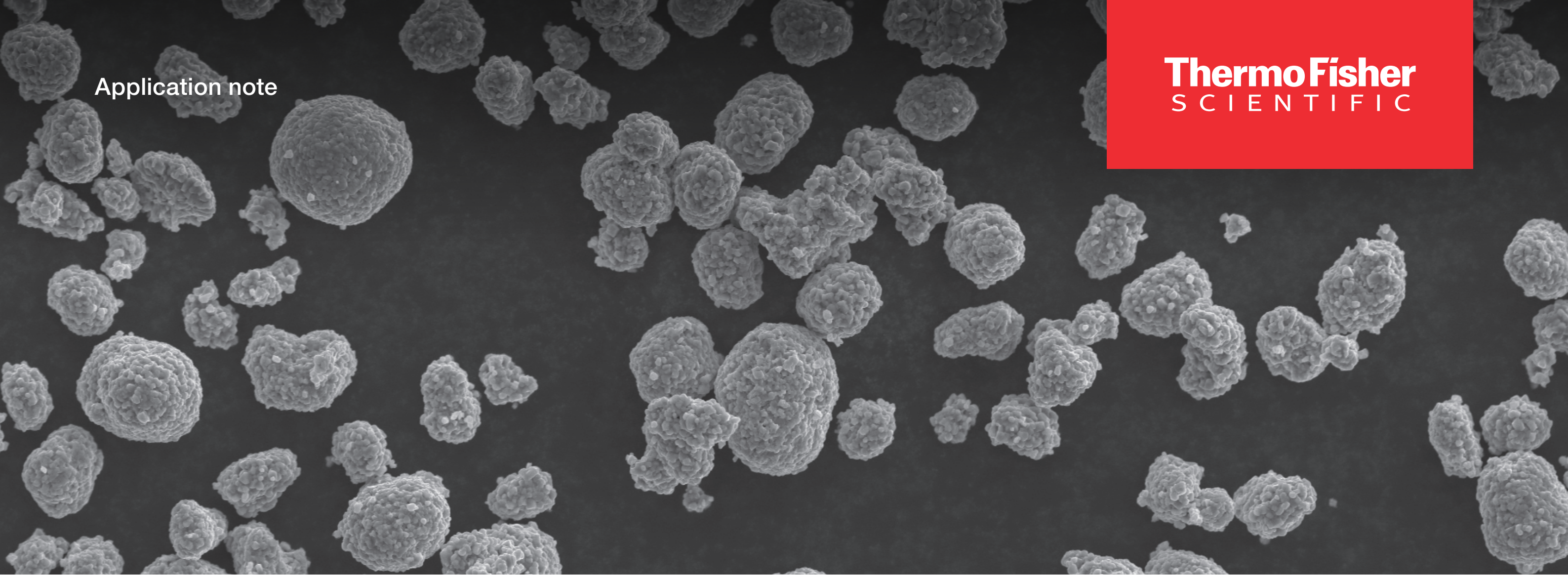
Application note



Automated impurity analysis for lithium-ion batteries with Perception Software

Authors

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This article discusses using Thermo Scientific™ Perception™ Software and scanning electron microscopy (SEM) to automate analysis of impurity particles during lithium-ion battery manufacturing. The automated process generates customized reports that present impurity particle data through various charts and graphs, enabling a visual assessment of whether the analysis outcome aligns with quality control standards.

