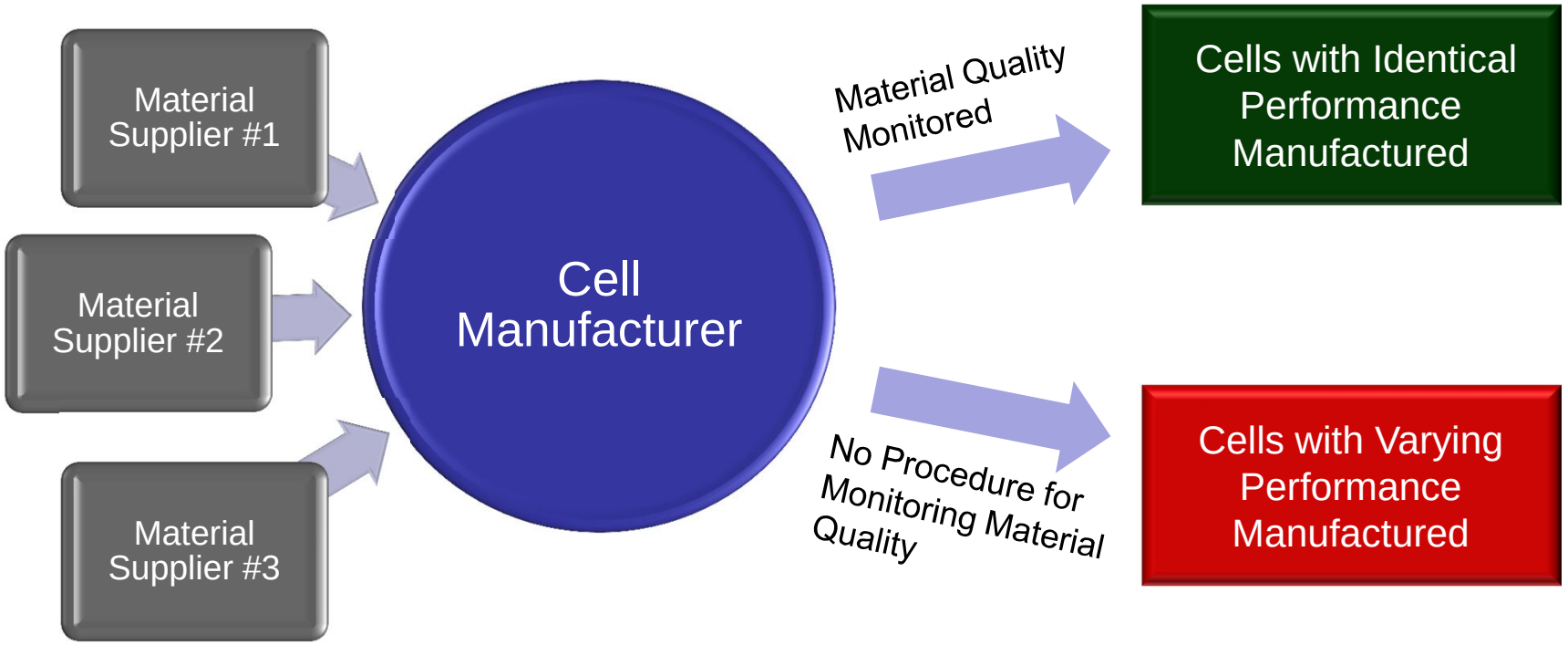
Total Ion Image, Sample A
MC: 302; TC: 9.952e+006

