Grain Boundary Analysis with Atom Probe Tomography
LEAP® Analysis of Ni-based Super Alloy 600

Application Note #APT-02

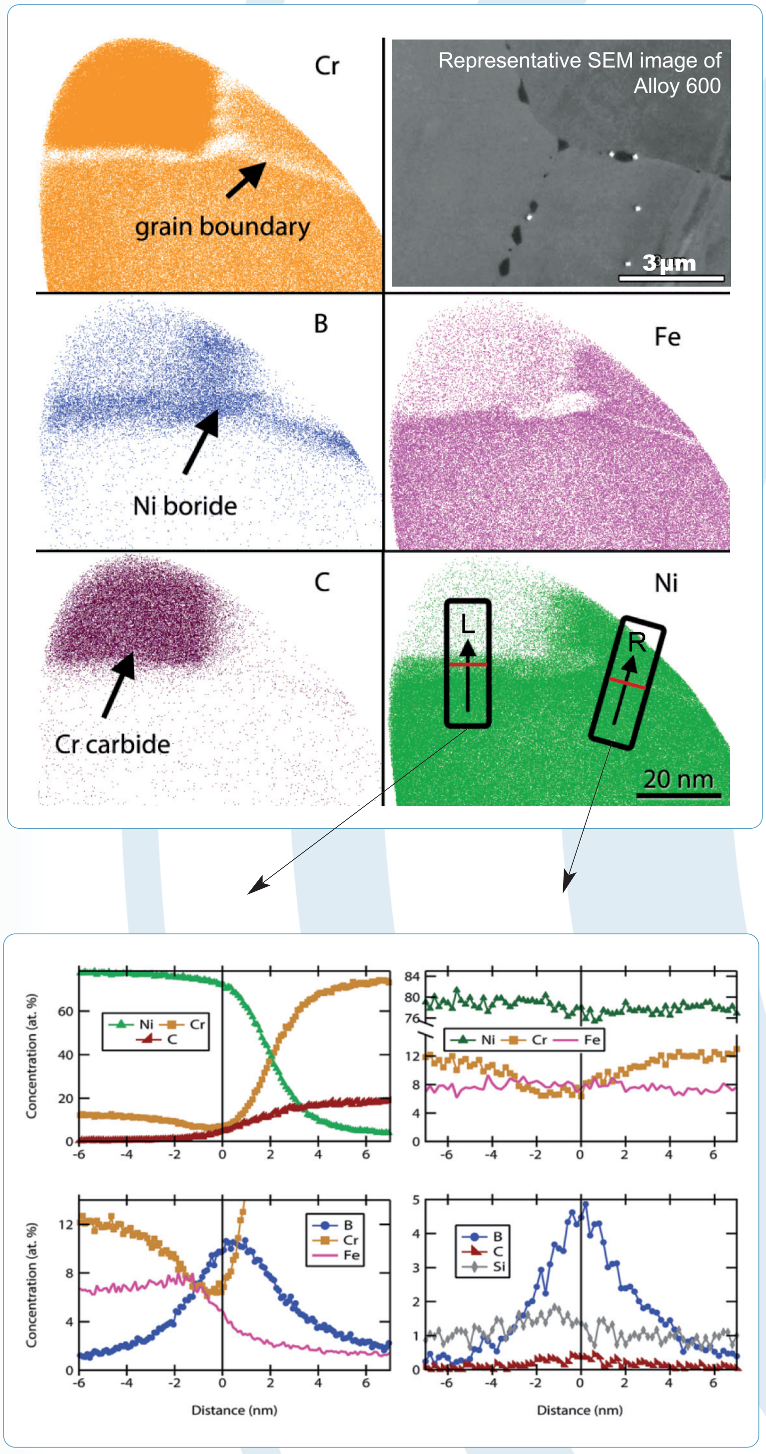
Atom Probe Tomography (APT) is a particularly adept technique for analyzing 3D grain boundary segregation of all species, including light elements such as boron. It has several advantages over analytical transmission electron microscopy and scanning Auger microscopy for the measurement of segregants particularly in small concentrations at grain boundary or precipitate interfaces. The speed of modern APT and the latest sample preparation techniques now enable studies previously impossible or impractical.

Detailed grain boundary analysis has been demonstrated for a wide variety of alloy systems including this study from Pacific Northwest National Laboratory of a structural material used in the safety systems of nuclear reactors. Although the strengthening effects of intergranular carbides on stress corrosion cracking (SCC) has been well documented, the effect of nanoscale grain boundary segregation of these materials had not. This work demonstrates that grain boundary segregation is key to the alloy's strength and toughness during critical events.

