Correlative Analysis with Atom Probe Tomography
Carbon Segregation with LEAP® and STEM

Application Note #APT-01

Grain boundary (GB) segregation leads to nano-scale chemical variations that can alter a material's performance by orders of magnitude (e.g., embrittlement). To accurately understand this phenomenon, a large number of grain boundaries must be characterized in terms of both their interface parameters and their atomic-scale chemical composition.

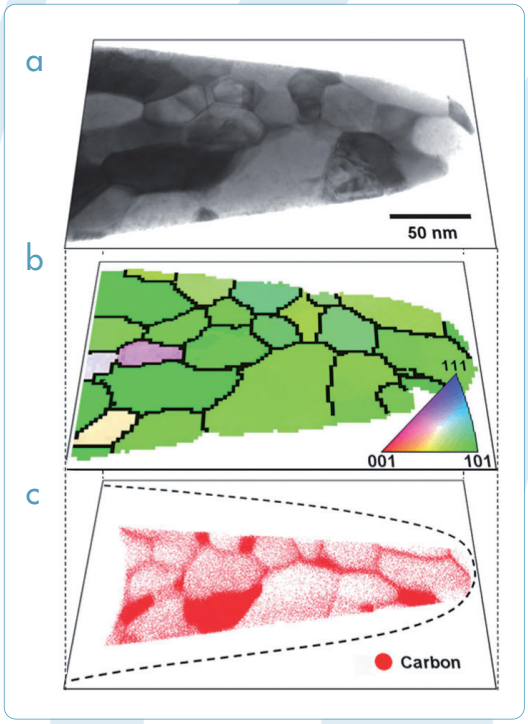
GB segregation is commonly analyzed by Auger electron spectroscopy, Secondary Ion Mass Spectroscopy, and analytical electron microscopy, but these techniques typically provide only average values or lack crystallographic information, chemical and/or spatial resolution, or statistics. Additionally, 3D quantification of carbon segregation in steels by analytical TEM/EELS is confounded by the 3D nature of the interfaces and carbon contamination.

To solve this fundamental problem, this study shows an approach that combines the accuracy of structural characterization by Transmission Electron Microscopy with the 3D subnanometer chemical sensitivity of Atom Probe Tomography (APT) applied to a pearlitic steel with nanoscale columnar grains.

In this correlative study, scanning transmission electron microscopy (STEM) is used to map GB orientation while APT is used to accurately identify up to 80% of the atoms in 3D. The application of these complementary techniques to the same volume is the ideal way to understand the relationship between grain boundary mis-match and segregation levels.

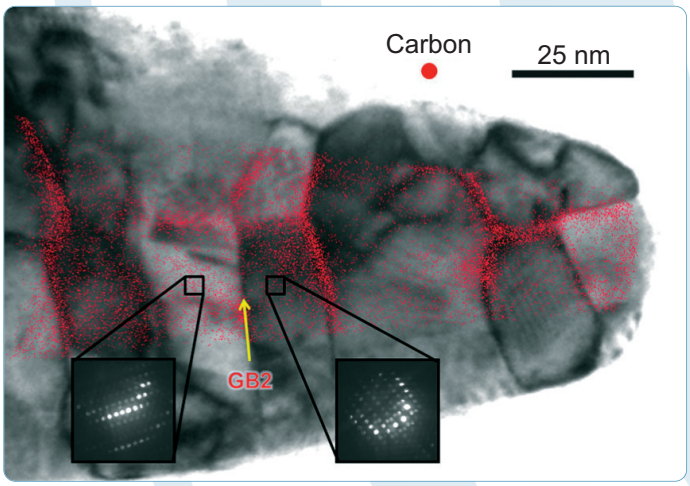
Analyses of GB segregation were completed on 121 grain boundaries in less than six days. With a large number of measurements, trends can be observed between the misorientation angle, the deviation from ideal misorientations and carbon excess. A linear trend was revealed between carbon segregation and the misorientation angle ω for low angle grain boundaries in ferrite.

This method is generally applicable to any nanocrystalline material. It demonstrates that APT in conjunction with standard STEM analysis is a robust, fast, and reliable technique to quantify local chemical compositions and for the first time, provide a precise as well as statistically robust analysis of the grain boundary segregation space.



