CAMECA SCIENCE & METROLOGY SOLUTIONS / AMETEK

APPLICATION NOTE #EIK-02

Grain Boundary Engineering
3D Subnanometer Analysis with EIKOS™ Atom Probe

A better understanding of the structure and chemistry of grain boundaries is essential for developing a thorough knowledge of materials and their performance. Attaining detailed information on grain boundary chemistry has long been a very challenging task for material scientists, due in part to challenges identifying and preparing samples for analysis while ensuring that the grain boundary remains in the analyzable volume.

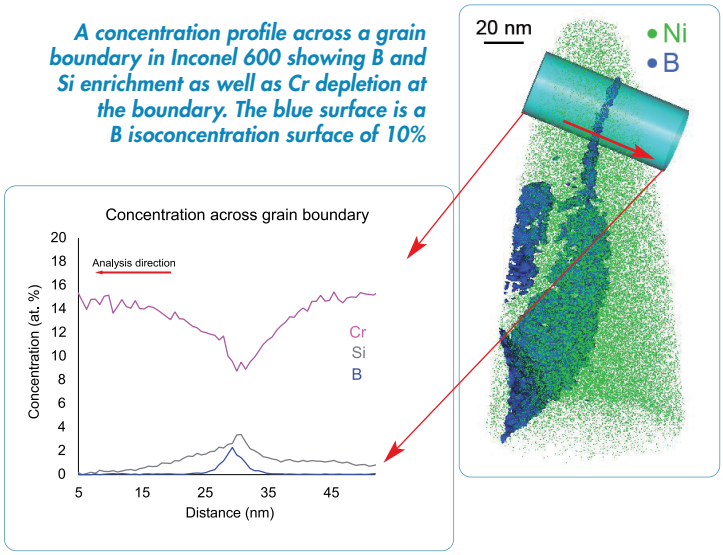
However, the use of a three-dimensional Atom Probe Tomography (APT) provides unique information, which is sufficiently powerful to yield quantitative 3D subnanometer chemical information in and around the grain boundaries allowing a window into understanding how chemical segregation may affect performance such as weldability. Chemical segregation to the grain boundary is an important aspect of materials science that APT is extremely well suited to explore.

A grain boundary in a commercially-available Inconel 600 alloy was identified by transmission EBSD and a sample was prepared using standard FIB liftout techniques such that the grain boundary was positioned in the near-apex region. Subsequent EIKOS atom probe analysis reveals strong boron segregation to the grain boundary.

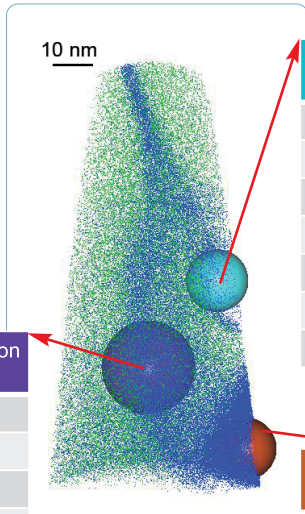
Several precipitates are seen near the grain boundary as shown in the figure bottom right. Using spherical regions of interest, the composition of these regions shows both boron-rich (orange) and titanium-rich (blue) precipitates near the grain boundary compared the bulk alloy composition (purple). Multiple types of precipitates are observed along a single grain boundary, providing a deeper understanding of the material. By analyzing a region across the grain boundary with a 1D concentration profile (top right) the grain boundary itself shows enrichment in boron and silicon, as well as depletion in chromium, some of which have previously been observed in APT analysis of this material.

Advances in alloy development can be made faster and more accurately with the understanding that atom probe data provides about nanoscale segregation of solutes.

A concentration profile across a grain boundary in Inconel 600 showing B and Si enrichment as well as Cr depletion at the boundary. The blue surface is a B isoconcentration surface of 10%



Bulk atomic concentrations from the bulk region (purple), Ti-rich precipitate (blue), and B-rich precipitate (orange).



