BNL-112534-2016-JA

File # 93466

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Submitted to Microscopy and Microanalysis

February 2016

Photon Sciences Department

Brookhaven National Laboratory

**U.S. Department of Energy
USDOE Office of Science (SC),
Basic Energy Sciences (BES) (SC-22)**

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Three-Phase 3D Reconstruction of a LiCoO₂ Cathode via FIB-SEM Tomography

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Abstract:

Three-phase three-dimensional (3D) microstructural reconstructions of lithium-ion battery electrodes are critical input for 3D simulations of electrode lithiation/delithiation, which provide a detailed understanding of battery operation. In this report, 3D images of a LiCoO₂ electrode are achieved using focused ion beam-scanning electron microscopy (FIB-SEM), with clear contrast among the three phases: LiCoO₂ particles, carbonaceous phases (carbon and binder) and the electrolyte space. The good contrast was achieved by utilizing an improved FIB-SEM sample preparation method that combined infiltration of the electrolyte space with a low-viscosity silicone resin and triple ion-beam polishing. Morphological parameters quantified include phase volume fraction, surface area, feature size distribution, connectivity, and tortuosity. Electrolyte tortuosity was determined using two different geometric calculations that were in good agreement. The electrolyte tortuosity distribution versus position within the electrode was found to be highly inhomogeneous; this will lead to inhomogeneous electrode lithiation/delithiation at high C-rates that could potentially cause battery degradation.

Keywords: Li-ion batteries, three-phase 3D reconstruction, FIB-SEM tomography, cathode, microstructure

1. Introduction

Lithium-ion battery electrode performance depends not only on chemistry but on microstructure, such that an improved understanding of the electrode microstructure-performance relationship is needed (Fergus, 2010; J. Vetter, et al., 2005). Three-dimensional (3D) characterization methods such as focused ion beam – scanning electron microscopy (FIB-SEM) and X-ray computed micro/nanotomography (XCT) have gained significant attention in the past few years due to their advantages in effectively quantifying electrode microstructural parameters including grain size, phase volume, surface area, phase connectivity, and tortuosity (Babu, et al., 2015; Chen-Wiegart, et al., 2013; Cooper, et al., 2014; Ebner, et al., 2013b; Ebner, et al., 2013c; Ender, et al., 2012; Ender, et al., 2011; Hutzenlaub, et al., 2012; Liu, et al., 2013; Shearing, et al., 2010; Wang, et al., 2014; Wilson, et al., 2011; Wu & Jiang, 2013). Moreover, measured 3D electrode microstructures can be used as input for 3D model simulations of lithiation/delithiation (A. H. Wiedemann, et al., 2013; Hutzenlaub, et al., 2014; Malavé, et al., 2014; Yan, et al., 2013), providing more detailed information and more accurate predictions of electrode electrochemical processes compared with well-known low-dimensional models (e.g. Newman-type models (M. Doyle & Newman, 1995; M. Doyle, et al., 1993)) or 3D models based on ideal spherical particles (Goldin, et al., 2012).

Typical Li-ion battery electrodes contain three phases with different functions. The active material acts as reservoir for lithiation/delithiation; the electrolyte-filled pores serve as Li-ion pathways, while the carbon and binder (CB) phase provides electronic conductivity. Thus, the ability to resolve all three phases in 3D microstructure reconstruction is critical for accurately analyzing electrode processes (Hutzenlaub, et al., 2014; Zielke, et al., 2014a). In initial reports describing 3D battery electrode microstructure, the active oxide particles were resolved but the electrolyte and carbon/binder phases could not be separated since the electrolyte space was filled with a carbon-based epoxy during imaging (Liu, et al., 2013; Wilson, et al., 2011). Recently, a few methods have

