doi:10.1017/S1431927619005063
Microsc. Microanal. 25 (Suppl 2), 2019
© Microscopy Society of America 2019

# Focused $Ne^+$ Beam for Improved SIMS Analysis of Lithium Ion Batteries

Fouzia Khanom[1*], U. Golla-Schindler[2], Timo Bernthaler[2], Gerhard Schneider[2], and Brett Lewis[1]

[1.] Carl Zeiss SMT, Inc. PCS Integration Center, One Corporation Way, Peabody, MA, USA.
[2.] Materials Research Institute (IMFAA), Aalen University, Aalen, Germany.
* Corresponding author: fouzia.khanom@zeiss.com

Using a high brightness gas field ion source (GFIS) with helium (He) or neon (Ne) gas, the ZEISS ORION NanoFab generates secondary electron (SE) images with high resolution, high contrast, and large depth of field [1]. With unique contrast and surface sensitivity, this tool can produce secondary electron (SE) images with sub 0.5 nm spatial resolution for He and 1.9 nm for Ne. This powerful, multi-ion beam instrument is also capable of fabricating sub-10 nm structures via ion beam sputtering, lithography, and beam-assisted chemical etching or deposition.

To enable surface analytical ability, a custom designed Secondary Ion Mass Spectrometer (SIMS) has been integrated onto this instrument [2,3]. Using Ne as the primary ion beam to produce Secondary Ions (SI), this new spectrometer identifies all elements and isotopes and even some small compounds and clusters. Since the NanoFab's neon beam can be focused to a probe size to under 2 nm, it enables analysis of regions limited only by the lateral distribution of the surface sputtered atoms. It has been demonstrated that NanoFab SIMS can produce elemental maps with a lateral resolution down to 15 nm [3-6]. In contrast, conventional SIMS instruments are limited by focused probe sizes larger than 50 nm, due to the low brightness of their ion sources [3,4]. Alternate techniques such as X-ray mapping have additional limitations when detecting lighter elements like lithium (Li), as well as the lateral resolution limitations. Therefore, NanoFab SIMS is an excellent technique for the study of Li battery samples, which often have features requiring a high lateral resolution and light elements. Moreover, NanoFab SIMS uniquely allows *in-situ* correlative imaging, combining high resolution SE images with elemental information from SIMS [3,7]. Furthermore, with this SIMS add-on for the NanoFab, it is now possible to follow the chemical composition in real time during milling applications such as end-pointing.

In this report, we present SIMS elemental analysis on a commercial graphite / NMC($Li[Ni_{1/3}Mn_{1/3}Co_{1/3}]O_2$) pouch cells to observe degradation due to storage and the cycling processes [8, 9]. Figure 1 shows the elemental mapping of (a) $^{6}Li$, (b) $^{12}C$, and (c) $^{28}Si$ while (d) represents a correlative imaging example with a He ion induced SE image, overlaid with the SIMS signal from $^{6}Li$ and $^{28}Si$. All images are taken from the anode side of the separator foils. It is obvious from the mapping that all the newly created features (precipitates) on top of the separator foil show an enrichment in the lithium content. This result was previously unattainable via conventional EDS. Results from other battery samples will also be presented.

References:

[1] G Hlawacek and A Gölzhäuser in "Helium Ion Microscopy", (Springer, Switzerland).
[2] T Wirtz, N Vanhove, L Pillatsch, D Dowsett, S Sijbrandij, and J Notte, Appl. Phys. Lett. **101** (2012).
[3] D Dowsett and T Wirtz, Anal. Chem. **89** (2017), pp. 8957-8965.
[4] T Wirtz, P Philipp, J-N Audinot, D Dowsett, S Eswara, Nanotechnology **26** (2015), p. 434001.
[5] P Gratia, et al., J. Am. Chem. Soc. **138** (2016), pp. 5821–15824.

https://doi.org/10.1017/S1431927619005063 Published online by Cambridge University Press

[6] P Gratia, et al., ACS Energy Lett. **2** (2017), pp. 2686-2693.
[7] F Vollnhals et al., Anal. Chem. **89** (2017), pp. 10702-10710.
[8] U Golla-Schindler, D Zeibig, L Prickler, S Behn, T Bernthaler, G Schneider, Micron **113** (2018), pp. 10-19.
[9] The authors from Aalen University want to acknowledge C. Guenther and M.A. Danzer for the samples and Federal Ministry for Economic Affairs and Energy (BMWi) Germany, for financial support

