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Nanoscale and Microscale Research Centre

nmRC CASE STUDY

Understanding the structure and composition of battery materials

nmRC_CS_18

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Structure and composition of Nickel Manganese Cobalt (NMC) cathode materials

FIB-SEM-EDS case study

INTRO TO NMC AND LI ION BATTERIES

- Li-ion batteries are **rechargeable batteries** that have become ubiquitous in modern technology due to their **high energy density and relatively low self-discharge rate**. They are used in everything from smartphones to electric vehicles.
- Li-ion batteries consist of three primary elements: **a cathode** (positive electrode), **an anode** (negative electrode), and **an electrolyte**.
- A promising material for the cathode are **nickel manganese cobalt oxides** (**NMC**, $LiNi_xMn_yCo_{1-x-y}O_2$) as they offer **larger capacity**.
- However different synthetic routes can **vary the composition** and therefore structure so it is important to analyse both to understand the properties.

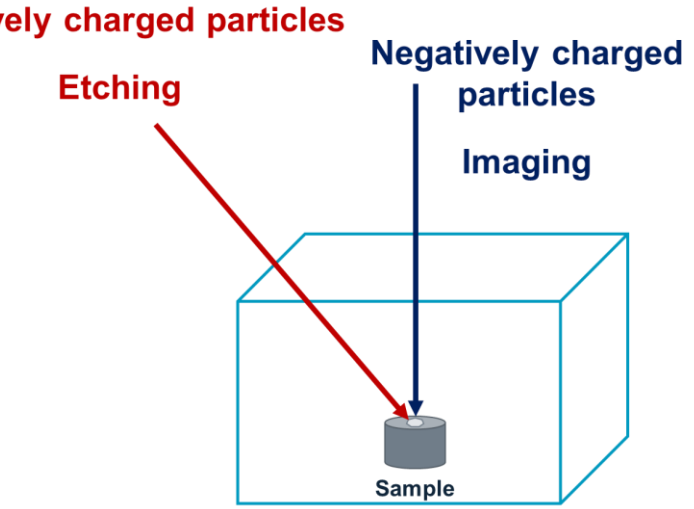
CHARACTERISATION

- To examine the structure and composition of these materials, we employed **F**ocussed **I**on **B**eam **S**canning **E**lectron **M**icroscopy (FIB-SEM) with in-situ energy-dispersive X-ray analysis (EDS).

- Traditional SEM images a sample using a beam of electrons which are scanned across the surface. FIB uses a **second beam of ions** (in this case Ga^{+}) to mill away at the sample. This allows the **internal structure** of a material to be investigated to analyse the homogeneity, or reveal hidden features.

- EDS is able to map the **chemical composition** of a material to qualitatively and quantitatively evaluate it. This was performed before, during and after FIB milling to verify homogeneity.

Focussed
Ion
Beam
Scanning
Electron
Microscopy



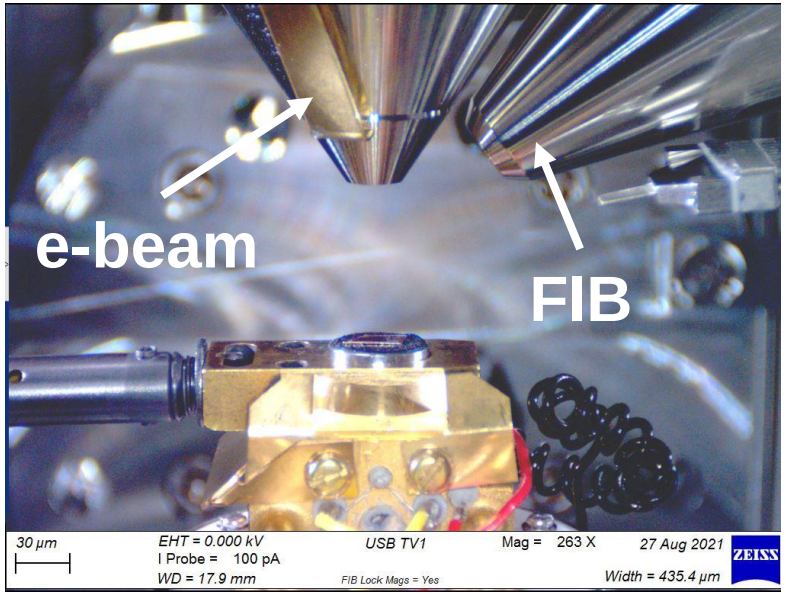
The Zeiss Crossbeam 550 scanning electron microscope is equipped with a range capabilities including: FIB, lift-out, Cryo, STEM imaging, EBSD, EDS and WDS.