been proposed for three-phase reconstructions, but each has limitations during data acquisition and processing (Babu, et al., 2015; Chen-Wiegart, et al., 2013; Ender, et al., 2012; Ender, et al., 2011; Hutzenlaub, et al., 2012). In one case using X-ray computed tomography, the three phases were resolved via Zernike phase contrast imaging (Babu, et al., 2015; Chen-Wiegart, et al., 2013). However, such data are hard to segment due to the low contrast between each phase, thus limiting quantitative analysis. For FIB-SEM tomography, there are two approaches for three-phase reconstruction to date. Hutzenlaub et al. performed serial-sectioning directly on the electrode without using any filling materials (Hutzenlaub, et al., 2012). This technique is an efficient method in terms of sample preparation and phase contrast is observable between empty pores and surrounding materials. However, severe electron charging on the FIB-milled surfaces, together with curtaining effects caused by the rough top surface, can induce image artifacts, and hence, require significant post-processing effort in manual segmentation. The alternative method is to infiltrate filling materials into the porous electrode for contrast enhancement. Instead of using carbon-based epoxy resin, which does not allow differentiation between resin-filled pores and CB phase, silicone resin is employed to obtain considerable atomic Z-contrast (Ender, et al., 2012; Ender, et al., 2011). This method effectively avoids the overcharging and curtaining effects. However, attempts to use this relatively high viscosity silicone resin with the present low-porosity electrodes always led to incomplete pore infiltration. In addition, the overlap between carbon and resin gray scale frequency peaks may cause uncertainty during image segmentation. Therefore, a method that incorporates the advantages of both FIB methods, namely, clear phase contrast with minimal artifacts, is needed for acquiring high-quality three-phase 3D data sets.

In the present work, an improved method was developed utilizing a low viscosity silicone resin as a filling material and three-phase 3D reconstruction was demonstrated via FIB-SEM on a commercial LiCoO_2 cathode. The low viscosity resin enables full infiltration of low porosity electrodes and

provides good contrast among phases, the latter of which allows for automatic segmentation. However, it is difficult to mechanically polish samples filled with such a low viscosity material. Thus, a straightforward sample preparation method using a triple ion-beam cutter was developed to guarantee a smooth top surface, minimizing image artifacts during data collection. Microstructural parameters such as phase volume fraction, surface area density, and feature size distribution were extracted from the as-obtained 3D reconstruction. In addition, electrolyte connectivity, tortuosity, and tortuosity distribution (tortuosity along the planar section within the electrode) were determined. The results are quite different than those obtained when the electrolyte and carbon phases are not resolved, confirming the importance of performing full three-phase imaging of battery electrodes.

2. Materials and Methods

2.1 *Sample preparation for FIB-SEM tomography*

In present report, a LiCoO_2 cathode from a commercial cylindrical 18650 energy cell (Molicel ICR 18650J, Taiwan) was investigated. In order to obtain clear contrast between carbonaceous materials (including the conducting carbon and binder, CB) and electrolyte space, a commercial two-part silicone resin (ELASTOSIL RT 604, Wacker, Germany) was used as filling material to infiltrate the pore regions remaining after the electrolyte was removed from the cathode. It is noteworthy that one of the biggest advantages of using RT 604 is its low viscosity (800 mPa•s), compared to RT675 viscosity (50,000 mPa•s) used in a previous report (Ender, et al., 2012), which allows the resin to fully penetrate the entire open pore space. This is critical when infiltrating electrode samples with small pore sizes. The pristine cell was opened in an argon-filled glove box. After separating the LiCoO_2 cathode from the carbon anode and separator, the cathode was rinsed with dimethyl carbonate (DMC) to remove the electrolyte as well as any salt residue. The rinsed cathode was then slowly dried in a glove box for two days and cut into a $1 \times 1 \text{ cm}^2$ sample with a razor blade for FIB-SEM tomography. The low viscosity silicone resin was used for filling the porosity in the electrodes.