Ion	Concentration (at. %)
Si	1.3%
Cr	15.5%
Ni	72.0%
Fe	8.6%
C	0.04%
B	0.03%
Ti	0.16%

Ion	Concentration (at. %)
Si	2.8%
Cr	14.5%
Ni	61.8%
Fe	7.2%
C	2.4%
B	0.5%
Ti	6.0%

Ion	Concentration (at. %)
Si	0.3%
Cr	5.1%
Ni	70.0%
Fe	4.0%
C	0.6%
B	17.0%
Ti	0.2%

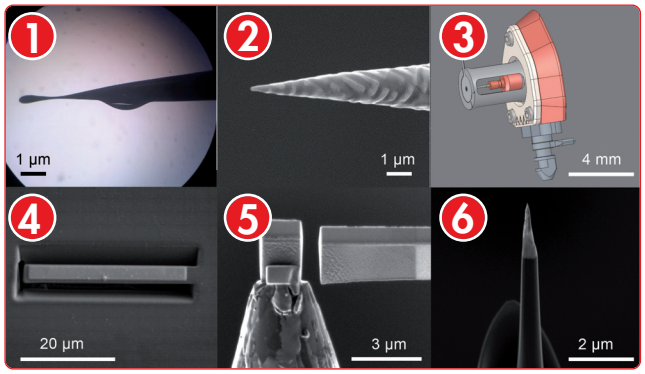
M. Thuvander, M.K. Miller, K. Stiller, Materials Science and Engineering: A. 270 (1999) 38–43.

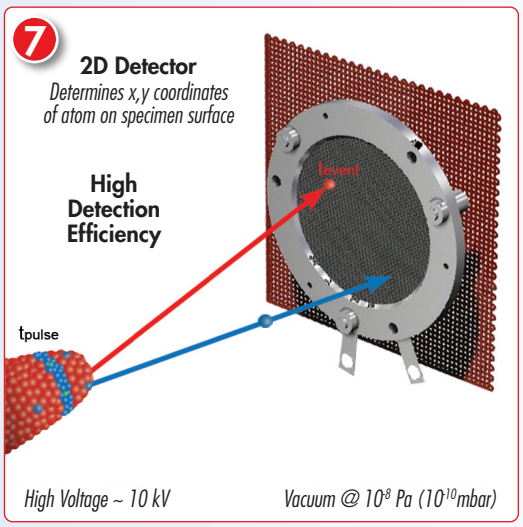
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Three Steps to 3D Nanoscale Analysis with the EIKOS-UV™ Atom Probe

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. Very similar to standard TEM preparation, the needle shape can be created using commercially available electropolishing or Focused Ion Beam systems. At the right, standard electropolishing (1, 2) and sample mounting in an integrated electrode puck (3) and standard FIB liftout (4) mounting, and sharpening (5) of a specimen to a carrier needle (6).





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).

With the EIKOS-UV™ Atom Probe microscope, a high magnification is created from the ~ 80nm tip being projected onto an 40mm detector, resulting in a magnification of approximately 5E10^5.

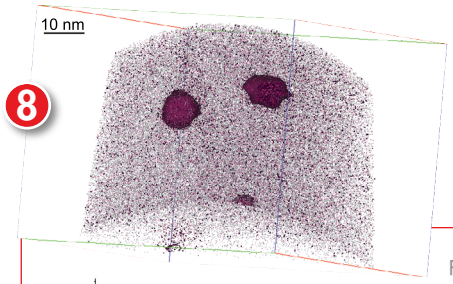
Atoms are identified by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

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 40% of the collected ions, independent of ion mass.

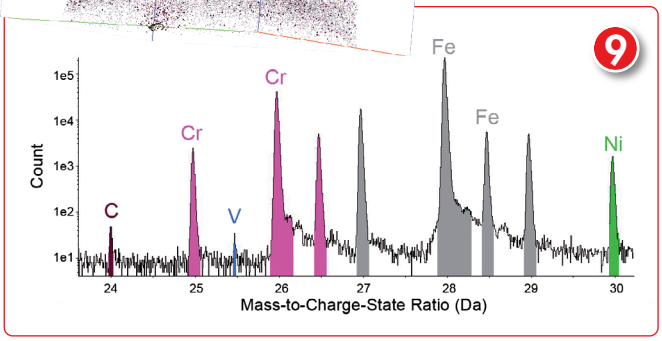
Step 3: Data Visualization & Analysis

Examples of data output are illustrated by a 3D atom map of a stainless steel (8), and a mass spectrum of the entire data set (9).

The image shows the positions of the individual atoms in the alloy with subnanometer resolution). From the reconstructed data set many types of useful analysis are possible. These include 3D visualization, 2D atom mapping, 1D depth profiling and line scanning as well as mass spectra and compositional analysis from user-selected or chemically distinct volumes.



Stainless steel analysis obtained with CAMECA EIKOS-UV™ , clearly revealing the MoN and C rich precipitates that add strength and corrosion resistance to this alloy.



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