

Leveraging Complementary Electron Microscopy and Microanalysis Techniques to Reveal Localized Structure, Chemistry, and Functionality in Compound Semiconductors

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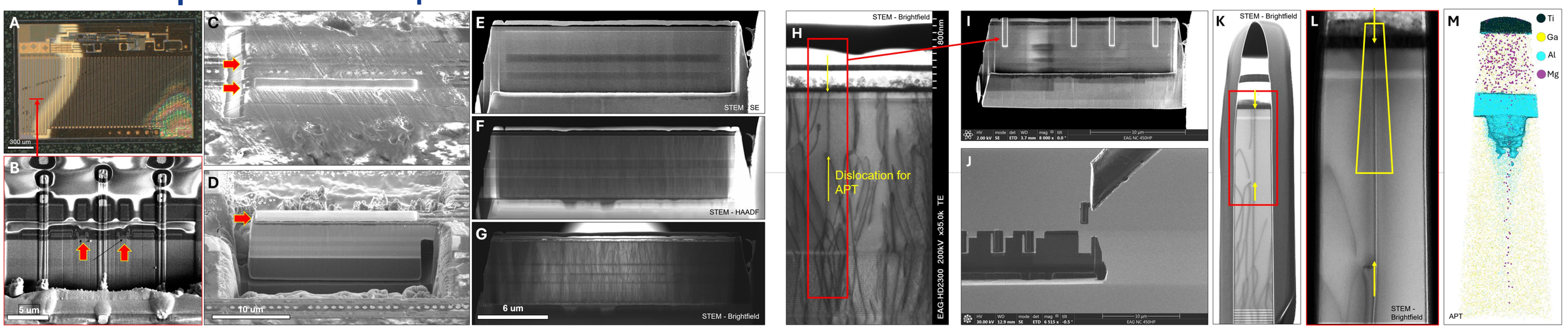
Introduction

The performance of advanced compound semiconductor materials and devices is strongly influenced by highly localized variations in crystal structure, defect populations, dopant distributions, and impurity incorporation. These features often span multiple length scales and can significantly impact device functionality despite being difficult to detect using conventional characterization techniques alone. As compound semiconductor architectures continue to increase in complexity, establishing direct relationships between material structure, composition, and functionality requires the integration of multiple complementary analytical techniques.

Modern electron microscopy has evolved into a multimodal characterization platform capable of evaluating structural, compositional, and functional information within a common workflow. Electron imaging, diffraction, spectroscopy, and cathodoluminescence (CL) data can be utilized to guide further site-specific specimen preparation using focused ion beam (FIB) methods and advanced microanalytical techniques such as atom probe tomography (APT). Together, these approaches enable targeted investigation of defects, interfaces, and localized functional responses across length scales ranging from complete devices to individual atomic defects.

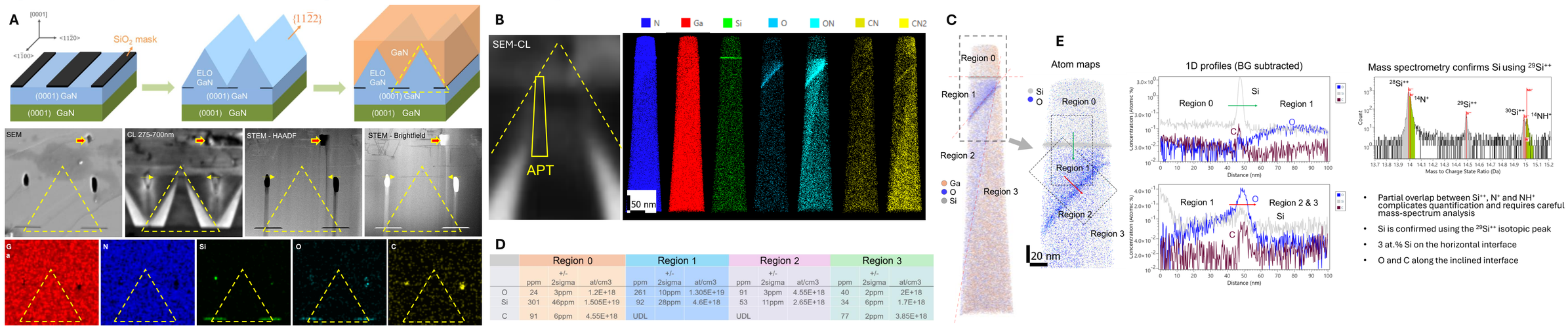
This work highlights two representative examples demonstrating the power of correlative characterization for compound semiconductor analysis. In the first example, optical microscopy, scanning electron microscopy (SEM), FIB, scanning transmission electron microscopy (STEM), and APT are combined to identify, classify, and chemically characterize an individual threading screw dislocation within a commercial GaN power device. In the second example, SEM-cathodoluminescence (SEM-CL), STEM with energy dispersive X-ray spectroscopy (STEM-EDS), and APT are used to investigate anomalous luminescence behavior in Facet-Controlled Epitaxial Lateral Overgrowth (FACELO) GaN epitaxy. By integrating complementary structural, chemical, and functional measurements, these workflows establish direct relationships between crystallographic defects, impurity distributions, epitaxial growth processes, and device-relevant behavior that would be difficult or impossible to obtain through any individual characterization technique alone.

