1976
doi:10.1017/S1431927620019996

Microsc. Microanal. 26 (Suppl 2), 2020

npSCOPE: A New Instrument Combining SIMS Imaging, SE Imaging and Transmission Ion Microscopy for High Resolution In-situ Correlative Investigations

Olivier De Castro[1], Antje Biesemeier[1], Eduardo Serralta[2], Nico Klingner[2], Gregor Hlawacek[2], Peter Gnauck[3], Serge Duarte Pinto[4], Falk Lucas[5], Cecilia Bebeacua[5] and Tom Wirtz[1]

[1]Luxembourg Institute of Science and Technology (LIST), Belvaux, Grevenmacher, Luxembourg, [2]Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Sachsen, Germany, [3]Carl Zeiss Microscopy GmbH, ZEISS Group, Oberkochen, Baden-Wurttemberg, Germany, [4]Photonis Netherlands B.V., Roden, Drenthe, Netherlands, [5]ETH Zurich, Zurich, Zurich, Switzerland

In various research areas ranging from materials science to life sciences it becomes more and more important to be able to analyze the structure as well as the chemical composition at the nano-scale. For example, the size of electronic components becomes smaller and smaller increasing the need of having techniques to precisely follow dopant distributions with high spatial resolution. In the field of renewable energy devices, e.g. solar cells and batteries, the performance typically depends on the chosen material composition and distribution. Linking the underlying structure and composition at the nano-scale to the device's performance is therefore of utmost importance [1,2]. Similar needs for having high spatial resolution and high-sensitivity chemical information can be found in life sciences [3]. In nano-toxicology for instance, it is important to be able to reveal sub-cellular structures and simultaneously determine their chemical, elemental or isotopic composition in order to better understand relevant processes [4]. In most of the afore mentioned studies a number of different instruments is nowadays used to perform these investigations using correlative approaches. Being able to do such correlative studies in one single instrument is definitely beneficial for reducing the analysis time, speeding up the throughput as well as for facilitating the precise localization of the region of interests on the investigated samples.

Therefore, we developed a multimodal nano-analytical platform allowing in-situ analysis of a same sample using different information channels. The instrument is equipped with the ultra-high resolution Gas Field Ion Source (GFIS) technology [5] allowing the sample to be irradiated with very finely focused He^+ and Ne^+ primary ion beams. This allows sub-nanometer spatial resolution when working with the secondary electron (SE) detection mode as imaging mode. Furthermore, the instrument incorporates a compact secondary ion mass spectrometer (SIMS) for chemical analysis of samples with excellent sensitivity and high dynamic range. The mass spectrometer is based on a double focusing magnetic sector design and allows sub-20 nm chemical imaging resolution [6-8]. Moreover, the SIMS system incorporates a new kind of detector for parallel mass detection providing a full mass spectrum for each analyzed pixel. The third newly developed detection mode available within the instrument is a position sensitive transmission detector located at the backside of the sample in order to detect the transmitted He beam. This scanning transmission helium ion microscopy (STHIM) mode provides further in-situ structural/compositional data with tomographic capabilities.

In order to optimize the analysis of biological samples, one further key feature of the instrument is a 5-axis cryo-stage along with cryo-capabilities for sample transfers. This cryo-capability allows biological samples to be analyzed in a frozen-hydrated state, thus avoiding artefacts caused by classical sample preparation (e.g. chemical fixation) used for HV or UHV imaging of biological specimens at room temperature. Moreover, the cryo-mode can be beneficial for analyzing beam sensitive samples in materials science such as OLEDs and polymers.

In this work we will present the npSCOPE concept and the instrument's overall setup, report on the performance of the different detection modes and discuss the correlative microscopy capabilities. We will present results from case studies in different fields, with a particular focus on nanoparticles (see Figure 1) [9].

