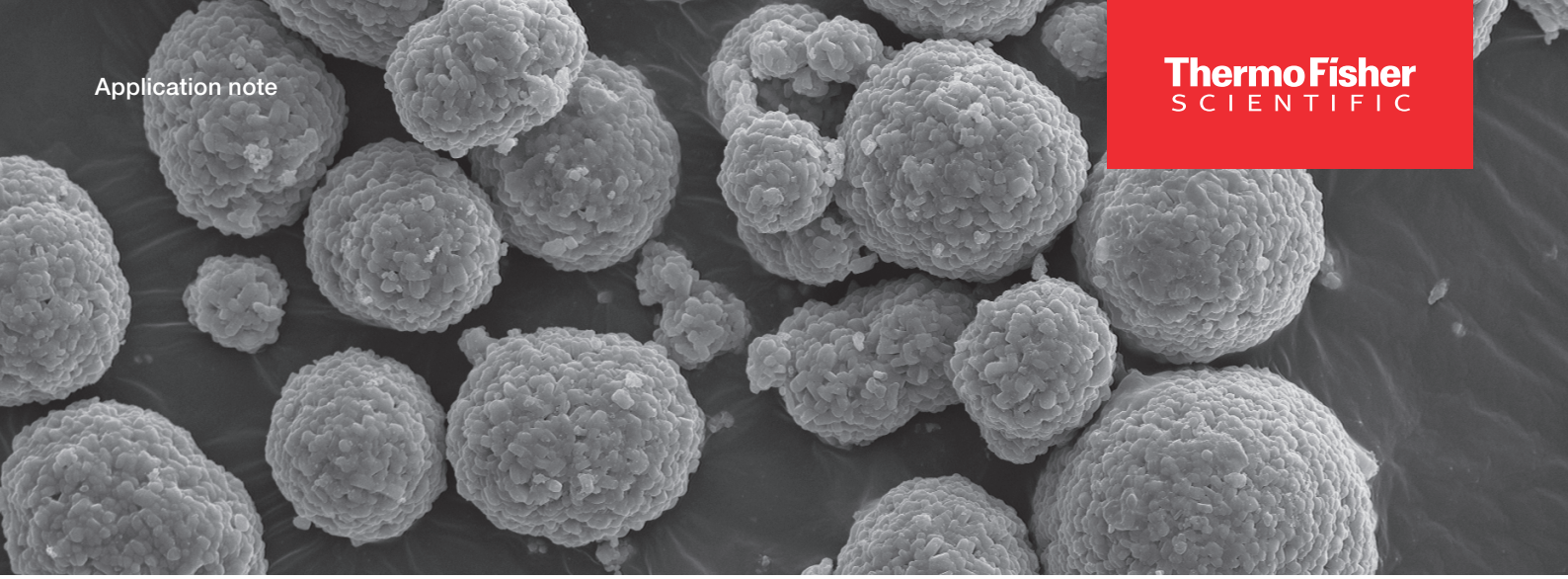

Application note

Accelerate battery cathode material quality control with automated SEM imaging and analysis

Introduction

An electric vehicle battery pack consists of thousands of individual cells, and the electrodes of these cell each contain millions of particles. During charging and discharging, it is important that these particles are working together; each individual particle must perform as expected, and together they must maintain consistent electrochemical characteristics to fully utilize the capacity of the battery.

The particle size distribution and microstructure of cathode materials and their precursors are critical to the energy density and safety of batteries, which means that the quality of these particles needs to be strictly monitored during the production process. Scanning electron microscopy (SEM) is used in manufacturing process control to identify quality fluctuations of raw materials as well as their intermediate products. By offering intuitive morphological results with statistics, SEM plays an essential role in the cathode particle quality control process.

In this application note, a time-effective SEM automation method is used on an NCM (nickel cobalt manganese oxide) cathode and its precursors, showcasing how this approach can help cathode materials manufacturers accelerate their quality check (QC) procedures. This automation solution promises to bring major cost savings in manufacturing and raw material by increasing factory productivity.

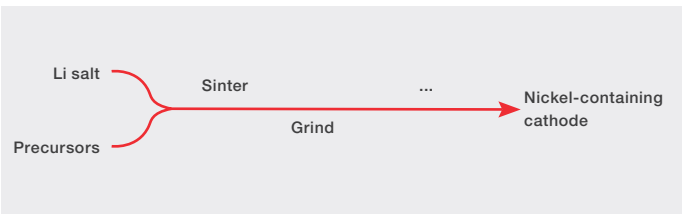

Figure 1. Schematic diagram of the manufacturing process for nickel-containing cathode materials.