Secondary Ion Image
Lithium



Li+ ion image, Sample A
MC: 9; TC: 5.721e+005

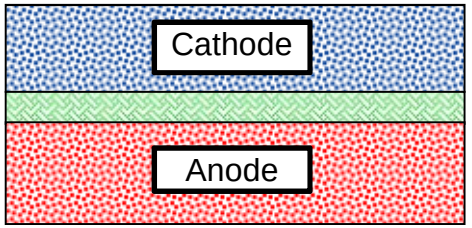
Raw Material Control



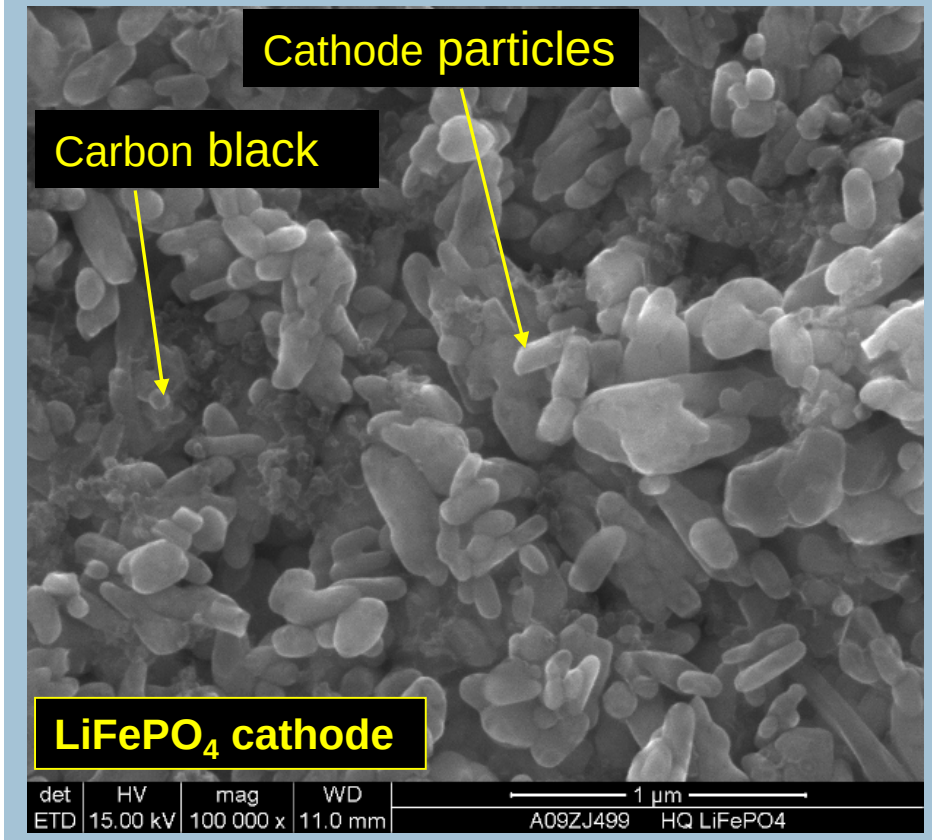
Variation in material grade from one supplier to another or even batch to batch (from the same supplier), should be monitored to ensure identical performance of manufactured cells.

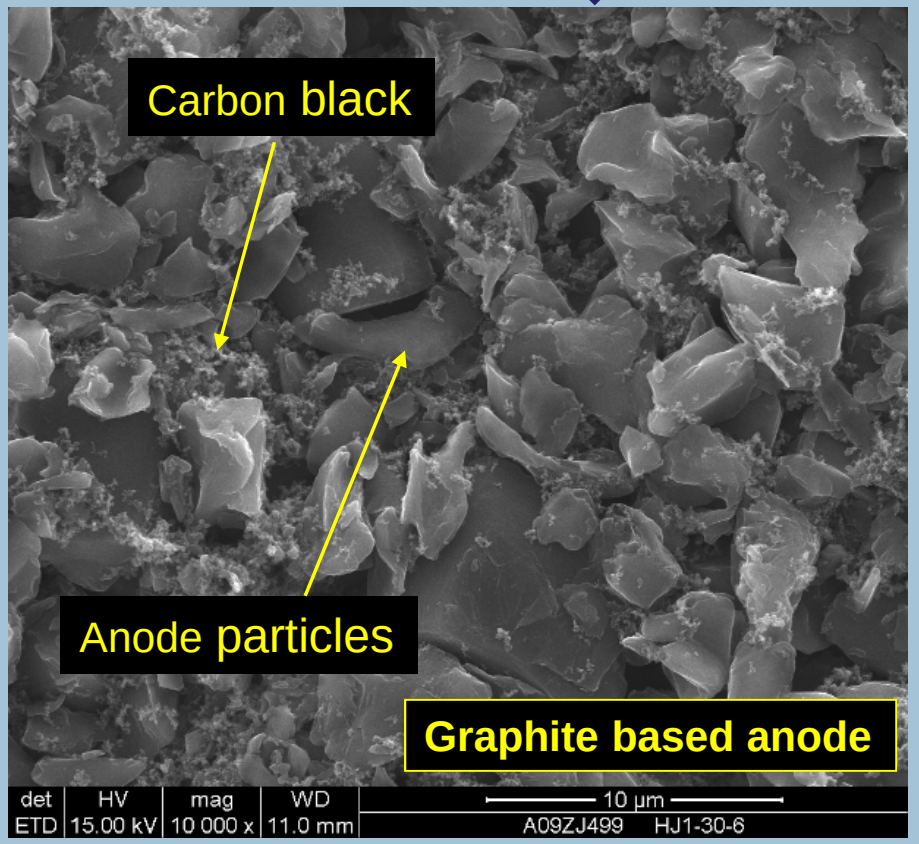
Understanding Particle Size and Distribution

