doi:10.1017/S1431927622001039
Microsc. Microanal. 28 (Suppl 1), 2022
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SIMS Performed on Focused Ion Beam Instruments : In-situ Correlative Structural and Chemical Imaging

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Engineered nanomaterials have become the focus of intensive research in diverse areas, including microelectronics, aerospace, production and storage of energy, toxicological studies and medical applications. The development of new characterization methodologies and instruments is a key factor to push forward materials' research and development, and thus to make improvements in product performance and reliability. Analytical challenges include the analysis of very small features in the range of 10 nm, leading to a trade-off between analyzed volume and detection limit. Secondary Ion Mass Spectrometry (SIMS) is a powerful surface analysis technique, in particular to its ability to detect all elements with excellent sensitivity and high dynamic range and to differentiate between isotopes. SIMS allows to acquire mass spectra, to perform depth profiling, and 2D and 3D imaging. The development of new ion sources installed on the latest generation of FIB platforms, e.g. the Gas Field Ion Source (GFIS), the Cs^+ Low-Temperature Ion Source (LoTIS) or multi-species Liquid Metal Alloy Ion Source (LMAIS), open up new possibilities for the analysis of nanosized objects. Adding SIMS capability to FIB instruments offers not only imaging with highest resolution and sensitivity, but also a tool for in-situ process control during patterning and milling [1,2].

We developed compact magnetic sector SIMS systems specifically designed and adapted for the ZEISS ORION NanoFab Helium Ion Microscope (HIM) [1-3], Thermo Fisher DualBeam platforms [4] and the ZeroK FIB:ZERO, a FIB platform with a Cesium LoTIS [5]. These instruments are capable of producing elemental SIMS maps with sub-15 nm lateral resolution, while maintaining the performance of the FIB platform in terms of secondary electron (SE) imaging and nanomachining. These instruments have the capabilities to provide structural and chemical information with a unique spatial resolution and analytical sensitivity. Moreover, these FIB-SIMS platforms allow for correlative imaging in a single instrument by combining SIMS with other analytical or electron microscopy data available on these tools (SE, STEM, EDS, BSE, EBSD). This allows to go beyond traditional data acquisition workflows and processing methods. To this end, complete acquisition workflows and multimodal image processing approaches were then specifically developed for the afore-mentioned FIB platforms [6,7].

Here, we will present our SIMS systems to solve current and future analytical challenges in a variety of different fields. On a microelectronics sample, small domains were analyzed, both in depth and with 2D imaging. By stacking consecutive 2D image planes, a 3D volumetric representation was created. In petrochemistry, we studied the modification of chalk from sub-marine oil reservoirs after exposition to a $MgCl_2$ fluid, for future enhancement of the oil recovery (EOR) process. Thus, the chemical and structural mineralogical phases were linked to the exposed surface of the micro-sized coccoliths (Figure 1). In life science, FIB-SIMS was used to localize environmental (toxic) chemicals inside cells. The

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https://doi.org/10.1017/S1431927622001039 Published online by Cambridge University Press

relation between cellular viability and the local accumulation of these substances was investigated to demonstrate the effect of the pollutant concentration on the cell metabolism [8].

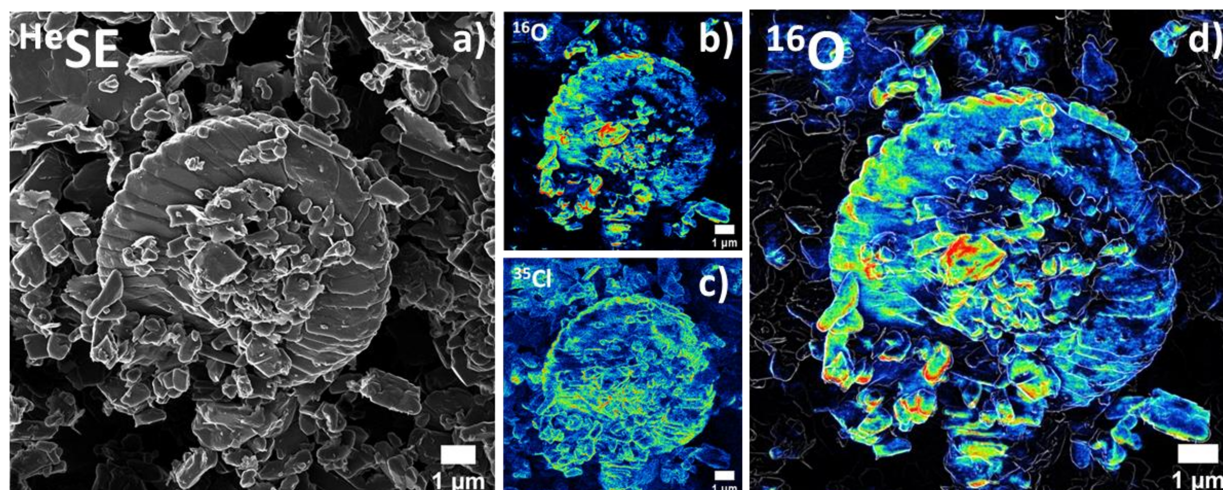

Figure 1. HIM-SIMS analysis of chalk exposed to a $MgCl_2$ fluid. a) Secondary Electron (SE) image obtained with a 25 keV He^+ beam with a coccolith fragment in the center of the image. b) and c) SIMS images of Oxygen and Chlorine acquired with a 25 keV Ne^+ beam. d) Correlation of the SE and ^{16}O SIMS image by pixel fusion allowing a correlation between the sample structure and its chemical composition [2,6].

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[8] The authors gratefully acknowledge support by the Luxembourg National Research Fund (FNR) under grant no. INTER/DFG/17/11779689, and grant no. INTER/ANR/18/12545362.