APPLICATION NOTE

Assessment of contaminants within battery materials via Axia ChemiSEM

Introduction

Contamination is a major issue in the battery manufacturing process. From production of the cathode, anode, and battery cell to battery module assembly and testing, contaminants are a concern at every stage of the process. The existence of contaminants in the battery can cause a wide range of problems—lowering materials usage efficiency, accelerating cell degradation, and even causing internal shorts. As a result, it is essential for materials scientists to obtain a thorough understanding of the contaminants that enter the battery manufacturing process.

The combination of a scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS) can be used to probe both structure and elemental information of contaminants in battery materials. However, battery contaminants generally have a low concentration level, and image acquisition times can be long when studying these contaminants using conventional EDS. In this application note, we introduce a fast and simple method for characterizing these contaminants via the Thermo Scientific™ Axia™ ChemiSEM, a new SEM platform designed to bring speed and simplicity to materials micro-structural analysis and defect discovery.
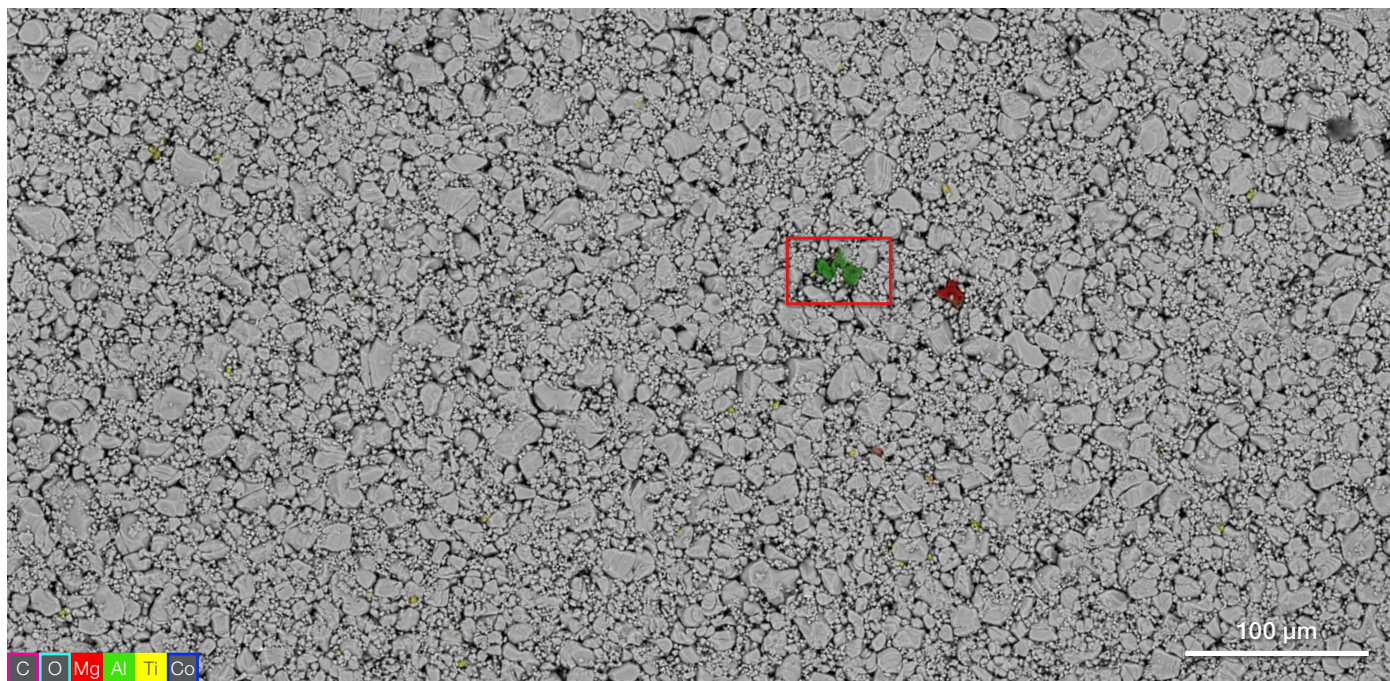

Figure 1. Large-scale navigation montage image obtained by collecting neighboring frames to generate a low magnification image for point-and-click navigation. 750 μm x 370 μm. Acquisition parameters: acc voltage 20 keV, beam current 0.13 uA.

