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Scanning Electron Microscopy (SEM) for Battery Electrode Analysis

SEM enables failure analysis of aged cells, quality control of manufactured electrodes, and fundamental research on material degradation mechanisms.

Introduction

Battery performance, lifetime, and safety are heavily influenced by the morphology and microstructure of the electrode materials. Scanning Electron Microscopy (SEM) provides high-resolution imaging and elemental analysis capabilities, making it a critical tool for evaluating both pristine and cycled battery electrodes.

This application note outlines how SEM can be used to characterize battery electrodes, highlights key preparation methods, imaging techniques, and typical analysis goals.

SEM is used to investigate the following properties of battery electrodes:

- Surface morphology and porosity
- Particle size and shape distribution
- Electrode coating uniformity and thickness
- Interface degradation (e.g., between active material and binder/current collector)
- Formation of surface layers (e.g., SEI on anode)
- Elemental composition using Energy Dispersive X-ray Spectroscopy (EDS/EDX)
- Structural changes after cycling (cracking, delamination, etc.)

Experimental

Effective SEM analysis requires proper sample preparation. The lithium-ion battery was first disassembled in an argon-filled glovebox to prevent exposure to air or moisture, which could alter the sample. The electrodes were carefully isolated, rinsed with solvents like DMC or DEC to remove residual electrolyte, and dried within the glovebox. Subsequently, the prepared electrodes were mounted on conductive tape and transferred to the SEM in an airless vessel for imaging.

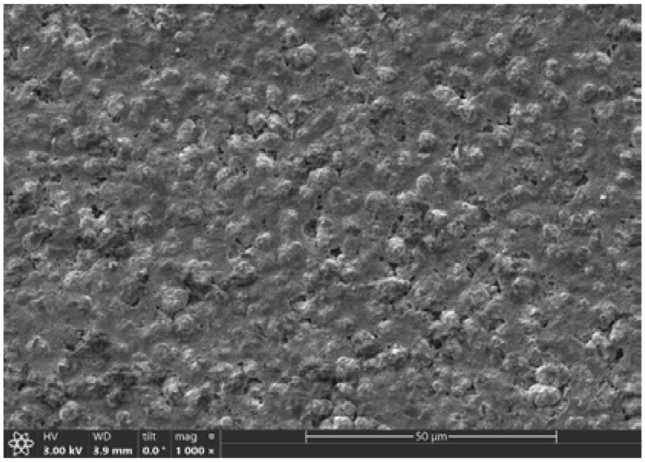
Results and Discussion

SEM Analysis

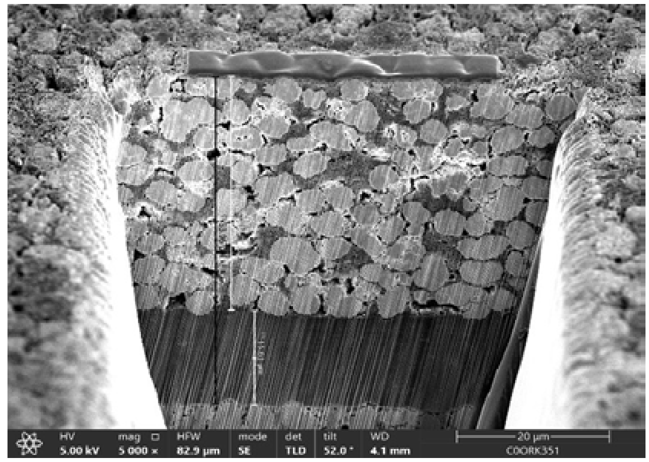
SEM analysis of the cathode electrode provided detailed topographical information. As shown in Figure 1A, a top-down SEM image reveals the surface morphology of the cathode, where active material particles are clearly visible within the electrode film. A cross-section of the electrode, prepared using a Dual Beam Focused Ion Beam (DB-FIB), as shown in Figure 1B, allowed for the precise measurement of the cathode film and current collector thicknesses.

Different electron signals yield distinct types of information.