This analysis of alloy 600 prior to and after exposure to simulated PWR primary pressure vessel conditions gives new understanding into intergranular attack (IGA) mechanisms and the altered composition in the metallic grain boundaries ahead of the IGA front. Substantially elevated B concentrations were found to be consistent with SCC susceptibility.

Samples were prepared using standard FIB Liftout and APT was performed using a CAMECA LEAP system equipped with a 355 nm wavelength laser.

Modern LEAP systems and standard sample preparation techniques developed by CAMECA and our partners enable unique analysis of grain boundaries on important real-world applications. Atom Probe Tomography is the ultimate in 3D sub-nanometer compositional analysis required to understand the underlying science behind the strengths and weaknesses of advanced alloys.



Top: Slices of the 3D APT data showing nanoscale grain boundaries revealing an intergranular chrome carbide and strong boron segregation. Bottom: compositional measurements across the different boundaries (left and right) demonstrate the different levels of the boron segregation and chrome depletion depending upon the type of boundary.

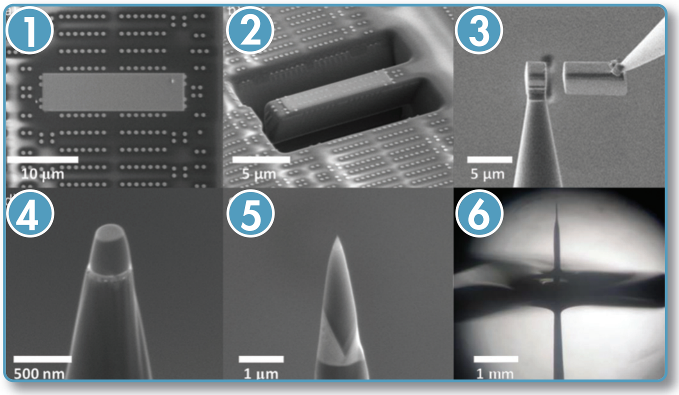
Adapted from D.K. Schreiber et. al. Microsc. & Micro 19(3) p. 676, 2013.

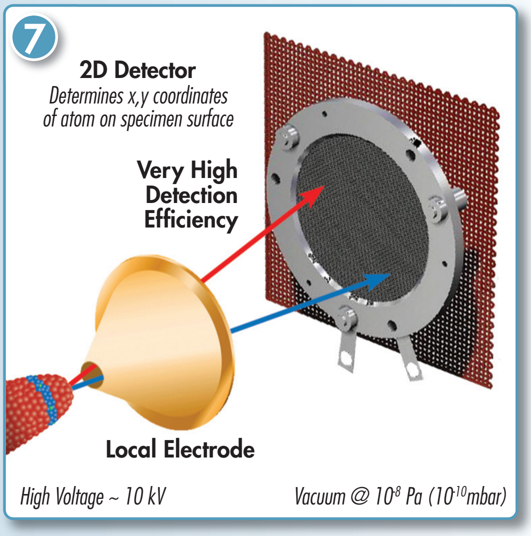
AN#APT-02 - Ni sup-alloys - Jan 15

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.





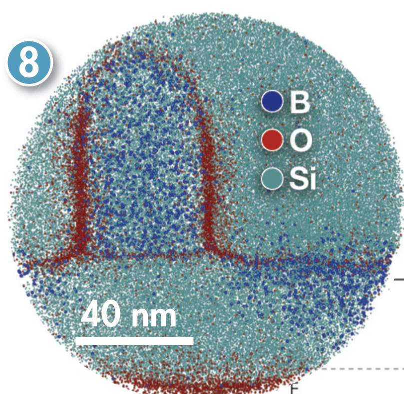
Step 2: Data Collection

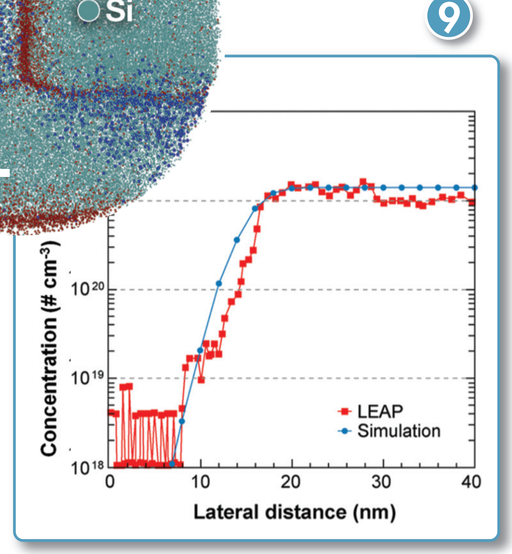
An atom probe produces images by field evaporating atoms from a needle-shaped specimen and projecting the resultant ions onto a detector 7.

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.*