Efficiency and consistency of the slurry mixing process is evaluated by SEM by checking particle distribution and agglomeration

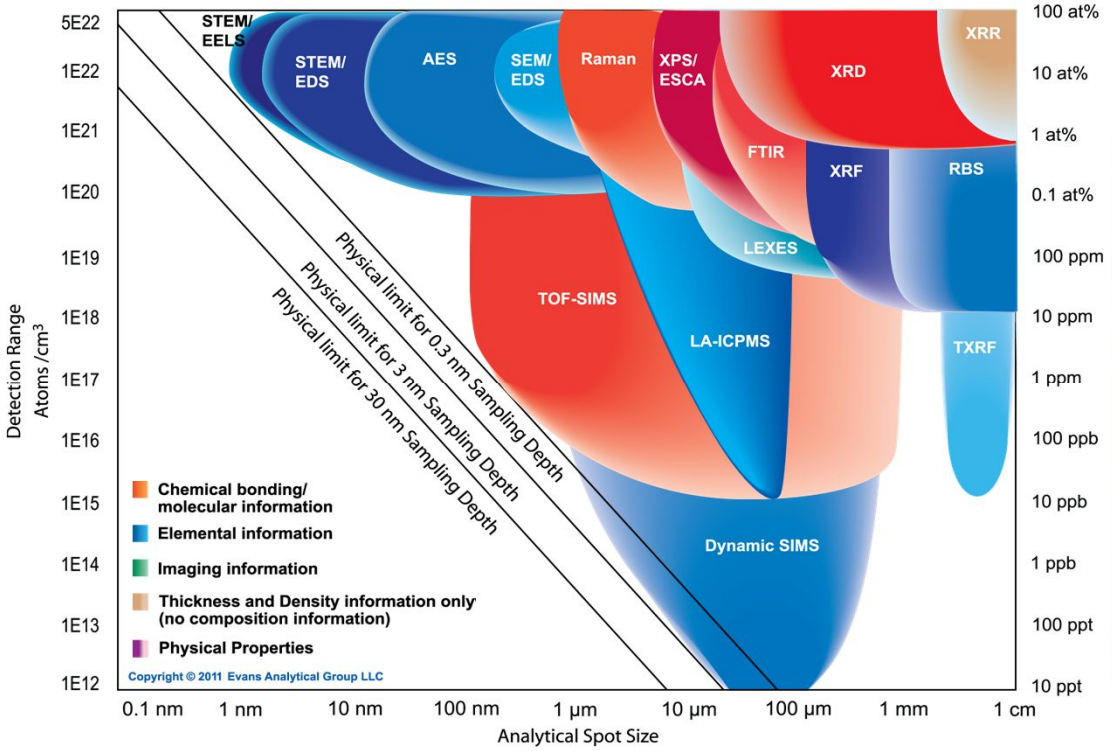


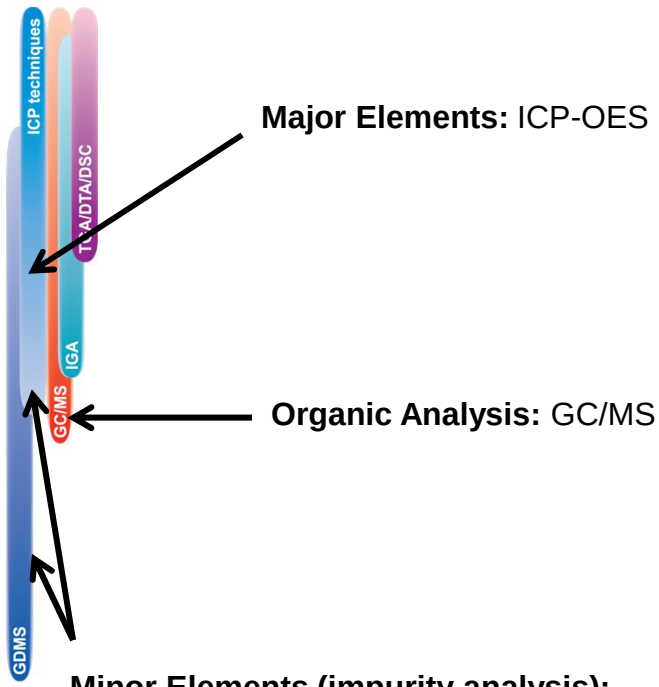
~300nm size





Manufacturing Control of Electrode Materials





Major Elements: ICP-OES

Organic Analysis: GC/MS

Minor Elements (impurity analysis):