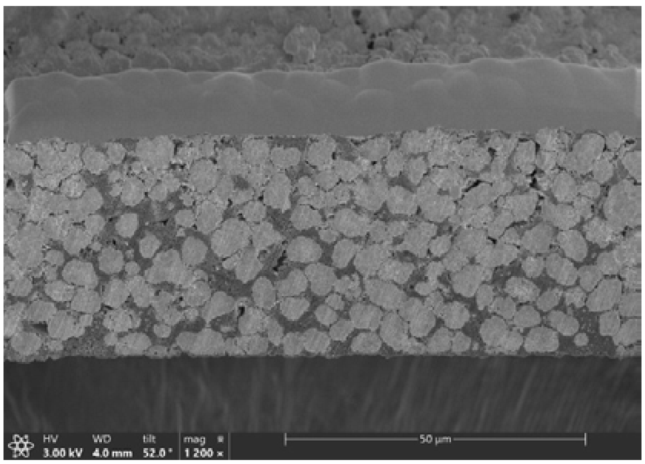
- Secondary Electron (SE) images (Figure 1C & 1E) offer high-resolution information on the sample's surface topography due to the surface-sensitive nature of low-energy secondary electrons.
- Backscattered Electron (BSE) images (Figure 1D & 1F) provide compositional contrast, with areas of higher atomic numbers appearing brighter. BSE images are valuable for distinguishing different materials, complementing the surface detail provided by SE images.