Large-scale navigation

With the Axia ChemiSEM, identification of the presence of contaminants can be rapidly achieved within a large field of view—thanks to the system's full integration of different imaging modalities including live-quantitative elemental mapping and conventional SEM imaging. The image in Figure 1 shows a navigation image acquired on the surface of a lithium cobalt oxide (LiCoO$_2$) cathode via Axia ChemiSEM.

This large-scale overview of the sample with EDS data included is collected within 15 minutes. Traditionally, a grayscale image with morphological information and contrast based on backscattered electron signal cannot provide enough information to identify regions of interest for contamination analysis. The main reason for this limitation is that a backscattered electron image only provides a compositional contrast based on the atomic number, and oftentimes the compositional contrast between two different elements is too similar to be observed in grayscale.

With the quantitative elemental information provided by Axia ChemiSEM, however, the large-scale overview already shows some foreign elements, such as magnesium (Mg), aluminum (Al), and titanium (Ti), as well as their position. The large-scale overview serves as a navigation image that can be used to easily move to the region of interest where the contaminants are present to run a more detailed characterization.

Combined SEM-EDS analysis

Using the navigation image as a reference, the user can simply click on the point of interest to drive the stage to it. This process significantly decreases time-to-data for each suspected contaminant. To further demonstrate this ability to hone in on specific regions, the characterization of one area of interest highlighted in Figure 1 is presented in Figure 2. Using conventional SEM imaging, the backscattered electron image, which offers a first level of feedback on the composition, does not provide enough information to identify the contaminant.

By contrast, Axia ChemiSEM provides near instant access to quantitative elemental information every time a grayscale image is acquired since X-ray detection is always on. X-rays are acquired and processed in the background during the acquisition of the grayscale image to obtain quantitative elemental information, different from the raw signal usually acquired from a traditional EDS gross counts mapping analysis. This constant access to elemental information translates to a seamless characterization experience where no time is wasted waiting for data.

The quantitative elemental map presented in Figure 2 was collected simultaneously during the conventional SEM image acquisition. To view this result, the user simply needs to activate the quantitative elemental view. There is no need to re-acquire the data as one would need do with a conventional EDS system.
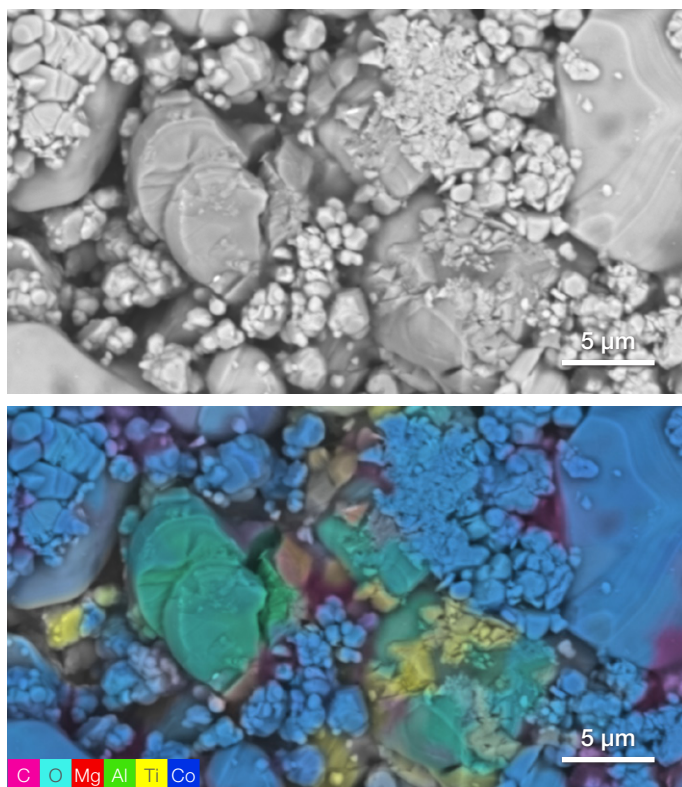