Correlative workflow for observation of Mg diffusion along an individual threading screw dislocation in a lateral GaN power device: Optical + SEM + FIB + STEM + APT



A commercial GaN power device extracted from a consumer phone charger was mechanically deprocessed by parallel lapping to remove the packaging and expose the active device structure. Optical microscopy and SEM imaging within a DualBeam FIB-SEM (A-D) were used to establish the device architecture and select a specific region for further defect analysis. A cross-sectional lamella aligned along the gate foot was prepared by FIB and initially thinned to approximately 500 nm thickness for subsequent STEM characterization of threading defects and conversion to APT samples. STEM characterization and diffraction-contrast g·b analysis (E-H) were performed to classify threading defects within the region of interest as edge-, mixed-, or screw-type dislocations, enabling selection of an isolated threading screw dislocation for further investigation. The targeted dislocation was subsequently located within a three-dimensional FIB-prepared pillar on a TEM/APT support grid using a proprietary correlative marking and navigation technique (I-L). Following APT needle fabrication, three-dimensional atom probe reconstruction (M) revealed localized Mg enrichment extending along the dislocation line, providing direct evidence of screw dislocation-mediated Mg transport in GaN epitaxial structures. By continuously tracking a single defect from device-scale inspection through atomic-scale chemical analysis, this multimodal workflow establishes a direct relationship between an atomic-scale defect structure and dopant transport.

Spatially Resolved Dopant Level Analysis of GaN FACELO: SEM-CL + FIB + STEM + EDS + APT



D

	Region 0			Region 1			Region 2			Region 3		
	ppm	+/- 2sigma	at/cm3	ppm	+/- 2sigma	at/cm3	ppm	+/- 2sigma	at/cm3	ppm	+/- 2sigma	at/cm3
O	24	3ppm	1.2E+18	261	10ppm	1.305E+19	91	3ppm	4.55E+18	40	2ppm	2E+18
Si	301	46ppm	1.505E+19	92	28ppm	4.6E+18	53	11ppm	2.65E+18	34	6ppm	1.7E+18
C	91	6ppm	4.55E+18	UDL			UDL			77	2ppm	3.85E+18

- Partial overlap between Si^{++}, N^{+} and NH^{+} complicates quantification and requires careful mass-spectrum analysis
- Si is confirmed using the $^{29}Si^{++}$ isotopic peak
- 3 at.% Si on the horizontal interface
- O and C along the inclined interface

The FACELO (Facet-Controlled Epitaxial Lateral Overgrowth) growth sequence and resulting epitaxial structure are illustrated schematically (A, top row). FACELO utilizes selective-area epitaxy and lateral overgrowth from inclined crystal facets to control strain, improve crystal quality, and facilitate heteroepitaxial integration. The SEM-CL dataset was acquired from a cleaved cross-section and subsequently converted via FIB for STEM analysis of the same location. Correlative SEM, SEM-cathodoluminescence (SEM-CL), STEM-HAADF, and STEM bright-field images are shown in (A, middle row). The dashed yellow triangle denotes the approximate ELO growth geometry and enables direct comparison of corresponding features across the different imaging modalities. A common void feature (red arrow, yellow outline) is used as a fiducial marker to identify the same location throughout the SEM, SEM-CL, STEM datasets, while a horizontal planar feature (yellow arrows) is observed in both the SEM-CL and STEM images, enabling direct correlation of optical and structural information. This feature was subsequently targeted by APT and corresponds to the interface between Regions 0 and 1 in the reconstructed atom maps. Corresponding STEM-EDS elemental maps of Ga, N, Si, O, and C are shown in (A, bottom row). While the STEM-EDS data reveal the expected structural and compositional features of the epitaxial growth, the pronounced luminescence variations observed by SEM-CL are not fully explained by the measured elemental distributions, indicating the presence of functionally distinct regions below conventional STEM-EDS detection limits. SEM-CL and STEM observations were subsequently used to guide site-specific APT specimen preparation from the apex region of the ELO GaN containing the planar feature and exhibiting strong luminescence variance. The resulting APT analysis volume and corresponding three-dimensional atom maps are shown in (B-C), while quantitative compositional analysis of Regions 0-3 is summarized in (D). The same horizontal feature identified in the SEM-CL and STEM datasets was subsequently analyzed by APT, where higher-resolution concentration profiles and mass-spectral analysis (E) confirmed localized Si enrichment at the interface between Regions 0 and 1, together with O and C segregation along the inclined growth facet. The combined SEM-CL, STEM, EDS, and APT workflow establishes direct relationships between luminescence behavior, growth morphology, and nanoscale impurity incorporation, revealing previously unrealized impurity segregation associated with the FACELO process.

Discussion-Conclusions

No single characterization technique can fully describe the complex relationships between structure, composition, and functionality. Each technique provides a unique perspective while requiring tradeoffs in spatial resolution, chemical sensitivity, sampling volume, and functional information. The true value of correlative characterization lies in leveraging the unique strengths of each technique and integrating their datasets. By careful analysis of the same feature across multiple modalities and length scales, direct relationships can be established between device-scale performance, microscale structure, nanoscale defects, and ultimately atomic-scale structure and composition, enabling a level of understanding that would otherwise be unattainable through any individual characterization method.

In both examples presented here, electron microscopy served as the critical bridge between device-scale observations and atomic-scale chemical analysis. Techniques such as SEM, SEM-CL, and STEM were used to identify, classify, and precisely localize the defects, interfaces, and growth features governing material behavior, enabling APT to be applied to the specific volumes of interest. By leveraging the complementary strengths of electron microscopy and advanced microanalysis, the rich chemical information obtained from nanometer-scale analysis volumes can be directly related back to overall device structure and performance. When integrated with device-level electrical, optical, and reliability measurements, these workflows provide a pathway toward true root-cause failure analysis by linking observed device behavior to the underlying nanoscale materials mechanisms responsible for that behavior.