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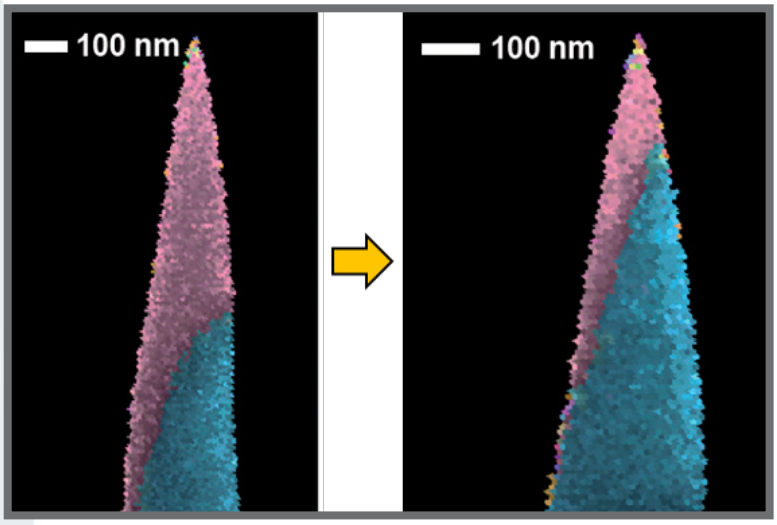

Technical Note TN-09

Transmission EBSD of APT Specimens

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INTRODUCTION

Transmission electron backscatter diffraction (tEBSD), also known as Transmission Kikuchi Diffraction (TKD), is grain texture analysis technique similar to reflection electron backscatter diffraction (EBSD). Transmission electron diffraction patterns from a sample are collected by an EBSD camera and mapped and indexed to crystallographic orientations by commercial software. For atom probe tomography (APT) sample preparation, this technique can be used during standard specimen preparation process, aiding in the accurate identification and positioning of grain boundaries near the tip apex. This approach improves the likelihood of collecting data from targeted regions-of-interest (ROIs) during APT analysis. Adding tEBSD information adds only a few minutes to the APT sample preparation process, and can be done without moving the specimen from it's location during milling.

In tEBSD, diffraction patterns are formed from the near-exit surface of the specimen.[1] Consequently, it may be difficult to map grains that are stacked along the beam direction. The presence of multiple diffraction patterns can cause difficulty in properly indexing overlapping grains, or identification of the near-surface grain only. It works well for micron-sized domains down to 50 nm, but for thin specimens, such as APT specimens, grain boundaries or nanoscale grains are typically targeted.[2,3] This technical note provides an overview of the technique and suggested best practices for EDAX TEAM™ EBSD systems. This is not intended to be a comprehensive guide. Also note that results can vary substantially based on material.

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STEP-BY-STEP PROCEDURE

1 Region of Interest Identification

Prior to doing specimen preparation via FIB liftout, it is useful to have a reflection EBSD map taken at low magnification so specific regions, grains, or grain boundaries can be identified. Procedures for mechanically polishing a surface are available in the literature.[4]

Once a grain boundary of interest has been identified from the EBSD map, the surface of the grain boundary can be marked by a line of electron beam deposited GIS-Pt, C, or W for better visibility as shown in Fig. 2. Special grain boundaries can be distinguished from random grain boundaries in the OIM Analysis™ software.

The grain boundary direction below the surface is unknown, so the liftout region may need to be adjusted toward the center of the grain boundary to maximize the grain boundary region within the wedge. To identify the direction of the grain boundary below the surface, make a cleaning cross-section cut at the end of the Pt marker as shown in Fig. 3. After inspecting the direction below the surface, a protective Pt region can be placed over the center of the grain boundary .