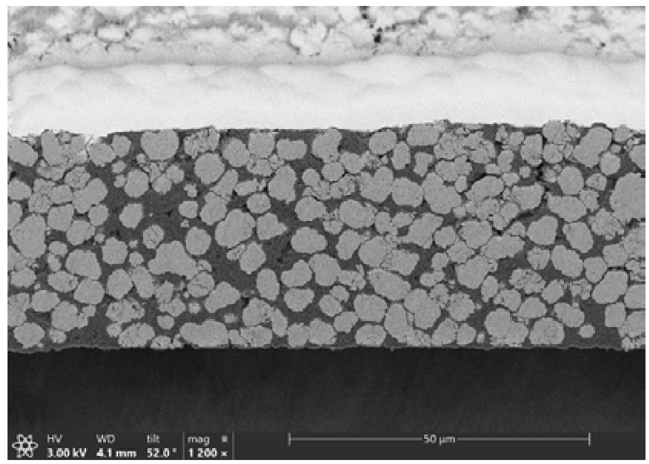
A. Topdown



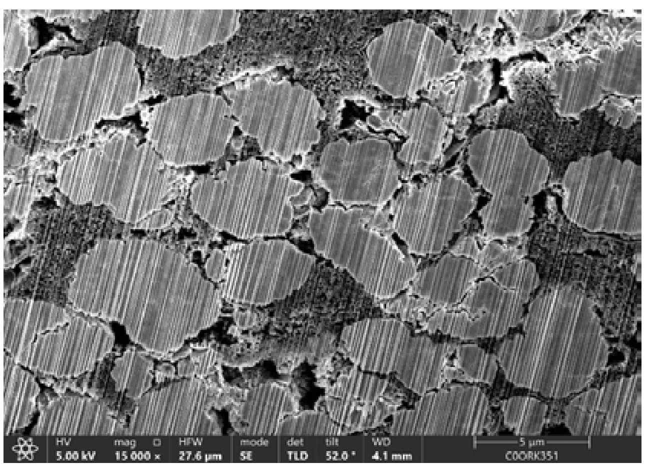
B. Cross-cutting



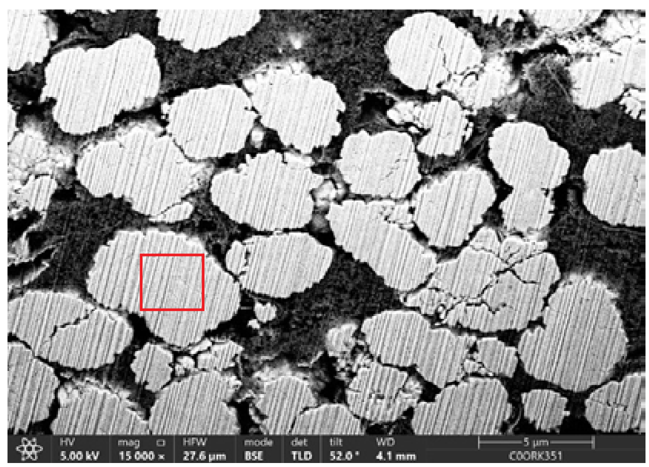
C. Cross-section SE image



D. Cross-section BSE image



E. Cross-section SE image



F. Cross-section BSE image

Figure 1: SEM images of cathode

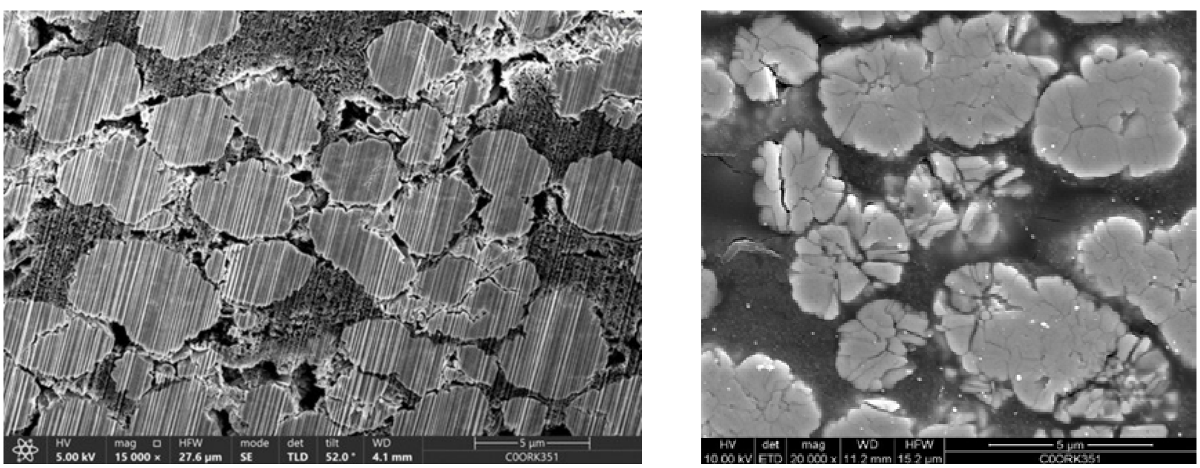
When coupled with energy-dispersive X-ray spectroscopy (EDX/EDS), SEM provides qualitative elemental analysis of the sample's surface. This is critical for evaluating new cathode materials, optimizing synthesis, monitoring coatings, and investigating degradation or contamination. The composition of cathode particle in Figure 1F was determined and shown in Table 1. The elemental results indicate that this cathode is NMC522.

Figure 2 highlights the difference between DB-FIB and mechanical cross-sectioning.

- DB-FIB (Figure 2A) delivers exceptional nanometer-scale precision, making it ideal for targeted, damage-free analysis of specific features.
- Mechanical cross-sectioning (Figure 2B) can introduce artifacts like scratches and deformed structures. Ion polishing can be used to refine the surface after mechanical cutting.

Table 1: SEM/EDS quantitative analysis of cathode

Element	Atom No.	Weight Conc. (%)
Nickel	28	50.68
Manganese	25	30.02
Cobalt	27	18.95
Fluorine	9	0.33
Aluminum	13	0.01
Carbon	6	0.00
Oxygen	8	0.01
	Sum	100.00

