Analytical STEM at 30 keV, EDS, EELS and CBED at the Nanoscale

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This paper will present state of the art results acquired with the new SU-9000EA dedicated analytical STEM that works at 30 keV and less. It has a resolution of 0,22 nm in bright field STEM imaging mode at 30 keV without an aberration corrector [1]. It is equipped with an Oxford Extreme SDD EDS detector that allows lithium detection. With EELS and EDS, results for Li detection will be presented and the challenges, in regards of quantification and beam damage, will be covered. Examples of EELS analysis at 30 keV for nanomaterials will be presented as well, including surface plasmon imaging. The SU-9000EA allows to perform electron diffraction and CBED patterns acquired at the nanoscale will be presented.

Figure [1-a] shows an EDS spectrum, acquired at 30 keV, of a LiO_2 compound that was damaged during electron beam irradiation. A small lithium peak is visible. The intensity of Li is significantly reduced in compounds, compared to pure lithium, because of the increase of the mass absorption coefficients and also of the possible reduction of x-ray emission because of bonding effects. With issues of beam damage that limits acquisition time, it is clear that quantitative x-ray microanalysis will be a challenge with this technology because of these low x-ray counts.

Figure [1-b] shows an EELS spectrum, acquired at 30 keV and only background subtracted, of the same material. Metallic Li as well as possibly lithium carbonates can be identified in different region of the material. The LiO_2 might possibly have reacted under the beam with some carbon, coming from the grid holey film or from carbon contamination, thus forming lithium carbonate. Another possible reaction might have happened where some oxygen was removed, leaving the remaining lithium in metallic form. The number of counts of the EELS spectra is much higher than x-ray spectra, owing to the fact that all ionizations are detected while for EDS, the fluorescence yield of Li is of the order of 10^{-4}. This is clearly a strong advantage of EELS compared to EDS and also, the 0.5 eV resolution of the EELS spectrometer of the SU-9000EA alloys to perform spectroscopy to determine the nature of the bonds of the materials. The drawback of EELS at 30 keV is that specimens in the 20 to 50 nm must be prepared for Li compounds. Clearly, low voltage SEM will continue to improve for materials characterization [2].

References:

[1] H. Demers, N. Brodusch and R. Gauvin (2017), Microscopy and Microanalysis, 23, S1, 1044 - 1045.

[2] N. Brodusch, H. Demers and R. Gauvin (2018), Scanning Electron Microscopy, New Perspectives in Materials Characterization, Springer.

a

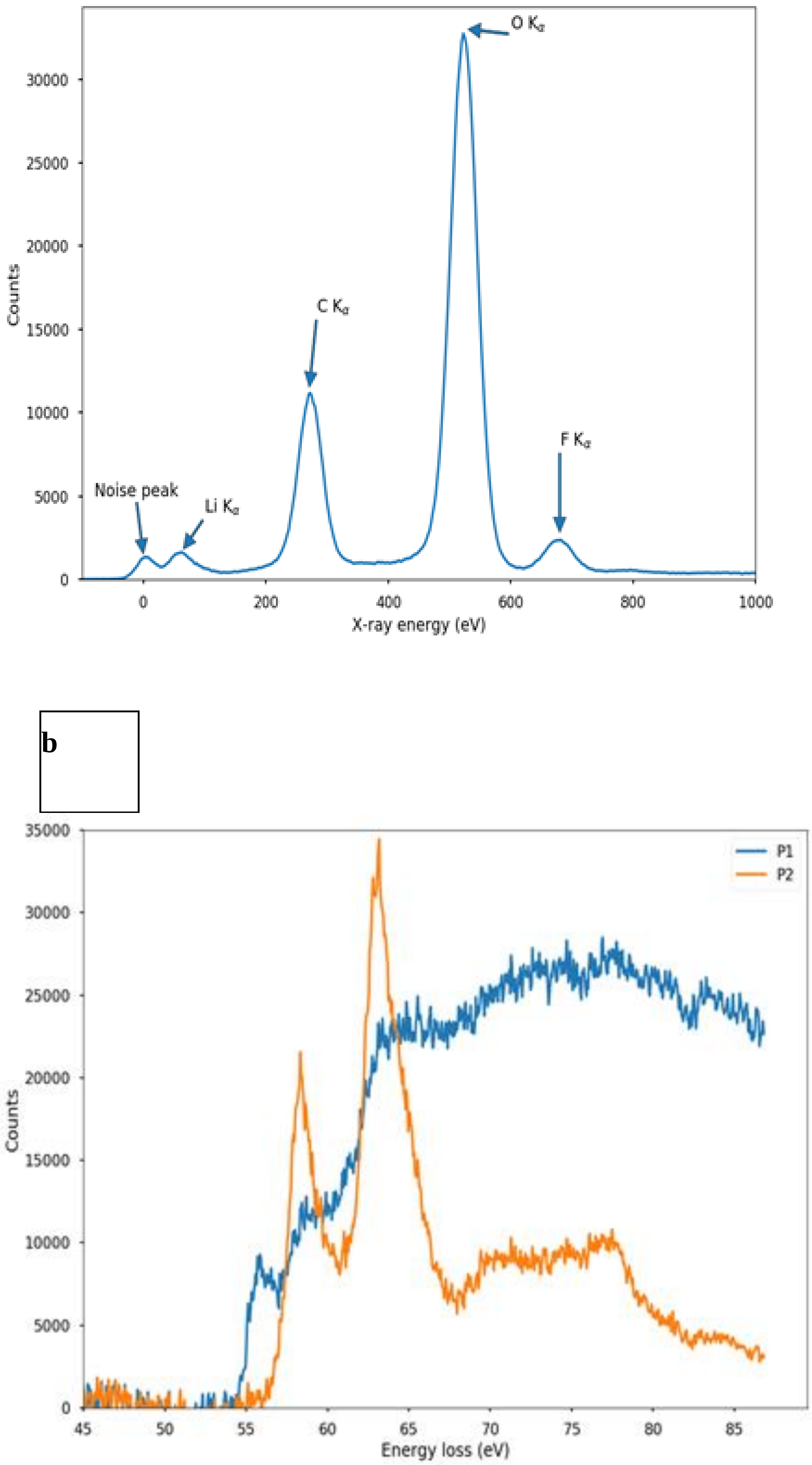

Figure 1. a) EDS and b) EELS spectrum acquired from Li_2O particulates at 30 keV with the Oxford Extreme EDS and Hitachi High-Technologies EELS system with the SU-9000EA.