

## A Better Insight Into Battery Materials Using A Correlative Approach

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Continued demand for electrification of transport, portable consumer electronics and even grid level storage continues to drive rapid developments of battery technology. Although each use case has its own relative performance criteria there is an overall need to increase charge and power density, cell cycle lifetime and safety while continuing to decrease cost per cell. For NMC cathodes particle size and material consistency has a large impact on final cell performance. Material inconsistencies decrease lifetime and can even result in catastrophic failure. Rapidly determining consistency while performing detailed characterisation of identified irregularities enables significant improvements in the next generation of materials.

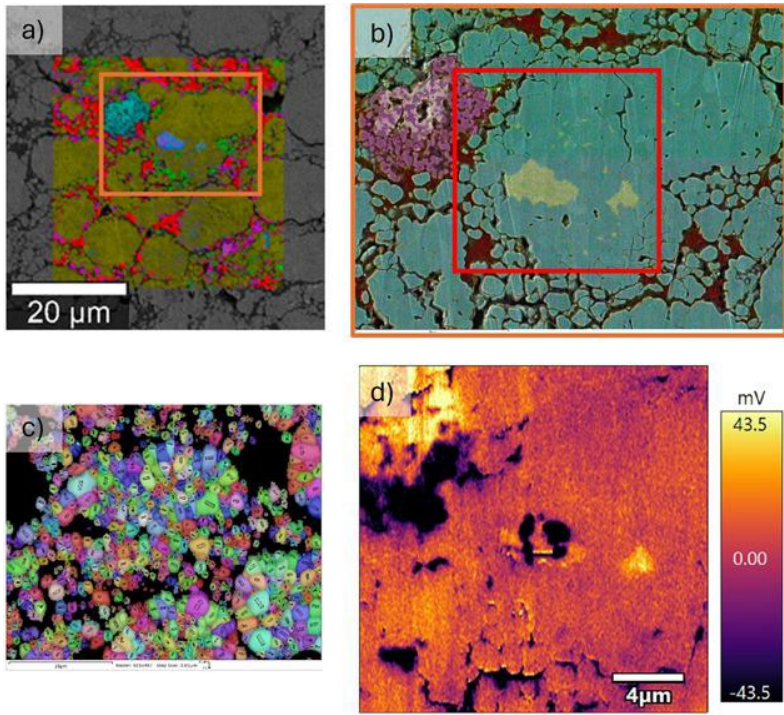
Here we show how using multiple techniques can deliver both that rapid assessment as well as detailed understanding of the material. However, it can be difficult and time consuming to know where on the sample to begin analysis. The first step is to create an overview of the sample, which can subsequently be used to identify areas for further analysis. A rapid (~2 min) survey using backscatter electron and x-ray (BEX) imaging identified areas of titanium (Ti) and sulphur (S). Where the chemistry was sufficiently divergent from NMC, that further analysis was required.

Once an interesting area with both Ti and S containing phases was selected, further detailed analysis was carried out. In this sample, it was the larger particles that were of interest to understand the chemical consistency. As some of the features identified in the BEX image were small and lithium is present, analysis at reduced electron energy (low kV) is preferred; firstly, to improve spatial resolution and surface sensitivity and secondly, to reduce beam damage of the sample. Windowless energy dispersive x-ray spectrometry (EDS) was used because the technique is designed for low kV analysis. Identifying molecules, particularly lithiated ones, is critical in beginning to understand the charging properties of the cathode. EDS is only sensitive to elements and not molecular structure, additionally Li can be difficult to detect using EDS in compounds, therefore Raman was used to identify lithiated and non-lithiated molecules. Raman can also be used to visualize where the molecules are within the NMC particles and characterise the degree of lithiation, which will dictate the electrical properties. To understand the structure, texture and grain boundary information, which will also indicate the electrical properties and charging efficiencies, electron backscatter diffraction (EBSD) was carried out. In cathodes, grain boundaries

are typically found to be a source of failure in large particles where electrolyte impregnation can cause cracking and degradation. To characterise how both structure and composition relate to the electrical properties of the cathode particles, Kelvin Probe Force Microscopy (KPFM), an electrical mode in atomic force microscopy (AFM), was used to measure localised electrical potential. Fig.1 shows the complementary nature of individual techniques when spatially correlated. The Raman results showed that the Ti was associated with lithium titanate (LTO, teal) and the S was associated with a lithium sulphate compound (blue). The EDS was used to aid in identifying and qualifying the Raman spectra. Structurally it was found that a high percentage of the unit cells were aligning around the perimeter of the crystal. For optimum speed of charge and discharge it is preferable to align perpendicular in the 0001 direction with the particle radius, in this case it was found to be perpendicular to the radius thus improving the motion of Li<sup>+</sup> ions. The KPFM results showed that the small S area had a higher electrical potential than the surround particle. However, the larger S particle had zero electrical potential; this was because the S areas were damaged by the electron beam during EDS acquisition which caused bubbling of those areas, which would cause a hollow zone and would measure as zero. New regions containing S will be measured to correctly measure the electrical properties.

This work shows that a correlative workflow works well for battery cathodes and enables critical insights. Individually, the techniques deliver useful information that can help in understanding battery materials and improving processes. When the multiple techniques are correlated, they enable new conclusions, reduce uncertainty and in some cases stop the incorrect conclusion being drawn.

**Graphic:**



**Keywords:**

Battery, correlative, multiscale