Faraday Institution FSP 8-Month Review

Postdoc-Focused Session

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WP: Degradation WP3
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THE FARADAY INSTITUTION

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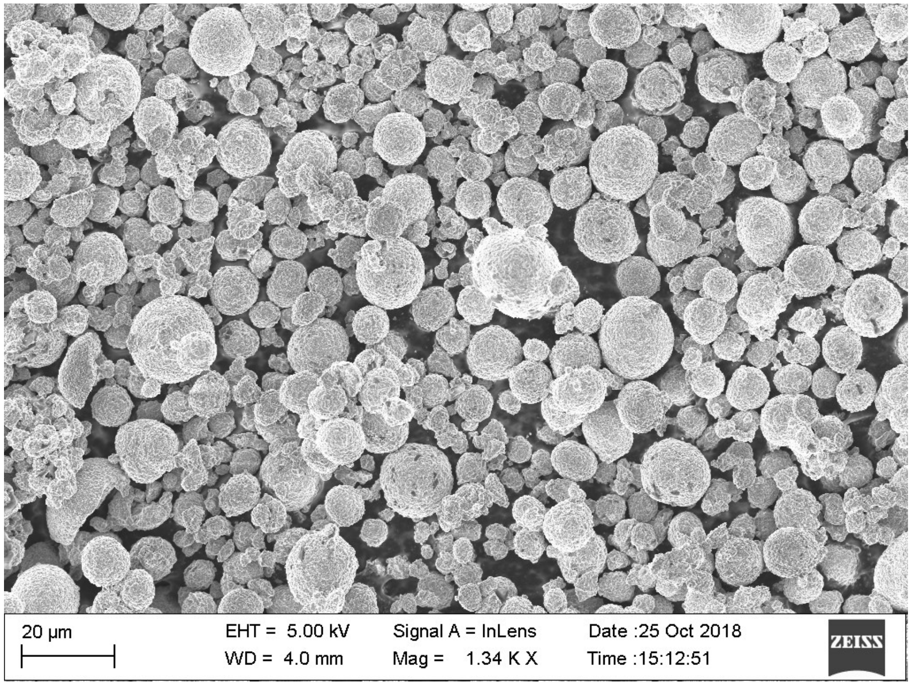
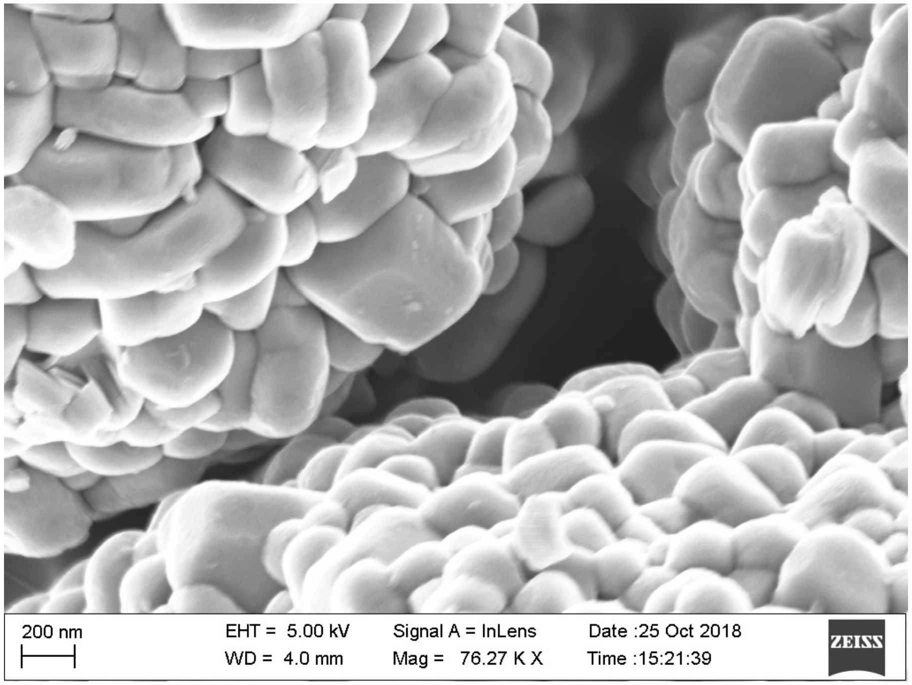
Current Research and Future Plans

