Transmission Electron Backscatter Diffraction (t-EBSD) Assisted Specimen Preparation of APT Specimens

Technique Challenge

Atom Probe Tomography (APT) is a high spatial resolution chemical analysis technique that allows elemental identification with subnanometer resolution. Atoms are evaporated from the apex of a sharp needle-shaped sample and both the atomic number and the original position of the atom are determined.

APT is uniquely suited to the study of segregation to subsurface grain and phase boundaries. However, this high spatial resolution also severely limits the volume of material that may be analyzed. Typical specimens are needle-shaped with tip diameter < 200 nm and a typical analysis depth < 500 nm.

Specimen preparation is relatively straightforward if the question does not require site-specific analysis, but a targeted sample preparation of a specific feature requires high resolution imaging methods to ensure that the area of interest is present near the tip of the needle.

Comparison with Existing Techniques

Conventional specimen preparation to produce the sharp needles required for APT analysis is typically done via a Focused Ion Beam (FIB-SEM) instrument. This can be combined with Transmission Electron Microscope (TEM) imaging to verify positioning of a specific feature near the tip of the needle. Electropolishing is occasionally used as an initial preparation step, but is generally not applicable for site specific analysis.

Electropolishing

- Conventional electropolishing of small (10 x 0.3 x 0.3 mm^3) rods that are pre-cut mechanically works well for "bulk" analysis.
- This method does not allow for accurate positioning of a specific site near the tip apex.
- Grain boundaries may be lost during processing.

FIB Preparation

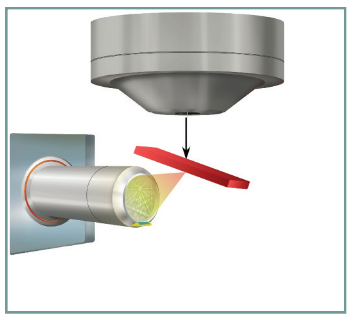
- A selected region of interest can be lifted out of a bulk sample and positioned on a prepared post, much like TEM sample preparation.
- Annular FIB milling is used to shape and sharpen the sample into the desired geometry for APT analysis.
- As feature sizes decrease through preparation, detecting grain boundary position becomes difficult due to low imaging contrast.
- Grain boundaries in the top 200 nm of the sample are invisible.

TEM Position Verification

- When the FIB contrast becomes insufficient to locate the feature accurately, the contrast may be improved by viewing the needle in a TEM.
- Diffraction contrast in TEM images allows easy identification of boundaries, but images need to be compared to identify the location again when the sample is returned to the FIB-SEM.
- This requires moving the specimen between instruments several times which is time consuming and risks damaging the sample during re-mounting.

Optimization of site specific APT specimen preparation using t-EBSD

- t-EBSD utilizes the electron transparency of the sample to collect diffraction patterns of the needle using a standard EBSD detector.
- Analysis can be done in-situ in the FIB-SEM, the sample can remain in vacuum.
- Indexing the patterns allows for accurate orientation determination with up to 5 nm spatial resolution.
- In addition to locating the feature of interest, the orientation measurements may also be used to characterize the crystallographic character of the boundary, e.g. if it is a low-angle, high-angle, or twin boundary.

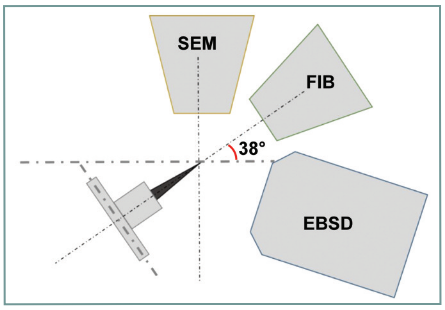


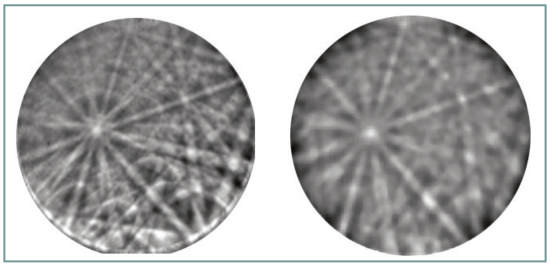
t-EBSD Principle

In t-EBSD the electron transparent specimen is mounted at an angle between 0 and -40°. In this configuration the EBSD patterns are projected onto the EBSD detector from the bottom surface of the sample. This allows improved spatial resolution and identification of electron transparent areas.

