

# Mitigating radiation damage in beam sensitive battery materials by adapting scanning parameters

**Mme. Hannah Nickles Jäkel**<sup>1</sup>, Mr Eric Gautron<sup>1</sup>, Mr Maurice Peeman<sup>2</sup>, Mr Philippe Moreau<sup>1</sup>, Ms Patricia Abellan<sup>1</sup>

<sup>1</sup>Nantes Université, CNRS, Institut des Matériaux de Nantes Jean Rouxel, IMN, 44000 Nantes, France, <sup>2</sup>Thermo Fisher Scientific, Eindhoven, Netherlands

## Background incl. aims

Electron-beam sensitivity of battery materials hinders the full potential of high-resolution STEM-EELS insights on battery failure mechanisms. It is therefore important to understand how electron beam induced alterations can be minimized. Beam damage mitigation in STEM can be achieved by adjusting electron probe properties such as beam size, current and acceleration voltage, by changing dwell time, but also by changing the scan pattern and thus, exploiting the spatial component of beam damage (1). For the latter, many different approaches have shown great promise. Alternative scanning techniques, such as leapfrog scanning, which explores the effect of increasing pixel spacing (2), or sparse scanning, which irradiates only a percentage of pixels (3), can reduce damage compared to conventional raster scanning. Since beam damage is a process that is highly dependent on the type of material being irradiated, damage mitigation techniques require material-specific investigations to ensure effective adaption of the scanning parameters. Here we are interested in Li-containing battery materials, where for LiF, for example, radiolysis is more dominant than knock-on damage (4). Methodical experiments are necessary to understand how irradiation conditions like acceleration voltage, electron dose, and scan pattern can be optimized for specific electron beam sensitive battery materials.

## Methods

In order to determine the optimum acceleration voltage, damage cross-sections for different acceleration voltages are calculated for various battery materials. To gain a better understanding of the damage mechanisms involved, damage is systematically induced on Li-containing battery materials and monitored with STEM-EELS ((S)TEM Themis Z 3G). The effect on materials damage of conventional scan patterns is compared to different “alternative” scan patterns.

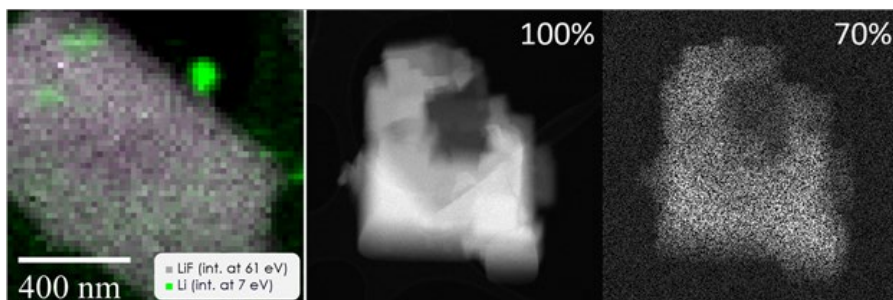
## Results

The calculated damage cross-sections show, that avoiding knock-on damage by imaging below a threshold voltage is not applicable for (S)TEM measurements of lithium-containing battery materials. STEM-EELS observations provide insights on the degradation of LiF to metallic Li, being consistent with radiolysis being the dominant damage mechanism. EELS spectra of LiF over radiation time show that the LiF bulk plasmon peak and the Li K-edge for LiF at 61 eV decrease, while the bulk plasmon peak for metallic Li appears at 7 eV. Lastly, scanning variations give insights on damage delocalization in battery materials. Damage imposed by conventional scanning is compared to different alternative scan patterns, by quantifying electron beam induced alterations in image intensity.

## Conclusion

With the gained understanding for radiation damage in specific battery materials, optimized acceleration Voltage and scan pattern can be chosen to reduce electron beam induced alterations. Optimized scanning parameters enable higher imaging flexibility and could provide new insights into battery failure mechanisms.(5)

**Graphic:**



**Keywords:**

beam-damage, scan pattern, battery materials

**Reference:**

- (1) Boniface, M. (2017). Nanoscale evolution of silicon electrodes for Li-ion batteries by low-loss STEM-EELS, Ph.D. thesis in Material Physics. Université Grenoble Alpes.
- (2) Egerton, R. F. (2017). Scattering delocalization and radiation damage in STEM-EELS. *Ultramicroscopy*, 180, 115–124.
- (3) Nicholls, D., Lee, J., Amari, H., Stevens, A. J., Mehdi, B. L., & Browning, N. D. (2020). Minimising damage in high resolution scanning transmission electron microscope images of nanoscale structures and processes. *Nanoscale*, 12(41), 21248–21254.
- (4) Hobbs, L. W. (1987). Electron-beam sensitivity in inorganic specimens. *Ultramicroscopy*, 23(3), 339–344.
- (5) Acknowledgements: The authors thank the OPINCHARGE project, which received funding from the European Union’s Horizon Europe research and innovation programme under grant agreement No 101104032. We acknowledge Battery2030+ for their support to the OPINCHARGE project. Measurements were performed using the IMN’s characterization platform, PLASSMAT, Nantes, France.