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APPLICATION NOTE

Strategies for accurate imaging on battery separator structure

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Introduction

As one of the major components in a Li-ion battery, the battery separator plays a key role in preventing physical contact between electrodes as well as facilitating the ion transport within the cell [1]. Because the battery separator structure is closely correlated with its performance, an in-depth understanding of its structure is essential [2]. 2D and 3D imaging are effective approaches to characterize the separator structure; however, due to the intrinsic beam sensitivity of the separator, appropriate imaging techniques are needed to accurately characterize its structure [3-4]. In this application note, strategies for using scanning electron microscopy (SEM) and DualBeam™ technology, also known as focused ion beam-scanning electron microscopy (FIB-SEM), to image the battery separator structure are presented.

Discussion and Results

A commercial polypropylene (PP) separator was used for SEM imaging. The accelerating voltage effects on imaging are explored on the Thermo Scientific™ Apreo SEM (Figure 1). At a 1.5 keV acceleration voltage, clear beam damage effects are observed on the PP separator, where the polymer shows melting and distortion of the pore shapes. Decreasing the voltage to 100 eV minimizes the beam damage and maintains pore structure and features on the polymer. This result indicates the importance of optimizing accelerating voltage in imaging the battery separator. Low-energy imaging is recommended to preserve the separator structure for accurate characterization.

Besides characterizing the separator surface morphology, the cross-section of the separator is also of interest for understanding the structure of the component. Broad ion beam (BIB) polishing under cryogenic temperature is a well-accepted method for 2D cross-section preparation of separator components. Sample preparation to final image collection usually takes 6–8 hours per sample. Compared to BIB polishing, the DualBeam instrument performs both milling and imaging at the same location, which reduces the sample transfer steps. In addition, since the focused ion beam can effectively cut a separator cross-section with tens-of-micron width as a representative sample, it opens up fast access to the separator cross-section within an hour.

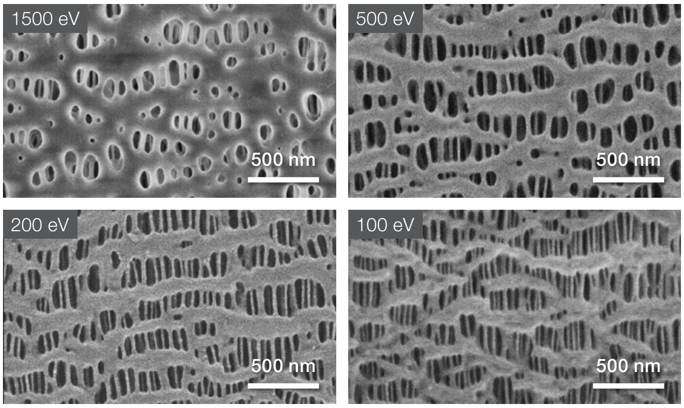

Figure 1: Accelerating voltage effects on battery separator morphology.

Figure 2 shows the ceramic-coated composite separator cross-section prepared at different temperatures using the Thermo Scientific Scios™ 2 DualBeam. The cross-section preparation at room temperature shows severe ion beam damage during the FIB milling process (Figure 2 (a)). The entire polymer structure is deformed, and there is a delamination between the ceramic layer and polymer layer on the edge. Figure 2 (b) and (e) shows the separator milled and imaged at -80°C via cryo-FIB milling. At this temperature, the separator structure is well maintained, and the contrast among different phases is clearly visible. However, when decreasing the cryo FIB-milling temperature to -180°C, inferior image quality due to redeposition on the cross-section has been observed, where the contrasts among phases is no longer clear.