ICP-MS or GDMS

-GDMS had advantage of measuring all elements in period table rather than a select number of elements as in the case for ICP-MS.
-ICP-MS provide better accuracy over GDMS.

Impurity Analysis of $LiFePO_4$

Study

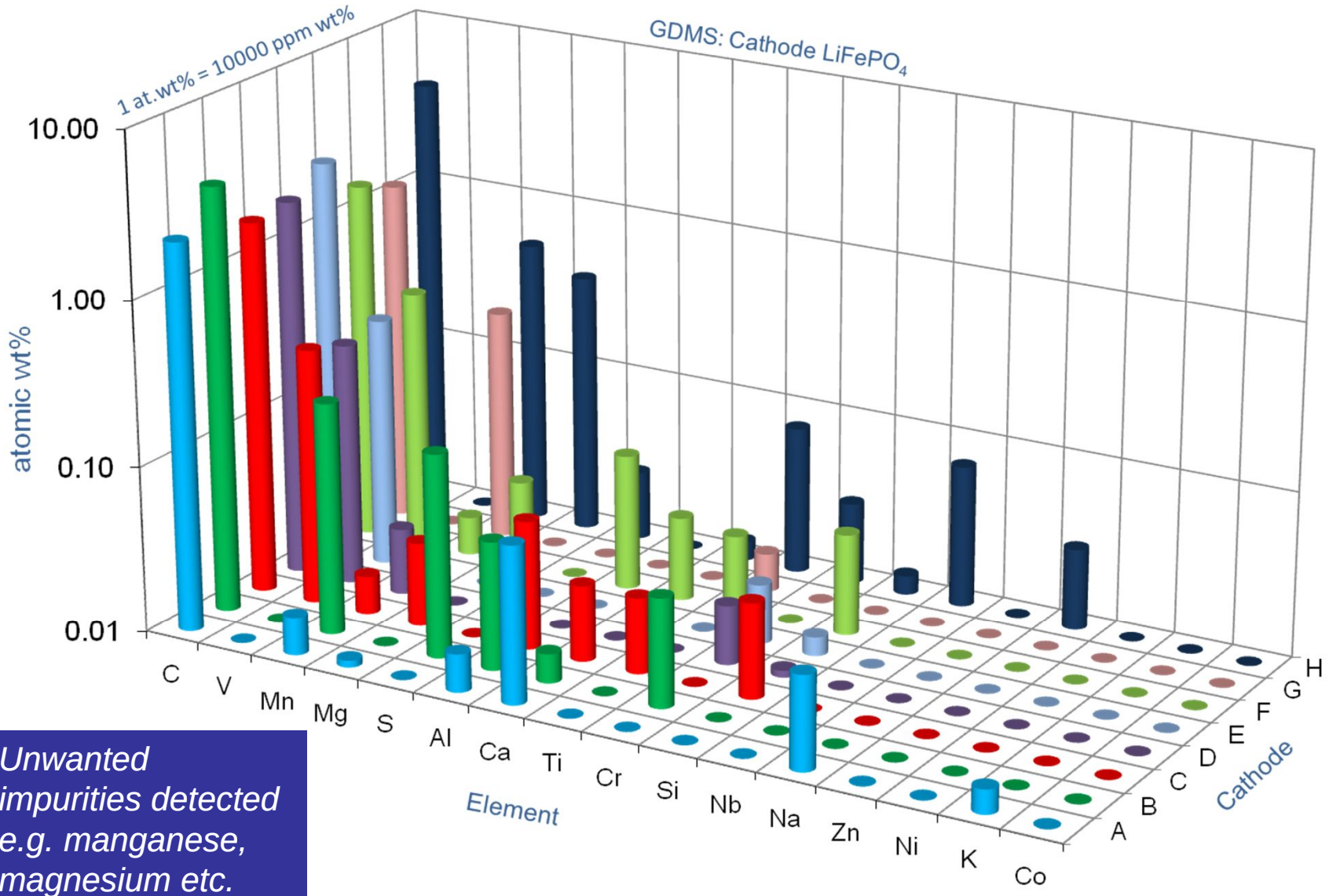
- Eight $LiFePO_4$ batches ready for cell manufacture
- Measure impurities

