KNMF Laboratory for Microscopy and Spectroscopy

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is available only in less than 1.000 industrial and academic laboratories worldwide. We can look back on almost 100 publications of KNMF projects including ToF-SIMS studies published in the last 5 years and several collaborations with industrial partners. Most of this work is related to polymers and organic compounds, together with a focus on biological applications, but also semiconductors, metal organic frameworks on other scientific fields are included in our expertise. SIMS is – complementary to XPS – a surface analysis technique providing elemental and molecular information at high lateral resolution. A focused high energy ion beam is used to bombard the surface of the sample releasing characteristic fragments of the material to be analyzed. Secondary ions are mass separated and counted resulting in a mass spectrum of the sample (information depth approx. 2 nm). The lateral distribution of chemical functionalities can be obtained by rastering the primary beam and the sample itself. ToF-SIMS is ideally suited for the analysis of polymers, or thiol self-assembled monolayers, as well as surfaces from technical applications and environmental studies. Depth profiling and 3D imaging is performed by applying a sputter ion source eroding the sample with cesium, oxygen, or argon cluster ions. Charge compensation on insulating samples is facilitated by an electron flood gun.

Several data processing tools allow for the analysis of complex sample chemistries. These approaches include principal component analysis of the multidimensional spectra or images and "gentle-SIMS" to correct for some fragmentation effects.

Contact

See KNMF website or contact the KNMF User Office.

Equipment

ToF.SIMS^5-100, ION-TOF GmbH, equipped with a liquid metal cluster ion source, and several sputter sources.

Features

- Bi/Mn Primary Ion Source (Bi^+, Bi_3^+, Bi_3^{++}, Mn^+)
- Mass resolution: up to 11000 m/Δm @ 29 amu (bunched mode)
- Spatial resolution < 150 nm (collimated mode)
- Surface sensitivity < 5 nm
- Cs thermion source and O_2 EI source for sputter depth profiling, Zalar-rotation possible
- Argon cluster ion source for analysis and sputter depth profiling of organic samples
- Transfer vessel for atmosphere contact free sample transport from glove boxes to the spectrometer
- Sample heating and cooling in UHV
- Max. sample size: 6×7 cm

Limitations/constraints

All elements and isotopes are detectable, the sample has to be solid at RT and stable under vacuum conditions. Most biological samples require fixation, (freeze-)drying or other preparations. Quantification requires standards or calibration based on complementary techniques available within the KNMF. Detection limits: 10 ppm for elements, sub-fmol for molecules. Dynamic SIMS is destructive because particles are removed from the surface.

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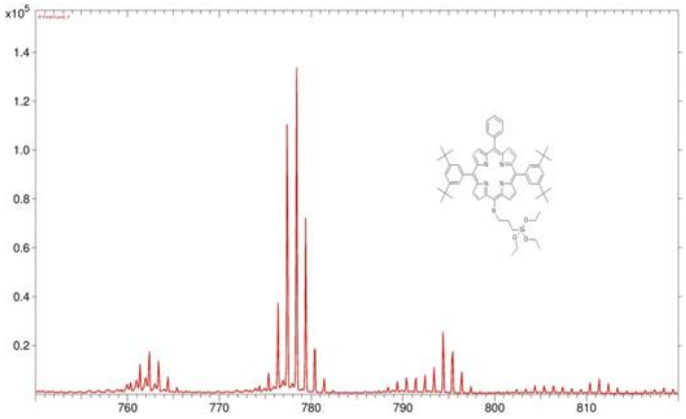
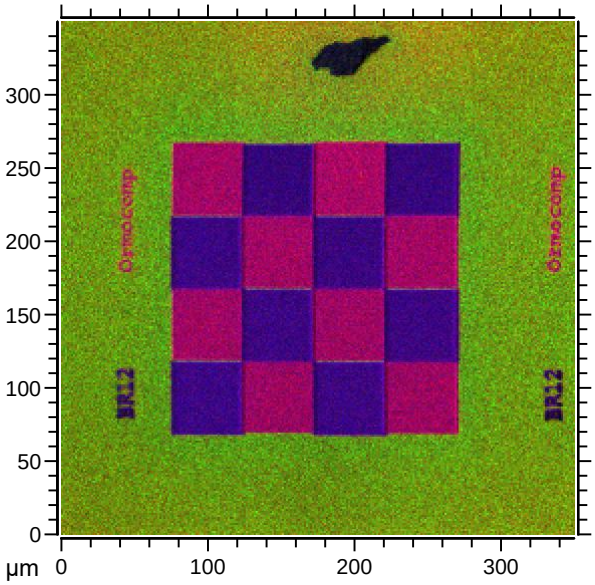
Typical results