The infiltration process was performed at vacuum levels lower than ~50 mbar to further improve infiltration. After curing the resin for 24 h at room temperature, samples were roughly cut into cubes before further polishing. Because the cured silicone resin is too soft for mechanical polishing (Hardness Shore A 25, ISO 868), the triple ion-beam cutter (Leica EM TIC 3X) was used to obtain a smooth top surface required for the subsequent FIB milling. During the polishing process via the triple ion-beam cutter, each ion gun was operated at 4 KV accelerating voltage and 2 mA ion current for 8 hours of milling. Figure 1 (a) shows the schematic view of polishing the silicone resin infiltrated cathode sample via the triple ion-beam cutter. The sample was mounted ~50 μm above the mask to expose enough space for milling. Triple ion-beams formed a ~100° wide milling section after cutting the exposed sample and created a smooth region, as shown in Figure 1 (b), magnified further in Figure 1 (c). Based on these images, the electrode is estimated to be ~ 60 μm thick and the aluminum current collector is ~20 μm thick. It is clear that the top surface satisfied the requirement on the surface smoothness, which effectively avoided most of the curtaining effect during the subsequent FIB milling. After obtaining a smooth top surface with the triple ion-beam cutter, a ~30 nm osmium coating was deposited on the sample to minimize charging during FIB-SEM data collection.

2.2 FIB-SEM Tomography

An FEI Helios FIB-SEM (FEI Company, OR, USA) was used for serial sectioning and data collection. A trench (50 x 30 x 15 μm^3) was first milled to expose side walls for serial-sectioning (trench not shown). The pixel size of the SEM images is 31.25 nm, while the slice thickness is 150 nm. A through-the-lens detector (TLD) was used to collect the backscattered electron signal so as to obtain atomic Z-contrast among phases. As indicated in Figure 1 (c), a typical serial-section area of 50 μm x 45 μm was selected; the slicing direction was parallel to the current collector. Recognizing the spatial location of the current collector relative to the as-obtained 3D electrode structure is useful

in characterizing directional connectivity and tortuosity. In the present case, the current collector is adjacent to the left YZ plane of the 3D data set. Overall, 268 consecutive images were collected. After data collection, the 2D image sequences were aligned, cropped and stacked into a 3D microstructure using a method previously described (J. R. Kremer, et al., 1996; J. R. Wilson, et al., 2006). A total 3D volume of $\sim 35,000 \mu\text{m}^3$ was reconstructed for further analysis.

2.3 *Image Processing:*

In order to quantitatively analyze the 3D reconstructed volume, image segmentation was needed to attribute different gray scale intensity values to different phases. In the current case, white (gray scale intensity: 255) is assigned to LiCoO_2 particles, with gray (127) to resin-infiltrated porosity and black (0) to carbon and binder. Firstly, ImageJ software (National Institutes of Health, MD, USA) was used (Abramoff, et al., 2004) in which a combination of gray scale histogram equalization, background subtraction, and median filtering was performed to alleviate the shadowing of the FIB-milled trench wall and any curtaining effects. Then a multi-level image threshold method, Otsu's method (Otsu, 1979), was applied to segment a three-phase image. In brief, the as-collected SEM image was assumed to contain three classes of pixels that follow tri-modal distribution. The threshold value is then determined by finding maximum inter-class variance between CB/electrolyte and electrolyte/ LCO phases. Finally, the noise filter built in ImageJ was used on images that needed further noise reduction. Once segmented, the images were imported into Amira 5.5.0 (FEI Visualization Sciences Group, MA) for 3D visualization.