Examples of SEM applications in cathode materials QC

Figure 1 shows the production process of an NCM-type cathode powder. NCM cathode materials are sintered after mixing Li salt with additional precursors, which are usually prepared by hydrothermal treatment followed by co-precipitation. After sintering, the agglomerated particles are crushed into the desired granulometry via a grinding step.

QC of NCM cathode precursor particles

The final NCM particle morphology and granulometry depend on the granulometry of the precursor particles as well as the sintering process, which means that it is critical to check the quality of the precursors during their production. Quality engineers are looking for two primary structural characteristics during precursor QC: size distribution and surface structure. Typically, precursors with narrow particle size distributions can be lithiated in less time, leading to better crystallinity. A narrow particle size distribution and a well-defined layer structure also lead to better electrochemical performance.1,2 Figure 2 shows SEM images of precursor particles produced via different synthesis processes. A precursor sample with broad particle size distribution is shown in Figure 2a, ranging from 4.5 to 13.6 µm in diameter. Figure 2b shows a narrow-span-distribution precursor with a porous surface structure. Inset graphs show the particle size distributions as determined by Thermo Scientific™ Phenom™ ParticleMetric Software.

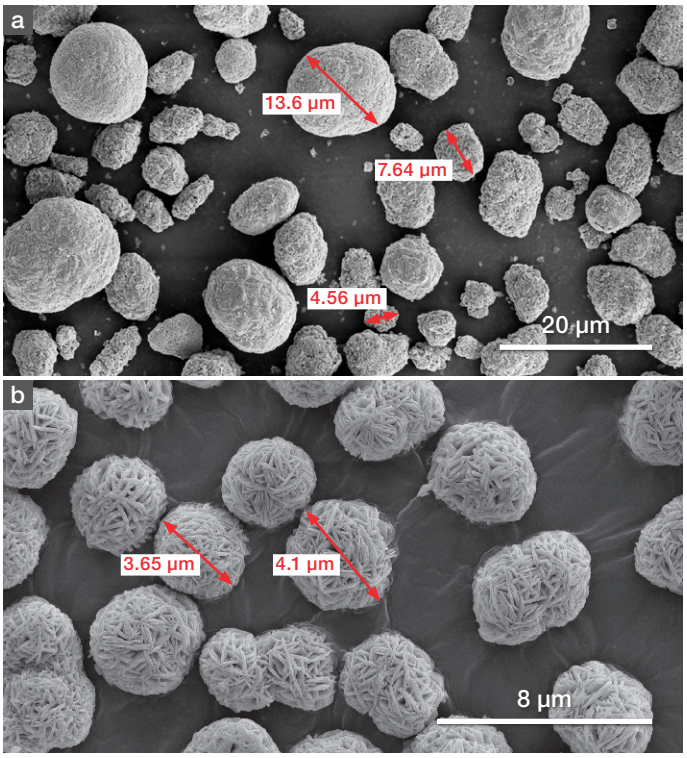

Figure 2. NCM precursors produced under different synthesis conditions. a) Precursor particles with a wide particle size distribution. b) Precursor particles with a narrow distribution and porous structure.

QC of NCM cathode materials

Characterization of primary and secondary particle characteristics is an essential step during QC of the NCM cathode. As shown in Figure 3, NCM cathodes are normally synthesized as spherical polycrystalline particles (called secondary particles) composed of many primary crystals.

