Atom Probe Tomography (APT)

Atom probe tomography (APT) is a nanoscale materials analysis technique that provides 3D (three-dimensional) spatial imaging and chemical composition measurements with high sensitivity.

PRINCIPLES

The technique relies on ionization and subsequent field evaporation of individual atoms/atomic clusters from a specimen surface. The specimen is prepared in the form of a conical tip with an apex radius < 100 nm. The field evaporation occurs as a result of a base DC voltage bias (typically between 1-10 kV) and a pulsed voltage (conductive samples) or pulsed laser (semiconducting and insulating samples). The specimen is also cryogenically cooled down to between 25-80K to suppress thermal lattice vibrations and improve on spatial positioning.

Field evaporated ions are collected onto a position sensitive detector (PSD) to accurately identify x and y position. The sequence of ions collected is used for z position. The x, y and z together provide the spatial imaging of the specimen tip. The time of flight of the ions is also measured and converted to a mass to charge ratio for chemical composition measurements. The spatial resolution is up to 0.3 nm in depth and 0.5 nm laterally, which is limited to metallic samples. The resolution for semiconducting and insulating samples is lower, typically ~ 1 nm at best. The chemical sensitivity for APT is 10 ppm.

With analysis areas that are~ 30-50 nm in x and y and between 100-500 nm in z based on material type, this technique is particularly useful for cases where [a] 3D structures/buried interfaces are present, [b] low atomic number (Z) elements are present, either as dopants or in the bulk and [c] nanometer sized clusters are part of the structure. While APT does not provide the same chemical sensitivity as SIMS (secondary ion mass spectrometry), it enables identification of elements within 3D structures that is not possible with SIMS. It also provides higher chemical sensitivity than TEM/STEM (scanning/ transmission electron microscopy) related techniques such as EDX (energy dispersive X-ray spectroscopy) and EELS (electron energy loss spectroscopy). 3D Compositional analysis of nanoscale clusters is routinely carried out using APT, as it is not possible to measure using TEM/STEM.

The LEAP 5000XR at Nanolab has the latest in instrument capability, with highest detector efficiency, higher signal to noise ratio and larger volumes captured and analyzed compared to older versions of the instrument. These enable capture of low concentration elements in 3D that are not possible to measure using other techniques.

IDEAL USES OF APT

- 3D structure analysis such as FinFET, PMOS cap layer, 3D NAND, curved or non-uniform grain boundaries in photovoltaics.
- Nanoscale precipitates and matrix analysis for composition and chemical identification such as metallic alloys (Al 7075, shape memory, Ti).
- Light element analysis- Be, B, Li, C and Al such as Li-ion batteries.
- Low concentration dopants in 2D and 3D for LEDs, FinFETs, 3D NAND memory and surface coatings of Li-ion batteries.

STRENGTHS

- Small volume dopant mapping.
- Light element (e.g. Li and B) detection and mapping.
- Ability to identify isotopes.
- Equal probability of detection of all elements (H-U).
- Cluster analysis for nanoscale precipitates.
- High mass resolution to identify various elements present in the specimen.

LIMITATIONS

- Significant sample preparation time (~4-6 hours).
- Small sampling volumes.
- Low data acquisition yield as some materials may not be stable in the high electric field.
- Optimized parameters need to be obtained for each material type and structure.
- Low quantification accuracy for certain cases containing different elements with overlap in mass spectrum.
- Spatial resolution and composition accuracy can be limited for heterojunctions or complex geometries due to trajectory aberrations.

APT TECHNICAL SPECIFICATIONS

- Analysis type: 3D
- Material type: Metals, semiconductors, oxides, ceramics
- Elements Detected: H-U
- Detection Limits: ~10 ppm (~5E18 atoms/cm^3)
- Imaging/Mapping: Yes
- Maximum Lateral Resolution: 0.5 nm in lateral and 0.3 nm in depth

Atom Probe Tomography (APT)

- Analysis area: 50*50 nm^2
- Analysis depth: 100-500 nm
- Detected Ion Type: Positive Charged
- Isotope identification: Yes

TYPES OF ANALYSIS

1. Elemental Maps: Observation of individual chemical species in the material/device for homogeneity/heterogeneity and 2D or 3D location is an important aspect of APT analysis. Typically 2D maps and 3D movies are used for this analysis.

a. 2D maps (device level): These are used to provide information about the device structure and to correlate with other techiques.
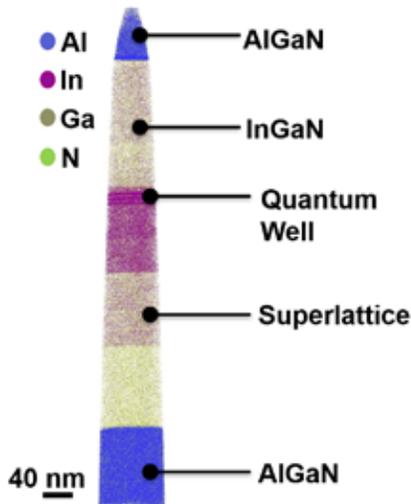

b. 2D maps (material level): Individual element maps can provide information about regions of high and low composition. Individual maps can be taken in any 3D orientation and are based on requirements of the device structure.