2.4 *Microstructural Quantification*

Microstructural parameters including volume fraction, surface area density, feature size distribution, connectivity, and tortuosity (τ) were calculated to quantitatively evaluate the electrode microstructure characteristics (Chen-Wiegart, et al., 2014; J. R. Wilson, et al., 2006; Liu, et al., 2013; Münch &

Holzer, 2008; Shanti, et al., 2014). Phase volume fraction (V_f) was calculated by counting voxels of each phase within the 3D data set. For surface area, both interfacial surface area density (SA_I) and specific surface area density (SA_s), also known as the volume specific surface area, were calculated using a built-in function in IDL software (EXELIS, CO, USA). The SA_I is determined by normalizing the surface area of each phase with total reconstruction volume, while SA_s is calculated by dividing the surface area of each phase by the volume of corresponding phase. Continuous feature size distributions of all three phases were calculated using the method introduced by Münch and Holzer (Münch & Holzer, 2008). This algorithm measures the particle size by filling the 3D volume with a sphere of a given radius. The cumulative size distribution is calculated by incrementally decreasing the sphere radius, and thus, filling increasingly larger volumes until the volume of interest is fully occupied. The size distribution of the analyzed volume can also be calculated by taking the derivative of the cumulative size distribution. This algorithm was implemented via a lab-made MATLAB code.

Connectivity and tortuosity are two important parameters that represent transport properties of the electrode, and realistic values of these can only be measured after resolving contrast between carbonaceous materials and electrolyte regions. The connectivity was determined using the function “bwlabeln” in MATLAB software (Mathworks, MA, USA), which defines the regions connected to the opposite face of the current collector (current collector is touching the YZ plane on the left) as “Percolated”. Those regions which touch other faces but not the percolating volume (not the opposite face of current collector) are labeled as “Unknown”. Finally, regions that are completely isolated from the percolating and unknown regions are “Isolated”. By segmenting the electrolyte phase with this definition, the directional connectivity towards the current collector was calculated via dividing the segmented phases by total electrolyte volume.

For tortuosity calculations, two different methods were used, namely the path length ratio method (PLR) (Shanti, et al., 2014) and the distance propagation method (DP) (Chen-Wiegart, et al., 2014). (Schematics of the two algorithms are shown in supplemental information, Figure S1 and S2). The calculations are limited to the interconnected regions only. Both methods are based on the geometrical calculations of the effective actual path (L_{eff}) and the Euclidean distance (straight distance, L) ratio using the simple geometrical definition of tortuosity, τ :

$$\tau = \frac{L_{eff}}{L} \quad (1)$$

For the PLR method, the 3D microstructure of the phase of interest was skeletonized into a matrix containing nodes and pathways that connect neighboring nodes. L is determined by calculating the straight distance between randomly selected node pairs, and L_{eff} is determined using Dijkstra's algorithm (Dijkstra, 1959). In this report, five sets of 1000 nodes per set were selected to measure τ and its standard deviation. In contrast, using the DP method, a propagation distance map was first generated within the phase of interest starting from the “seed plane”. After labeling all voxels by defining specific neighbors (the city-block method was used in current report), the correlations between L_{eff} and L are then built for extracting τ based on the “average” method (Chen-Wiegart, et al., 2014). It is noted that both geometrical methods are less computationally intensive than the well-developed diffusion method (J. R. Wilson, et al., 2006), while still obtaining comparable tortuosity values.

3. Results and Discussion

3.1 *Three-phase 3D reconstruction*

Figure 2 summarizes images, chemical identification, and segmentation of the data from the LiCoO_2 electrode. A typical 2D cross-section from the 3D data set is shown in Figure 2 (a). With the smooth top surface created by triple ion-beam cutter, the curtaining effect is much reduced compared to samples without any filling materials (Hutzenlaub, et al., 2012). The three phases demonstrated clear

contrast, namely, white (LiCoO_2 active materials), gray (infiltrated silicone resin), and black (carbonaceous materials including conductive carbon and binder (CB)). Note that the resin-infiltrated porosity corresponds to the regions that were occupied by the liquid electrolyte, prior to its removal upon battery disassembly; hence this region will be referred to as either silicone resin or electrolyte in the following text. No nano-porosity was observed within the CB phase in these images collected at ~30 nm resolution. Nanoporosity was observed within the CB by utilizing a combination of a multi-length scale imaging technique and 3D modeling (Stephenson, et al., 2011; Zielke, et al., 2014a; Zielke, et al., 2014b). However, the emphasis of the present 3D reconstructions was to measure a large total volume in order to obtain excellent statistics in the larger-scale microstructure quantification, rather than to resolve nano-scale features.