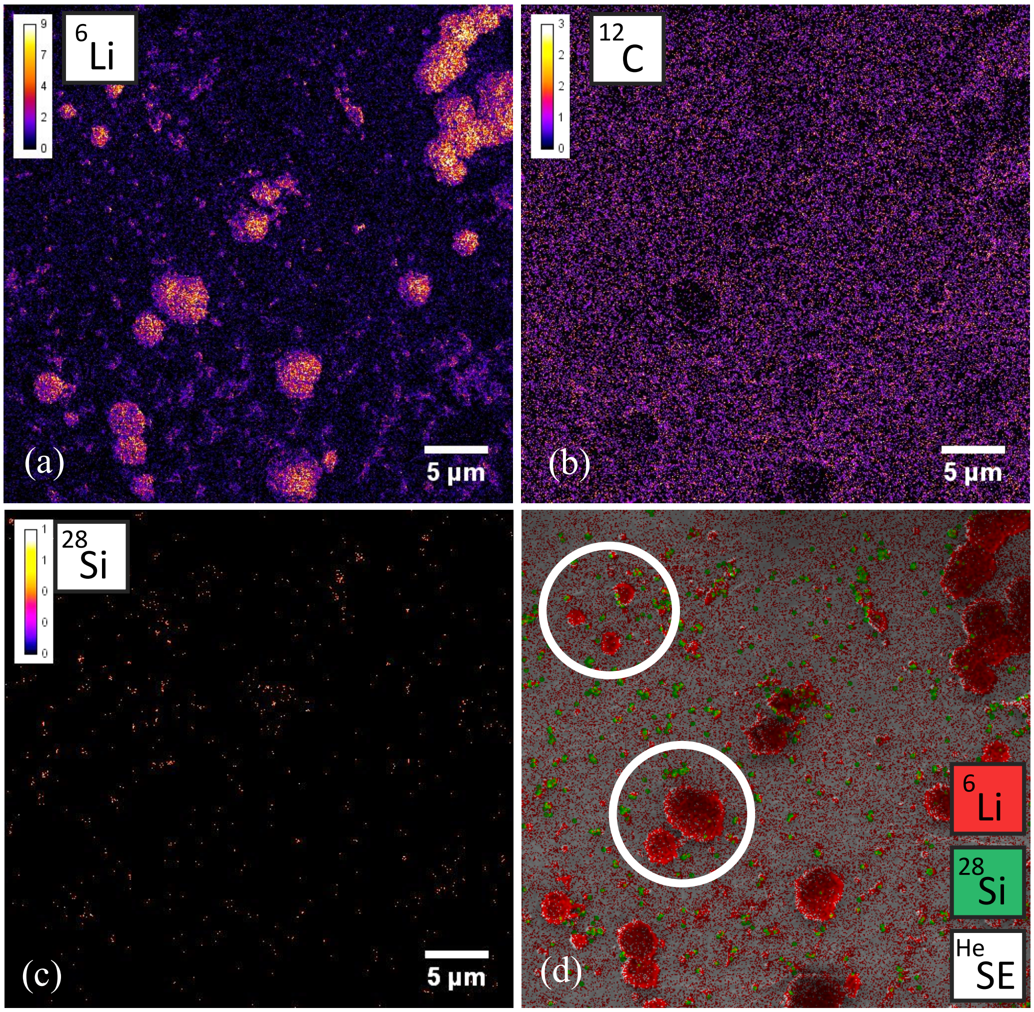


**Figure 1.** Investigations of a separator foil (anode side) from an aged battery cell with the Zeiss ORION NanoFab SIMS: (a) $^{6}$Li, (b) $^{12}$C, and (c) $^{28}$Si distribution images. Image (d) represents the correlative He beam induced Secondary Electron (SE) image with the $^{6}$Li and $^{28}$Si SIMS distribution overlay. Grown precipitations formed by degradation effects (examples of precipitates are in white circles) show enrichment from $^{6}$Li.

https://doi.org/10.1017/S1431927619005063 Published online by Cambridge University Press