Samples A and B: expected to be similar

Samples C, D, E and F: expected to be similar and to have higher levels of vanadium

Samples G and H: from two different overseas suppliers

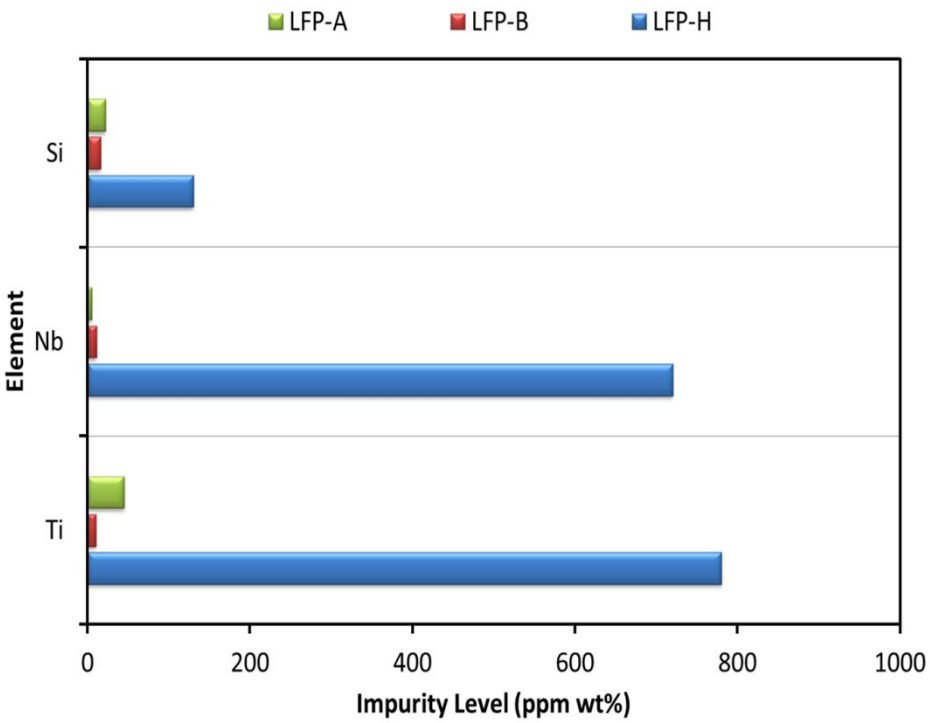
Elements >0.01 at. Wt%

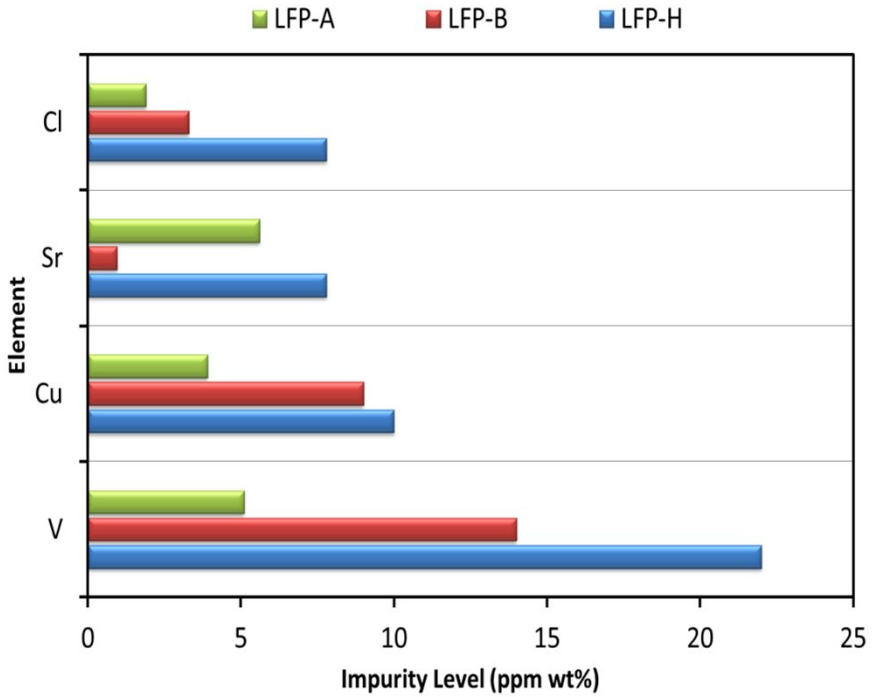


Unwanted impurities detected e.g. manganese, magnesium etc.