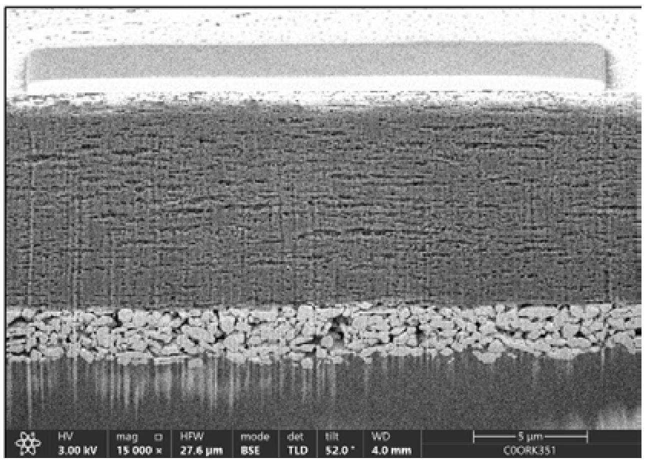


A. Dual Beam cutting

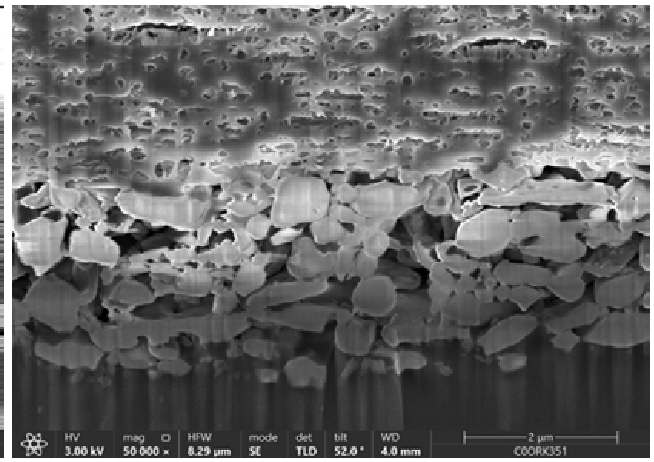
B. Mechanical cross-sectioning

Figure 2: Comparison of different cutting techniques

The separator layer, isolated from the jelly roll, was also analyzed. Figure 3A shows a cross-section of the ceramic-coated composite separator prepared via DB-FIB. By optimizing the cutting temperature, the separator's delicate structure was preserved, revealing clear contrast between different phases. A high-resolution image of the ceramic coating (Figure 3B) provides detailed information on particle size and distribution.



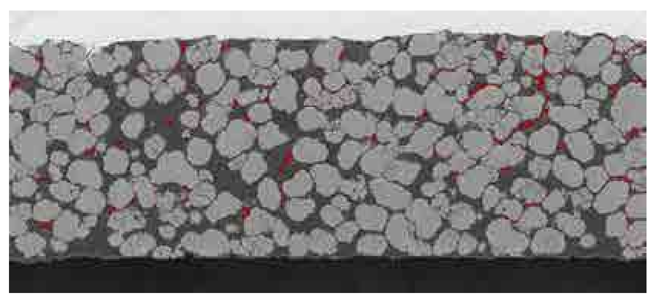
A.



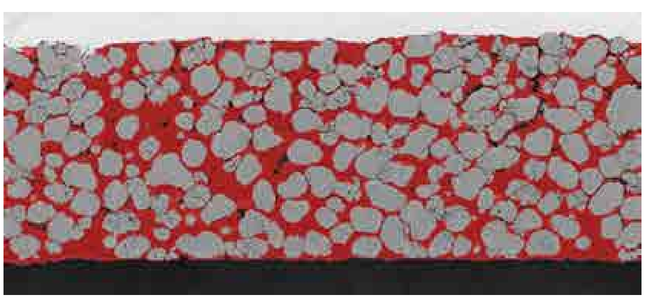
B.

Figure 3: SEM images of separator cross-section by DB-FIB

A. Particle



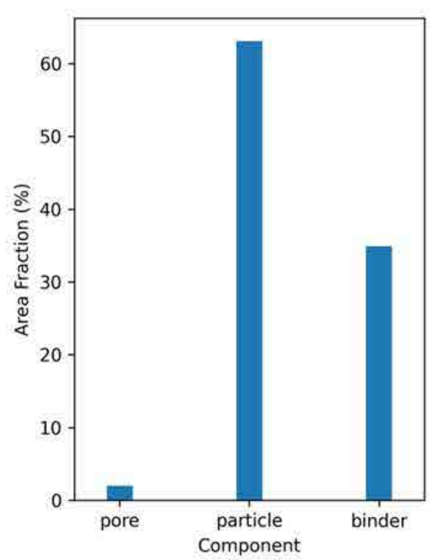
B. Pore



C. Binder

Figure 4: Cathode component percentage

D.