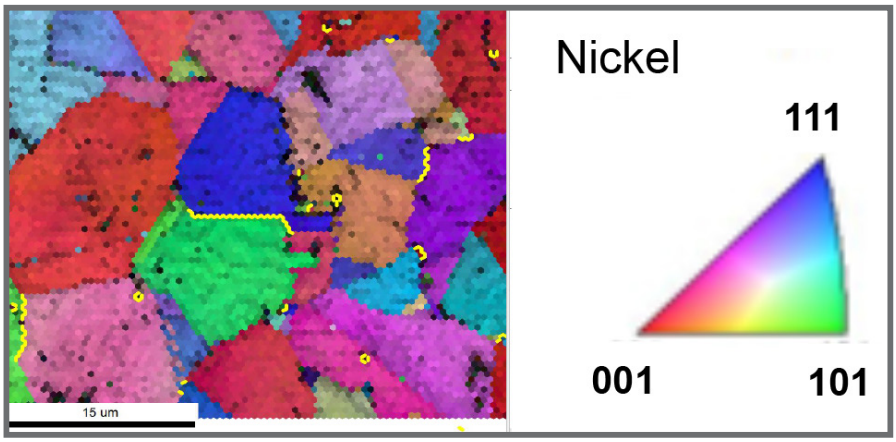

Fig. 1: Reflection EBSD map to find specific grain boundary, IPF triangle.

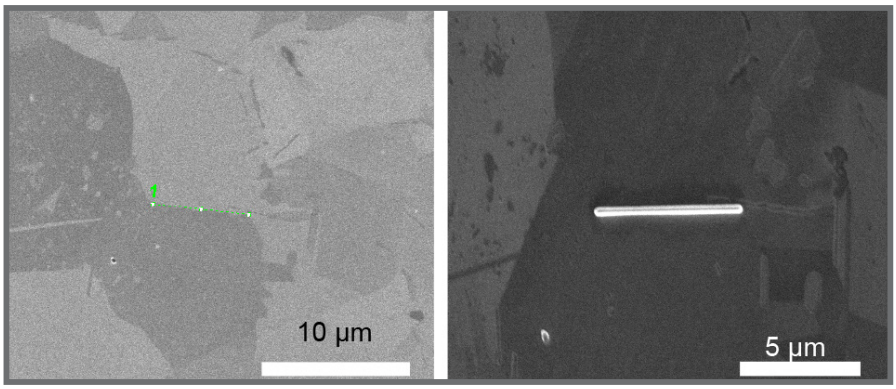

Fig. 2: Identified grains visible in SEM image.

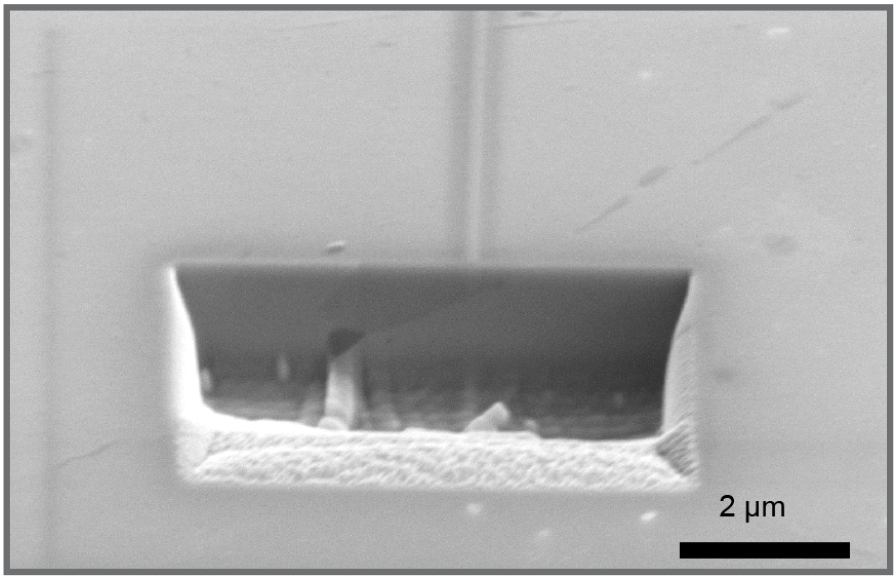

Fig. 3: Sub-surface inspection using a cross-section cut.

STEP-BY-STEP PROCEDURE

2 Mounting Specimens

Proceed with standard liftout process described in TN-01. Specimens should be mounted such that the triangular wedge face is oriented toward the long side of the coupon substrate (consistent with TN-01). To minimize reflection of the transmitted beam off the coupon surface, preferentially mount to the outside rows of a microtip coupon (blue arrows in Fig. 4). Specimens on any available tip can be successfully mapped, however the specimen will need to be tilted to a steeper angle to map the tips on interior rows. Use of 36-tip flat tops microtips are not recommended for tESBD because the microtips are further from the edges, increasing the likelihood for reflection patterns from the substrate.