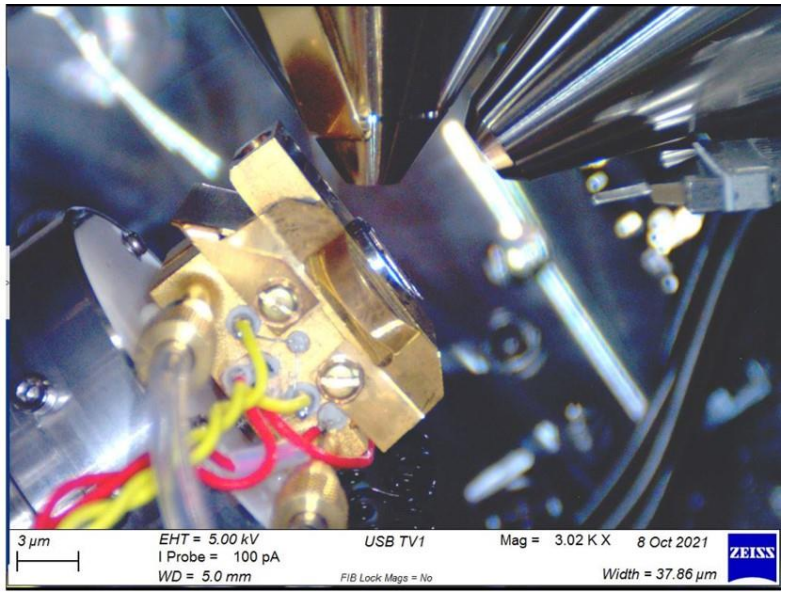
EXPERIMENTAL

To investigate the structure using **SEM**, the following parameters were used to minimise damage by the electron beam:

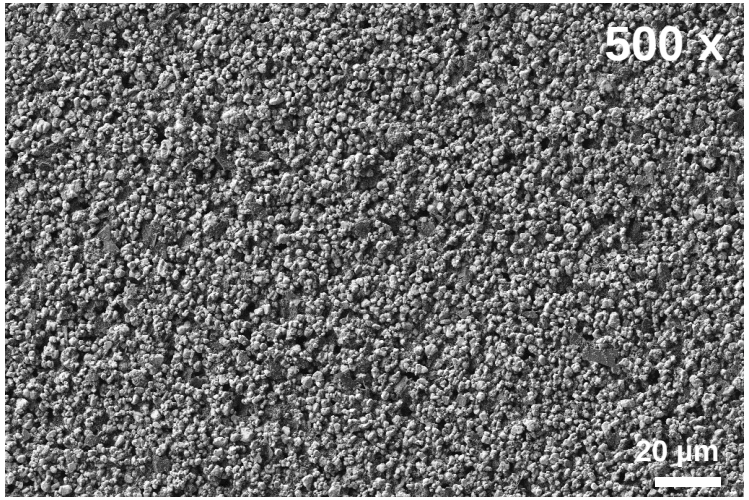
- Accelerating voltage: 2 kV
- Probe size: 200 pA
- Work distance: 8 mm

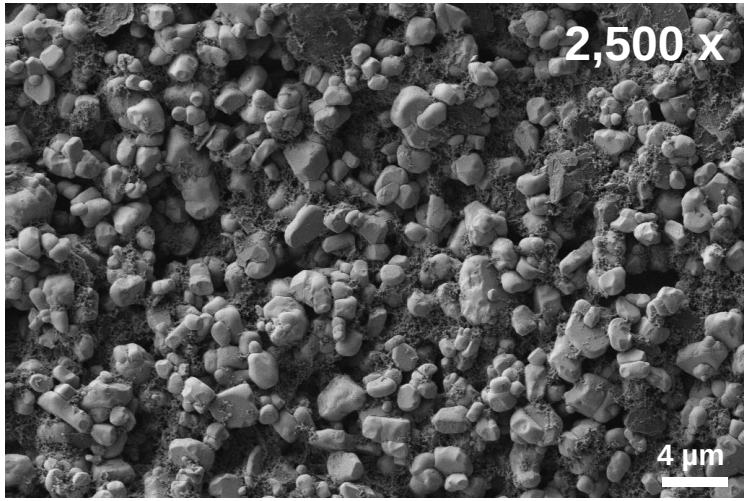
For **FIB measurements**, the sample was tilted to match the geometry of the ion beam and the work distance was also shortened to 5 mm.