- Microstructural Characterisation ☑
- Chemical Analysis on Pristine Materials – Surface Analysis ☑
- Cell Assembly and Electrochemical Cycling – *on going*
- Post mortem Analysis – Surface and Bulk Analysis – *future plan*
- In situ analysis – *future plan*

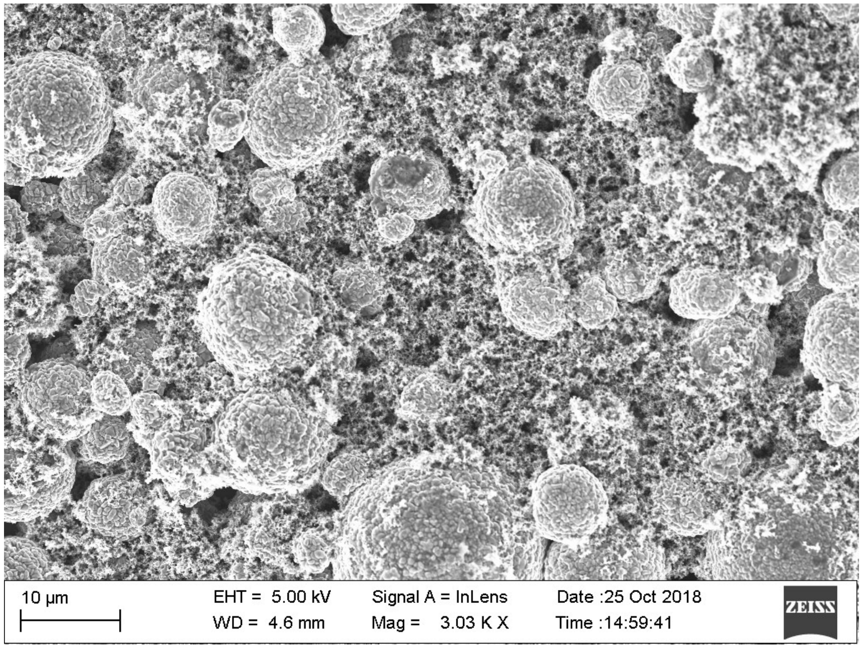
Microstructural Characterisation
NCM 811 Pristine Powders