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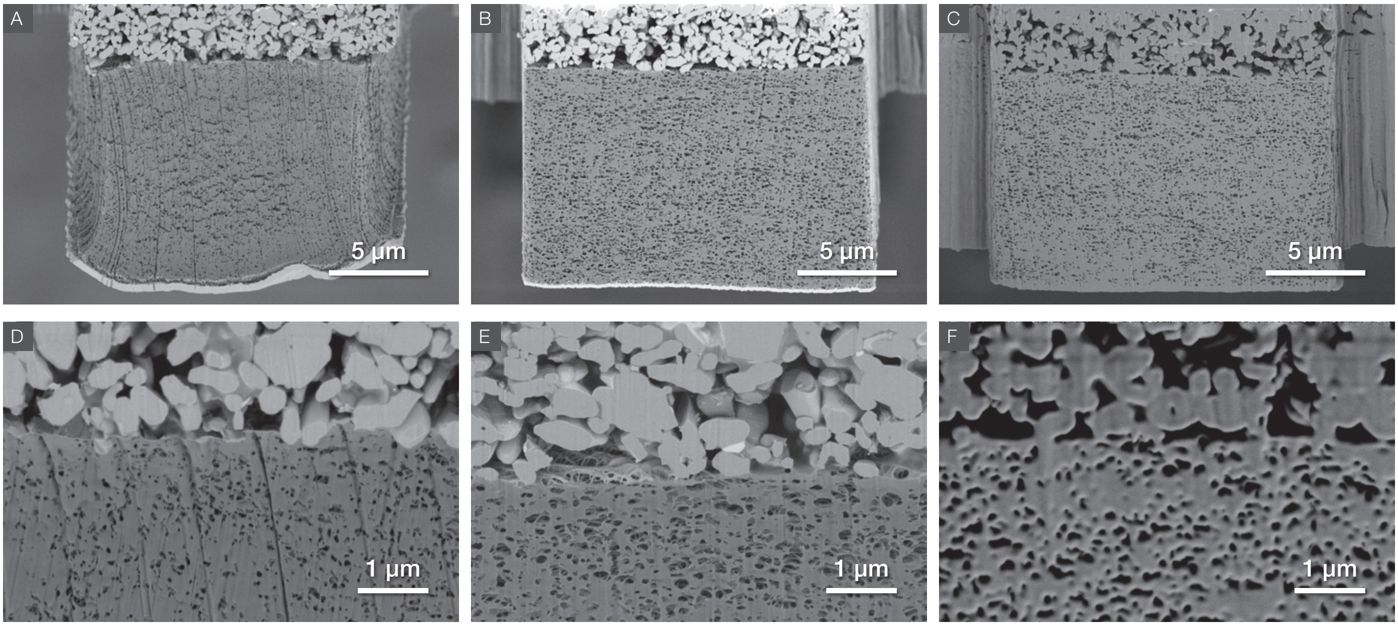

Figure 2: FIB cross-section preparation at different temperature (a) and (d) room temperature; (b) and (e) -80°C; (c) and (f) -180°C.

These results show the critical role of cryo-FIB milling for preparing separator sample cross-sections and defining the optimized temperature range for best imaging quality.

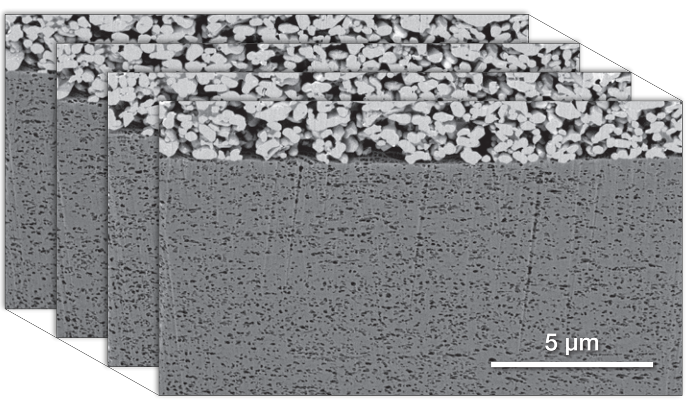

Figure 3: A series of 2D cross-section images collected via Auto Slice and View 4 Software for 3D reconstruction and analysis.

In addition to 2D cross-section imaging, the cryo-DualBeam can perform 3D imaging analysis via FIB serial sectioning tomography (FIB-SST). Figure 3 shows a stack of 2D images automatedly acquired via Thermo Scientific Auto Slice and View™ 4 Software for 3D data analysis. The 3D analysis on this data set allows for further extraction of key microstructural parameters such as closed/open pore volume fraction, pore connectivity, and tortuosity for separator transport property analysis.

Conclusion

The intrinsic beam sensitivity of the battery separator causes characterization challenges for the electron microscope; however, these challenges can be overcome by employing strategies such as low-energy imaging and cryo-FIB milling to accurately characterize the structure of the battery separator. The as-developed strategy for separator imaging will help scientists and engineers to make battery separator with safer feature and higher performance to enable better batteries.

Reference

[1] P. Arora, J. Zhang et al. Chemical Reviews, 2004; 104, 10, 4419-4462
[2] N. P. Deng, et al, Journal of Power Sources, 2016, 331, 132-155
[3] M. F. Lagadec, et al. Nature Energy, 2019, 4, 16-25
[4] M. F. Lagadec, et al. Journal of Electrochemical Society, 2016, 163 (6) A992 – A994

Key words
Li-ion battery, Separator, SEM, DualBeam, Cryo-FIB, 3D imaging, FIB-SST, SST, Serial sectioning tomography

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