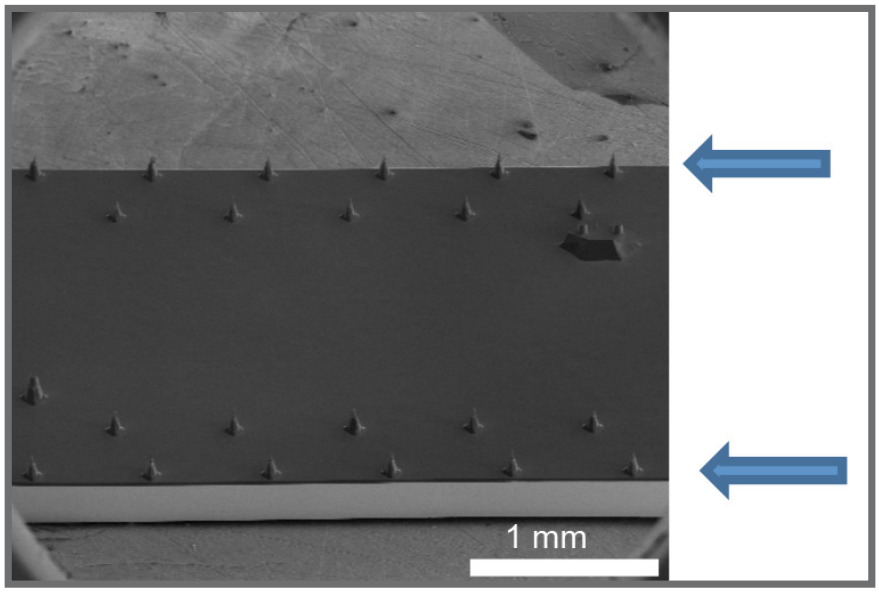

Fig. 4: Outside rows of the coupon minimize reflections.

STEP-BY-STEP PROCEDURE

3 Experimental Setup

Transmission EBSD can be performed on FIB-liftout specimens mounted to wires, TEM grids, or microtip coupons. The samples may also originate from electropolished wires where the grain boundary positions/types are unknown. The exact setup can vary based on the specific FIB configuration. Specimens need to be mounted such that the beam can freely pass through the specimen and not reflect off any substrate or holder material before reaching the detector. Wire specimens can be mounted directly to the stage as shown in Fig 5 to allow for free rotation to map any angle of the specimen.

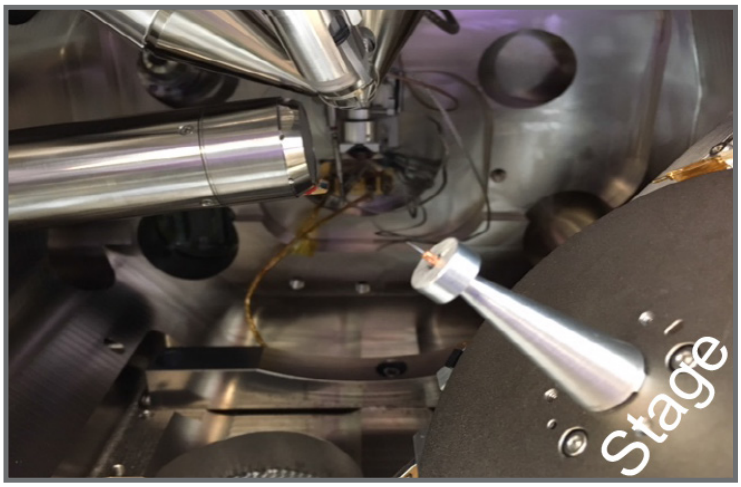

Fig. 5: Wire specimen mounted directly into the stage.