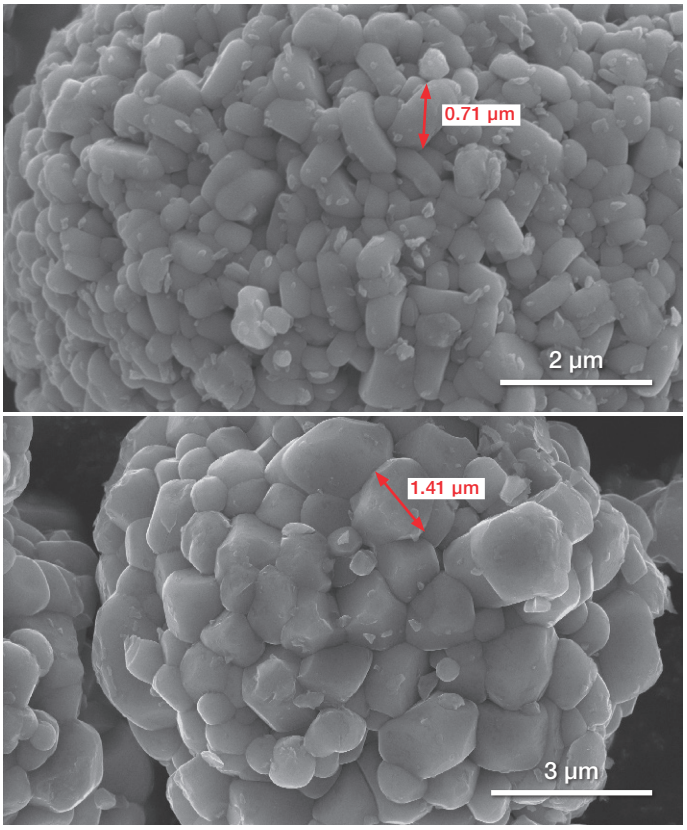

Figure 3. Polycrystalline NCM particles with different primary crystal sizes.

Cracking of secondary particles occurs during charging and discharging of the cathode material, when each primary crystal undergoes intercalation and de-intercalation of lithium ions. The volume of each primary crystal changes during this process, which is the primary cause of particle cracking. Cracked particles exacerbate internal cell side reactions and reduce the battery life cycle, making primary crystal characterization vital for overall NCM analysis.

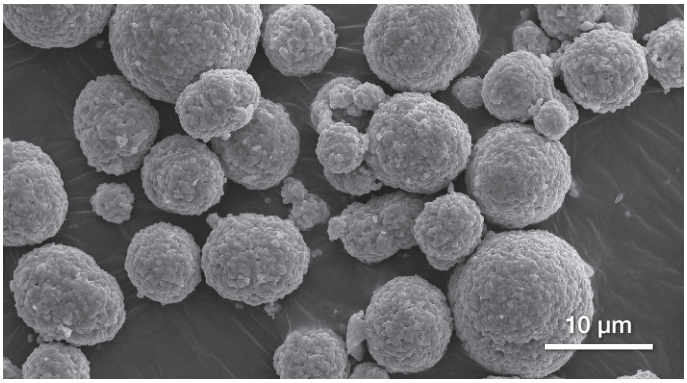

Figure 4. Polycrystalline NCM particles with inset graph showing a broad secondary particle size distribution, as determined by Phenom ParticleMetric Software.

Figure 4 shows NCM particles with a broad secondary particle size distribution, which results in lower tap and energy density. Overall, verifying that the precursor granulometry is within expected values improves the odds that the final cathode powder will be in line with specifications. At the same time, precursors that do not meet the QC criteria can be recuperated and reprocessed, reducing manufacturing costs. SEM provides information on both primary and secondary granulometry, helping manufacturers optimize key parameters during sintering.

After sintering, agglomerated particles are crushed and ground into individual particles.

Figure 5a shows an example of insufficient particle dispersion, whereas Figure 5b shows an instance of excessive crushing, resulting in fragmented particles. Figure 5c shows an example of high particle agglomeration, a result of the increased sintering temperature used to make single-crystal cathode materials. This agglomeration makes the particles more difficult to disperse than polycrystalline materials. Either insufficient dispersion or excessive fragmentation can have negative impacts on the electrochemical properties of the particles due to a lack of homogeneity. SEM can clearly visualize particles after grinding, helping to optimize this process and produce particles of uniform size.