Figures showing (a) bright field STEM, (b) nanobeam difraction analysis, (c) APT data of the same volume showing the carbon segregation to grain boundaries and the carbon rich phases identified in the APT data

Overlay of projected 3D atom map and brightfield STEM micrograph. Grain orientations, determined by nanobeam diffraction in the TEM highlight GB2, a Σ3 coherent twin, with significantly lower carbon segregation than the average grain boundary



Adapted from M. Herbig et al. Atomic-Scale Quantification of Grain Boundary Segregation in Nanocrystalline Material, Physical Review Letters 28 March 2014

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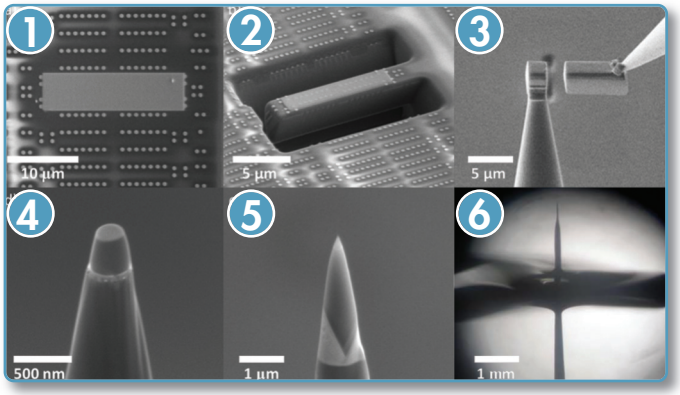
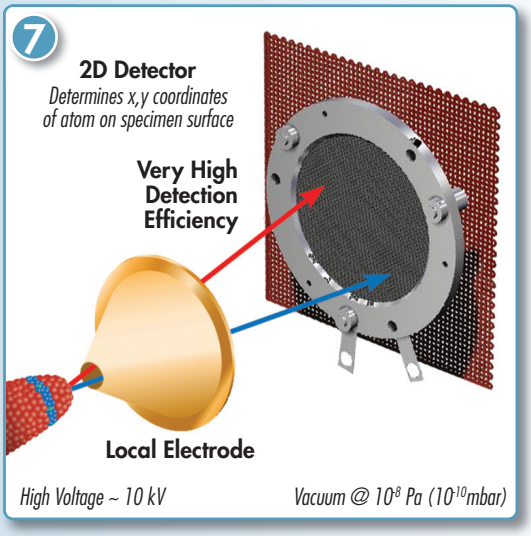
Three Steps to 3D Nanoscale Analysis
An Introduction to Atom Probe Tomography

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.

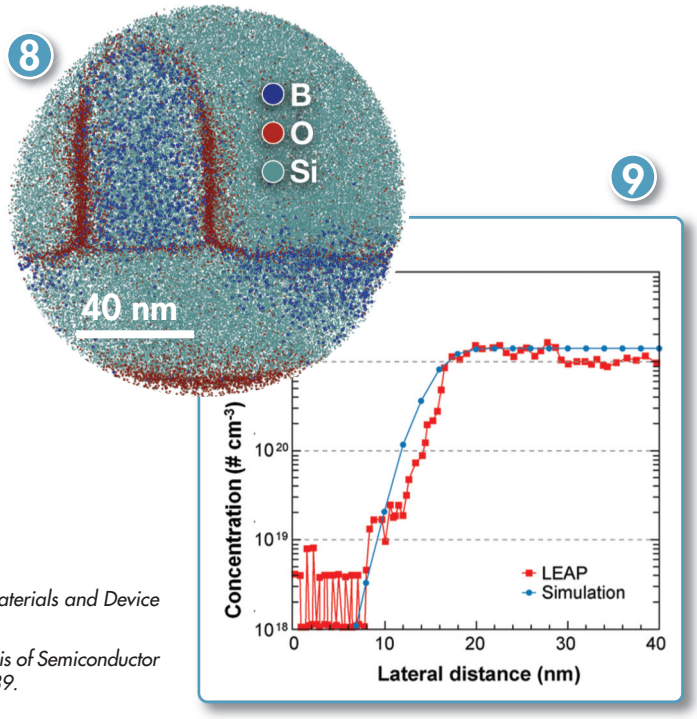
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately 10^6.

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.*