IN-SITU STUDY OF NMC CORE-SHELL PARTICLES AS CATHODE MATERIALS IN LI-ION BATTERIES

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Amongst the most promising materials used as cathodes in Lithium-ion batteries (LiBs), Ni-Mn-Co (NMC)-based compounds with core-shell structure are gaining increased attention owing to their high specific capacity and thermal stability **[1]**. The characterization of these materials requires the employment of the advanced microscopy and spectroscopy techniques, including in-situ and in-operando techniques, in order to achieve deeper comprehension of the phenomena governing the battery performance **[2,3]**.

In this work, NMC core-shell microparticles have been studied by means of a combination of techniques including scanning electron microscopy (SEM), X-ray diffraction (XRD), thermo-XRD, energy dispersive X-ray spectroscopy (EDS), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS), as well including in-situ characterization. The combination of these techniques allows to achieve insights in the morphological, structural, compositional, and electronic properties of the NMC microparticles under study. The analysed microparticles exhibited a rounded appearance with an average diameter of 4 µm and variations in their morphology and properties as a function of the synthesis route, as observed by SEM and XRD. The presence of the Ni-rich core and the Mn-rich shell with submicrometric dimensions have been confirmed by EDS. The formation of oxides presenting the usual layered structure of NMC (R-3m) together with the appearance of spinel phases as a function of the synthesis route have been assessed by means of XRD and Raman spectroscopy. This layered structure is directly related to the high capacity and structural stability of the NMC cathodes when the battery is cycled. Further characterization, including in-situ-thermo XRD, in-situ XPS under variable atmosphere and temperature, as well as in-situ SEM with variable temperature up to 900 ºC have been also carried out. These measurements confirm the formation of NiO as the temperature increases up to 900 ºC, as well as the promotion of changes in the morphology of the microparticles. Finally, in-situ XPS measurements at temperatures up to 500 ºC and a variable oxygen-rich atmosphere, indicate slight variations in the Ni3+/Ni2+ ratio, as well as in the Mn oxidation states as the temperature rises up. Variations in the valence band region were also observed as a function of the temperature.

The combination of these results, including the in-situ characterization, can allow to achieve a deeper knowledge about the properties of the NMC microparticles and lead to LiBs with improved performances.

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