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A Correlative and Multi-Modal Approach to Analyzing Microscopic Particulate Contaminants in Carbon Black

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Carbon based materials such as Carbon Black (CB) and graphite powders are used extensively in battery and fuel cell applications due to their unique chemical, structural and electronic properties. In Li Ion Battery (LIB) cathodes, carbon black based conductive additives are mixed with the active electrode material and polymer binder to ensure efficient electronic transport [1]. For this application, conductive carbons of high purity are required. While trace impurities, particularly transition metal contaminants, degrade the energy density, cycling stability and calendar life of the cell, larger contaminant particles have the potential to puncture the battery separator creating safety concerns in addition to local short circuits [2]. The impurities are also non-uniformly distributed making quality control of bulk powder samples or analysis of individual contaminant particles using spectroscopy based analytical techniques difficult. X-ray based imaging techniques offer a fast and reliable way to detect contaminants in carbon black [3]. In this study we describe a correlative workflow that combines high resolution 3-D X-ray tomography with Laser/Focused Ion Beam (FIB) milling and Energy Dispersive X-ray Spectroscopy (EDS) for chemically analyzing individual particulate contaminants in carbon black powder.

CB powder samples of tap density of 0.075 $g.cm^{-3}$ were used for this study. About 5g of powder was encapsulated in slow curing, cold-mounting epoxy and imaged using a ZEISS Xradia 520 Versa with a 150kV/10W X-ray beam and pixel size of 20 μm. Due to the relatively low mass-attenuation coefficient of carbon, the metal-rich particles could be observed after grayscale segmentation as shown in figure 1. At this stage of the workflow, the 3-D tomography data was used to measure the contaminant particle size distribution. Figure 1 also shows the different sources of contrast in the tomography images. To analyze selected contaminant particles identified by tomography, Laser-FIB milling was employed. The milling was performed using a modified ZEISS Crossbeam 340 with a *Trumpf* TruMark 6350 nanosecond pulsed laser system with an average power of 20 W. First, from the 3-D tomography data, the coordinates of particles close to an exposed surface were identified with respect to a fixed surface feature on the sample. The coordinate system from the 3-D tomography data and FIB setup were then linked using the ZEISS Atlas 5 system by overlaying and matching Secondary Electron (SE) images of the sample surface with the 3-D map. Once the coordinates were registered on the Laser-FIB system, the nanosecond laser, set to previously optimized process parameters, was used to remove material from the surface at a maximum rate of ~$3x10^5$ μm^3 per second till a hole of the desired depth was made. SE images of a hole milled on the sample surface using laser parameters of 50% peak power, 14.64 kHz frequency and 81.99 mm. s^{-1} raster velocity is shown in figure 2. Once exposed, final stages of milling and polishing the particle could be performed using the Gallium ion beam on the Crossbeam FIB. Finally, EDS (Oxford Instruments) was used to analyze the composition of the now exposed particles. Using this combination of techniques, it is possible to identify impurities that are otherwise below the surface, as demonstrated by the image of an iron rich particle found in an in-purified sample of carbon

black, shown in figure 3. This identification provides valuable information when determining potential sources of contamination and remediation strategies.

This multi-modal, correlative workflow using the ZEISS Atlas 5 system can be applied to other material systems where a single analytical tool or more traditional characterization techniques are not suitable for analyzing specific regions of interest in powder samples or sub-surface features in solid samples [4].

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[4] The authors acknowledge funding and support from Cabot Corporation.

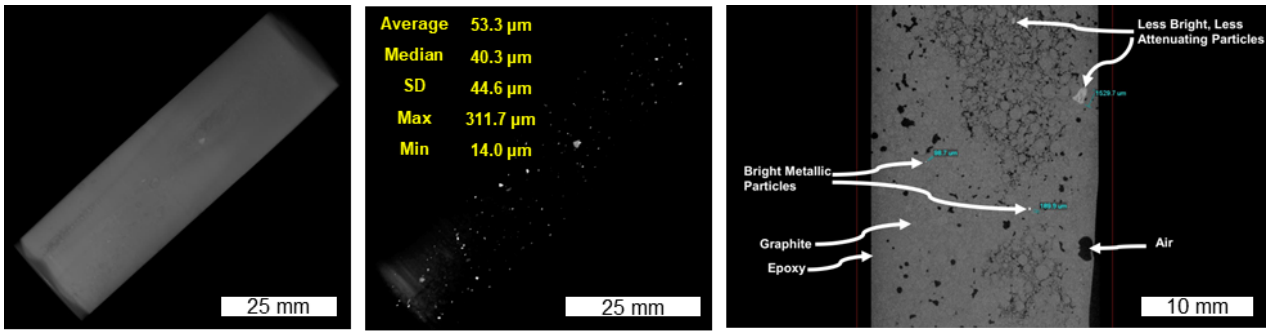

Figure 1. 3-D X-ray data sets of CB encapsulated in hardened epoxy (left). Same data set rendered with carbon and epoxy hidden (middle). A digital cross-section showing various sources of contrast (right).

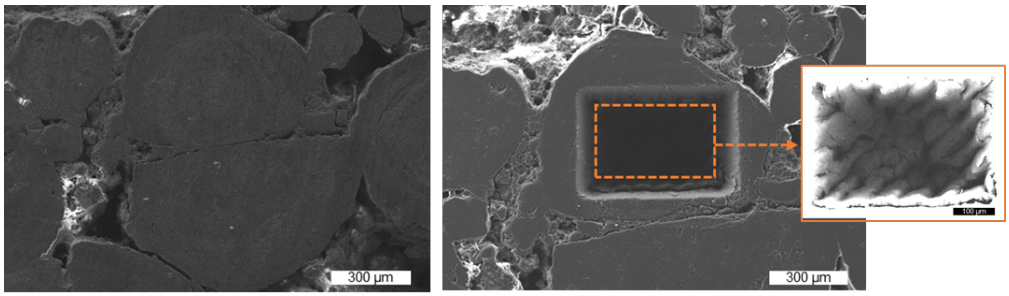

Figure 2. SE images of sample surface before (left) and after milling using nanosecond pulse laser. Inset shows SE image of bottom of laser-milled trench, ~600 µm below surface.

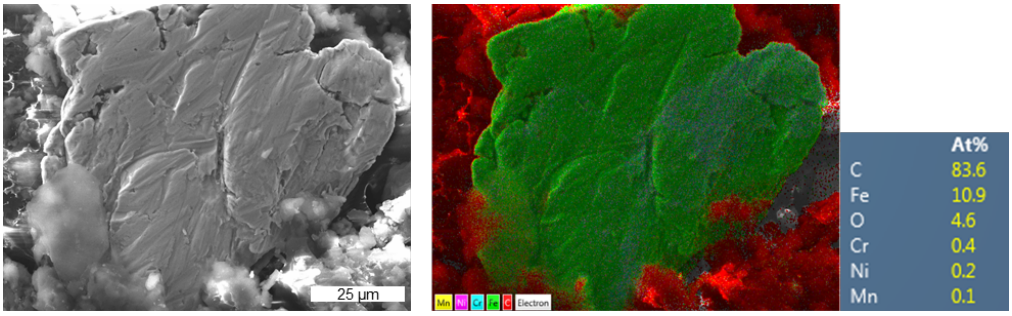

Figure 3. SE image (left) and EDS map of iron rich particle analyzed using the correlative workflow.

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