BIO Web of Conferences **129**, 25026 (2024) https://doi.org/10.1051/bioconf/202412925026
EMC 2024

Quantification of Lithium in State-of-the-Art low Voltage STEM

Pr. Raynald Gauvin[1], Mr. Nicolas Brodusch[1], Mrs. Stéphanie Bessette[1]
[1]Mining and Materials Engineering,McGill University, Montréal, Canada

Background incl. aims
This paper will present new achievements and challenges in performing quantitative EDS and EELS maps of lithium-based materials in a state-of-the-art low-voltage scanning transmission electron microscope. With the energy revolution coming because of global warming, Lithium-Ion Batteries of higher energy densities and much lower cost must be developed to allow the transition of fuel-based vehicles to be replaced with electrical vehicles. This will only become a reality if we can quantify lithium at the Nanoscale to see where it becomes lost during the electrochemical cycling of the batteries.

Methods
State-of-the-art results acquired with the Hitachi SU-9000 dedicated STEM will be presented. This microscope has EELS capabilities that allow Li detection [1], being the first to be sold by Hitachi High-Technologies. It is also equipped with the Extreme EDS system from Oxford Instruments that can detect the K line of lithium [2]. The SU-9000 has a resolution of 0,22 nm in bright field STEM without aberration correctors and this allows lattice imaging. A cryogenic specimen holder allows to analyze Li based materials at 77 K.

Results
Figure 1-a shows an EELS spectrum of Li2FeSiO4 taken with the Hitachi SU-9000 at 30 keV. Even if X-ray emission was not detected with the EDS detector for this material for the same reasons stated above for Li2CoSiO4, the ionization edges are visible for Fe, Li, and Si. This shows the strong advantage of EELS over EDS (or WDS) for Li quantification. This is seen in figure 1-b which shows a Li EELS edge-to-background ratio map of an Al-Li 2099 alloy recorded with the three-detectors system on the Hitachi SU-9000 at 30 keV. This map shows the round δ’ precipitates (Al3Li) of 5 to 20 nm and the T1 plates (AlCuLi) that have thicknesses between 1 to 2 nm. Results obtained with a cryo-holder to evaluate possible minimization of beam damage will be presented.

Conclusion
It is not always possible to detect Li with EDS because its 3S electron can be bonded with another atom. EELS is superior to EDS since it is always possible to detect the ionization edge if the beam damage can be minimized with a cryo-holder. The downside of EELS is the need for a transparent specimen.

Graphic:

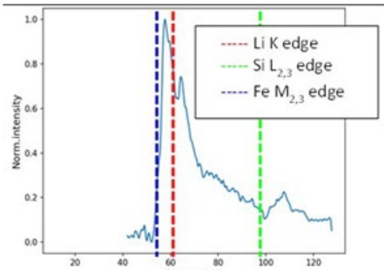

Figure 1-a
EELS spectrum of Li_2FeSiO_4 taken with the Hitachi SU-9000 at 30 keV.

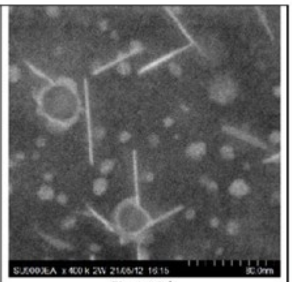

Figure 1-b
Li EELS edge-to-background ratio map of an Al 2099 alloy taken with the Hitachi SU-9000 at 30 keV.

Keywords:

Electron Energy Loss Spectroscopy, Lithium

Reference:

[1] N. Brodusch, H. Demers, A. Gellé, A. Moores, R. Gauvin (2019), Ultramicroscopy, 203, pp.1-36.
[2] P. Hovington, V. Timoshevskii, S. Burgess, H. Demers, P. Statham, R. Gauvin, K. Zaghib (2016), Scanning, 38, 6, pp. 571 – 578.
[3] R. Gauvin et al. (2021), Microscopy and Microanalysis, 27, pp. 1868-1869.