Microtip coupon substrates require orienting the row of tips being mapped so that they are closest to the EBSD camera, thus avoiding any opportunity for transmitted electrons to reflect off the substrate. Mounting any sample type in a pre-tilted holder provides additional flexibility for available tilt angles. Figs. 6a and b, show possible mounting options for different sample carrier types. Fig. 7 shows a schematic of the angles/configuration used for most ThermoFisher DualBeam setups. In ThermoFisher DualBeams, eucentric height is the recommended working distance. Based on the chamber/stage configuration, achieving this working distance may or may not be possible. If the specimen is too close to the pole piece, a larger working distance can be used for tEBSD mapping. These positions can be saved in the FIB to provide easy switching between milling and mapping positions. Remember to retract the EBSD camera when milling.

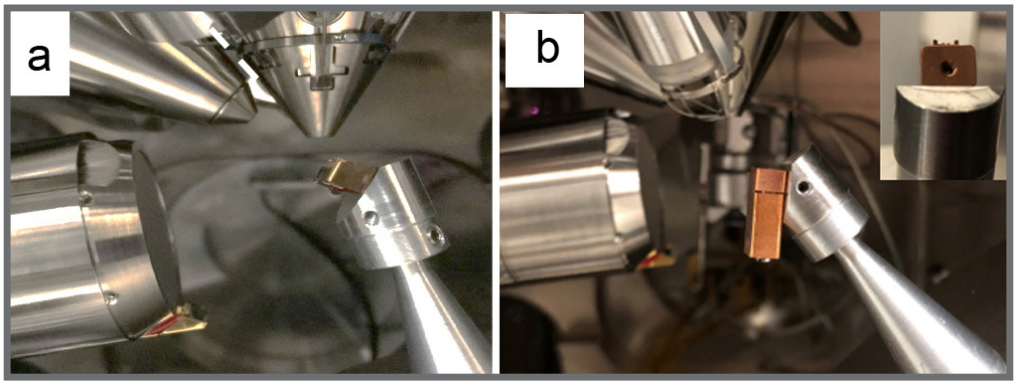

Fig. 6: (a) microtip coupon mounted to pretilt holder.
(b) CAMECA LEAP-compatible grid holder mounted to pretilt holder, PN 24242. Inset shows top view of holder with TEM half grid.

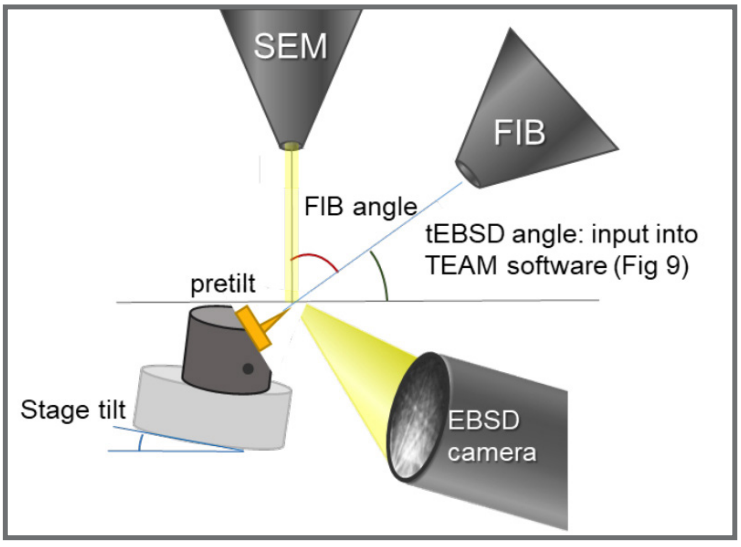

Fig. 7: Schematic of angles used in tEBSD mapping.

STEP-BY-STEP PROCEDURE

4 Setting Sample/Camera Positions

Set the microtip to eucentric height if possible. Again, orient the specimen such that the electron beam can freely pass through the specimen while avoiding any opportunity to reflect off any substrate or holder material to avoid reflection diffraction patterns from interfering with tEBSD patterns. Fig. 8a illustrates a clear path for the electron beam to pass through the specimen and over the side of the coupon without reflecting off the coupon flat. Fig. 8b illustrates a mapping position that may cause reflections due to the position of the microtips coupon substrate behind the tip apex.

