Synergistic t-EBSD/TKD and Atom Probe Tomography
Fast and Accurate Grain Boundary Analysis

Application Note #APT-13

The strength of Atom Probe Tomography (APT) is to give three dimensional, nanoscale chemical information on sub-surface phase and grain boundaries, but site specific sample preparation to collect such data can be challenging. Many sample preparation approaches have been used to position the region of interest as required near (< 500nm) the apex of the needle shaped APT specimen, but in some cases this can require multiple, time-consuming attempts. In the case of grain boundary analysis, the bulk sample often provides sufficient secondary electron channeling contrast to observe the grain boundary, but as the sample is reduced in volume to an APT specimen, the boundary is no longer resolvable. In these cases, experience, luck, and trial and error are relied upon to capture the region of interest in the analyzed volume.

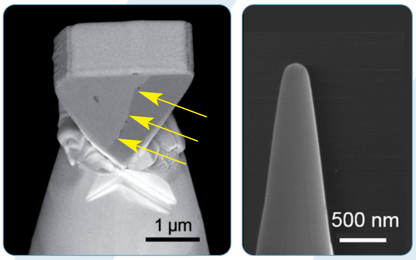
Transmission Electron Backscatter Diffraction (t-EBSD) / Transmission Kikuchi Diffraction (TKD) can be used to rapidly characterize and target specific phases or grain boundaries during APT sample preparation while providing improved spatial resolution on samples of much smaller sizes compared to conventional reflection EBSD. The combination of this chemical information provided by APT with crystallographic information from t-EBSD creates a more complete picture of the character of the boundary.

Although the shape and variable thickness of an atom probe sample can make t-EBSD challenging, new software available from EDAX, Atom Probe Assist (APA), makes positioning and characterizing GBs simple and fast. The standard Focused Ion Beam sample preparation techniques for APT create a specimen ideally suited for t-EBSD such that with just a few extra minutes, the atom probe sample can be mapped and the region of interest verified to be close enough to the specimen apex.

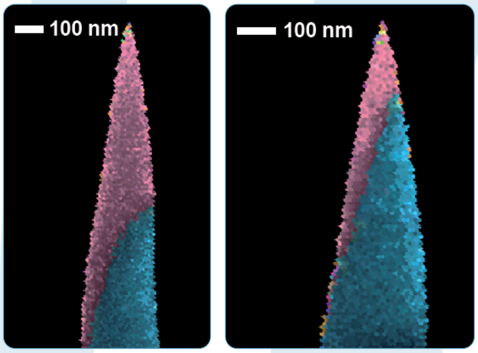
In the example at top right it is clear that high resolution SEM imaging is not sufficient to confidently center the GB with respect to the apex of the APT specimen. The EDAX orientation maps (here with IQ overlay) collected using the APA software make it easy to verify when the APT specimen is ready for analysis, saving substantial time over trial and error. GB character can have a significant effect on chemical species segregation, and using APA mode is a fast and easy way to collect grain boundary information to correlate with APT data. In the example at the bottom right, grain boundaries are identified with t-EBSD and matched with APT data showing Zr segregation.

Adapted from Y. Chen et al., Microscopy and Microanalysis, 2015 - Submitted.

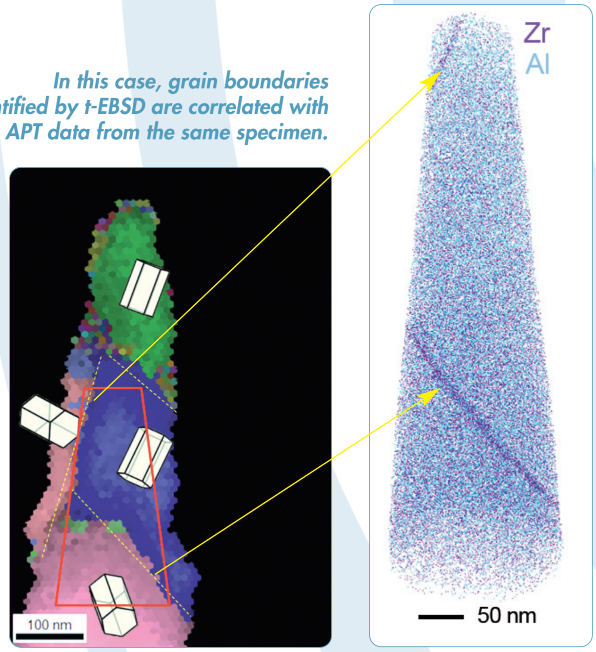
Although during the early stages of APT sample preparation the grain boundaries are often visible (Left), as the tip is sharpened, contrast is often lost, even with high resolution SEM imaging (Right).



