LED Device Engineering with Atom Probe Tomography

A Case Study in Competitive Analysis

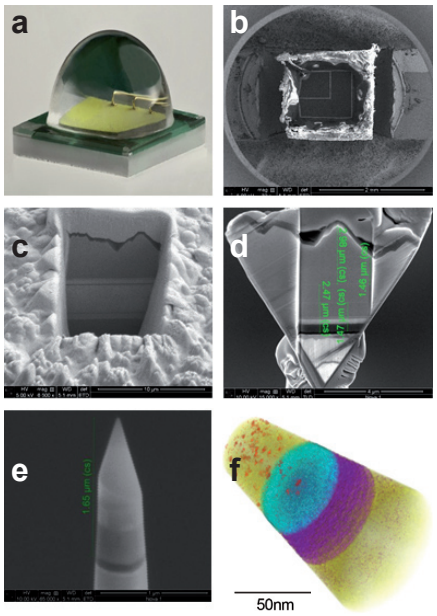
The nanometer scale and embedded nature of semiconductor devices, such as LEDs, means that there has been little recourse for physically confirming suspected intellectual property infringement. However, advancements in microscopy techniques over the past decade now enable new types of analysis as well as unprecedented levels of detail in characterization. One of the principle tools that has enabled this revolution in nano-analysis is the Local Electrode Atom Probe (LEAP). The ability to determine the 3D structure and composition of a material, at near-atomic scale allows the ultimate in "competitive analysis".

In this work, a commercial, high-performance white LED (OSRAM Golden Dragon® Plus) was purchased, depackaged, and prepared for atom probe tomography (APT) using standard lift-out and mount techniques very similar to focused ion beam-based sample preparation for transmission electron microcsopy (TEM).

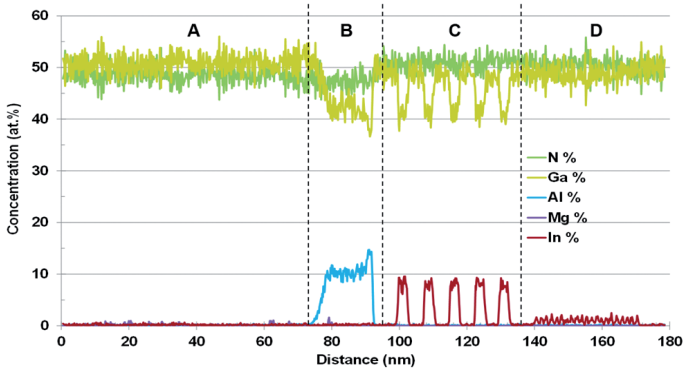
Data from the entire active area of the complex gallium nitride- based semiconductor device was collected on a LEAP system in laser pulsed mode and the resultant data analyzed using the standard, commercially available, software. Analysis of the data reveals four main regions of the device; a region doped to approximately 0.1 at. % Mg (p-type GaN), a graded concen-tration AlGaN region known as an electron blocking layer, and two active regions containing the indium-rich multi quantum well structure and a superlattice structure with 21 single atom thick layers of indium.

This extraordinary analysis demonstrates the now straight-forward process of using standard sample preparation techniques to prepare an 'off the shelf' device for 3D sub-nanometer analysis. Once the sample preparation and data collection is complete, the data can be visualized and analyzed using quantitative methods that allow direct comparison to similar devices with different manufacturing processes to understand what conditions are responsible for the LED's color and performance.

LED device through various stages of deprocessing and APT specimen preparation: (a) purchased device, (b) after depackaging (c) inspection by FIB, (d) lamella mounted onto a APT microtip carrier, (e) formed specimen ready for APT analysis, (f) 3D analyzed volume. See below for a quantitative depth profile.



Four distinct regions are evident from the profile:
A is composed of 0.07% Mg ions, making it the P side of the PN junction.
B contains 10% Al ions, and acts as an electron blocking layer.
C contains a high concentration (7.2%) of In ions in a periodic structure known as a multiple quantum well.
D contains 21 single atom thick structures known as a superlattice.



Adapted from A.D. Giddings et al., Reverse Engineering at the Atomic Scale: Competitive Analysis of a Gallium-Nitride-Based Commercial Light-Emitting Diode. Microscopy Today Vol. 22, Issue 05, September 2014, pp 12-1.

