doi:10.1017/S1431927620014944
Microsc. Microanal. 26 (Suppl 2), 2020

Secondary Ion Mass Spectrometry (SIMS) and Atom Probe Tomography (APT): Powerful Synergetic Techniques for Materials Scientists

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Engineers and scientists in both academic and industrial environments currently rely on a fleet of analytical methods to gain full understanding on technologies and processes, or to explore novel research ideas. However, all the characterization techniques populating the resolution-sensitivity landscape have their own strengths and shortcomings [1]. It is in this context that using a synergetic approach between two complementary techniques such as Secondary Ion Mass Spectrometry (SIMS) and Atom Probe Tomography (APT) could magnify the power of these techniques.

Beside its extremely sensitive elemental and isotopic surface analysis capabilities, coupled with its imaging and depth profiling functions, dynamic SIMS offers great depth resolution (DR) and lateral resolution (LR) depending on the models. For instance, using a 100 to 150eV impact energy, a 0.7nm DR is achieved for a modest LR of about 5µm with a CAMECA IMS SC Ultra, a 2nm DR is reached for a great LR of about 1µm with an IMS 7F Auto. This LR can be improved by a factor of 20 (50nm) using the NanoSIMS®, at the expense of the DR (10-20nm) for a 16keV primary ion impact energy.

The time-of-flight based LEAP® and EIKOS® APT systems both use UV laser capabilities to field evaporate ions and allow 3D image reconstruction of a needle-shape material at the atomic level along with the determination of chemical composition. Ion efficiencies as high as 80%, 0.1 to 0.3nm depth resolution and 0.3 to 0.5nm lateral resolution, provide great complementarity to the dynamic SIMS systems.

We will illustrate the benefits of these two techniques in correlative studies on Gallium Nitride (GaN) semiconductors used in high power and high efficiency light-emitting diodes (LEDs). First, SIMS was used to rapidly monitor the incorporation of impurities into GaN crystals during a sodium flux growth [2,3]. Quantum wells (QWs) and superlattice (SL) regions are quantified to the ppb range using the IMS 7F Auto, as shown in Figure 1. In a second study, APT analysis enabled 3D exploration of structures such as a Mg p-GaN layer, an Al-rich electron blocking layer (EBL), an In-rich multi QW region, as well as an In-based SL structure [4]. V-defects, unexpected features not easily detectable using SIMS, were unveiled using UV-laser APT in other GaN systems. A V-defect is illustrated by a 5at% Al isosurface in the YZ plane from a 110-nm atom map slice of the volume in the X direction, as shown in Figure 2. These results could help deciphering a root-cause for the failure of these type of LED devices.

The final case pertained to the research and development of silica-based optical fibers containing dielectric nanoparticles (DNPs). NanoSIMS®, ZEISS Orion NanoFab® and APT instruments were used to guide glass technologists and optical scientists in their quest for controlling the spectroscopic properties of their glass, based on the quantified chemical composition and size evolution of these DNPs, especially with respect to rare-earth (RE) elements. Although it was initially believed that the DNPs should be as small as possible in order to limit losses by light scattering, experimental results demonstrated that the smallest DNPs had compositions close to that of the matrix from which the fiber was derived, and were therefore not as important as initially thought for modifying the luminescence properties of RE ions. In addition, it was demonstrated that the first amorphous nuclei grew while changing composition, until they later evolved to the level necessary for crystallization. The validation of these correlated SIMS/APT results for several material systems currently allows theoreticians to refine their models and offers a much greater visibility to these glass scientists working with DNPs ranging from 2 to 200nm [5].

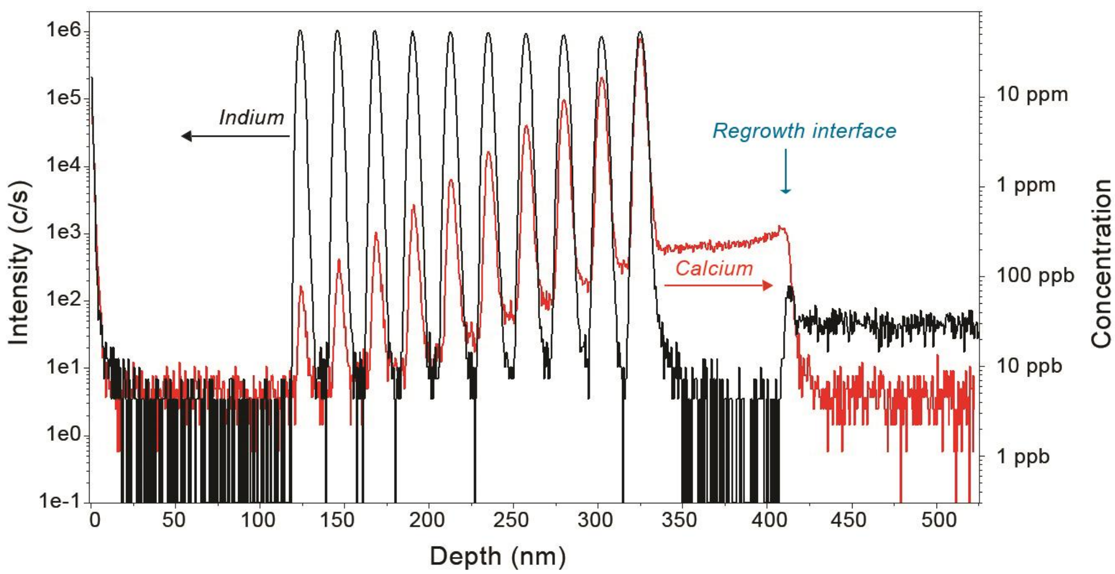

Figure 1. SIMS depth-profiles of QWs in GaN devices. In and Ca impurities are quantified in the ppb range.

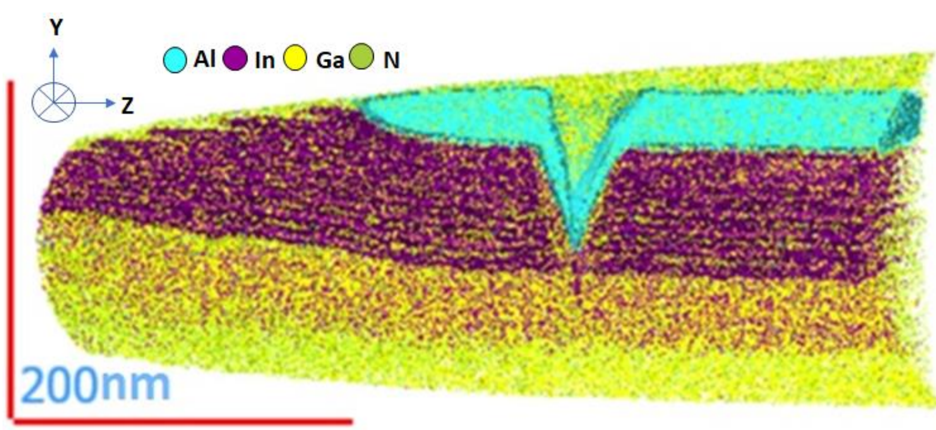

Figure 2. Atom map of GaN device analyzed by APT. The V-defect, shown by the 5at% Al isosurface, has affected the EBL and QWs regions.

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https://doi.org/10.1017/S1431927620014944 Published online by Cambridge University Press