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Beam damage in synchrotron radiation based operando characterization of battery materials

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Operando synchrotron radiation-based characterization techniques applied to energy storage materials are becoming a widespread characterization tool as they allow for non-destructive probing of materials with various depth sensitivities through spectroscopy, scattering, and imaging techniques. Moreover, they allow for faster acquisition rates, variable penetration depths, higher spectral or spatial resolution, or access to techniques that are only possible with a continuous tuneable source over a wide photon energy range. Compatibility between the electrochemical cell designs and the experimental setups may force some specific design features and care must be taken to ensure that these do not perturb the electrochemical response of the materials under investigation. The use of *operando* techniques has intrinsic advantages, as they enable the detection of metastable intermediates, if any, and ensure characterization under real conditions avoiding the risk of ex situ sample evolution during its preparation. *Operando* experiments are thus crucial for both the elucidation of redox mechanisms in new technologies and also for understanding of failure and aging processes for already commercial concepts. However, they do not come with the extent of risks. The interaction of the synchrotron radiation with the sample especially in the complex context of an operating electrochemical cell can give rise to abnormal behavior of the sample at the measurement point, compromising the reliability of the experiment. Here we will present a study that aims to assess the beam-induced effects under variable experimental conditions in terms of radiation energy and dose on commercial NMC and LPF electrodes and present a series of measures that can be taken to evaluate and minimize or suppress beam-induced hindrance in battery *operando* experiments.

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