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Characterization Technique for Advanced Materials for Lithium Batteries in an SEM

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The scanning electron microscope (SEM) is a powerful instrument to provide nanoscale surface detail and chemical information on a wide range of materials [1, 2]. The development of a new generation of lithium batteries needs advanced material like ceramic electrolytes, sulfite electrolytes, polymers, transition metal oxide, and lithium metal. These materials present various challenges for high quality results in an SEM: poor electronic and thermic conductivity, element migration [3] and phase change under an electron beam, high sensitivity to humidity or oxygen.

Figure 1 shows the effect of 1 hour acquisition x-ray map on garnet Li7La3Zr2O12 (LLZO) solid electrolyte powder [4]. The dark phase (BSE micrographs) at the surface of the LLZO disappear after the acquisition. LLZO is a ceramic with poor electric and thermic conductivity. The heat generated by the interaction of the electron and the sample change the phases present in the sample. Furthermore, small particles are observed (SE micrographs) on the edge of the acquisition area. Using a Peltier cool stage at -45 ºC allows long acquisition without removal of the dark phase, but did not prevent the formation of the small particles.

Figure 2 shows the effect of the accelerating voltage on the phases observed. At 15 kV, the LLZO powder seems to be homogenous in the BSE micrograph. However, the same area observed at 5 kV shows dark phase that was barely observed on the 15 kV micrograph. The observation of battery material, different accelerating voltage should be used to completely characterize the material.

Various characterization and preparation techniques are needed for the characterization of advanced materials for lithium batteries like cryo-microtome and cryo ion milling cross section for post-mortem and transversal observation; low accelerating voltage imaging with a low voltage BSE detector and x-ray microanalysis with a windowless EDS; cold and cryo stage to prevent beam damage and cryo FIB; surface analysis with TOF-SIMS; sample preparation and transport in controlled atmosphere (dry room and glove box). Different characterization techniques will be presented for observation and analysis of these materials in an SEM.

