High-resolution 3D chemical imaging of light elements by time-of-flight mass spectrometry

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Background incl. aims
It is a common practice to correlate multiple imaging techniques to achieve a comprehensive understanding of the interplay between material structure and function. Beyond the geometric structure, the chemical composition, particularly at the nanometer scale, plays an essential role. However, there is a shortage of instruments capable of imaging light-mass elements with sufficiently high spatial resolution and sensitivity. This gap is addressed by TOFWERK's fibTOF platform, offering high spatial resolution and 3D chemical imaging of all the elements of the periodic table, including light-mass elements. This capability is demonstrated through the application examples below, which are taken from the battery and metallurgy sectors.

Methods
fibTOF is a Secondary Ion Mass Spectrometry (SIMS) detector designed to be attached to a Focused Ion Beam (FIB) Scanning Electron Microscope (SEM). fibTOF extends microscopic analysis to probe the chemical composition of solids at the nanometer scale with sensitivities down to the ppm range. The properties of a FIB as a primary ion source to erode and ionize material is used. For each pixel probed by the FIB, the released secondary ions are extracted and characterized. The combination of the mass spectra/pixel, results in an intensity map of elements for a mass range of 0-500 Th. As the FIB repeatedly scans the region of interest (ROI), a 3D data set is generated.

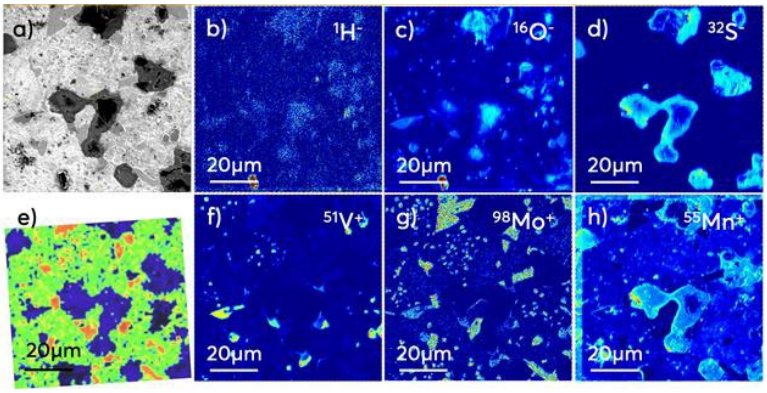
Results
The value of the fibTOF is demonstrated by the investigation of a Solid-Electrolyte Interface (SEI) composition at the anode after galvanostatic cycling. We explore how various molecular additives affect its stability. Lithium ions have been observed at various penetration depths, indicating differences in SEI formation efficacy based on the additives used. The lower SEI formation efficiency explains the diminished battery performance due to ongoing electrolyte degradation. In addition, measurements of Mn and Ni on the anode corroborate irreversible phase changes and metal dissolution of the cathode.
The use of the fibTOF has been shown as well in correlation with hardness tests done by nanoindentation of an M3 high-speed steel alloy. Certain regions of the sample, chemically characterized as hard phases, exhibited soft

mechanical properties. These regions also showed the presence of hydrogen, hinting at a potential link to hydrogen embrittlement. Notably, both case studies were carried out using elevated sputtering and detection rate of the fibTOF.

Conclusions

The studies presented demonstrate fibTOF's efficacy in light-mass element imaging, showcasing its versatility and reliability in analyzing diverse materials and complex microstructures. Furthermore, the demonstrated capability of fibTOF to manage enhanced signals bolsters its potential for a broad spectrum of applications.

Graphic:



Keywords:

FIB-SIMS, high-resolution imaging, lithium, battery

Reference:

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Nano-chemical Imaging and Spectroscopy at the Single-molecule Level

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Background
Biological processes rely on a wide class of biomolecular and macromolecular machines that have nanoscale physical dimensions and whose function emerges from a correlation between their chemical and structural properties. A fundamental objective of modern analytical methods is the comprehension of how physical-chemical properties and heterogeneity of single biomolecules underlie their role in cellular function and disease. While innovative nanoscale imaging methods have been developed to characterise biomolecules, imaging microscopies are to the most part chemically blind; thus, hampering the characterisation of inhomogeneous and complex systems.

Methods
The introduction of photothermal infrared nanospectroscopy (AFM-IR) has revolutionized the field of nano-chemical analysis in a wide-open range of fields, including biological, material and polymer sciences. Here, we will present an overview of our latest development and application of AFM-IR in combination with advanced spectroscopic analysis and chemometrics, as a real breakthrough for the analysis of heterogeneous (bio-)molecular systems and materials down to the single molecule level.

Results
To illustrate our path towards single-molecule AFM-IR, we first show the achievement of single protein molecule detection of infrared absorption spectra and maps by introducing off-resonance, low power, and short pulse ORS-nanoIR. [1] This approach enables the accurate determination of the secondary structure elements of single proteins and amyloids in the amide band I region. We will then showcase the application of this unprecedented single molecule sensitivity to: i) unravel molecular structure and interactions of protein and organic molecules [2]; ii) origin of chirality in click chemistry polymers [3]. Finally, we illustrate the application of this sensitivity to probe the surface and structural properties of functional materials, such as artificial model membranes [4] and functional protein self-assemblies [5].

Conclusion
Overall, our aim is to expand the capabilities of analytical nanoscience to shed light on the structure-activity relationship of biomolecules and functional materials design.

Keywords:

Nano-Imaging;Spectroscopy;Atomic Force Microscopy; Supramolecular Assembly

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