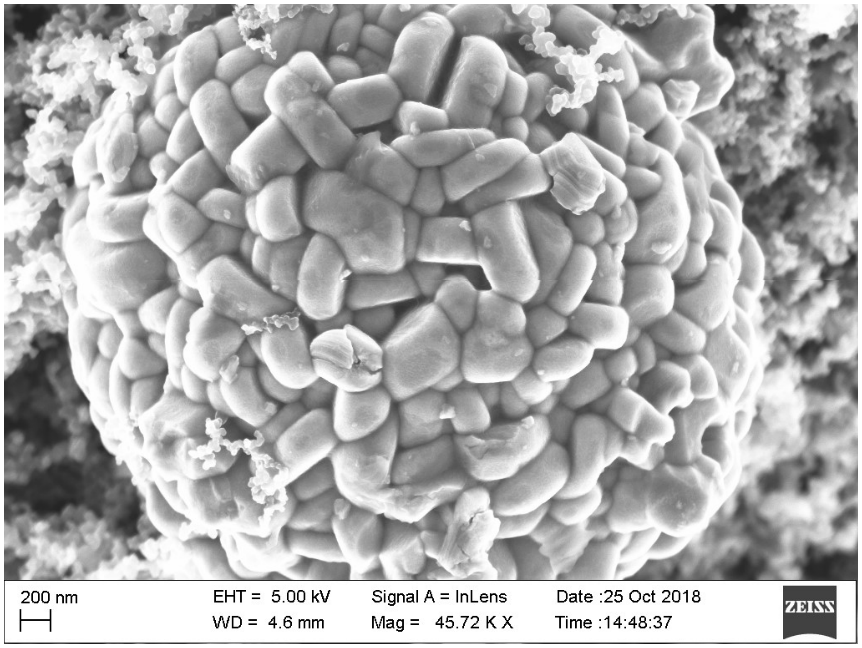




Courtesy of Mohammed Koronfel

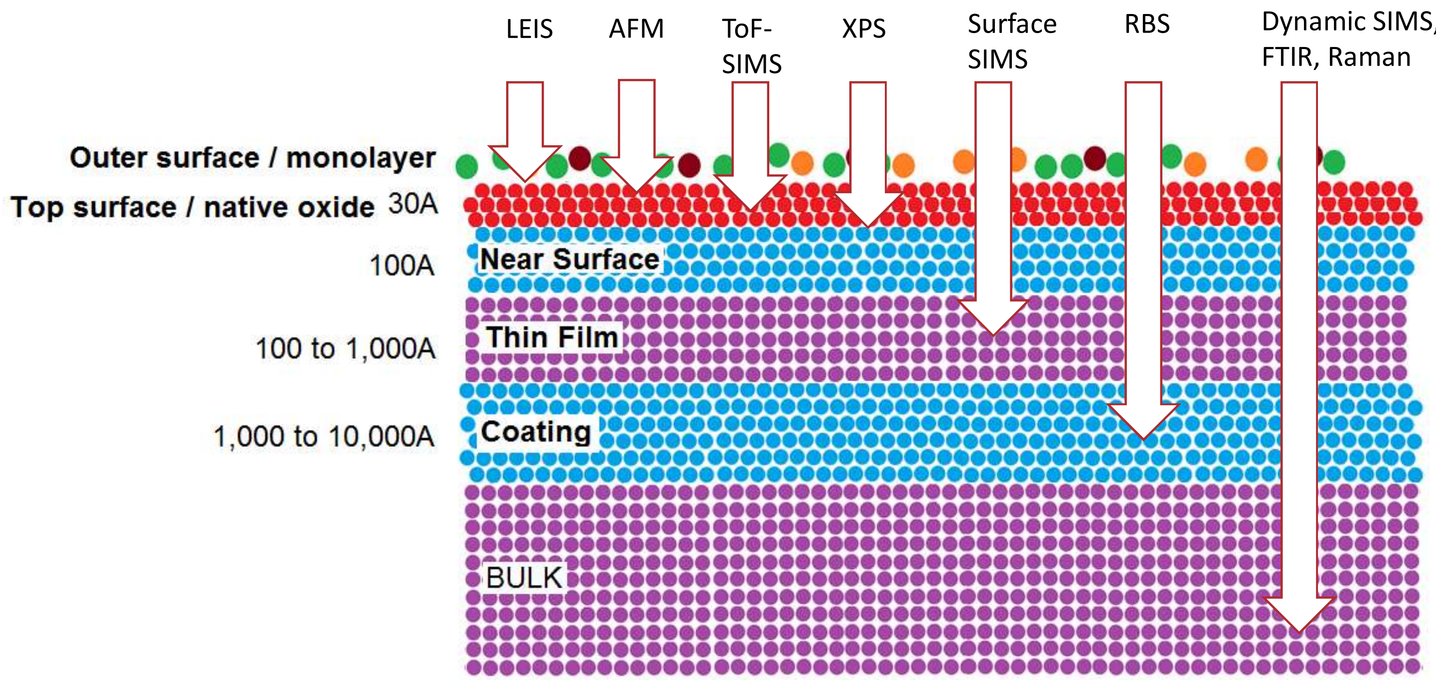
Microstructural Characterisation NCM 811 Pristine Electrodes