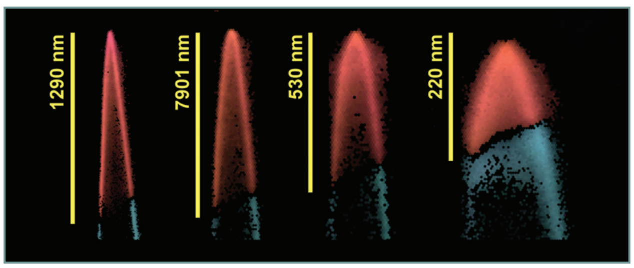
APT Specimen Preparation Geometry

The sample is mounted in the FIB-SEM such that the needle is parallel to the FIB beam. This configuration allows milling, SEM imaging and t-EBSD mapping without moving the specimen. Minor variations from this positioning can be made to optimize t-EBSD pattern quality.





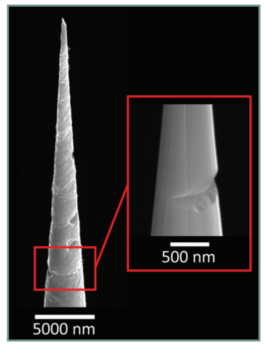
The diffraction patterns originate from the side of the specimen that faces the EBSD detector. The thickness variation across the needle tip affects the contrast in the t-EBSD patterns such as these collected at the tip of the specimen (left-thin) and further into the needle (right-thick). Enhanced image processing ensures optimum band detection and indexing over a range of specimen thickness up to a thickness of ~300 nm. The t-EBSD mapping can be used to collect orientation snapshots and determine grain boundary position between the successive FIB milling steps. The individual maps were collected in 3 minutes.

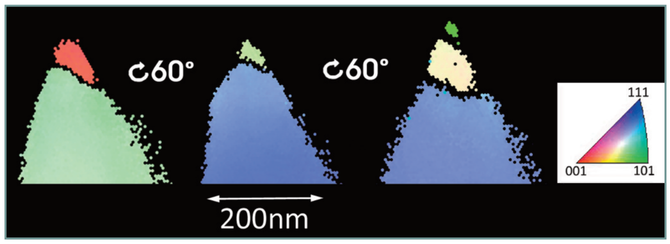


Example: grain boundary analysis

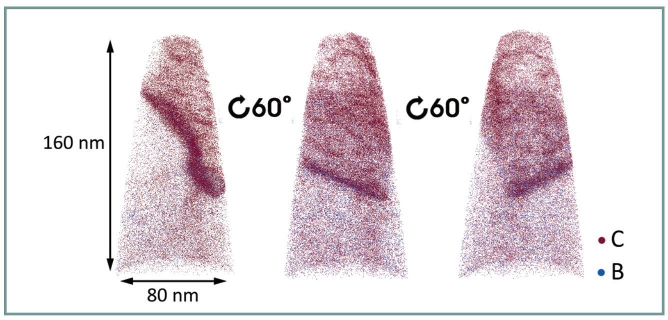
The presence of boron segregation on a prior austenite grain boundary was investigated by APT. The grain boundary of interest was identified in an electropolished APT sample by applying a picric acid etch, as seen in the larger SEM image (top right). The target boundary was then positioned closer to the tip apex using the in-situ t-EBSD and FIB-SEM milling technique. The grain boundary is still visible after some FIB-SEM preparation, as seen in SEM image insert.

EBSD maps showing the crystal direction orientation relative to the bottom surface of the specimens and showing the grain boundary position within 200 nm of the tip apex were collected from 3 directions by rotating the needle to illustrate the interface of the boundary in the tip of the needle prior to APT analysis.





Reconstructed APT compositional maps were generated from the APT data. B and C segregation to the prior austenite grain boundary is observed. The trace of the prior austenite grain boundary in the orientation maps can be matched with the B and C segregation in the APT maps.



Acknowledgements

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Recommended EDAX Solution

The EDAX Atom Probe Assist tool is recommended to help Atom Probe users prepare APT samples more efficiently and provide quantitative information on grain boundary position and character to accelerate research and understanding of grain boundary segregation. This product includes both a Hikari Plus EBSD camera, which provides fast and accurate collection of EBSD and t-EBSD patterns for crystallographic characterization of APT samples, and TEAM™ EBSD software with a dedicated Atom Probe analysis mode with customized features for the unique analysis of APT samples.