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Unique in-situ characterization workflow of Cathode Components using AFM-in-SEM

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Background incl. aims
As the demand for efficient and sustainable energy storage solutions grows, a lot of effort is dedicated to comprehending the battery components' functionalities. Not only exploring new configurations but also new materials are essential for improved efficiency, safety, lifetime, etc. of the new-generation lithium-ion batteries [1]. However, such materials require precise sample surface preparation and handling to avoid unwanted reactions or surface changes which then influence understanding of the battery component features.

Methods
In this work, we focus on the in-situ study of Cathode Active Materials (CAM) and present a unique measurement workflow that preserves the sample surface from oxidation without compromising sample preparation quality, moreover, enabling valuable insights into chemical and electrical properties. The CAM tape samples are prepared in a glovebox on a proper holder, cross-sectioned using Broad Ion Beam (BIB) polishing, and further moved to the Atomic Force Microscope in a Scanning Electron Microscope (AFM-in-SEM) for electrical and chemical characterization. The sample transfers between all instruments are realized using a safe sample transfer system under a controlled environment, so the sample is never exposed to air or humidity, and thus, the surface remains clean and preserved from chemical reactions.

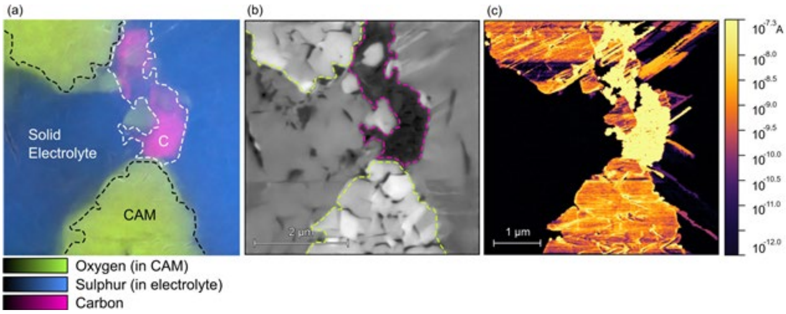
Results
With such a workflow, we studied Nickel-Cobalt-Manganese (NCM) tape with solid electrolyte, see Fig. 1. We determined the electronic conductivity using Conductive AFM (C-AFM) and distinguished the individual elements using Energy Dispersive X-ray Spectroscopy (EDS), all under in-situ conditions. We see that the NCM grains are well conductive, while the solid electrolyte remains non-conductive, which is correct, as the solid electrolyte only has an ionic conductivity (the dark part of Fig. 1c). The most conductive seems to be the secondary particles interface (the brightest part of Fig 1c), which perfectly correlates to the carbon additive observed in EDS map (Fig. 1a). Such precision would be unlikely possible or very challenging without the in-situ analysis.

Similarly, we observed a CAM tape with different grains, Nickel-Cobalt-Aluminum (NCA) and NCM, in a liquid electrolyte. The connection of the EDS map with electronic C-AFM shows that the conductivity of the grains differs 1000 times. From understanding such grains and their conductivity, the difference should be much lower. As we see in the SEM image, the NCM grain had probably been delaminated from the collector, and the conductive connection with the NCM particle was established via the inter-particle interface through the NCA particle.

Conclusion
These results emphasize the need for advanced measurement workflow and instrumentation, which could greatly help in understanding the cycling effects, durability, or lifetime of both primary and secondary particles of battery components. The above-mentioned characterization workflow is convenient, time-efficient, and suitable for a proper understanding of failure analyses and quality control of the battery components. Our findings were obtained using AFM LiteScope, which is compact enough to be used in SEM, in this case,

Helios 5 Hydra DualBeam, CleanMill for fine polishing, and CleanConnect for sample transfer [2].

Graphic:



Keywords:

in-situ, batteries, AFM, SEM, conductivity

Reference:

1. Itani, Khaled, and Alexandre De Bernardinis. "Review on New-Generation Batteries Technologies: Trends and Future Directions." Energies 16.22 (2023): 7530.
2. We would like to thank Libor Novák and Petr Zakopal from Thermo Fisher Scientific for their assistance with sample surface preparation.