Courtesy of Mohammed Koronfel

Surface Analysis Characterisation



Courtesy of Sarah Fearn

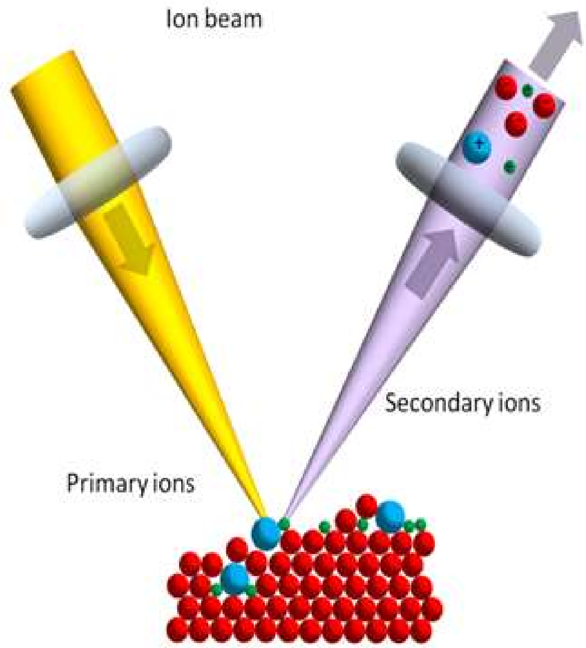
Surface Analysis Characterisation – ToF SIMS

1 **- Ion beam bombardment**

An energetic ion beam is rastered over sample surface.

3 **– Collection and analysis of emitted secondary ions**

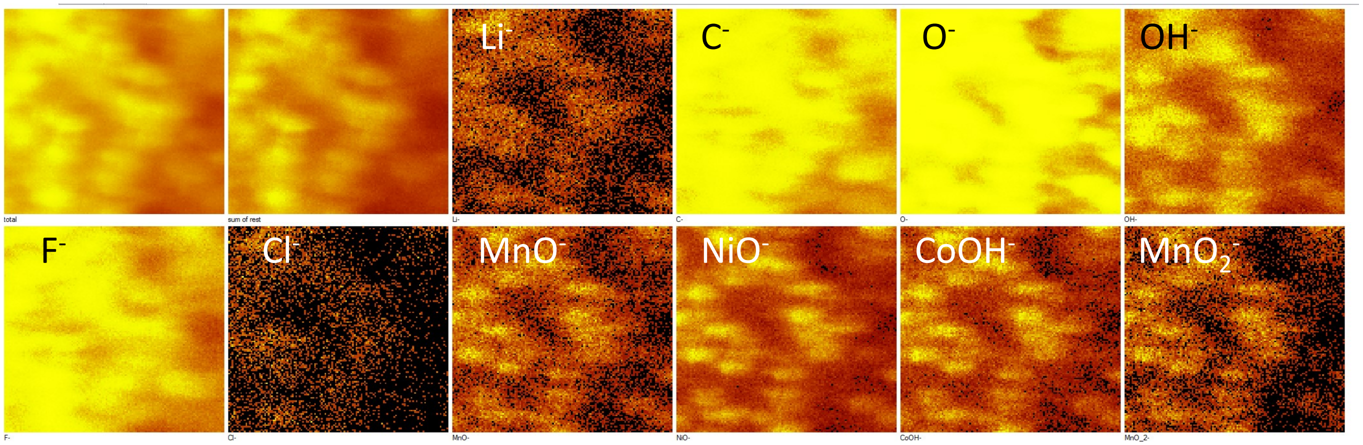
The desired secondary ions are extracted into mass analyser



