Investigating Surface and Interface Phenomena in LiFeBO₃ Electrodes Using Photoelectron Spectroscopy Depth Profiling

The formation of surface and interface layers at the electrodes is highly important for the performance and stability of lithium ion batteries. To unravel the surface composition of electrode materials, photoelectron spectroscopy (PES) is highly suitable as it probes chemical surface and interface properties with high surface sensitivity. Additionally, by using synchrotron-generated hard x-rays as excitation source, larger probing depths compared to in-house PES can be achieved. Therefore, the combination of in-house soft x-ray photoelectron spectroscopy and hard x-ray photoelectron spectroscopy (HAXPES) enables reliable and non-destructive depth profiling. Thus, detailed investigation of compositional gradients at electrode surfaces and interfaces from a sub-monolayer to several nanometer length scales can be performed. As this depth region is especially relevant for both electronic and ionic transport processes, the presented combinatorial approach is a very valuable tool in characterizing battery electrodes and their interfaces. We apply this technique to lithium iron borate (LiFeBO₃) battery electrodes at different cycling conditions to study the surface and interface layer evolution. Due to its high theoretical capacity (220 mA h g⁻¹) LiFeBO₃ is a promising cathode material for lithium ion batteries. However, its application is limited because of significant degradation due to surface reactions upon exposure to moisture and air. Therefore, detailed compositional characterization of LiFeBO₃ electrode surfaces is essential to gain further understanding of the degradation processes. To study these properties for the pristine material, the electrode fabrication conditions were carefully controlled to limit contact to air and moisture. The investigation of the iron valence state in dependence of the electrode preparation as well as cycling conditions is presented as higher valence states of iron are most likely involved in the degradation process.