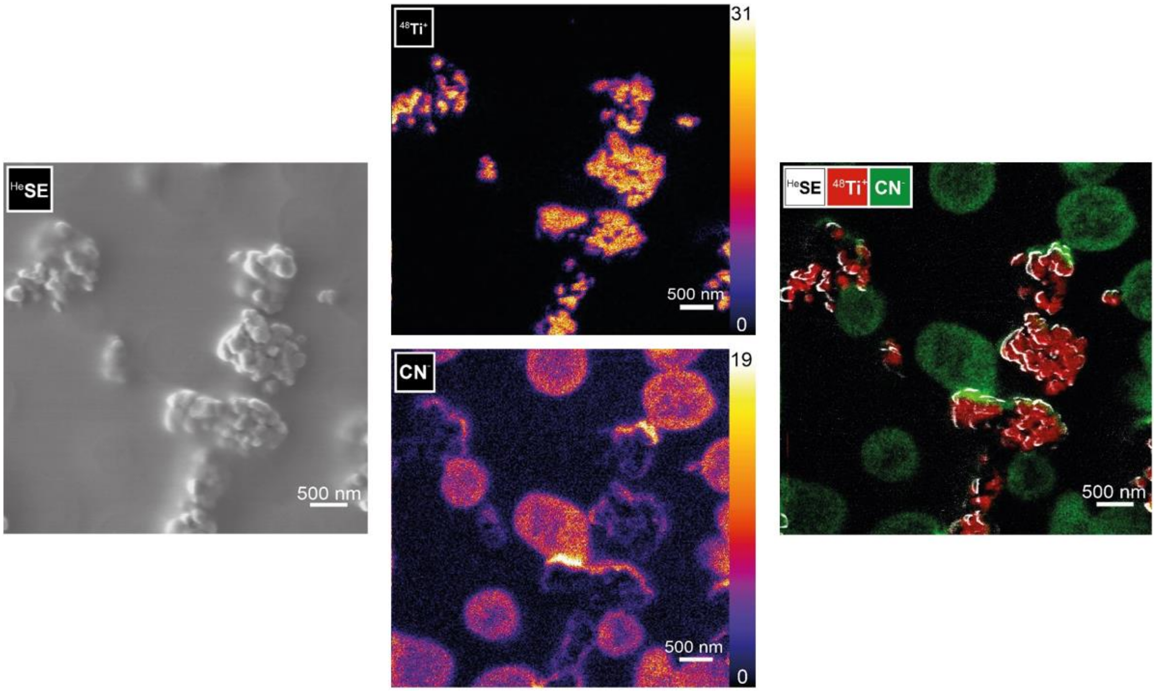

Figure 1. Food-grade TiO_2 nano-particles within *Escherichia coli* bacteria matrix**: Left: SE imaging mode (He+, 20 keV/6 pA); Middle: SIMS imaging mode for Ti+ and CN- signal (Ne+, 20 keV/5 pA); Right: Overlay image of SE imaging and SIMS imaging modes.

References

[1] S. Eswara, A. Pshenova, L. Yedra, Q. H. Hoang, J. Lovric, P. Philipp and T. Wirtz; *Appl. Phys. Rev.* 6, 021312 (**2019**).
[2] P. Gratia, I. Zimmermann, P. Schouwink, J.-H. Yum, J.-N. Audinot, K. Sivula, T. Wirtz and M. K. Nazeeruddin; *ACS Energy Lett.* **2017**, 2, p. 2686-2693.
[3] J. Lovric, J.-N. Audinot and T. Wirtz; Microsc. Microanal. 25 (Suppl 2), **2019**.
[4] D. R. Boverhof and R. M. David; *Anal. Bioanal. Chem.* 396 (**2010**), p. 953-961.
[5] B. W. Ward, J. A. Notte, N. P. Economou; *J. Vac. Sci. Technol. B* 24/6 (**2006**), 2871.
[6] T. Wirtz, D. Dowsett, P. Philipp; *SIMS on the helium ion microscope: a powerful tool for high-resolution high sensitivity nano-analytics;* Helium Ion Microscopy (**2016**), ed. G. Hlawacek, A. Golzhäuser, 297.
[7] D. Dowsett, T. Wirtz; *Anal. Chem.* 89 (**2017**), 8957.
[8] T. Wirtz, O. De Castro, J.-N. Audinot, P. Philipp; *Ann. Rev. Anal. Chem.* 12 (**2019**).
[9] This project has received funding from the European Union's Horizon 2020 Research and Innovation Programme under grant agreement No. 720964.
***The investigations on these samples have been done in the framework of a collaboration with Muriel Mercier-Bonin from Toxalim, Toulouse, France.*

https://doi.org/10.1017/S1431927620019996 Published online by Cambridge University Press