The energy dispersive X-ray spectroscopy (EDS) mapping (Figure 2 (b)) was performed using the Integrated Oxford EDS system (equipped on FEI Helios FIB-SEM) to confirm the microstructural chemical specificity of each phase. The signals from Co, C and Si elements ~~correspond well with~~ substantiated the above assignments of the LiCoO_2 (white), CB (black), and silicone resin (gray), respectively. A gray scale histogram of the highlighted region in Figure 2(a) is presented as Figure 2(c). The histogram with three well-separated frequency peaks is ideal for using Otsu's multi-level method to assign each phase with a specific gray scale intensity value, where white (gray scale intensity: 255) is assigned to LiCoO_2 particles, gray (127) to silicone resin and black (0) to CB.; the corresponding segmented image is shown in Figure 2 (d). The benefits of applying automatic segmentation in processing this data set are two-fold. First, it reduces the random errors introduced by manual segmentation and provides consistency to the entire image processing procedure. Second, the time required for the automatic segmentation process can be efficiently reduced to a several hours instead of days for manual segmentation.

Figure 3 shows the reconstructed 3D volume after segmenting the whole image volume and stacking 2D image slices. Microstructural parameter results, including volume fraction and surface area of each phase, are compiled in Table 1. It is apparent that the active LiCoO_2 material occupies most of the reconstructed volume, while the carbon phases have a relatively low volume fraction. In general, active materials are expected to be more than 70% of the volume in high-energy Li-ion 18650 cells to achieve high energy density within a single cell (Ender, et al., 2014). In addition, the volume fractions of CB (10.3%) and electrolyte (12.7%) satisfy the requirements for electron and Li-ion transport in this product, where the highest C-rate (1 hour for full charge/discharge) recommended is limited to 1C. The electrolyte volume fraction in such high-energy Li-ion cells is significantly lower than those of high power Li-ion cells where the electrolyte can occupy up to 38% of the volume to allow fast Li-ion transport (Ender, et al., 2012; Ender, et al., 2014).

One of the benefits of resolving all three electrode phases is that the interfacial surface areas between each phase can be accurately determined. If the electrolyte is assumed to only allow for Li^+ transport while the carbon-binder phase only permits electron transport, partial coverage of LiCoO_2 by carbon and binder can effectively enhance the electron transport while blocking the Li^+ reaction sites on the oxide particles, which can cause high transport loss (Hutzenlaub, et al., 2014). In the present case, the actual available oxide surface area density is $0.36 \mu\text{m}^{-1}$, but in cases where carbon was not differentiated as in prior 3D imaging (Liu, et al., 2013; Wilson, et al., 2011), the apparent oxide surface area density (the sum of electrolyte/LCO and CB/LCO interfacial areas) for the current data set, is nearly two times higher, $0.71 \mu\text{m}^{-1}$. Consequently, not considering CB coverage on active materials may lead to significant errors in simulating electrode properties. For example, the CB coverage presumably impedes the Li^+ charge transfer process by reducing the oxide-electrolyte interfacial area (Malavé, et al., 2014). This may result in an underestimation of the stress and local Li-ion concentration gradient within particles. In addition, the interface between active materials and

CB also plays an important role in generating surface heat during the battery cycling process (Yan, et al., 2013).

The specific surface area densities (SA_s) were also calculated. The largest SA_s among three phases is CB, while $LiCoO_2$ is the smallest, i.e., the CB phase possesses the smallest average feature size and $LiCoO_2$ the largest, which is also evident in the 3D reconstruction shown in Figure 3. The feature size distributions, shown in Figure 4, provide a more complete picture of feature sizes. The volume weighted feature size for $LiCoO_2$, CB and electrolyte are 2.94 μm , 0.64 μm and 0.93 μm , respectively. That is, the median feature size of $LiCoO_2$ is larger than that of electrolyte and CB, consistent with the SA_s calculations.