Figure 2. Traditional backscattered electron image (top) of the region of interest and quantitative elemental mapping (bottom) obtained with an acquisition of 80 seconds. (Acquisition parameters: acc voltage 10 keV, beam current 0.76 nA, dwell time 5 us).
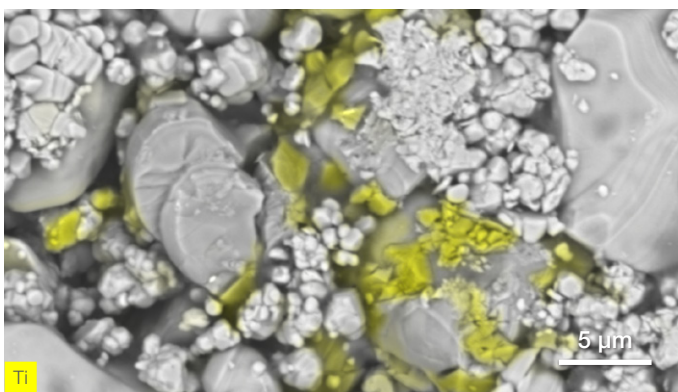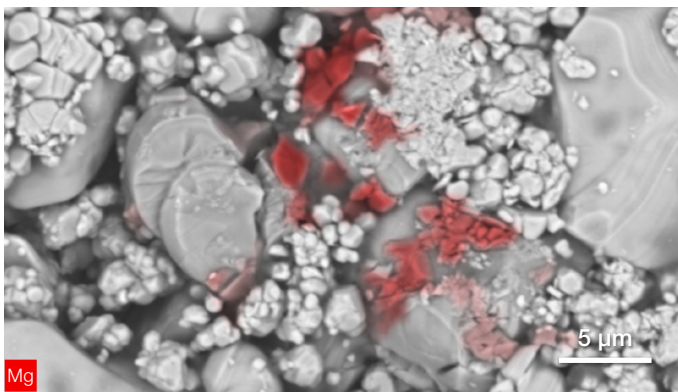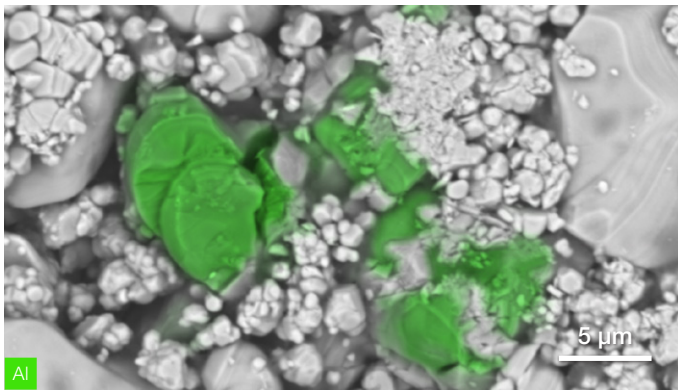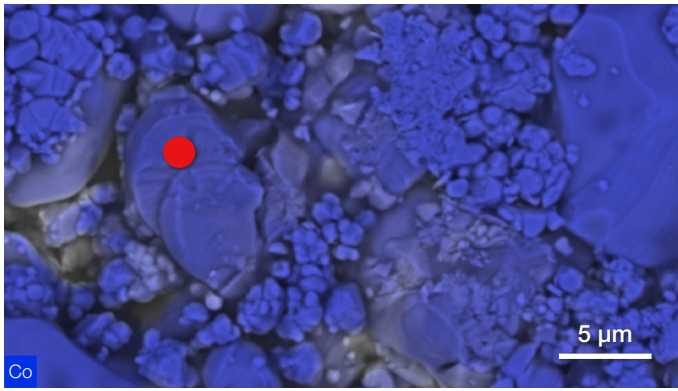
Figure 3. The first image on the top shows the distribution of the cobalt (Co) which is part of the battery matrix. The other three images highlight the distribution of aluminum (Al), magnesium (Mg) and titanium (Ti) contamination, respectively. The red dot in the top image shows the location where further analysis will be performed.

