

## Characterization of SEIs on Tin-based Nano-composite Electrode Films for Li-ion Batteries

### Abstract

The Thermo Fisher Scientific™ K-Alpha™ XPS instrument was used to characterize nano-particular tin-based electrode composites for Li-ion batteries in the pristine status and after polymeric SEI formation.

### Introduction

Referred to state-of-the-art graphite anodes, tin-based hydrocarbon nano-particulate composites are very promising materials to improve the performance of lithium ion batteries by increasing the specific capacity. Central point herewith is the formation of a polymeric SEI (solid electrolyte interphase) during the first electrical cycles of the battery and, therefore, the chemical composition of the SEI is essential for further developments regarding long-term and cycle stability. Post mortem X-ray photoelectron spectroscopy is one of the pivotal surface analytical methods to achieve the desired chemical information in a non-destructive manner.

### Experimental

For electrode synthesis porous  $\text{SnO}_2/\text{C}_x\text{H}_y$  nano-particular films were directly deposited onto preheated Nickel current collectors by means of the Karlsruhe Microwave Plasma Process (KMPP) using water-free  $\text{Sn}(\text{C}_4\text{H}_9)_4$  as a precursor in pure Ar carrier gas. For post mortem XPS characterization the electrodes were discharged from 2.8 V to 0.8 V (expected reduction of  $\text{SnO}$  and electrolyte) and from 2.8 V down to 0.25 V (expected alloying of tin with lithium) using commercially available LP30 electrolyte and vinylene carbonate (VC) as additive.

For XPS measurements freshly prepared samples were mounted within the glove-box directly attached to the load lock of the Thermo Scientific K-Alpha instrument. All samples were analyzed using the micro-focused, monochromated Al K $\alpha$  X-ray source at 400  $\mu\text{m}$  spot size. The charge compensation system was employed during analysis to prevent any localized charge build-up. Data acquisition and processing were performed using the Thermo Scientific Avantage software. The spectra were fitted with one or more Voigt profiles (binding energy uncertainty:  $\pm 0.2$  eV). All spectra were referenced to the C 1s peak of hydrocarbons at 285.0 eV binding energy.



## Results

C 1s, O 1s, and Li 1s spectra of the pristine electrode surface and surfaces of two cells cycled down to 0.8 V and 0.25 V, respectively, are shown in Fig. 1. The improved electrochemical performance of VC containing cells is mainly due to the formation of polymeric surface species originating from VC, evidenced by the components at an exceptionally high binding energy of O 1s = 534.5 eV and C 1s = 291.0 eV. Beside the VC based polymer the formed SEI also comprises further decomposition products of the electrolyte after discharging from 2.8 to 0.25 V, indicated by the peaks at 286.6 eV, 287.6 eV and 289.0 eV, which can be attributed to alkoxy-, etheral-, carbonyl-, and alkylcarbonate species. The corresponding O 1s peaks at 531.6 eV, 532.1 eV and 533.0 eV corroborate these findings. These compounds result from the direct decomposition of the carbonates of the LP30 electrolyte. The additional decomposition of the conducting salt ( $\text{LiPF}_6$ ) is proven by the appearance of peaks attributed to lithium fluoride and several fluorinated lithium phosphate species (F 1s at 685.1 eV, and F 1s at 687.3 eV, P 2p<sub>3/2</sub> at 134-137 eV). The Li 1s spectra of both cycled electrode surfaces in Fig. 1 show rather broad main peaks, which were not further deconvoluted

due to the low sensitivity for lithium and only slight binding energy shifts between different lithium species. Nevertheless, the tailing at lower binding energies indicates the presence of  $\text{Li}_2\text{O}$  and is also confirmed by the corresponding O 1s component at 528.2 eV.

## Summary

XPS was proven as a powerful and indispensable tool to chemically characterize the SEI of Li-ion batteries in a non-destructive manner. Here XPS could evidence the forming of a mainly polymer consisting SEI based on the VC additive and further compounds originating from the decomposition of both the electrolyte and the conducting salt.

