CAMECA SCIENCE & METROLOGY SOLUTIONS / AMETEK

APPLICATION NOTE #EIK-03

Synergistic t-EBDS/TKD and Atom Probe Tomography Site Specific Analysis with EIKOS™

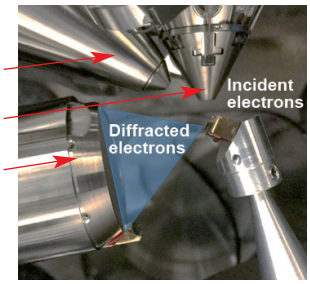
A strength of Atom Probe Tomography (APT) is the ability to give three dimensional, nanoscale chemical information on sub-surface phase and grain boundaries. Many sample preparation approaches have been used to position the region of interest as required near (< 500nm) the apex of the needle shaped APT 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. These techniques also provide 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 with existing EBSD equipment. 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.

A typical sample/instrument configuration is shown in the figure at top. In this analysis of an Inconel 600 sample, two t-EBSD maps with image quality overlay collected using the APA software are shown, demonstrating targeting of the grain boundary close to the apex during the milling process. Subsequent APT analysis from this specimen shows strong boron segregation to the grain boundary observed in the t-EBSD maps, as well as the presence of boron precipitates.

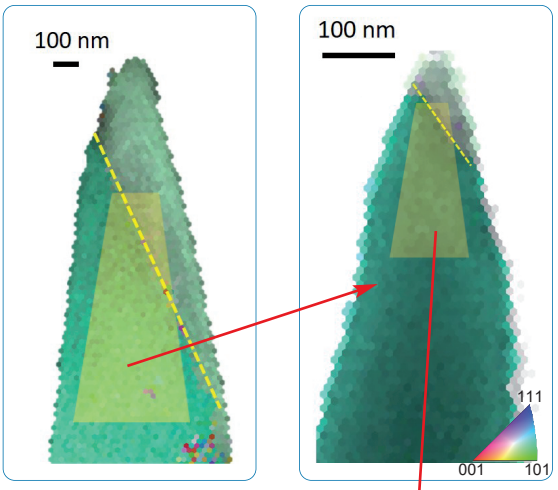
The EIKOS atom probe provides high quality, wide field-of-view 3D nanoscale compositional information. When combined with a technique such as t-EBSD, truly novel characterization is possible.



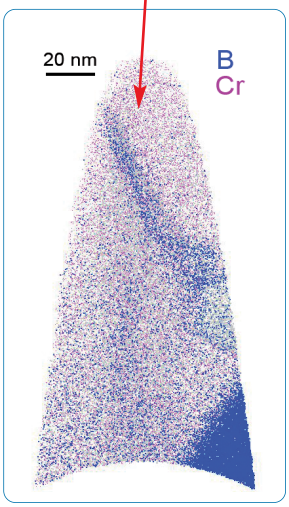
FIB/SEM configuration for t-EBSD of atom probe specimens.

Left: t-EBSD map in the early stages of milling, showing the grain boundary below the apex.

Right: t-EBSD map after final milling, showing the grain boundary in the near-apex region.



The orientation maps collected using the EDAX Atom Probe Assist (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 GB information to correlate with APT data.



A 20 nm thick slice of the 3D atom map (virtual TEM lamella) showing both boron segregation to the grain boundary as well as a boron-rich precipitate (over 17 at. % boron) in the lower right hand corner.

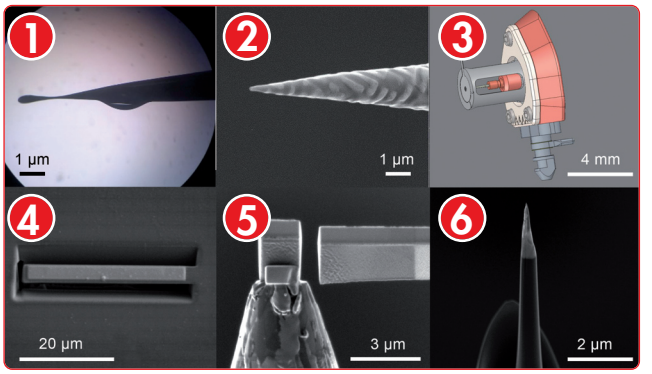
K.P. Rice, Y. Chen, T.J. Prosa, D.J. Larson, Microscopy and Microanalysis, Issue 24, May/June 2016

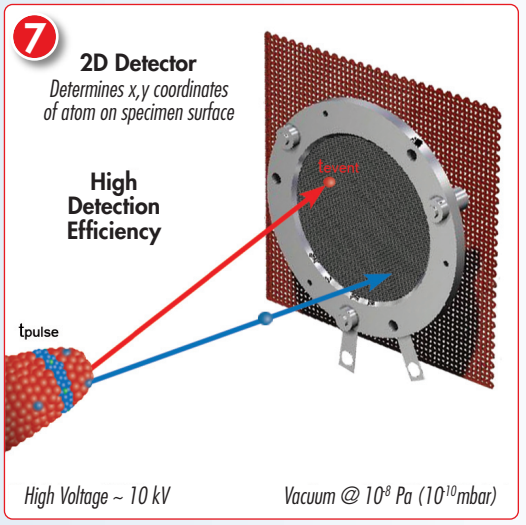
www.cameca.com • www.atomprobe.com
International Headquarters: 29 quai des Grésillons • 92622 Gennevilliers • France • cameca.info@ametek.com
Atom Probe Technology Center: 5470 Nobel Drive • WI-53711 Madison • USA • cameca.leap@ametek.com

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.

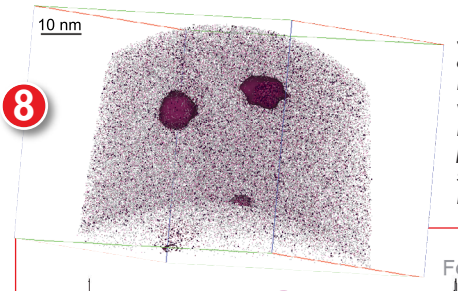
Atoms are identified by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

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.

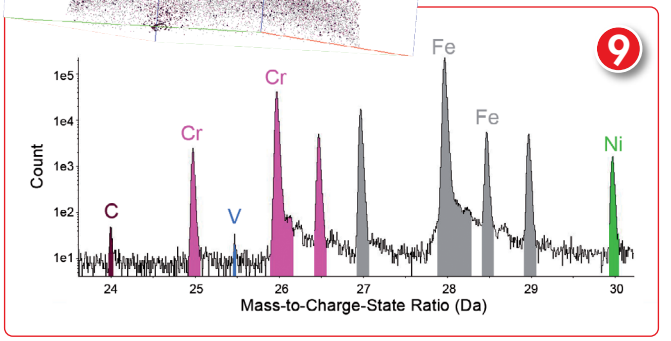
Step 3: Data Visualization & Analysis

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.



CAMECA SCIENCE & METROLOGY SOLUTIONS / AMETEK

www.cameca.com • cameca.info@ametek.com