To obtain a better view of the distribution of each element, the user can generate a complete set of images highlighting one element at a time, as shown in Figure 3. Besides Co, the main element from $LiCoO_2$, the results also show the presence of the elements Al, Mg, and Ti within the electrode, which are unexpected. These contaminants could have been introduced during the cathode materials synthesis, mixing, or coating processes as the battery was manufactured. The user stays focused on searching for the odd elements, which easily stand out, leading to a more intuitive and accurate discovery process.

Point analyses

To further identify these contaminants, point analyses have been executed to obtain the exact quantification of the elements present in the contamination with the focus on Al as an example. All of the conventional EDS functions are fully integrated into the Axia ChemiSEM user interface with no need to switch to a different software. (The location of the point analyzed is shown with a red dot in the first image from the top of Figure 3.)
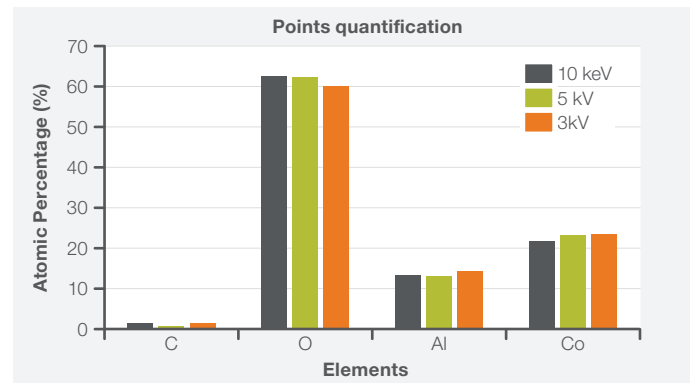

Figure 4. Contamination composition quantified at different acquisition conditions (10 keV and 0.76 nA, 5 keV and 0.28 nA, 3 keV and 0.16 nA).

In order to exclude the interaction volume effects on the quantitative results of the contaminant's composition, both accelerating voltage and beam current have been reduced and the same point analysis has been performed. Figure 4 compares the analysis of that point as a function of acceleration voltage.

Axia ChemiSEM's enhanced graphical user interface provides automated system alignments, allowing the user to change the analysis parameters without manual adjustments for fast and easy analyses. In short order, point analyses using three different characterization conditions have been acquired, lowering the accelerating voltage up to the minimum required to identify the contaminant's composition and excite the Al k-line (1.4866 keV), as shown in Figure 4. The obtained results are comparable, regardless of accelerating voltage.

The fact that the contaminant's composition remains consistent proves that a certain amount of Al is embedded in the $LiCoO_2$, which likely means that the Al element was reacting with the precursor used to synthesize $LiCoO_2$ during the sintering process. As a result of this analysis, the researcher would be able to determine that the raw materials for $LiCoO_2$ synthesis or the equipment involved in the sintering process need to be carefully examined to eliminate this contaminant.

Conclusion

A thorough assessment of the contaminants within battery materials is critical to assuring battery quality and performance. Using the Thermo Scientific Axia ChemiSEM, large-scale SEM-EDS mapping was employed to quickly and easily identify contaminants and then move directly into detailed quantitative EDS analysis to more precisely pinpoint the distribution of each contaminant. Point analyses were then performed to obtain the exact quantification of each element present in the contamination.

With the Axia ChemiSEM, SEM imaging and EDS are no longer separate workflows, but an integrated process designed to rapidly move from discovery to analysis that generates accurate results. Live EDS technology together with the Axia ChemiSEM's automatic alignment function allows for a smooth user experience and efficient characterization of the battery electrode. Using the Axia ChemiSEM for fast and simple SEM-EDS contaminant analysis, battery manufacturers can improve the efficiency of their research—reducing contaminants during the manufacturing process and improving battery performance.

Find out more at **thermofisher.com/Axia-ChemiSEM**