2.3 Connectivity and Tortuosity Calculations

Taking advantage of the capability to resolve carbonaceous materials and silicone resin enables the 3D reconstruction of an electrolyte network, important for simulating Li^+ transport to oxide particles. The directional connectivity and tortuosity of the electrolyte phase were determined from the 3D data. The connectivity of the percolated electrolyte volume toward the current collector is 97.7% with 1.3% unknown and 1% isolated regions (Figure S3, supplemental information). Considering the errors associated with data processing, the result indicates that there is no isolated electrolyte within experimental error.

Figure 5 shows electrolyte tortuosity values calculated via the distance propagation (DP) and path length ratio (PLR) methods (Chen-Wiegart, et al., 2014; Shanti, et al., 2014). Results from the DP and PLR methods values are in good agreement overall (the former was obtained as the average of values calculated in three orthogonal directions), yielding a tortuosity of 1.85. The directional tortuosity values calculated using the DP method suggest tortuosity anisotropy, with tortuosity in the

X and Z directions ~18% greater than that in the Y direction. The observation can be explained by the interplay between anisotropic particle shapes and anisotropic particle alignment during battery manufacturing (Ebner, et al., 2013a). The calendaring process presses the electrodes towards the current collector, which causes most of the particles to align along the Y direction, and therefore, reduce the tortuosity in this direction. It is noted that the absolute value of electrolyte tortuosity in the current report agrees with experimental or simulated values for similar (not identical) Li-ion cathodes obtained previously (Hutzenlaub, et al., 2013; Stephenson, et al., 2011; Zielke, et al., 2014b).

~~Clearly,~~ If one attempts to calculate the tortuosity directly from FIB-SEM images when the electrolyte and CB phases are not resolved, as is the case with carbon-based epoxies or unfilled samples, there is likely to be considerable error. To test this supposition, the tortuosity was calculated for the electrolyte-plus-CB volume, and was found to be 1.41 (supplemental information, Figure S4 and Table S1), substantially less than 1.85 when the electrolyte and CB phases are resolved. A similar outcome was noted by Hutzenlaub, et al. (Hutzenlaub, et al., 2013). Such underestimates are not expected with X-ray tomography or transport measurements.

In addition to the volume-averaged tortuosity, the 3D distance map and tortuosity distribution provides spatially resolved tortuosity information. Figure 6 (a) shows the 3D distance map of electrolyte along the X direction, *i.e.*, the direction of Li transport during battery operation. At some points near the current collector (furthest from the electrolyte), the longest Li diffusion pathway is as high as 85 μm , significantly larger than the electrode thickness 34.5 μm . Figure 6 (b) shows the electrolyte, tortuosity distribution on the YZ plane at $X = 34.5 \mu\text{m}$ (the plane adjacent to the current collector). Figure 6 (c) plots the corresponding tortuosity histogram. The tortuosity is quite heterogeneous with values ranging from 2.2 to 1.3. This large heterogeneity may result in

inhomogeneous electric-potential and Li content within the electrode, especially during battery operation at high C-rates. These variations could lead to large local potential deviations from the average cell potential and consequently large local currents that lead to degradation (A. H. Wiedemann, et al., 2013).

In order to further illustrate the importance of resolving the carbonaceous phases, we also determined the tortuosity distribution from the 3D images while intentionally ignoring the contrast between electrolyte and CB; this yields not only lower propagation distance and tortuosity values, but also much less heterogeneity, with τ ranging from 1.1 to 1.5 (see Figure S5, supplemental information). Thus, one cannot obtain meaningful local tortuosity information without resolving electrolyte and carbonaceous phases.