Using t-EBSD mapping, the grain boundary can be targeted during the milling process to vastly improve chances for collecting the exact ROI desired.



In this case, grain boundaries identified by t-EBSD are correlated with APT data from the same specimen.



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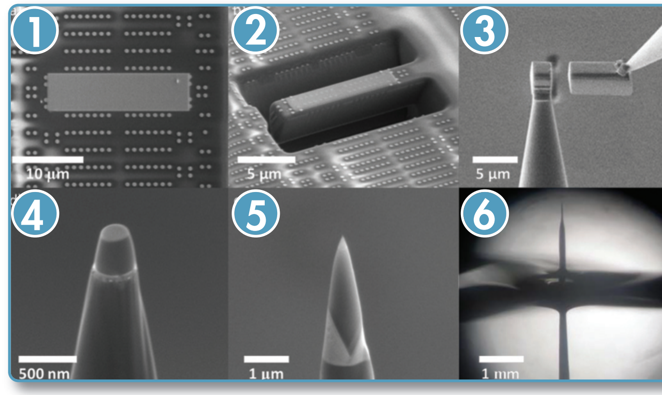
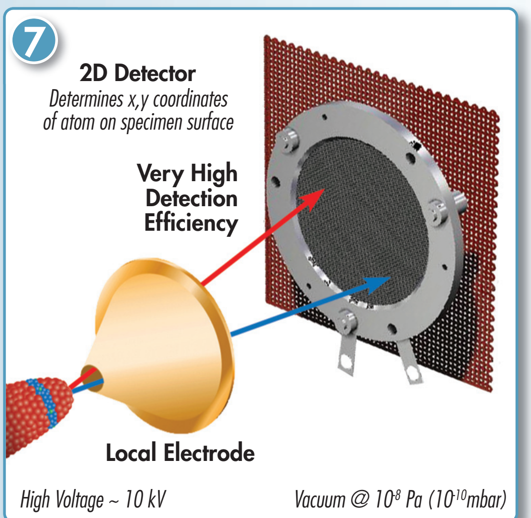
Three Steps to 3D Nanoscale Analysis
An Introduction to Atom Probe Tomography

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.





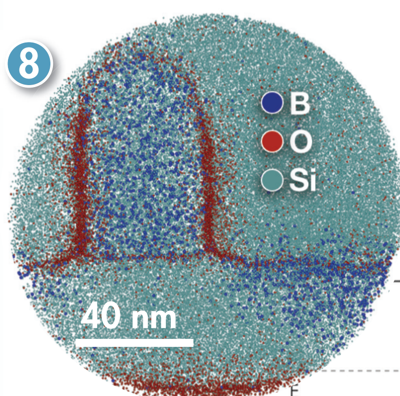
Step 2: Data Collection

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.

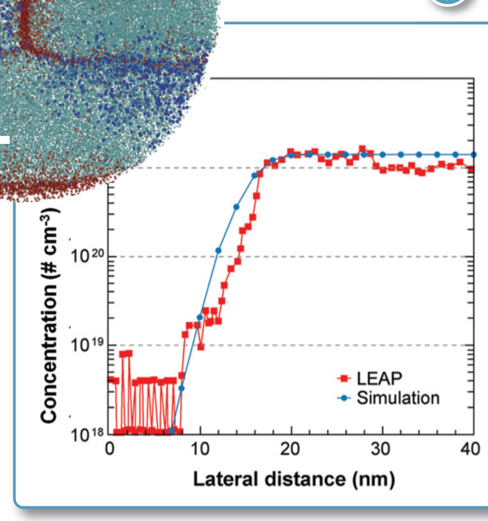
An atom probe identifies atoms by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

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.