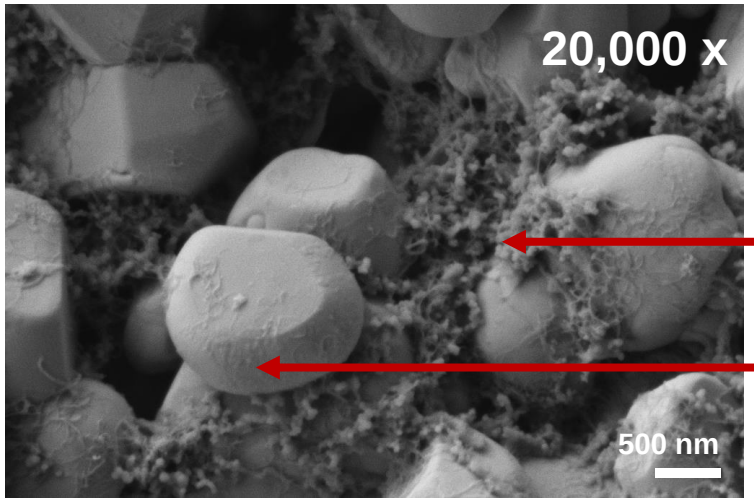


SEM IMAGING OF PRISTINE NMC





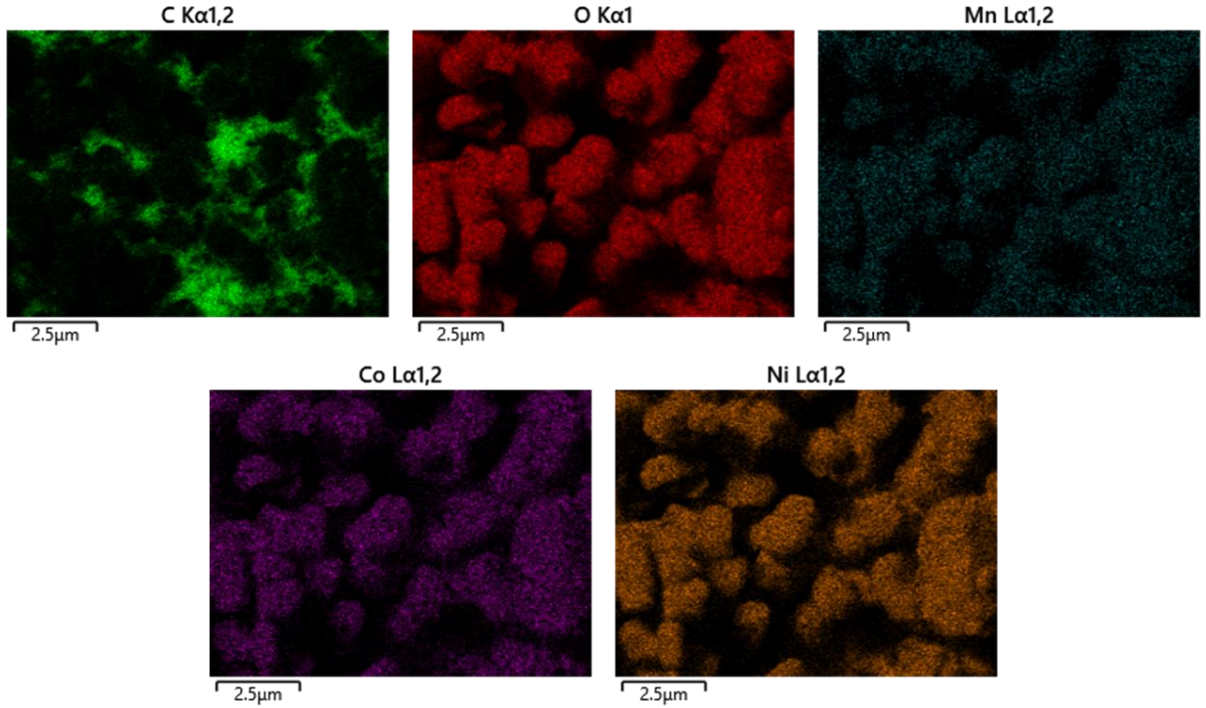
SEM images of the NMC Li-ion cathode material at various magnifications.



