

# Nanoscale PEEM spectroscopy combined with XPS to elucidate the surface reaction mechanism of cycled electrodes

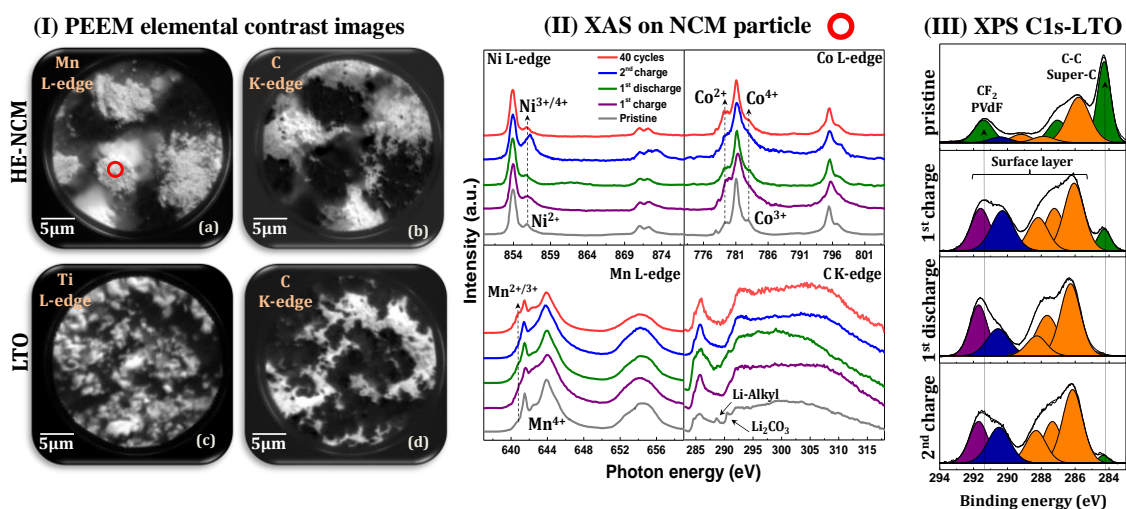
Daniela Leanza<sup>a</sup>, Carlos A. F. Vaz<sup>b</sup>, Alice Gillen<sup>a</sup>, Petr Novák<sup>a</sup>, Mario El Kazzi<sup>a</sup>

<sup>a</sup> Paul Scherrer Institute, Electrochemistry Laboratory, 5232 Villigen PSI, Switzerland

<sup>b</sup> Paul Scherrer Institute, Swiss Light Source, 5232 Villigen PSI, Switzerland

Daniela.Leanza@psi.ch

For the first time we explore the potential and feasibility of X-ray PhotoEmission Electron Microscopy (PEEM) to investigate commercial-like battery electrodes and to study separately, at the nanoscale level, the surface layer chemistry evolution on the active materials and on the conductive carbon. Two types of electrodes are investigated, LiM (M=Mn, Ni, Co)O<sub>2</sub>-Li<sub>2</sub>MnO<sub>3</sub> (NCM) and Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> (LTO), as promising positive and negative electrodes, respectively, for Li-ion batteries. Thanks to the PEEM technique, we are able to overcome the limited spatial resolution and poor sensitivity to transition metals of conventional X-ray photoemission spectroscopy (XPS). Furthermore, both techniques probe the same range of depth (< 8nm). The elemental contrast images in Figure 1.I attest the high lateral resolution of PEEM and its ability to distinguish single particles of the active materials, NCM or LTO, from the conductive carbon in typical commercial-like electrodes, without the need of using model systems. The local XAS spectra (Figure 1.II) at the Ni-, Co-, Mn-L edges and C-K edge acquired on NCM particles allow us to monitor their oxidation states as well as the chemical species present on the surface without interference from the signal arising from the conductive carbon. By combining PEEM with XPS (Figure 1.III) during the early stage of cycling together with long cycling, we can obtain a better insight on the mechanism behind the surface layer formation on the positive and negative electrodes. Moreover, the origin of the surface layer present on LTO was elucidated, alongside the impact of the high potential on the NCM structure stability.



**Figure 1:** (I) Element-specific PEEM contrast images performed on NCM (a, b) and LTO (c, d) pristine electrodes. (II) XAS evolution of the transition metals oxidation states as well as the surface layer formation acquired on one single NCM particle. (III) LTO surface layer formation probed by XPS C1s core level.