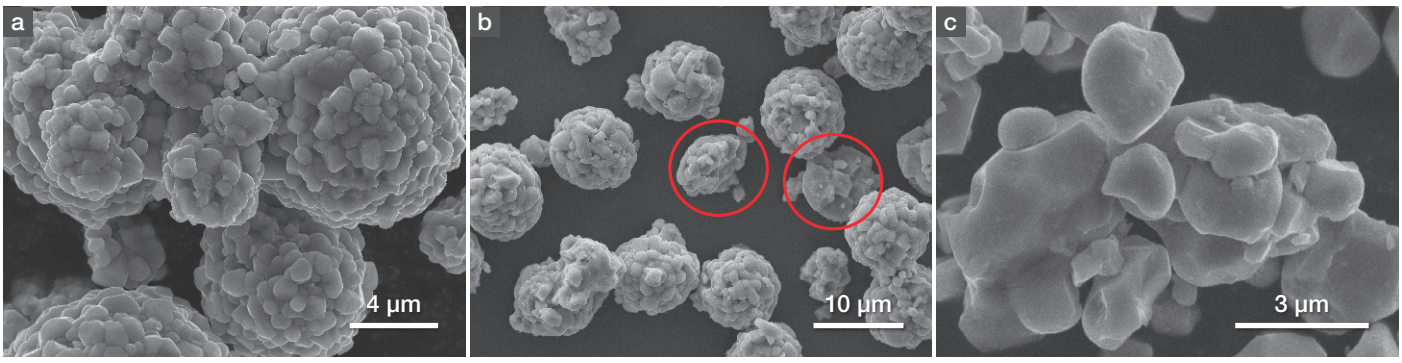

Figure 5. a) Agglomerated polycrystalline particles. b) Fragmented particles. c) Agglomerated signal-crystal particles.

Quality control with SEM

For standard SEM QC, multiple locations in one sample are checked to make sure the results are representative. Typically, SEM images at multiple magnifications are required, with high-magnification SEM images showing detailed microstructures (e.g., layered structures in precursors, primary crystals) while low magnification SEM images show overall particle characteristics (e.g., size, distribution, roundness, etc.). Acquiring these multiple images requires the following steps:

1. Loading the sample
2. Navigating to the desired position
3. Adjusting focus, brightness, contrast, etc.
4. Acquiring images at different magnification
5. Repeating Step 2–4 as needed

A manufacturing facility that produces several tons of material daily can potentially require hundreds of samples to be tested each day. This translates to hours of monotonous, manual work that can be prone to human error due to its repetitive nature.

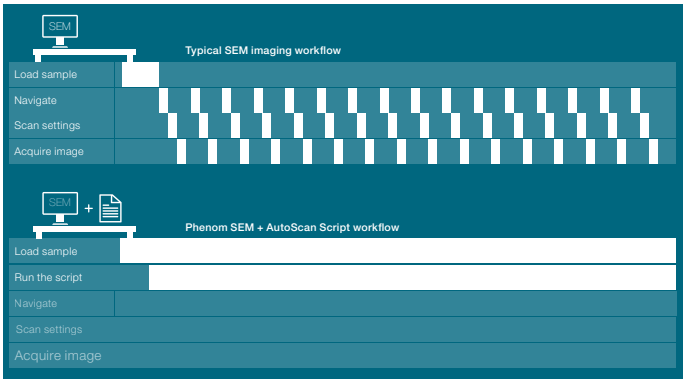

Figure 6. Typical SEM imaging workflow compared to the automated imaging workflow of the Phenom XL Desktop SEM.

Automated imaging workflow

The Thermo Scientific™ Phenom™ XL G2 Desktop SEM offers an automated imaging workflow that uses AutoScan Scripts to acquire data automatically after the sample is loaded. The instrument can hold up to 36 samples at a time, and each can be imaged in several different locations, at different magnifications. This entire procedure can be easily customized.

For instance, a standard quality control for cathode raw materials might require the analysis of 5 different locations on each sample at magnifications of 1k, 5k, and 10k. This would allow for clear observation of both primary and secondary structures. Performed manually for 36 samples, this would require the operator to repeat the steps shown in Figure 6 hundreds of times, potentially requiring 3–4 hours to complete. Automating this process would require only 10 minutes of user input, freeing up valuable time for other tasks. The SEM will run unattended, improving efficiency as the microscope will run more consistently, resulting in higher productivity with less error.

Automated imaging workflows with AutoScan Scripts

The AutoScan Script is based on the Phenom Programing Interface (PPI). With AutoScan Scripts, the Phenom Desktop SEM can acquire images automatically at multiple locations per sample as well as multiple magnifications per location, as specified by the user.