Conductive carbon mesh that binds the structure

Metal oxide particles

EDS MAPPING OF PRISTINE NMC

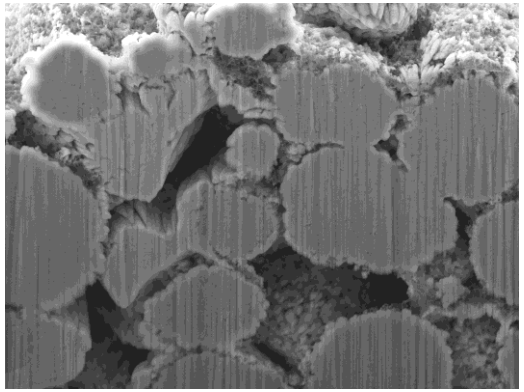


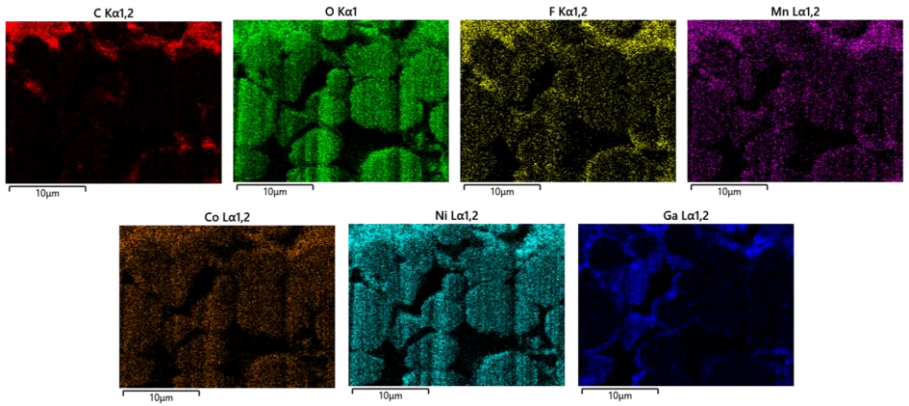
EDS mapping used to confirm the composition of the NMC material, finding it to be 80% Ni, 10% Mn, 10% Co.

Li has too low of an atomic number to be detected by EDS, hence why it does not appear.

