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4D Reconstructions from Microscale Photogrammetry: Correlation of 3D Surface Representations with SIMS to Link Microstructural Topography and Chemical Information

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Understanding materials' transformation processes at the relevant spatial scales requires the development of high-resolution 3D visualization methods for micro- and even nano-sized objects. The latest generation of electron microscopy (EM) instruments enables topographical imaging in 2D with a spatial resolution of down to 0.5 nm [1]. On the other hand, Secondary Ion Mass Spectrometry (SIMS) is a highly sensitive surface analysis technique with detection limits down to ppb. Combining EM structural images with chemical maps from SIMS allows to better visualize complementary information and provides a deeper understanding about the sample. While correlating microscopy and spectroscopy has been mostly done with 2D images so far [2], this methodology has been extended recently to 3 + 1 dimensions [3,4], i.e. by creating first a topographic 3D surface model of a region of interest (ROI) and then by overlaying it with chemical images acquired on the same area.

Here Secondary Electron (SE) images were taken with a Helium Ion Microscope (HIM). For the SIMS analyses, we used our SIMS system specifically developed for the HIM, allowing for high lateral resolution elemental imaging (< 20 nm) [5,6]. In addition, a CAMECA NanoSIMS 50L instrument was chosen for imaging of specific isotopes at highest mass resolving power. SE images were taken first in a series around a ROI. Subsequently, a SIMS map of the same ROI was acquired. For data processing, the SE images were implemented into a photogrammetry software and a 3D SE surface model was obtained. The SIMS image was then projected onto the 3D SE reconstruction to obtain a full 4D surface model. Using a numerical processing algorithm, topographical information was extracted from the reconstruction and linked to local intensity of the SIMS signal to better understand the intrinsic properties of the material.

In this contribution, we will present stepwise the workflow for 4D surface reconstruction. We will discuss the advantages of this visualization method compared to simple 2D images by showing applications in materials and environmental sciences. In a first example focussing on photovoltaics applications we will demonstrate how a 4D model of a copper indium gallium selenide (CIGS) sample was generated from experimental data and used to avoid artefacts in determining variations of the concentration of the species of interest. Such artefacts can result from local changes of the sputtering yield due to topography related variations of the incidence angle of the primary ion beam (Figure 1). In a second case study from the field of soil biogeochemistry the impact of surface topography on the deposition of organic matter in soil microaggregates will be presented (Figure 2). Thus, with this methodological development we demonstrate that we move beyond just improving sample visualization and provide a practical tool to study sample structure and chemistry correlatively [7].

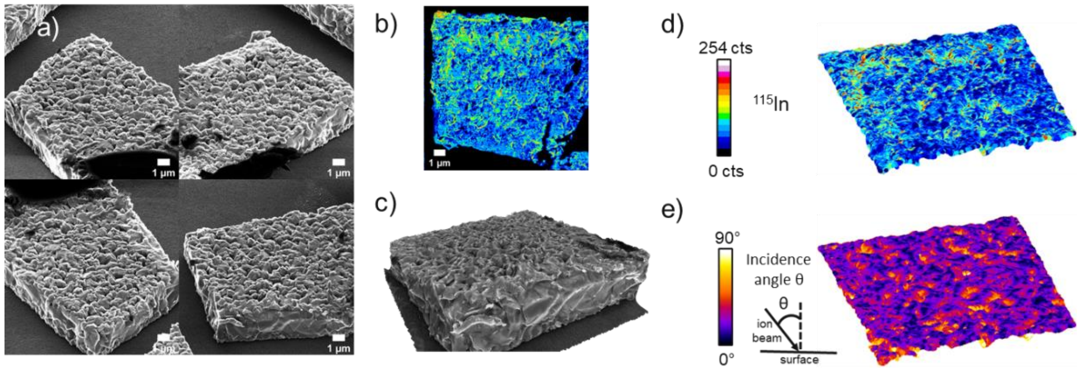

Figure 1. 4D surface reconstruction workflow illustrated on a CIGS structure. a) Serial SE image acquisition around the ROI (48 images in total). b) SIMS image of ^{115}In. c) 3D SE reconstruction by a photogrammetry software using the SE images in a). d) View of the 3D + 1D overlay of the 3D SE model (c) with the SIMS image (b). e) Visualization of incidence angle information obtained from the 3D SE model (c) to study local sputtering yield variations in SIMS with respect to the incidence angle of the primary ion beam.

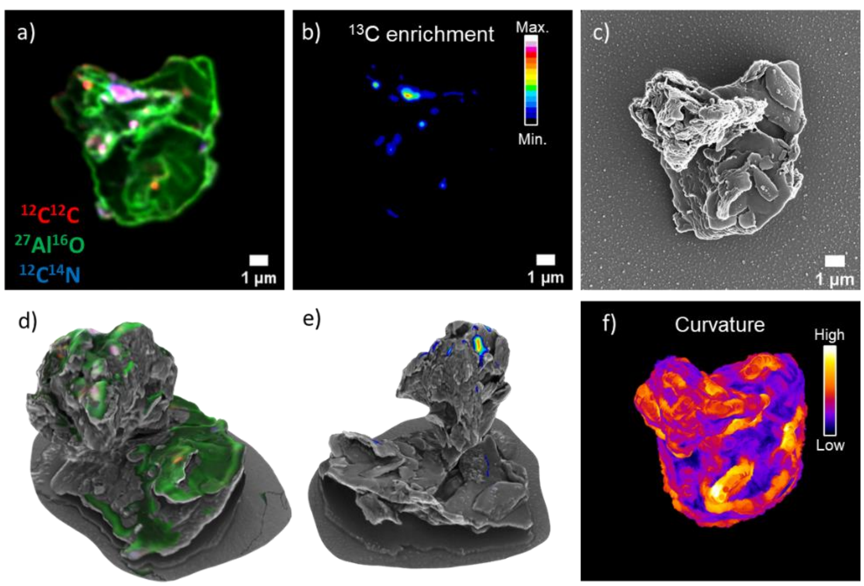

Figure 2. Topographical and compositional investigation of a soil microaggregate reconstructed in 4D (figure adapted from Ost et al. [4]). a) SIMS image of organic matter (red: $^{12}C^{12}C$, blue: $^{12}C^{14}N$) and mineral phase (green: $^{27}Al^{16}O$). b) SIMS image showing the isotopic ^{13}C enrichment. c) HIM SE image in top view. d), e) Overlay of the images in a), b), respectively, on the 3D SE model reconstructed previously from 35 SE images. f) 3D model (in top view) showing in color the local curvature information to study the topography of organic matter hotspots in the microaggregate.

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References:

[1] T. Wirtz et al., Annu. Rev. Anal. Chem. **12** (2019), p. 523. doi:10.1146/annurev-anchem-061318-115457

[2] F. Vollnhals et al., Anal. Chem. **89** (2017), p. 10702. doi:10.1021/acs.analchem.7b01256

[3] F. Vollnhals, T. Wirtz, Anal. Chem. **90** (2018), p. 11989. doi:10.1021/acs.analchem.8b02530

[4] A.D. Ost et al., Environ. Sci. Technol. **55** (2021), p. 9384. doi:10.1021/acs.est.1c02971

[5] J.-N. Audinot et al., Reports Prog. Phys. **84** (2021), p. 105901. doi:10.1088/1361-6633/ac1e32

[6] S. Kim et al., Anal. Chem. **91** (2019), p. 12142. doi:10.1021/acs.analchem.9b03377

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