2 – **Sputtering and ionisation of target material**

Courtesy of Sarah Fearn

ToF-SIMS on pristine NMC electrodes

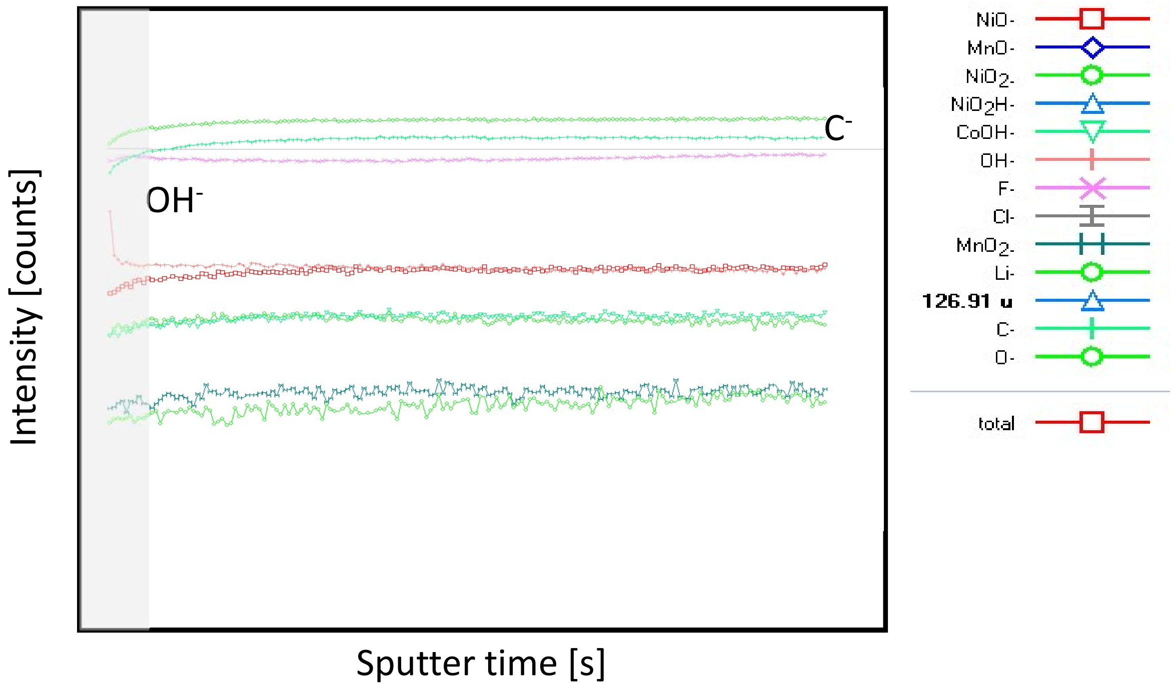


150 x 150 μm maps

- Ni, Mn and Co distribution highly inhomogeneous
- OH- appears to be present on the electrode surface.
- TOF-SIMS can not resolve elemental distribution within individual particles

No vacuum suitcase used! Sample exposed to air for few minutes

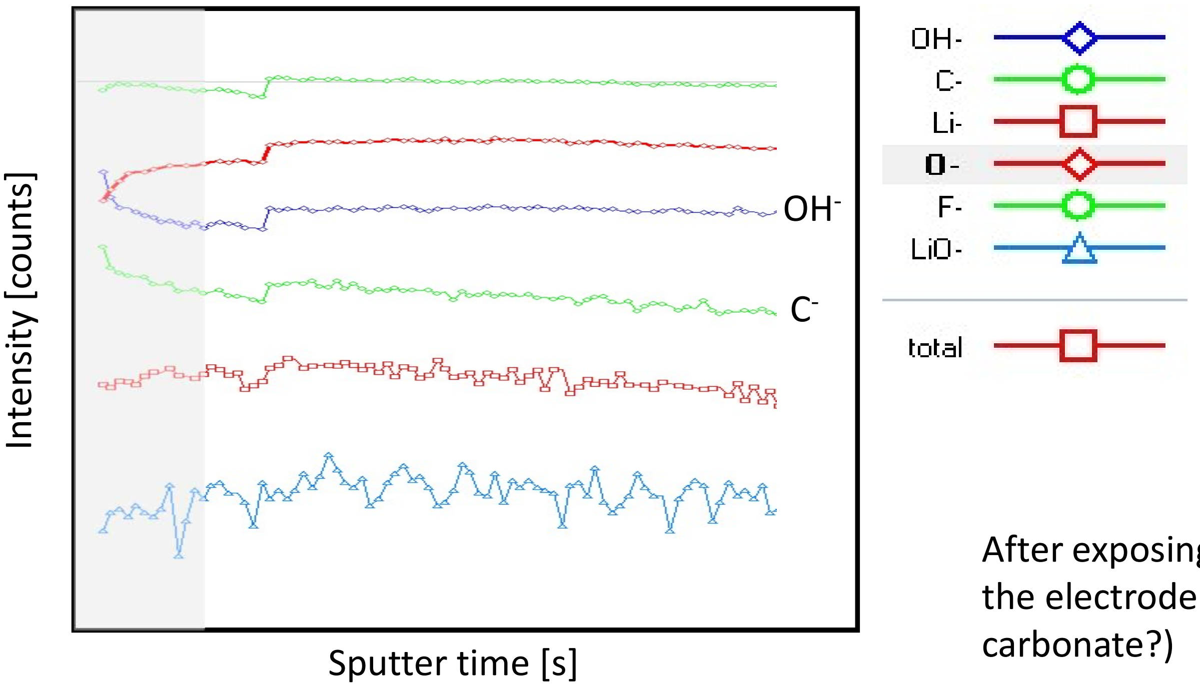
ToF-SIMS on pristine NMC electrodes – Transfer without Vacuum Suitcase