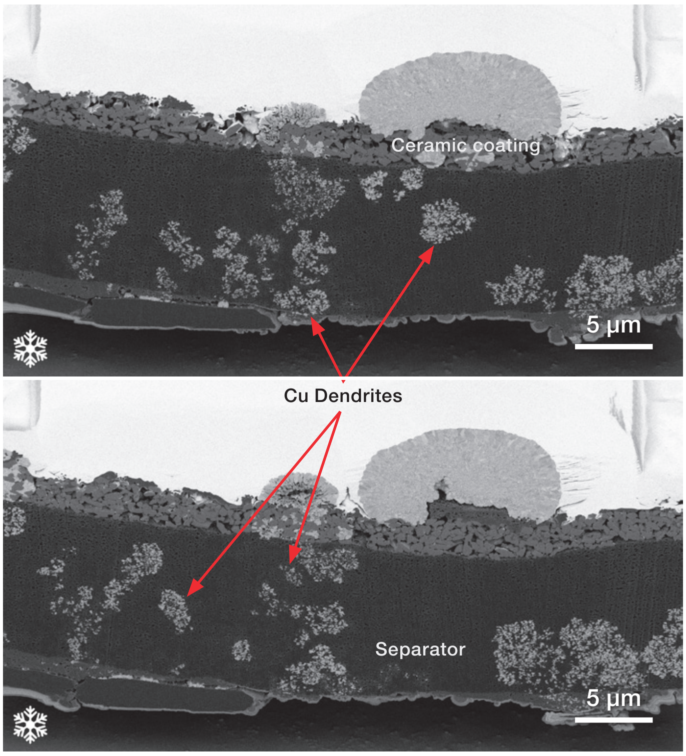


Figure 1: SEM cross section showing Cu dendrite precipitation within and penetrating the Li-ion battery separator. Sample preparation and imaging were done using the Thermo Scientific™ Scios™ 2 DualBeam™ FIB-SEM with cryo stage at –178°C. ***Sample courtesy of the Sichuan New Energy Vehicle Innovation Center.***

Introduction

Lithium-ion batteries are recognized for their high energy density and long cycle life, which make them popular for use in electric vehicles and renewable energy storage. However, battery safety and durability can be compromised by impurity particles that may cause internal short circuits. These short circuits may occur when large-size impurity particles mechanically pierce the separator or when metal impurity particles in the high-potential cathode undergo a dissolution-precipitation process.

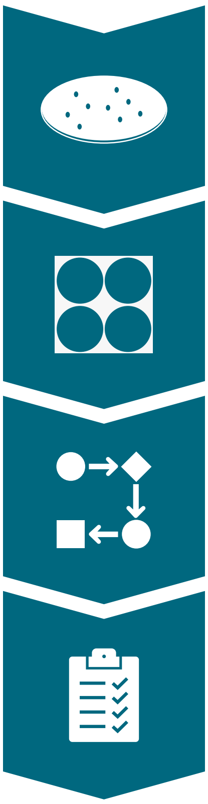
Among these impurities, copper (Cu), characterized by its low dissolution potential and commonly generated in raw material manufacturing, poses a high risk of dissolving on the cathode side, diffusing to anode side, growing and depositing along the separator's pores (as Figure 1 shows), connecting the anode and cathode, and causing micro short circuit. Other metal impurities have also been proven to affect the safety and performance of batteries.

Analyzing metal impurity particles, particularly in cathode powder material, has gained significant attention from battery manufacturers worldwide. To further enhance battery safety and standardize the testing process, China, where most lithium-ion batteries have been manufactured to date, drafted the GB/T 41704-2022 standard for battery impurity particle testing, which was implemented on February 1, 2023.

Although it is known that metal impurity particles are harmful to batteries, analyzing these impurity particles remains a big challenge for manufacturers. Conventionally, collecting these impurity particles involves using a filter membrane or transferring the particles to carbon tape for manual particle analysis. Each filter membrane or carbon tape sample typically contains tens of thousands of particles, making manual analysis or identifying impurity particles a time-consuming and labor-intensive task. To address this issue, Perception Software reduces the time per particle to less than one second, providing quick results and generating detailed, conclusive reports.

Methods

Perception Software integrates SEM control and automates particle analysis, facilitating the entire particle analysis process. Analyzing impurity particles with it takes only four steps:



Step 1. Load the sample into the sample holder

Step 2. Create a stage file to tell Perception Software where the sample is located and what area to analyze (you can also save the stage file as a template for future tests)

Step 3. Load the standard recipe for battery impurity particle analysis or a previously created recipe

Step 4. Initiate the analysis, which can run overnight and unsupervised, and receive an automatically generated report

Within the standard recipe for battery impurity analysis in Step 3, you can define the criteria for analyzing particles of interest:

- In the morphology tab, specify the size parameters (diameter, aspect ratio, etc.) of particles to be analyzed. For example, you can analyze impurity particles larger than 15 micrometers in diameter within LiFePO4, or particles larger than 5 micrometers in diameter within NCM powder. During runtime, particles falling outside the specified size range are automatically skipped.
- In the rule file tab, define the categorization of impurity particles based on their composition and morphology. You can also assign longer analysis times (secondary check) to critical or hazardous impurity particles, enhancing the reliability of results. These classified particle results help you find the source of particles and improve production process control.

Results

For our test, we prepared a sample by mixing copper and stainless-steel powders into NCM 811 single-crystal cathode powder. This mixed powder was then spread onto a one-inch aluminum stub with carbon tape. Next, we used Perception Software to analyze particles larger than 5 µm on the sample.