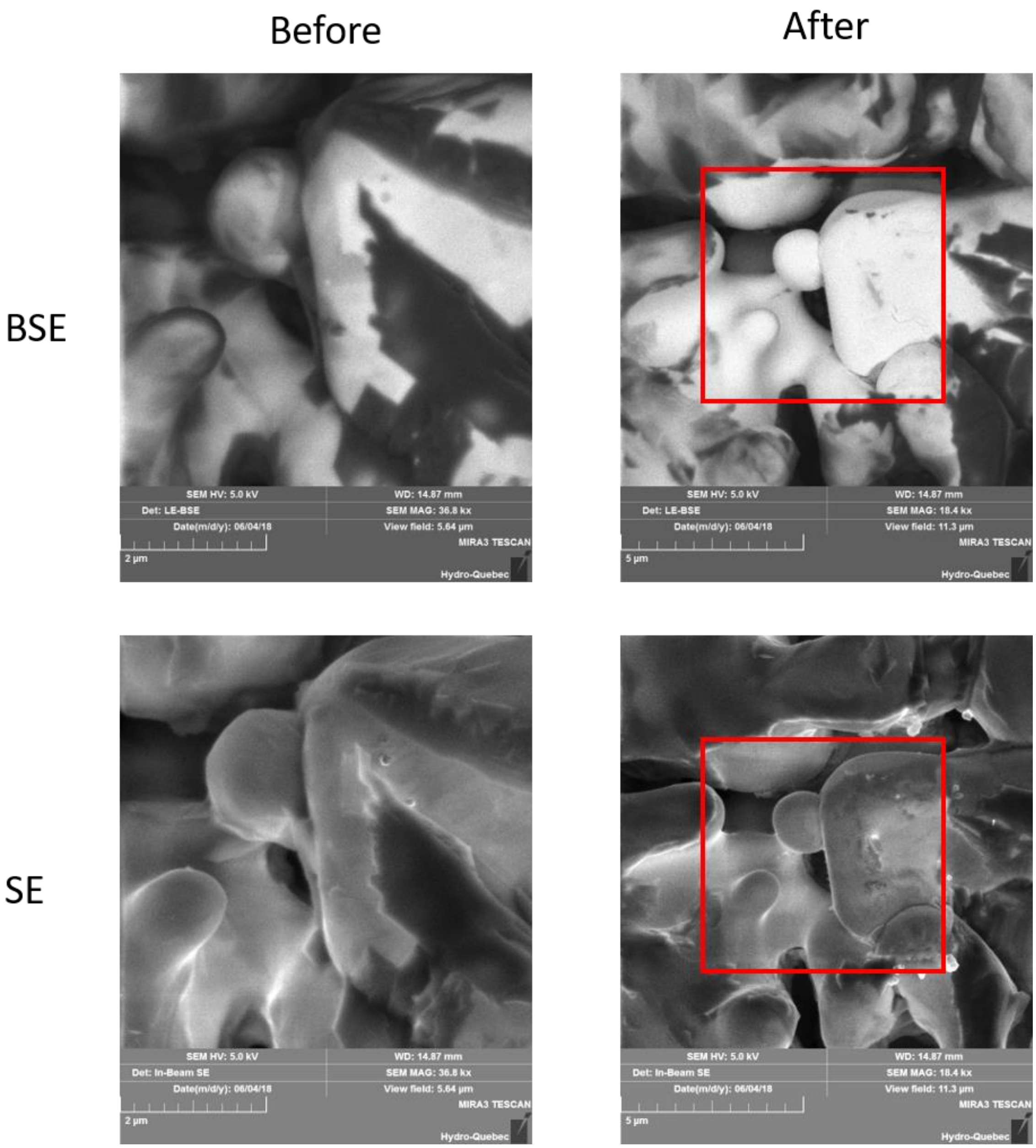

Figure 1. Effect of 1 hour acquisition on the LLZO-Ga sample. The red square shows the acquisition area. The BSE micrographs were obtained with low voltage BSE detector and show a compositional contrast of the sample. The SE micrographs were obtained within column SE detector which gives higher resolution topographic contrast, but it is more sensitive to sample charging.

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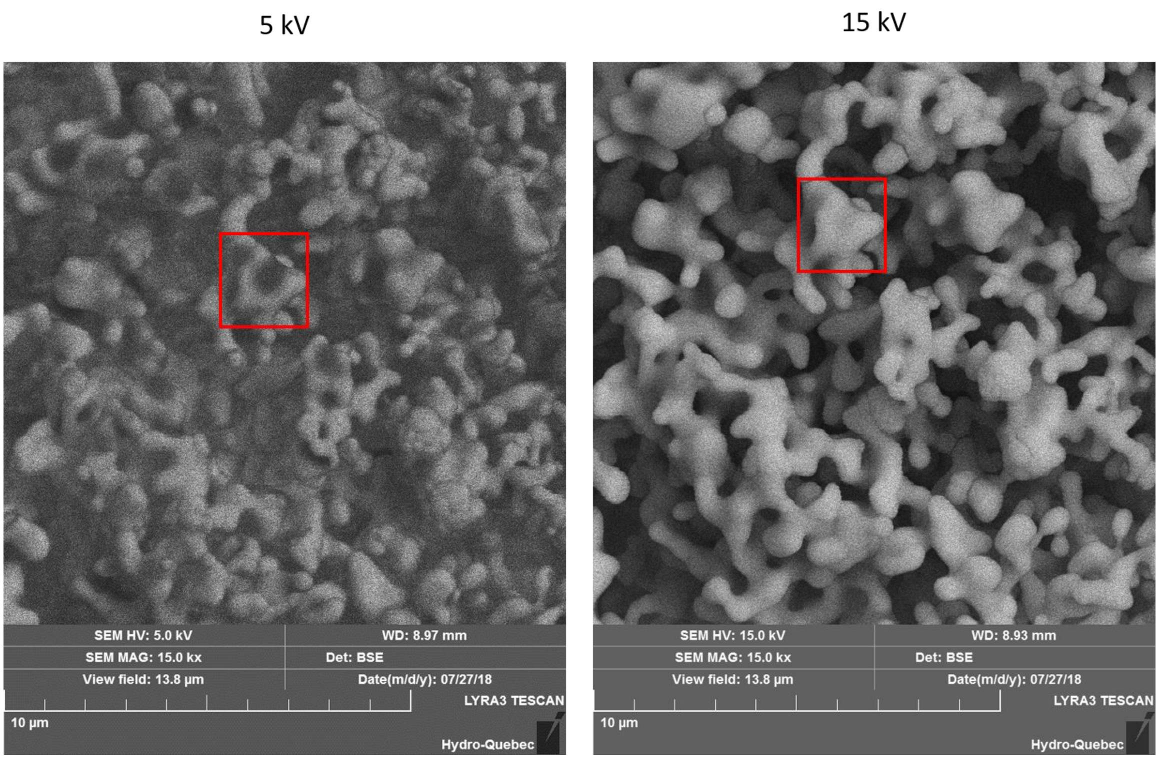

Figure 2. The effect of accelerating voltage is shown by comparing two micrographs of the same LLZO-Ga area at 5 kV and 15 kV. The micrographs were obtained with the low voltage BSE detector and show a compositional contrast of the sample. The red square shows a dark phase that is visible at 5 kV, but not at 15 kV.

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