## Acknowledgements

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For further details see:

G. Kilbarda et al., *Electrochemical performance of tin-based nano-composite electrodes for Li-ion cells*, *Journal of Power Sources*, 263 (2014) 145-153.

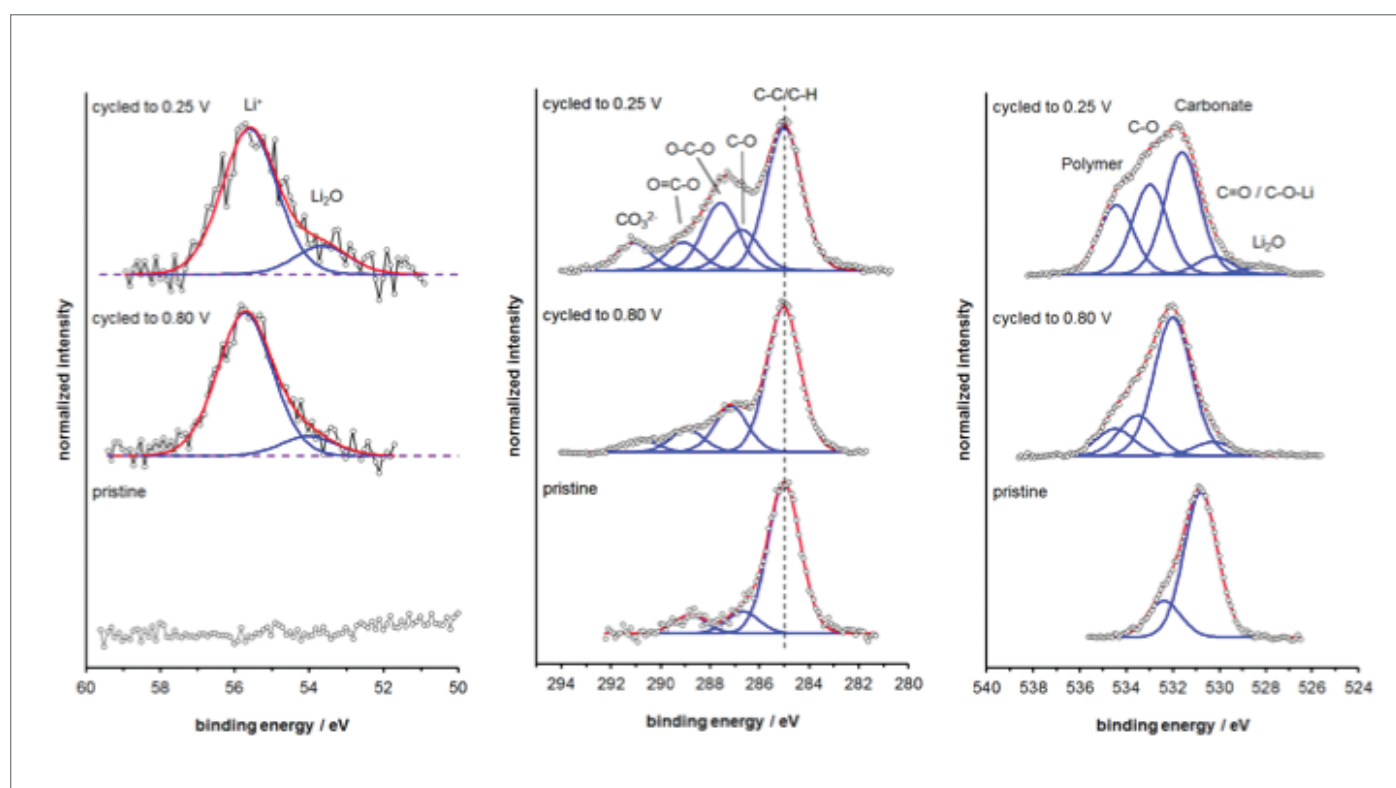


Figure 1: C 1s, O 1s, and Li 1s XP spectra of a pristine electrode surface and electrodes surfaces cycled to 0.8 V and 0.25 V, respectively.

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