High mass range of a positive polarity secondary ion spectrum of a porphyrin derivative immobilized via silanol groups onto a silicon wafer (Bi_3^+).

The multipletts reproduce the isotope distribution of this molecule and can be unambiguously assigned to the porphyrine headgroup, $C_{54}H_{58}N_5$, etc.



Imaging with high lateral resolution and good spectral quality by "delayed extraction mode". Functionalized polymer board. Red: $^{79}Br^-$ + $^{81}Br^-$, green: glass signals, blue: ethylene glycol signals.
[DOI: 10.1002/anie.201509937]

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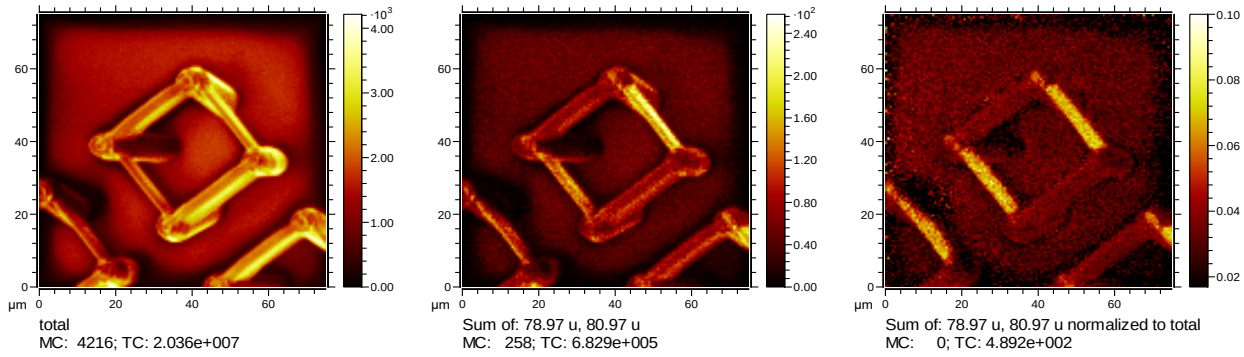

Imaging with high lateral resolution and good spectral quality by "delayed extraction mode". Two horizontal bars of the 3D polymer structure obtained by two photon 3D Direct Laser Writing, see KNMF home page (http://www.knmf.kit.edu/3D-DLW.php), a selectively post functionalized by bromination. [DOI: 10.1002/anie.201509937]

The video:
https://pubs.acs.org/doi/suppl/10.1021/acsami.8b11525/suppl_file/am8b11525_si_003.avi shows the 3d structure of a patterned poly(2-methacryloyloxyethyl phosphorylcholine) grafted on a SI-ATRP initiator film. [DOI: 10.1021/acsami.8b11525]

The video
https://pubs.acs.org/doi/suppl/10.1021/acsami.8b11525/suppl_file/am8b11525_si_002.avi shows the isosurface of the bromine signals of a PPX-EB sample prepared on a gold coated silicon wafer substrate irradiated for 20 minutes with UV. The sample was eroded with 5 keV argon cluster ions and repeatedly imaged for $^{79}Br^-$, $^{81}Br^-$ and several gold clusters in a high lateral resolution mode ("burst alignment"). Bromine levels were computed as an isosurface. X=Y=100µm, Z not to scale. [DOI: 10.1021/acsami.8b11525]

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