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## TOF-SIMS in-depth investigation of interphase between $\text{TiNb}_2\text{O}_7$ electrode and electrolyte in Li-ion batteries

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Originally proposed in 2011 by Han, Goodenough et al.[1], the  $\text{TiNb}_2\text{O}_7$  (TNO) anode-based battery has emerged as a promising alternative to graphite and  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  (LTO) anodes. This is attributed to its high specific capacity comparable to graphite and its high potential close to LTO, enabling the attainment of both high energy density and battery safety. Additionally, TNO exhibits suitability for fast-charging electric vehicle applications due to the transition metal oxides constituting its intercalation material.

While numerous studies have investigated TNO material properties, scant attention has been devoted to the passivating layer formed at the electrode/electrolyte interface known as Solid Electrolyte Interphase (SEI) [2][3], despite its significant influence on battery performance. Our study describes, for the first time, the TNO anode passivating layer using Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS). This technique enables precise characterization of the SEI molecular composition at the extreme surface as well as deeper spatial distribution of species of interest with etching experiment. Associated with other complementary techniques such as X-ray photoelectron spectroscopy (XPS) and Auger Electron Spectroscopy (AES), this work allows us to deeply understand the electrode/electrolyte interfacial behavior at various battery cycling stages and under different conditions (including distinct binders, temperatures, and cutoff voltages).

As depicted in Figure 1, ToF-SIMS experiments unveil an increased amount of LiF on the electrode surface at 60°C, originating from the electrolyte salt decomposition. These findings align with XPS and Auger analyses, revealing heightened fluorine levels within the Li-F environment at 60°C. The significant lithium consumption during battery cycling explains the substantial capacity loss observed at 60°C.

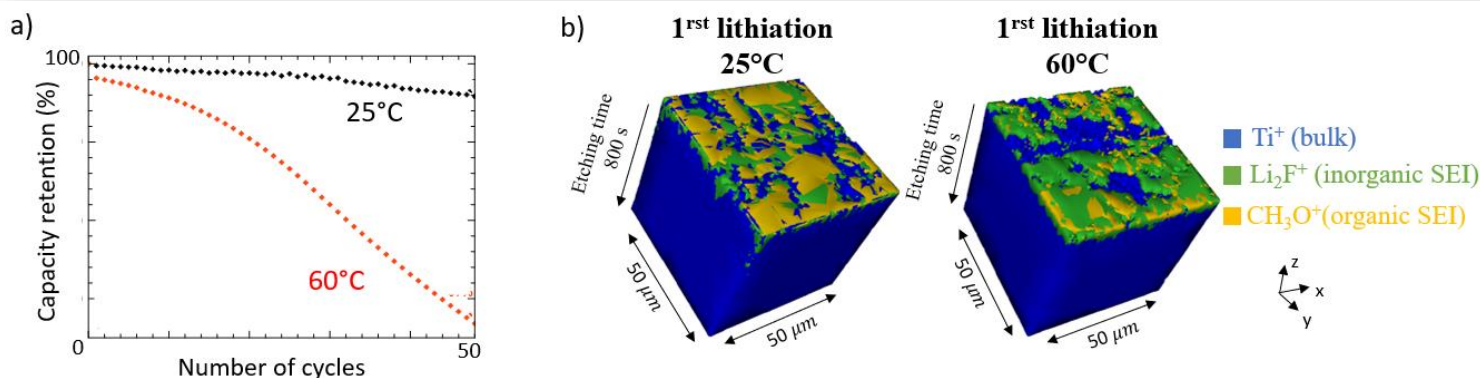


Figure 1 : a) Evolution of capacity retention during cycling (C/10) at 25°C and 60°C b) 3D representation of the SEI layer formed after one lithiation at 25°C and 60°C according to TOF-SIMS analysis

- [1] J. T. Han and J. B. Goodenough, "3-V full cell performance of anode framework  $\text{TiNb}_2\text{O}_7$ /Spinel  $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ ," *Chemistry of Materials*, vol. 23, no. 15, pp. 3404–3407, Aug. 2011, doi: 10.1021/cm201515g.
- [2] X. Wu et al., "Unravelling the Interface Layer Formation and Gas Evolution/Suppression on a  $\text{TiNb}_2\text{O}_7$  Anode for Lithium-Ion Batteries," *ACS Appl Mater Interfaces*, vol. 10, no. 32, pp. 27056–27062, Aug. 2018, doi: 10.1021/acsami.8b08425.
- [3] D. Parikh et al., "Operando Analysis of Gas Evolution in  $\text{TiNb}_2\text{O}_7$ (TNO)-Based Anodes for Advanced High-Energy Lithium-Ion Batteries under Fast Charging," *ACS Appl Mater Interfaces*, vol. 13, no. 46, pp. 55145–55155, Nov. 2021, doi: 10.1021/acsami.1c16866.