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Correlative analysis of beam and air-sensitive materials using TOF-SIMS, SEM and XPS

J-P Barnes^a, C. Guyot^a, P. Hirchenhahn^a, A. G. De Carvalho^{a,e}, N. Gauthier^a, T. Maindron^a, B. Gilquin^a, D. Ratel^a, C. Gaude^a, O. Renault^a, A. Galtayries^b, G. L. Fisher^c, C. Seydoux^d, P-H Jouneau^d,

a Univ. Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France

b Chimie ParisTech, PSL University, CNRS, Institut de Recherche de Chimie Paris, 75005 Paris, France

c Physical Electronics, Chanhassen, Minnesota 55317, United States

d Univ. Grenoble Alpes, CEA, IRIG-MEM, F-38000 Grenoble, France

e current address : Robert Bosch GmbH, 71272, Renningen, Germany

Time-of-flight Secondary Ion Mass Spectrometry (TOF-SIMS) is an essential analysis technique for materials and device development for semiconductor technology. However, to overcome limitations in lateral resolution or quantification issues the use of correlative analysis approaches may be necessary. Fortunately, semiconductor research facilities are able to produce well-controlled, bespoke samples that allow the development of such correlative approaches and the improvement in performance to be assessed. For example scanning probe measurements can be correlated with TOF-SIMS depth profiling data on the same sample to correct for sputter rate differences [1] or to measure mechanical or electrical properties. In a similar fashion, morphological and compositional data from the same sample volume can be obtained by performing X-ray tomography on a post-shaped sample prior to FIB-TOF-SIMS analysis [2].

The availability of cluster ion sources has meant that TOF-SIMS analysis can now be extended to organic and biological materials and meet the challenges of applications such as organic light emitting diodes (OLED) [3], implantable electrodes for decoding brain signals [4] or biomass production in a symbiotic context. In this specific case, the correlation of TOF-SIMS with SEM and AFM measurements and appropriate data treatment can help decipher metabolic interactions between symbiotic partners. For OLED and implantable electrode applications, TOF-SIMS can be correlated with photoemission techniques such as X-ray photoelectron spectroscopy (XPS) or X-ray photoemission electron microscopy (XPEEM) to understand the degradation mechanisms occurring. To ensure that the sample analysis conditions (air exposure, surface contamination, beam damage, ..) do not create sample modifications that could be confused with degradation from sample ageing a wedge crater protocol has been developed. This consists in milling a wedge crater in a sample with a very shallow angle using argon cluster sputtering. This allows exactly the same area of the sample to be analyzed by several technique whilst minimizing beam damage to deeply buried layers. The sample is transferred between instruments under vacuum or inert gas environment. Tandem mass spectrometry (tandem-MS) has been used to help confirm peak assignments in these complex organic samples. In an attempt to increase the signal available for tandem-MS, the effect of MALDI matrices on the secondary ion yield of characteristic secondary ions has been evaluated on simplified OLED samples.

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[1] M. A. Moreno et al. *Journal of Vacuum Science and Technology B*, vol. 36, MAY 2018.

[2] A. Priebe et al. *Ultramicroscopy*, vol. 173, FEB 2017.

[3] Sandrez et al. *Advanced Electronic Materials*, vol. 7, 2021.

[4] A. G. De Carvalho et al. *Biointerphases*, vol. 15, 2020.