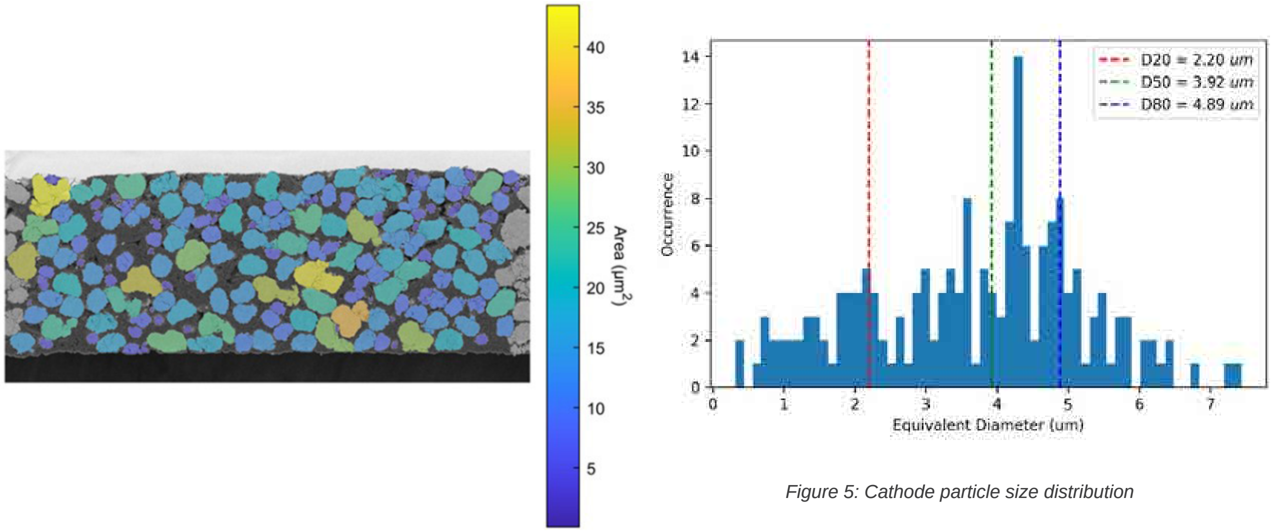


Figure 5: Cathode particle size distribution

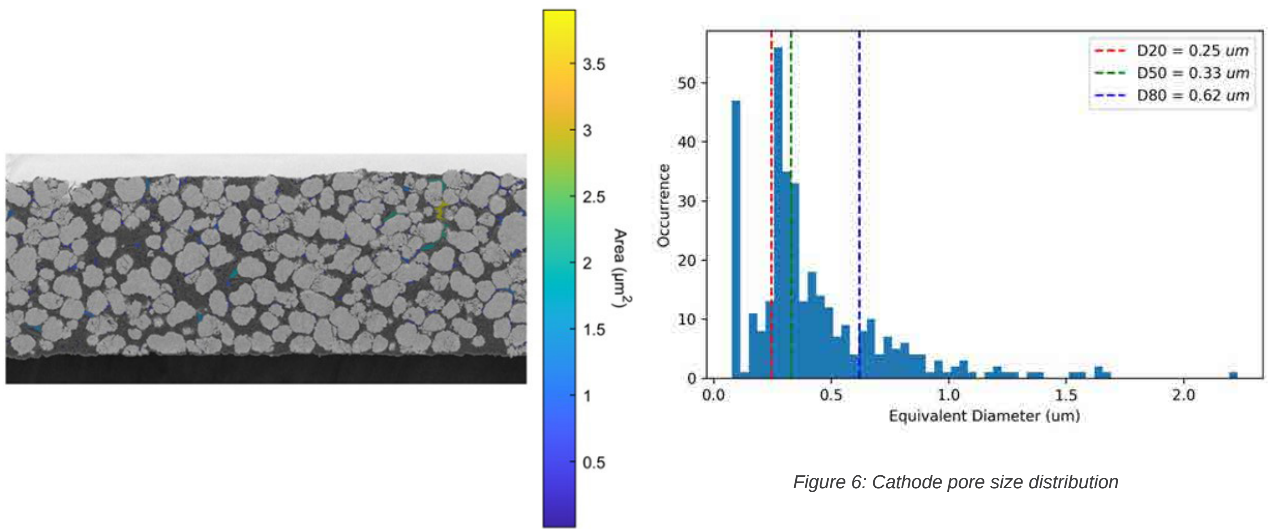


Figure 6: Cathode pore size distribution

Particle Size Distribution (PSD) Analysis

Other than the above morphology analysis, SEM images can also be used to determine the active material particle size and distribution, particularly for irregularly shaped or nanoscale particles that are difficult to measure with other methods. After capturing high-resolution SEM images of the cathode at various magnifications, the images were processed to enhance contrast and delineate particles from the background as shown in Figure 4. Particles were then detected and measured using automated methods as shown in Figure 5. Additionally, pore size (gap between active materials and binders) can also be determined by the same method as shown in Figure 6.

Conclusion

SEM is a vital tool in battery electrode analysis, offering insights into both morphological and chemical properties. With careful sample preparation and proper imaging strategies, SEM enables failure analysis of aged cells, quality control of manufactured electrodes, and fundamental research on material degradation mechanisms.

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References

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2. Wood, K.N., et al. "Operando SEM and EDS characterization of solid-state lithium batteries." *ACS Applied Materials & Interfaces*, 2017.
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