Size class		B	C	D	E
Size Range (µm)	Total	5≤X<15	15≤X<25	25≤X<50	50≤X<100
Cu-rich	1104	888	163	52	1
Iron-rich	1192	83	678	427	4
Al-rich	7	6	1		
Zn-rich	3	3			
Al-silicates	0				
Si-rich	400	394	5		1
Total counts	2706	1374	847	479	6
Clean level		11	10	9	3
Component clean code (CCC): V(B11/C10/D9/E3/F00/G00/H00/I00/J00/K00)					
Specifications: V(B12/C10/D8/E6/F0/G0/H0/I0/J0/K0) Does not pass					

Class	Color	Sub total
High conductivity		2296
Mid conductivity		10
Low conductivity		400

This table reports whether particles within different size ranges meet the specified standards. Rows represent particle categories, while columns display the size distribution of particles. Red, yellow, and green indicate particle conductivity. At the bottom of the table, “CCC” represents the defined cleanliness standard. For instance, “D8” indicates that for column D (25 µm < particle diameter < 50 µm), the upper specification limit is 28 = 256 particles. The result shows its cleanness level for column D is 9, exceeding our set value of 8. Therefore, the bottom section of the table is marked in red and displays “Does not pass specification.”

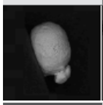
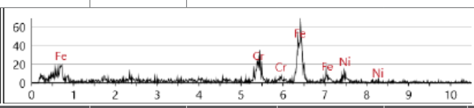
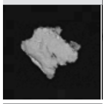
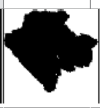
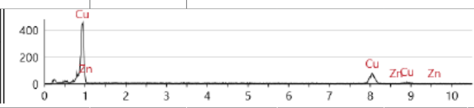
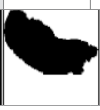
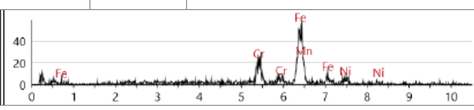
Particle limits

This table reports whether each particle category meets the requirements and can be customized by limiting the particle count. The first column of the table represents the particle classification while the following three columns indicate the number of detected particles, the minimum acceptable particle count, and the maximum acceptable particle count. The result shows that 1,276 iron-rich particles and 8,693 Cu-rich particles rich were detected, exceeding the maximum acceptable particle count of 100. As a result, the bottom row of the table is marked in red, indicating that it did not meet the requirements.

Rule class	Particle count	Minimum	Maximum
Iron-rich	1260	0	100
Cu-rich	8693	0	100
Zn-rich	85	0	100
Al-rich	29	0	500
Si-rich	1176	0	2000
Al-Silicates	10	0	500
FAIL			

This table reports whether each particle category meets the requirements and can be customized by limiting the particle count. The first column of the table represents the particle classification while the following three columns indicate the number of detected particles, the minimum acceptable particle count, and the maximum acceptable particle count. The result shows that 1,276 iron-rich particles and 8,693 Cu-rich particles rich were detected, exceeding the maximum acceptable particle count of 100. As a result, the bottom row of the table is marked in red, indicating that it did not meet the requirements.

Particle information

Rank	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge Roughness	ECD
1	246	1	53.702	30.757	Iron-rich	-7.477	0.742	1.751	1294.059	0.571	0.556	1.306	40.591
													
	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge Roughness	ECD
2	612	1	50.219	44.017	Cu-rich	-6.457	-5.879	1.177	1230.267	0.621	0.348	1.131	39.578
													
Rank	ID	Run ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form factor	Edge Roughness	ECD
3	522	1	48.919	25.796	Iron-rich	7.447	-3.004	2.199	860.571	0.458	0.502	0.745	33.102
													

If you are interested in investigating details on detected impurities, the particle information table includes representative particles rich in Cu or Fe, presenting particle images, mask images, particle positions in the sample, morphology information, and labeled spectra. In the Perception Software UI, you can reposition these particles for further analysis and confirmation.

The report software provides various table and chart plugins to display particle data in different ways. You can customize how the impurity particle data is presented in the report according to your needs and save it as a template integrated into the particle analysis recipe. Alongside the run, a .pxz file containing all particle information is generated, making it possible to integrate the analysis results into your data management software.

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Conclusion

Metal impurity particle analysis attracts increasing attention in the battery industry, especially after the implementation of the Chinese standard GB/T 41704-2022, which has standardized and streamlined the analysis process. By ensuring the quality and cleanliness of raw materials and the manufacturing process, potential internal short circuits caused by metal impurities can be effectively minimized.

Perception Software automates the impurity particle analysis process, enhancing measurement efficiency and data reliability. The information on impurity particle morphology, composition, and classification aids in tracing the source of impurities and improving production process optimization. Furthermore, Perception Software can automatically generate reports, providing immediate results on whether quality control requirements are met.

By adopting Perception Software's automated analysis solution, you can enhance your quality control processes and ensure the safety and reliability of lithium-ion batteries.

Learn more at **thermofisher.com/perception**

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