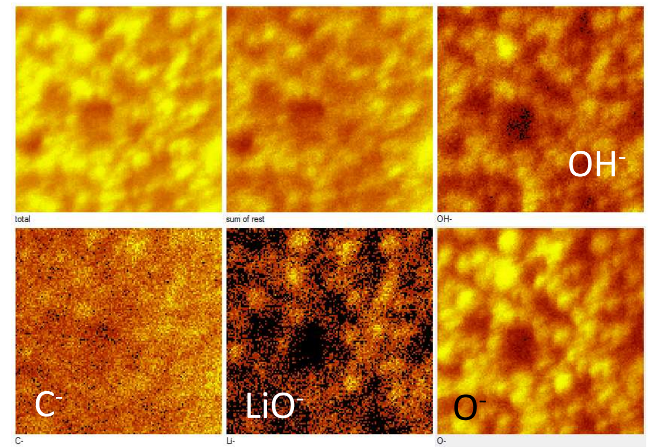


Depth profiles reveal OH- enrichment on the surface of the electrode, whereas other negative ions including C- appear to be depleted in the very surface of the electrode.

No vacuum suitcase used for these measurements!
Sample exposed to air for few minutes
Is the OH- coming from air exposure or are already present in the material received?

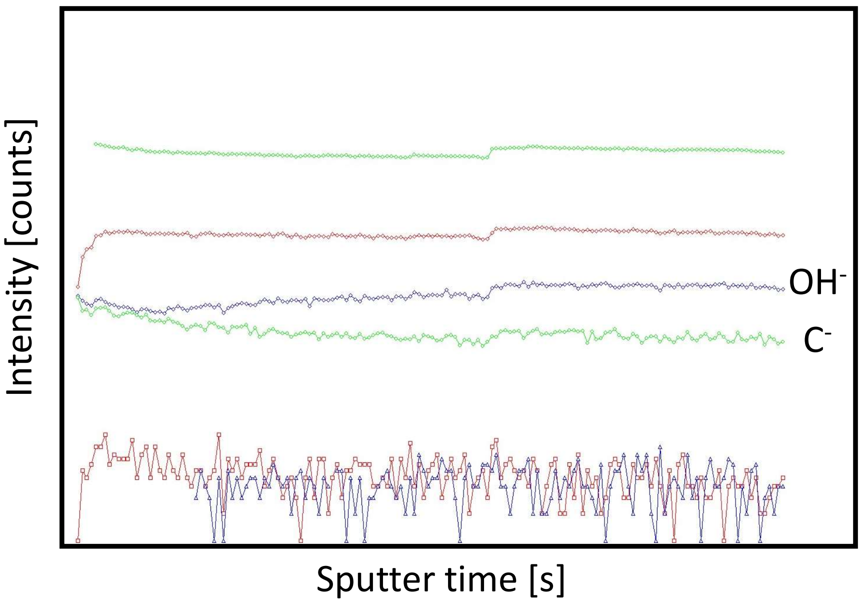
ToF-SIMS on pristine NMC electrodes – Sample Left in Air