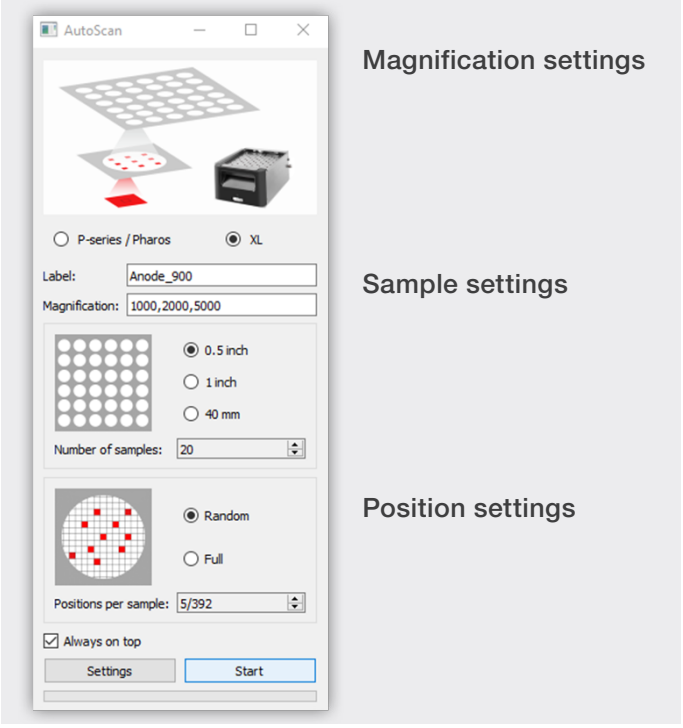

Figure 7. AutoScan Script user interface.

Automated procedures can run 24 hours a day, seven days a week. Automation also improves the overall accessibility of the Phenom Desktop SEM and gives users confidence in their data. Instead of gathering just a few images, users can collect large amounts of data, providing a robust statistical basis for their analysis.

Going beyond imaging with Phenom ParticleMetric Software

To further automate the analysis of particle size distribution, images can be imported directly into Phenom ParticleMetric Software, which can automatically analyze images and calculate particle morphology. Reports are generated immediately after analysis is completed and include various particle properties and statistics.

Figure 8 shows ParticleMetric Software's interface being used to analyze a single-crystal NCM sample. The automated particle size distribution indicated an average particle size of 2 µm.

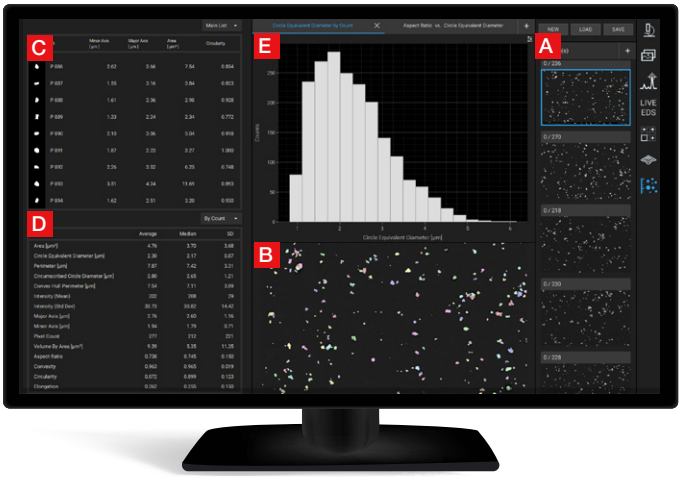

Figure 8. Phenom ParticleMetric Software's user interface showing the analysis of a single-crystal NCM sample. A) List of all images used in this project. B) Identified, colorized particles. C) List of identified particles. D) Calculated statistics for all particles. E) Custom user-defined graphs can be used to visualize the data.

Conclusions

In this application note, the role of SEM in the QC of cathode materials is shown. The automated imaging workflow available with the Phenom XL G2 Desktop SEM highlights how automated image acquisition and analysis can greatly speed up the QC process, reducing production costs and increasing productivity.

- The Phenom XL G2 Desktop SEM, combined with AutoScan Scripts, can be used to acquire numerous SEM images automatically.
- SEM images are analyzed in ParticleMetric Software, visualizing critical particle properties.
- Automated SEM imaging workflows can be applied to the QC of other raw materials used in battery production.

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3. Langdon, Jayse, and Arumugam Manthiram. "A perspective on single-crystal layered oxide cathodes for lithium-ion batteries." Energy Storage Materials 37, 143-160 (2021)

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