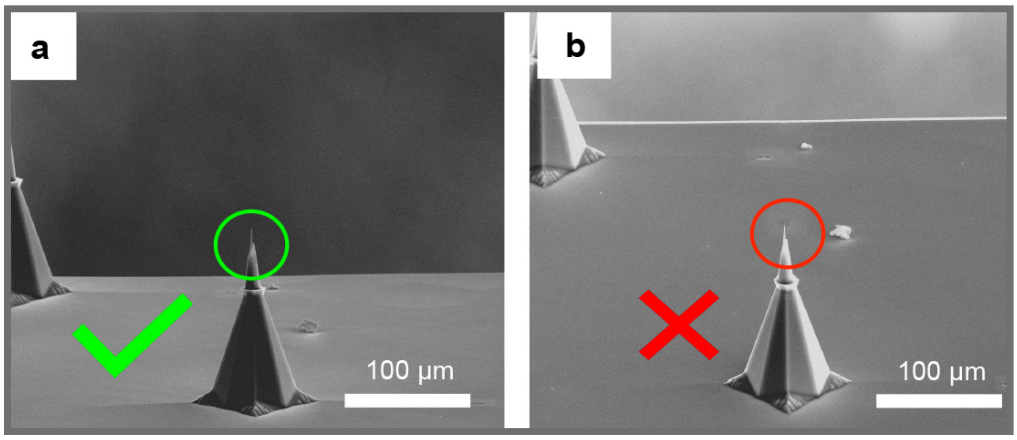

Fig. 8: (a) Example of clear diffraction path to camera from specimen apex, good. (b) Example of diffraction path blocked by coupon flat, bad.

Accurately inputting the correct tilt angles between the specimen and the camera is essential for determining accurate orientation information. Consider tEBSD maps of a specimen positioned at the same position/orientation used for ion milling and mounted on a pre-tilted holder, such as the one used in Fig. 8. The specimen starts at 45° relative to the electron column, with an additional 7° of stage tilt so that the specimen axis is aligned with the ion column. The specimen is then at 52° from vertical and (90°- 52°) 38° from horizontal. Select the Transmission Mode by ticking the box under the tEBSD/TKD pull down menu and input the tilt angle from horizontal (Fig. 9). Note, depending on the overall layout of your specimen relative to the substrate or other features, a larger stage tilt and smaller tEBSD angle may be needed to prevent reflections during mapping, especially for interior rows of a 22-tip microtip coupon.[5] These instructions are particular to an ion column oriented at 52°, but that angle may vary depending on FIB manufacturer/configuration.

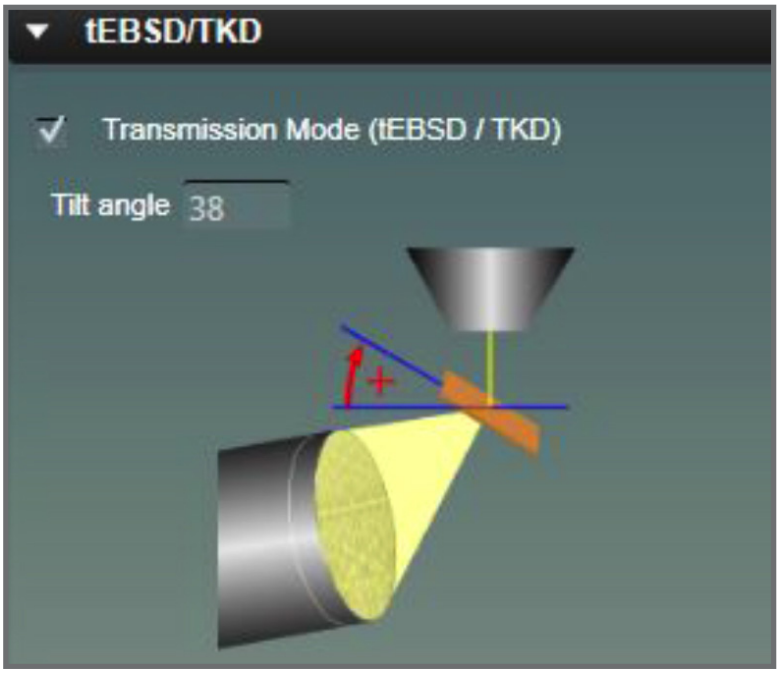

Fig. 9: tEBSD/TKD software pull down menu, showing the transmission mode checkbox and tilt-angle entry field.