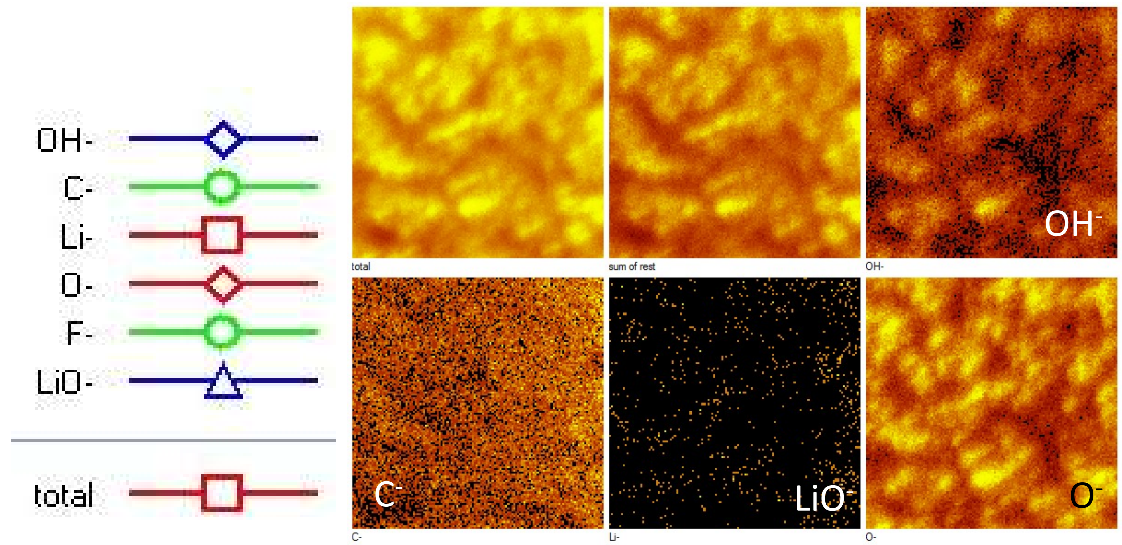




After exposing the NMC electrodes to air for two weeks, the electrode surface appears enriched in C- species. (Li carbonate?)

ToF-SIMS on pristine NMC electrodes – Pristine Sample (Minimum exposure to air)





Minimising the contact with air during transfer into the ToF-SIMS results in a more clean surface of NMC electrodes.
Development of a better vacuum suitcase is urgently needed!

Challenges and Future Plans

- Challenges:
  - Exposure to air leads to dirty electrodes surfaces – need of design a practical vacuum suitcase. ✕
  - ToF-SIMS does not provide enough lateral resolution to analyse the chemical composition within a single particle. ✕
- Future Plans
  - FIB-SIMS 3D reconstruction of pristine and cycled electrodes (***lateral resolution 25nm!!***)
  - Electrochemical cycling
  - Microstructural and chemical surface analysis of postmortem cells.

Hi-5 – Work in progress

- Main chamber at $100^{o}C$ to remove moisture, etc
- Current vacuum level: 10^{-7} / 10^{-8} mbar
- Target vacuum level: 10^{-10} mbar
- Antechamber with probes to be added

1. X
2. Y
3. Z
4. Chemical analysis: Dual detection of positive and negative ions simultaneously!
5. Processing within vacuum environment
   - Isotopic labelling
   - In situ electrochemical measurements on solid state devices

- Volume reconstruction
- ***Will resolve elemental distribution within individual NMC particles***

Spatially resolved dissolution of NMC

- *In situ* spectro-microscopy using X-ray Transmission Microscopy (TXM) and X-ray absorption spectroscopy (XAS):
- Spatially resolved chemical information as function of time and cycling conditions on NMC
- Chemical analysis of Cu/Graphite interface (XAS – Diamond proposal submitted)

Mohammed Koronfel

Mary Ryan

Probing gas evolution

- Ultra-sensitive and real time electrochemical mass spectrometry
  - Can measure submonolayer amounts of gases evolved, e.g. 30 nA/cm^2 O_2 evolution
- Ideal for model studies with low surface area, e.g. current collector or PLD-deposited NMC
  - Not used before in battery science

Daisy Thornton

Ifan Stephens

Conclusions

- Microstructural characterisation shows an inhomogeneous distribution of particle size ranging from few μm to tenth of μm

- ToF-SIMS analysis also shows an inhomogeneous distribution of NMC particles

- ToF-SIMS analysis suggests an enrichment in hydroxide species on the surface of the NMC particles as a result of exposure to air.

- Current lateral resolution is not high enough to allow chemical analysis within single particles.

- A unique FIB-SIMS is currently being commissioned at Imperial College and will allow chemical analysis within single particles.

Acknowledgments

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Thank you!

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Daisy Thornton

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