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Treading Lightly – Achieving Spectroscopy and Elemental Maps of Beam Sensitive Specimens in the SEM

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The versatility of the latest generation of SEMs continues to develop the possibilities for the imaging of beam sensitive specimens. Spectroscopy of these specimens lags behind imaging due to the perceived requirements for longer working distances, higher voltages and higher beam currents. The challenge for carrying out EDS, and particularly X-ray mapping, on beam sensitive specimens is achieving a reasonable number of X-ray counts for each unit time spent with the specimen exposed to the electron beam.

By using a high sensitivity windowless EDS detector in the SEM it is possible to achieve elemental analysis at or very close to the desired imaging conditions for beam sensitive specimens. By optimizing the X-ray detector geometry it is possible to achieve high solid angles (~ 0.2 srad) at the short working distances (< 7 mm) required for high resolution low kV electron imaging on most SEMs. The majority of beam damage arises from three sources, ionization of the specimen by the electron beam, electron induced heating, and specimen charging [1]. These damage mechanisms are primarily dependent on two factors, electron energy and beam current. To minimize specimen damage, a balance must therefore be achieved between current and electron energy for each particular type of specimen.

Next generation Lithium-ion batteries may contain solid electrolytes that are composed of a wide range of elements, including heavy elements such as lanthanum. Due to the mobility of Li in these materials they can be extremely sensitive to the electron beam and in particular the energy of incident electrons [2]. A specimen of LiLaAlZr oxide electrolyte material was prepared from a powder and low energy ion milled to produce a clean surface. At electron energies greater than 3kV the material was found to readily exfoliate Li to form surface dendrites. Dendrite formation after 5 minutes of continuous exposure to a 500pA, 5kV electron beam is shown in figure 1a. By using electron energies below 3 kV this formation can be reduced or stopped entirely. A different area of the same specimen, observed using a 100pA beam of 2kV electrons for 7 minutes, showed minimal change in surface properties (figure 1b). The windowless nature of the EDS detector used in this study significantly increases sensitivity to low energy X-rays (<1keV). This allows count rates of 5000 cps or greater at these low current, low energy beam conditions. As a result, an elemental map representative of the observed structures can be collected (figure 1c). For this material, X-ray peaks for all constituent elements are available even at electron energies of <1kV (figure 2): Li Kα 54eV, Al Ll 72eV, La $N^{4,5}O^{2,3}$ 81eV and Zr Mζ 151 eV [3]. Performing spectroscopy at 1kV further reduces specimen damage from the electron beam allowing for longer acquisition times, improved X-ray map resolution and spectroscopic data representative of the actual specimen and not the beam induced changes within that specimen.

This study has confirmed that by optimizing the geometry of the EDS detector and improving X-rays transmission, X-ray spectroscopy can be performed at the electron imaging conditions used for minimizing specimen damage. The ability to perform EDS under these conditions opens up the possibility of performing spectroscopy and elemental mapping on a range of other highly beam sensitive specimens such as zeolites and biological tissues.

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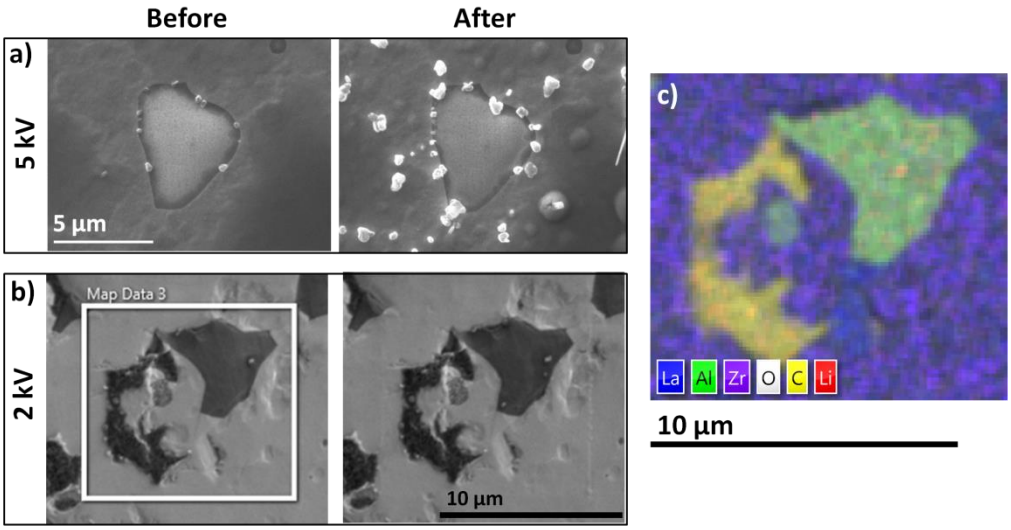

Figure 1. Electron images showing the stability of a LiLaAlZr Oxide electrolyte specimen under an electron beam at a) 5kV and b) 2kV. c) An elemental map collected for 7 minutes at 2 kV from the region shown in b.

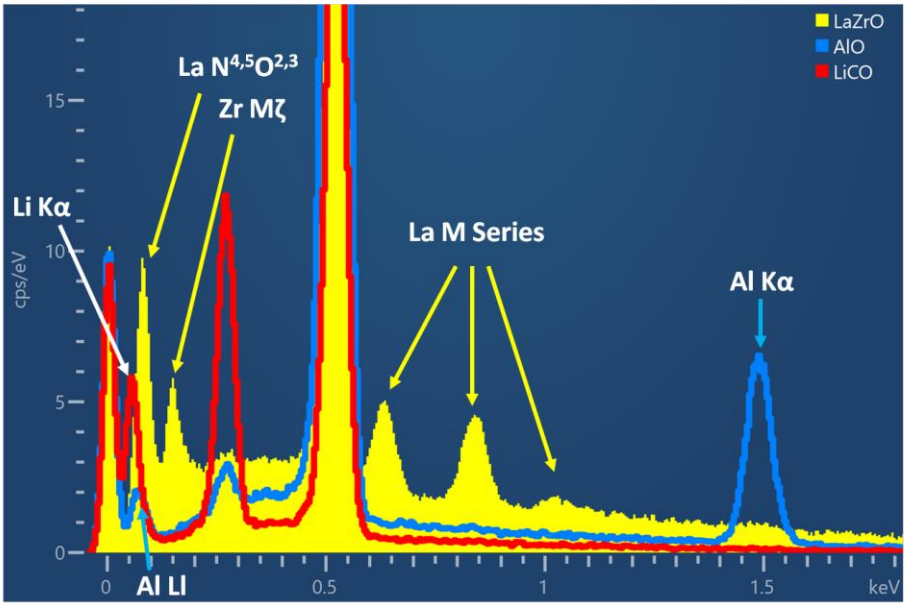

Figure 2. EDS spectra collected at 2kV for, LaZrO, AlO and LiCO regions of the specimen showing the availability of sub 1keV X-ray lines for complete sample characterization.

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