Enabling TOF-SIMS based correlative analysis with novel sample preparation and data treatment

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The high sensitivity, detection of elements and molecules and relatively good lateral resolution of time-of-flight mass spectrometry imaging has been exploited in many applications ranging from microelectronics to biology. However, it does suffer from limitations and artefacts that can be overcome by using correlative approaches. These can be used to improve quantification, add new information such as physical or electrical properties, correct sputter rate variations in 3D imaging, or supply higher resolution images of the same sample area.

For example, argon cluster sputtering can be used to generate wedge craters of OLED stacks that allows correlative TOF-SIMS and XPS analysis on exactly the same area with so little damage that this method may be used to characterise the degradation in the layers in order to understand device degradation[1]. Another example is in-situ FIB cutting that can be used to preserve 3D morphology better than top-down sputter profiling and even allows 3D reconstruction using a slice and view approach. Performing a FIB cut in-situ is particularly interesting for sensitive samples so that the sample can be analysed directly without any modification. This is well illustrated by recent our work using a tiltable in-situ cryo holder and argon cluster sputtering to cross-section biological samples whilst maintaining temperatures of below -140°C before performing TOF-SIMS imaging[3]. This approach is compared to results from samples that are sectioned ex-situ using cryo-microtomy and transferred and analysed under cryo conditions. Finally, the correlation of high-resolution TOF-SIMS images and SEM images from the same area will be presented alongside novel denoising algorithms to improve signal to noise ratio without sacrificing lateral resolution[4,5].

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[1]Guyot C*. et al Surface and Interface Analysis* **56**(3), 129 (2024)

[2]Priebe A*. et al Journal of Microscopy* **264**(2), 247 (2016)

[3]Seydoux C. *et al, J. Am. Soc. Mass Spectrom.* **37**, 1132 (2026)

[4]Seydoux C*. et al Analytical Chemistry* **96**, 19917 (2024)

[5]Seydoux C*. et al Analytical Chemistry* **97**, 12925 (2025)