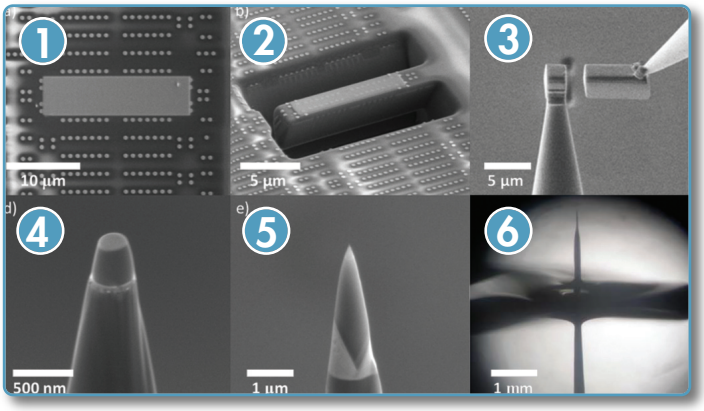
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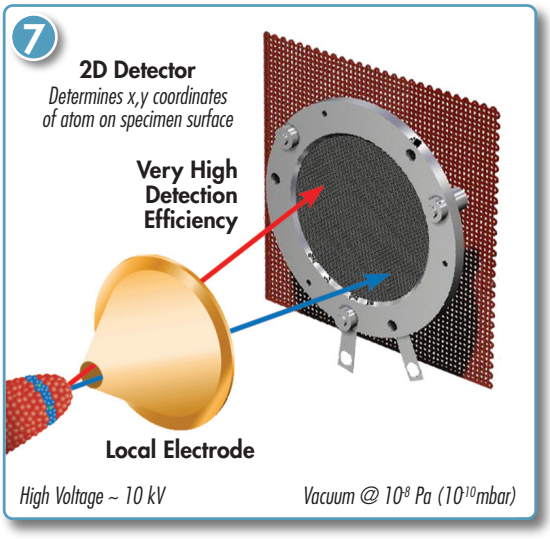
Three Steps to 3D Nanoscale Analysis

An Introduction to Atom Probe Tomography

Step 1: Specimen Preparation

An atom probe specimen usually has a nanoscale region of interest (ROI) requiring both 3D compositional imaging and analysis. The sample is formed into a needle shape containing the ROI. Common APT specimen preparation methods using electropolishing or a Focused Ion Beam system (FIB) are very similar to TEM methods except instead of forming a thin sheet, a needle shaped sample is desired. At the right, standard FIB liftout and mounting of a specimen (figures 1 through 3) and then sharpening the sample with the ROI left at the very apex (4 and 5). In 6, a wire geometry sample is being electropolished.





Step 2: Data Collection

An atom probe produces images by field evaporating atoms from a needle-shaped specimen and projecting the resultant ions onto a detector 7.

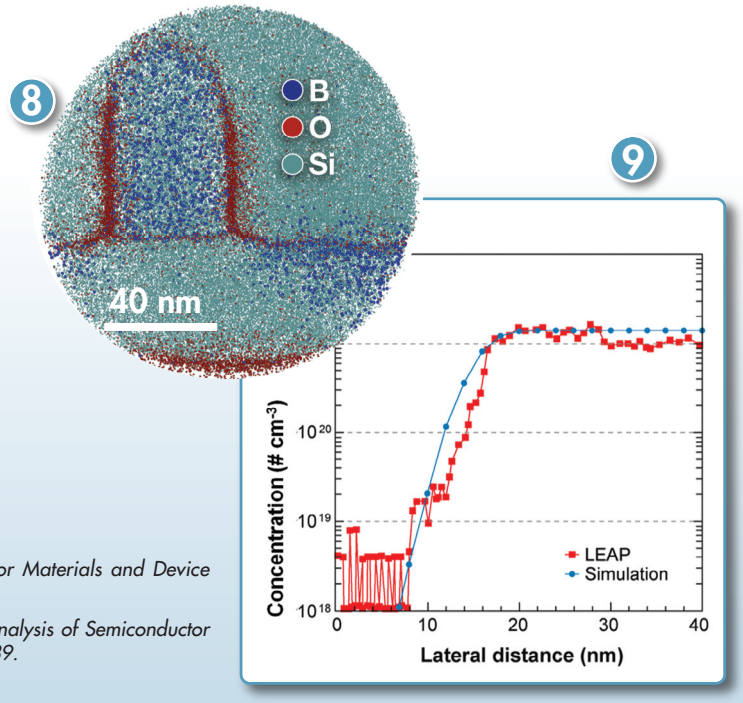
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately 10^6.

An atom probe identifies atoms by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

The specimen is held at approximately 50K to reduce surface diffusion during the experiment. The high electric field results in 100% ionization and the high speed detector is capable of measuring up to 80% of the collected ions, independent of ion mass.

Step 3: Data Visualization and Analysis

Examples of data output are illustrated by a slice of a 3D atom map of a transistor† 8, and a dopant composition profile‡ 9. The image shows the positions of individual atoms (oxygen is red and boron is blue) in the transistor with subnanometer resolution. From the reconstructed data set many types of useful analyses are possible. These include 3D visualization, 2D atom mapping 8, 1D depth profiling and line scanning 9, as well as mass spectra and compositional analysis from user-selected volumes.



† *Lauhon, L. J. et al, MRS Bulletin "Atom Probe Tomography of Semiconductor Materials and Device Structures" 34(10) (2009) 738.*

‡ *Moore, J. S.; Jones, K. S.; Kennel, H.; Corcoran, S., Ultramicroscopy "3-D Analysis of Semiconductor Dopant Distributions in a Patterned Structure using LEAP" (2008), 108, 536–539.*

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