4. Conclusions

A three-phase 3D reconstruction of a commercial LiCoO_2 cathode was demonstrated using FIB-SEM tomography. Clear contrast between electrolyte and carbonaceous regions was achieved by using low-viscosity silicone resin to fill the emptied electrolyte regions. Microstructural parameters including volume fraction, surface area, and feature size distributions of all three phases were determined. Moreover, the electrolyte connectivity, tortuosity, and tortuosity distributions were calculated and tortuosity was compared between electrolyte and electrolyte-plus-CB phases to emphasize the important role of the realistic electrolyte structure in evaluating the electrolyte transport property. For connectivity calculations, the electrolyte network was found to reach almost complete percolation (97.4%) towards the current collector. Based on tortuosity calculations, the electrolyte structure possesses a more tortuous 3D pathway than the electrolyte-plus-CB phases for Li-ion transport. In addition, the heterogeneous tortuosity distribution observed in the electrolyte

structure may result in inhomogeneous charge/discharge states, and consequently cause battery degradation.

The present study provides a straightforward approach to prepare electrode samples with three-phase contrast under SEM and can be easily transferred to other battery electrodes. This work demonstrates the importance of differentiating all three phases in extracting battery microstructure characteristics and analyzing the microstructure-performance correlations. The as-obtained 3D microstructures can further be used as exact templates for 3D simulations of battery transport and thermal properties.

Supplemental Information

Supplemental Information is available online or from the author.

Acknowledgements

The authors acknowledge the financial support from the Office of Naval Research Grant #N00014-12-1-0713 and Northwestern University Cabell Terminal Year Fellowship. We thank Dr. James W. Fleming for providing the battery samples studied in this report. We also thank Dr. Noah Shanti and Sarah Miller for help with the tortuosity calculations via the PLR method. The triple-ion cutting was performed at the Optical Microscopy and Metallography Facility at the Materials Research Center of Northwestern University. FIB-SEM (FEI) was performed in the EPIC facility of NUANCE Center at Northwestern University. NUANCE Center is supported by NSF-NSEC, NSF-MRSEC, Keck Foundation, the State of Illinois, and Northwestern University.

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Figure captions:

Figure 1. (a) Schematic view of the silicone resin-infiltrated LiCoO_2 electrode surface milled by a triple ion-beam cutter. (b) SEM image of the milled top surface. (c) higher magnification SEM image corresponding to the region indicated by the dashed rectangle in (b), a typical serial-sectioned region of electrode, where the arrow indicates the FIB slicing direction and the coordinate system is also shown.

Figure 2. (a) Cross-sectional SEM image of the fresh LiCoO_2 cathode sample after FIB milling. For the region corresponding to the red dashed rectangle region in (a), (b) shows EDS chemical mapping, (c) shows the gray scale histogram and (d), shows the segmented image

Figure 3. Three-dimensional reconstruction renderings of the electrode showing (a) LiCoO_2 , (b) carbon and binder, (c) electrolyte, and (d) all three phases superimposed.

Figure 4. Feature size distributions of (a) LiCoO_2 , (b) carbon-binder and (c) electrolyte phase; (d) cumulative feature size distributions of all three phases.

Figure 5. Electrolyte tortuosity values obtained using the path length ratio (PLR) method and the distance propagation (DP) method, including values in three directions and their average.

Figure 6. (a) Three-dimensional distance map of electrolyte and along direction to the current collector (along X direction); (b) Tortuosity distribution of electrolyte at $X = 34.5 \mu\text{m}$; (c) Histogram of the tortuosity distribution of electrolyte

Table 1 Microstructural parameters calculated from LiCoO₂ cathode 3D reconstruction

	V_f (%)	Surface Area Density, SA_I (μm^{-1})	Specific Surface Area Density, SA_s (μm^{-1})
LiCoO ₂	77.0	0.71	0.92
Carbon and Binder	10.3	0.43	4.17
Electrolyte	12.7	0.42	3.31
LiCoO ₂ /Electrolyte		0.36	
LiCoO ₂ /CB		0.35	
Electrolyte/CB		0.07	