Summary

Industry is progressively moving towards complex 3D architectures and using advanced materials and heterogeneous systems, which includes both organic and inorganic materials. Obviously, the performance of such complex 3D systems is also determined by the 3D elemental distributions, e.g. dopant distributions, or chemical compositions at the nanometric scale. Thus, 3D metrology should provide an accurate elemental and chemical measurement solution with a 3D spatial resolution (both lateral and depth) down to the nanometric scale in accordance with the needs of the industry (e.g. down to sub nanometer scale for the semiconductor industry). In this content, time-of-flight secondary ion mass spectrometry (ToFSIMS), grazing incidence X-ray fluorescence (GIXRF) and atom probe tomography (APT) are among the potential enablers to resolve such a 3D spatial resolution. Despite the recent improvements to push ToFSIMS and GIXRF as the reliable 3D measurement techniques, the metrological assessment of such analyses has not been yet well evaluated. This is mainly due to the absence of 3D reference materials and the calibration standards. On the other hand, APT is an inherent three-dimensional technique, which enables elemental identification and quantification at a near-atomic resolution. However, similar to the other aforementioned techniques, the metrological assessment of APT analysis is also hampered due to the absence of the suitable reference materials.

In this project, we have developed several well-characterized organic-inorganic 3D microstructures as the potential reference material (RM) for 3D ToFSIMS. To prepare the 3D nanostructures with the characteristic dimensions below 20 nm as a test vehicle for GIXRF analysis, we exploited the self-assembly of di-block copolymers (DBCs) as the lithography mask. We have also studied in detail the pattern transfer at sub 20 scale into the Si substrate.

In order to develop a potential reference material for APT, we have studied in detail the different aspects of the APT analysis, including ion trajectories, field-of-view (FOV) and the calibration of the different reconstruction parameters. We have studied in detail the FOV for both hemispherical and UV laser induced asymmetric tip shape. In addition, we suggested a new design for an APT specimen, which maximizes the FOV and allows to probe the entire specimen volume in APT (full tip imaging). We have proven the feasibility of full tip imaging both numerically (finite element analysis) and experimentally. To do so, a specimen preparation process was

developed based on standard lithography and etching technique which allows to prepare multiple APT specimens in a repeatable fashion and with a minimized tip to tip variations in view of the tip radius and the shank angle. The developed full tip imaging feature can pave the way for the uncertainty assessment for all the reconstruction parameters and potentially enables a more reliable 3D data reconstruction in APT with the quantifiable uncertainties. In the absence of the certified reference material for APT, we have developed a well-characterized (i.e. traceable) B doped SiGe reference system (i.e. piece of wafer). Relying on this reference, the accuracy and the repeatability of APT analysis in view of Ge and B quantification over the specimen volume has been evaluated using UV and green lasers as well as in the different experimental conditions (electric field).

In addition, the feasibility of APT analysis of an organic-inorganic system based a polyaniline (Pani) - porous silicon (PSi) nanocomposite was evaluated in detail. We demonstrated that such a complex system could be analyzed by APT, whereby the 3D compositional distribution (lateral and depth distribution) was identified according to the distribution of monoatomic ions. The remained challenges for such an analysis were addressed and a potential solution was proposed.