2. Compositional Variation: APT provides multiples ways to analyze composition, such as line profile, proxigram and iso-concentration surfaces that are based on depth or concentration.

a. Line profile: Compositional Analysis in 1D along a particular direction can be carried out using a 1D concentration profile. Since APT data is 3D in nature, a 3D volume is chosen and an analysis direction. Line profiles are particularly useful for distance-based analysis.
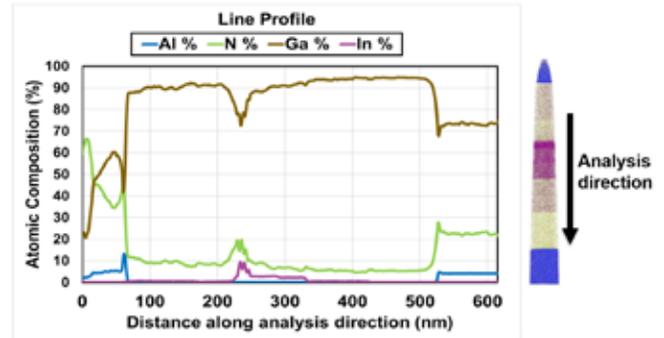

b. Iso-concentration surfaces: These are surfaces of uniform concentration that can provide lateral information on element distribution at specific or buried interfaces.
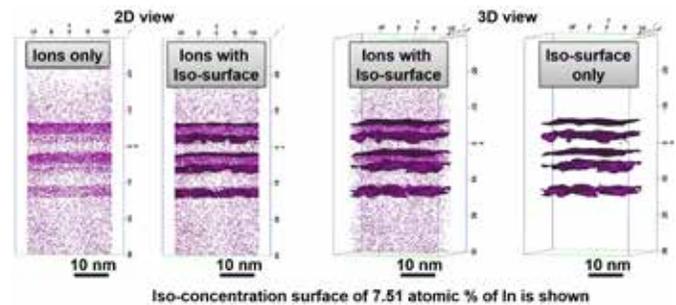
c. Proxigram: Nanoscale depth analysis across iso-concentration surfaces is also possible with APT. This can help to identify compositional changes as a function of distance from a uniform concentration surface.

- A proxigram depth analysis differs from line profiles since proxigram analysis is for the entire dataset versus a defined 3D volume within the dataset for the line profiles.

- Another feature is that line profiles determine compositions along a particular direction and representative of actual distances and depths in the device/material. Proxigram is based on a constant concentration surface which may not always be flat and hence representative of relative distances and not always actual distances in the device/material.

Atom Probe Tomography (APT)

Proxigram Depth Analysis — Ga — In — N

Iso-concentration surface of 7.51 atomic % of In is shown
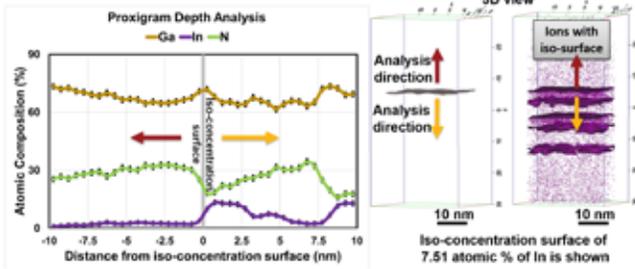
3. Cluster Analysis: APT has a strong suit in analysis for nanoscale (1-10 nm) clusters of elements within a matrix. The cluster size and composition for individual clusters of any shape and size can be analyzed with APT. With a large number of clusters, further quantitative analysis can be carried out.

4. Dopant Identification and Composition: Identification of dopant location in 3D, its chemical composition and number density (until ~10 ppm or 5E18 atoms/cm^3, in best cases) can be obtained using APT.

5. Correlative Study: In many cases, correlative studies with complementary techniques can help to answer our customer's specific material questions. STEM, EBIC, EBSD, Electron tomography and SIMS are all techniques that are routinely used to aid the interpretation of 3D APT data.

> **a. STEM and APT:** While STEM provides spatial information, APT provides chemical information which can be overlaid with the STEM data to better understand the device structure.
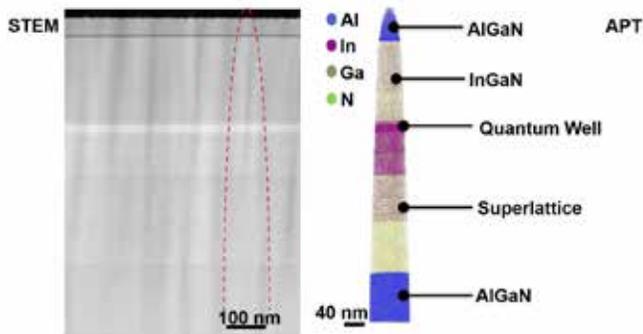
6. Specific Region Analysis: Based on customer requirements, a combination of different analysis can be used to better understand a specific region.

> **a. Superlattice in LED:** 2D maps and 1D line profile analysis helps to observe and quantify elemental composition across a superlattice.

In distribution across superlattice — In SL (%)
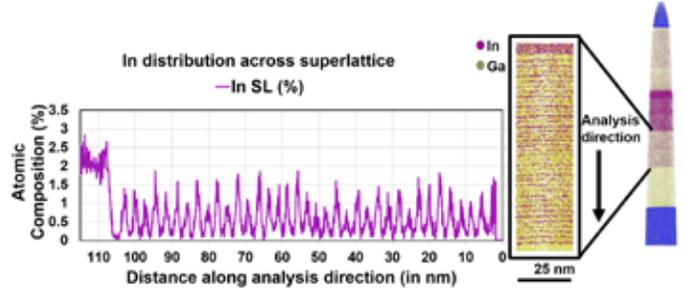
> **b. Quantum Well in LED:** The elemental composition of the quantum wells and bands in the LED controls the bandgap and hence light emission properties of the LED. APT can be used to observe how the composition changes using a 2D maps at the specific location, 1D line profile and iso-concentration surfaces for the lateral properties at the nanoscale.

QW thickness (from FWHM of larger peak)	
QW1	~ 3nm
QW2	~2.7 nm
QW3	~ 3nm

1D Line Profile across QW — In (%)

Bimodal nature of the element distribution is seen for 1st and 2nd QW