Comparison of GDMS Data

Sample LFP-H has significantly higher levels of Si, Nb and Ti compared to LFP-A and LFP-B.





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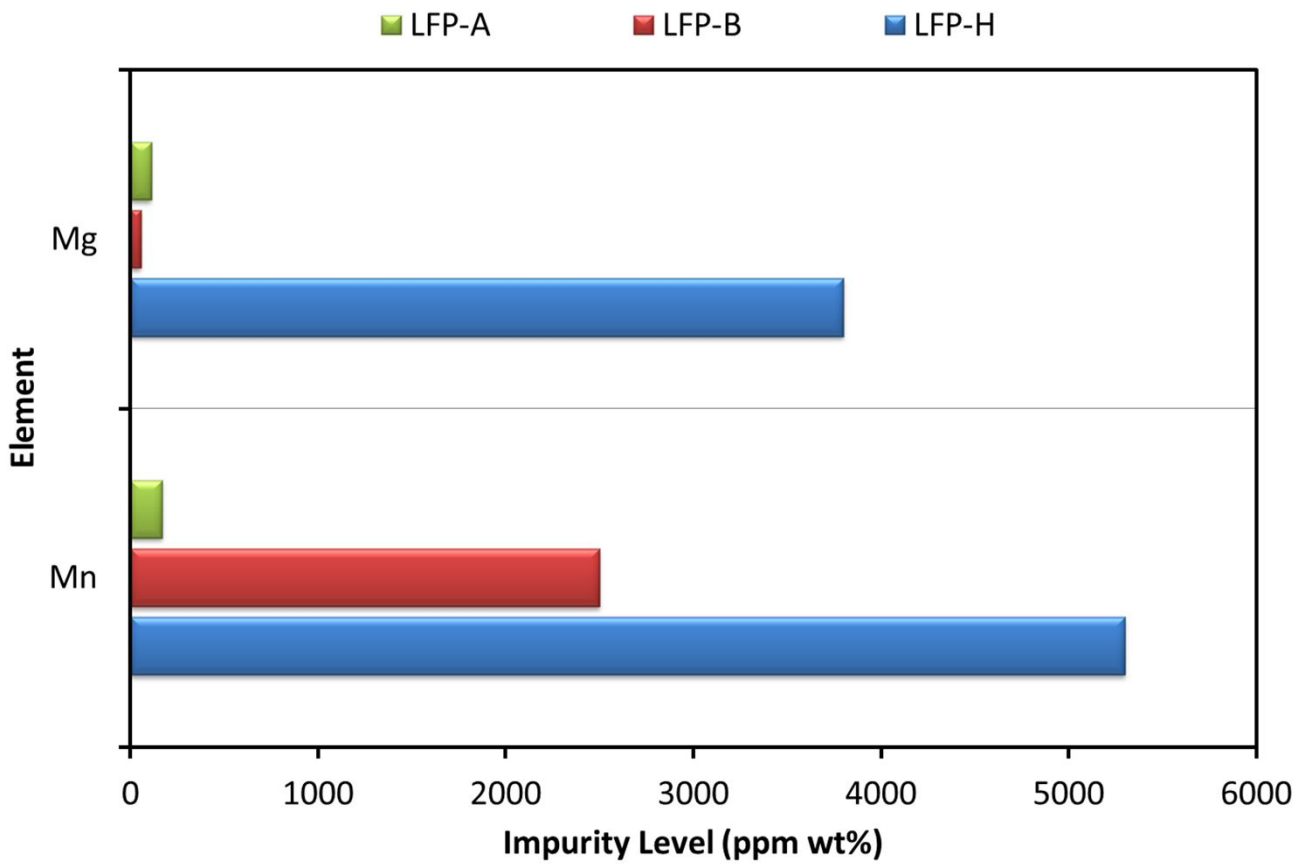
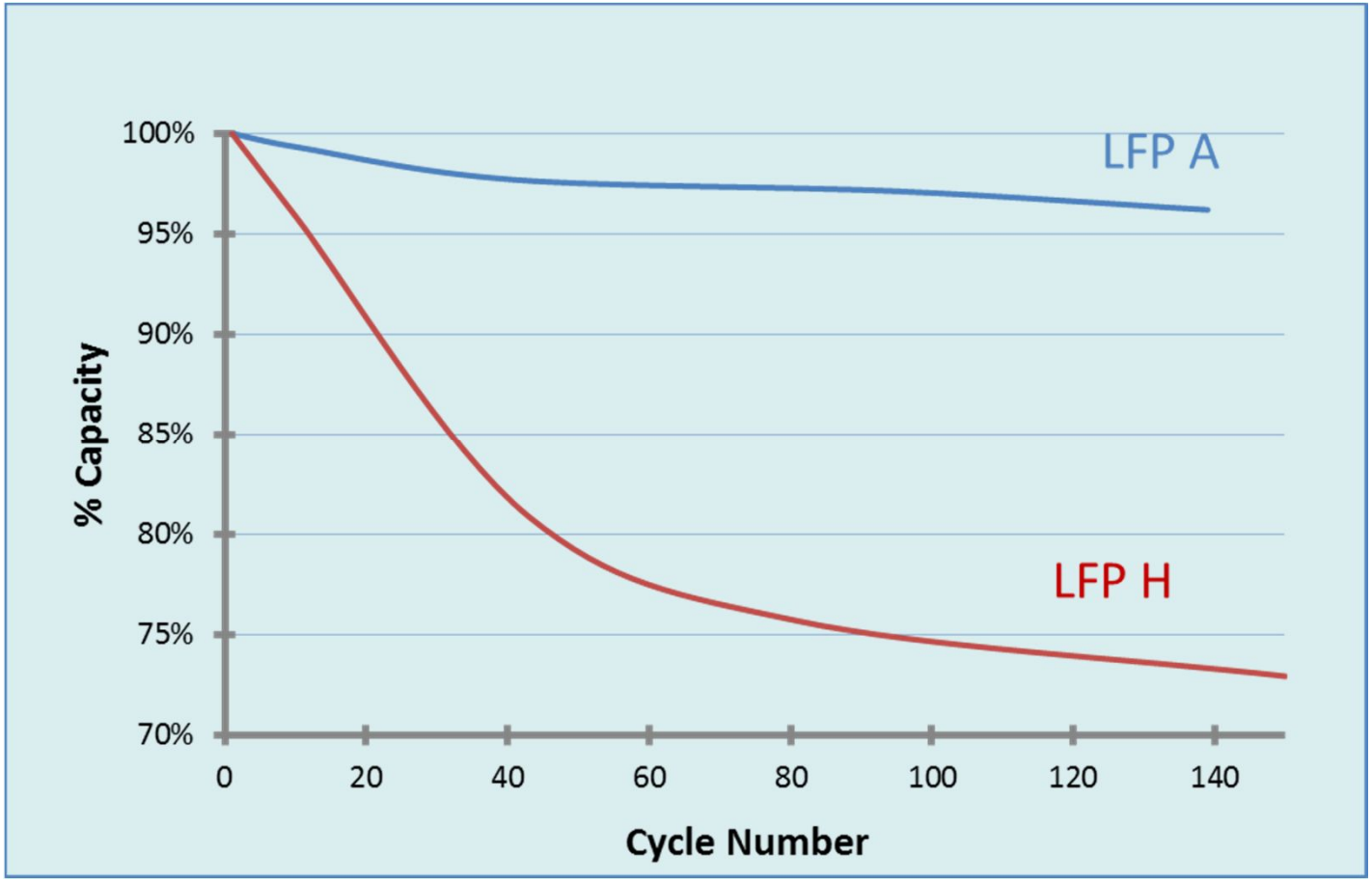


This chart shows higher levels of Mn and Mg in LFP-H compared to LFP-A and LFP-B.

Cell Cycling Data – Performance Variation

Two cells with very different characteristics – made from similar $LiFePO_4$ (according to supplier)



Summary

- Careful choice of analytical technique is essential for improving product development and good manufacturing control.
- Fast “turn-around” with optimized analytical method results in significant cost savings.
- With continued advances in battery materials, the analytical method requires changes/modification based on the material and information required.

Thank You!

Contact:

Sanjay Patel

480-239-0602

spatel@eag.com

www.eag.com