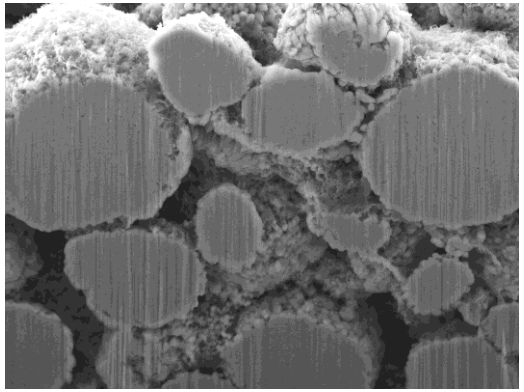
FIB-SEM-EDS IMAGING

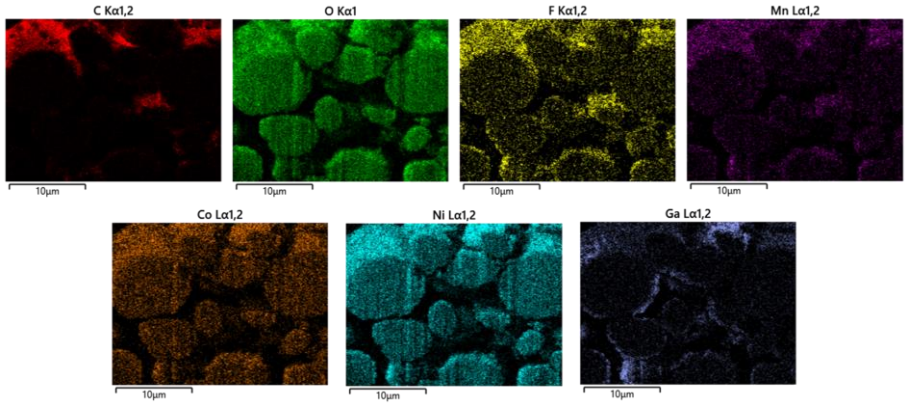
Before



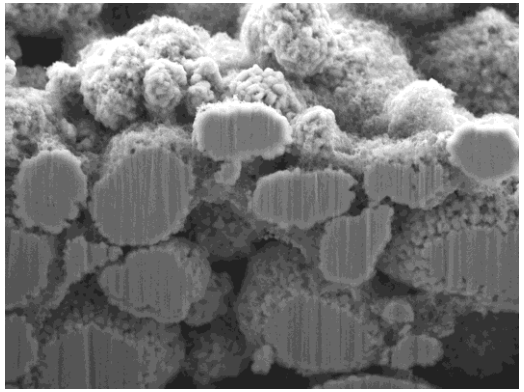


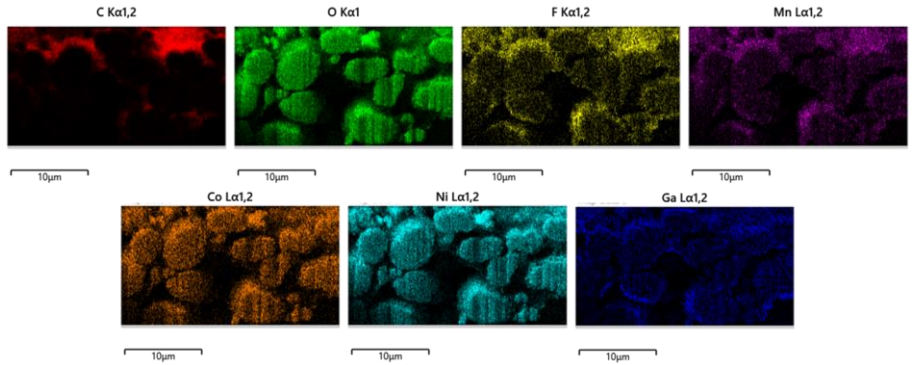
During





After 979 slices

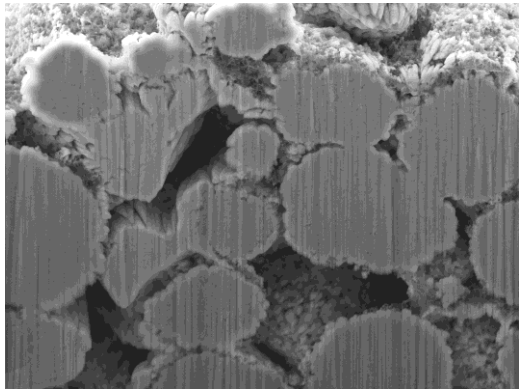




Scan/click me to view all 979 slices of the FIB-SEM process

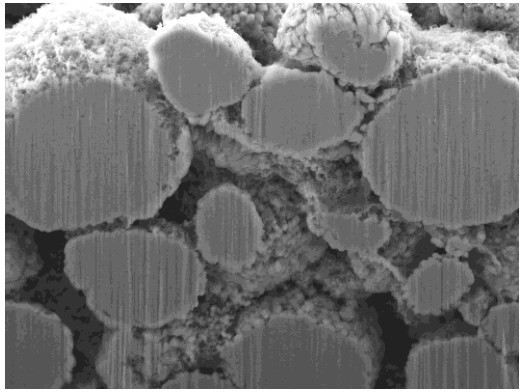
FIB-SEM-EDS IMAGING

Before



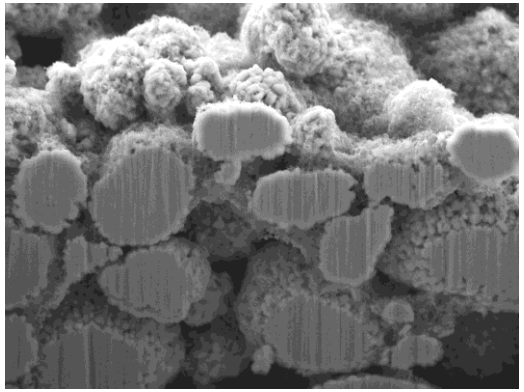
C	O	F	Mn	Co	Ni	Ga
4.4	12.3	0.9	6.6	8.2	63.4	4.2

During



C	O	F	Mn	Co	Ni	Ga
5.6	13.3	1.0	6.6	8.2	62.9	2.5

After 979 slices



C	O	F	Mn	Co	Ni	Ga
11.3	12.5	11.3	7.0	7.6	59.6	0.8

*Atomic percentages of elements. Note that Ga is present due to the Ga^+ ion beam used for FIB milling.

SUMMARY

- Initial SEM and EDS analysis allowed the pristine outer structure and composition to be examined.

- FIB-SEM then milled through the cross section to reveal the internal structure consisting of two primary components (metal oxide particles held together with conductive carbon mesh).

- EDS analysis was performed before, during and after FIB milling to examine if the chemical composition was homogenous throughout the particle.

REFERENCES / ACKNOWLEDGEMENTS

The FIB-SEM-EDS analysis documented here was performed at the Nanoscale and Microscale Research Centre (nmRC) at the University of Nottingham. www.nottingham.ac.uk/nmrc

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ADDITIONAL INFORMATION

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