STEP-BY-STEP PROCEDURE

5 Setting Background and Camera Parameters

Higher electron-beam voltages better penetrate thicker material, so 30 kV is optimal for tEBSD mapping during the early stages of tip milling, as described in TN-02. Beam currents of 2-10 nA can be used when available. Depending on the density and electron scattering characteristics of the material, initial milling may need to be done without mapping in order to thin the specimen enough to become electron-transparent. [5] For most transition metal specimens, thinning to 1-1.5 µ in diameter should produce meaningful Kikuchi diffraction at the exit surface. At this point, camera parameters need to be set for tEBSD mapping.

Insert the EBSD camera, making sure that the stage is in a safe position to do so. Use EDAX Atom Probe Assist software (here V4.5-RC7) to allow for automatic optimization of the camera and background settings. Under the Image Processing tab, select Atom Probe Assist in the Image Processing Mode dropdown list as shown in Fig 10. This will automatically provide a recipe for Dynamic Background Subtraction, apply a median smoothing filter, and normalize the intensity histogram. Dynamic Background Subtraction is especially useful for specimens where the thickness varies throughout the mapping areas and in the empty space off the specimen.[6]

Select Survey Mode and position the green dot in the center of the specimen (Fig. 11). Make sure the microscope is in external control. In the Camera tab, Select Atom Probe Assist under the dropdown menu and click Optimize, as shown in Fig 12. The camera will go through an optimization routine to select the best gain and exposure settings to capture high contrast Kikuchi diffraction patterns. The binning is set by default to 5x5 or 4x4. Manual background and camera parameters can also be set by selecting the Custom or Manual option.

.To manually adjust camera settings, select Custom under Image Processing Mode. This may be useful if you would like to increase pattern contrast to help the software index the patterns. By clicking Custom, the Image Processing Recipe Builder opens.

The recommended Image Processing recipe, shown previously in Fig. 10, and can be implemented by selecting these options from the list on the left side:

- Dynamic Background Subtraction
- Median Smoothing Filter
- Intensity Histogram Normalization

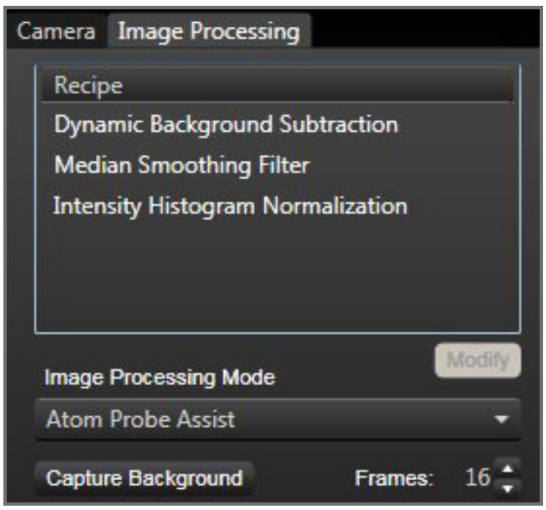

Fig. 10: Atom Probe Assist image processing recipe.

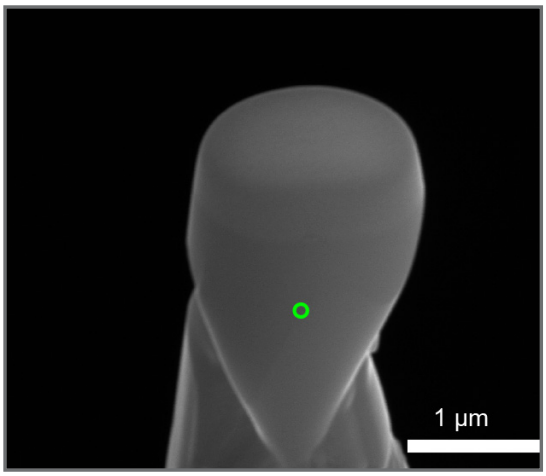

Fig. 11: Survey mode position for camera optimization.

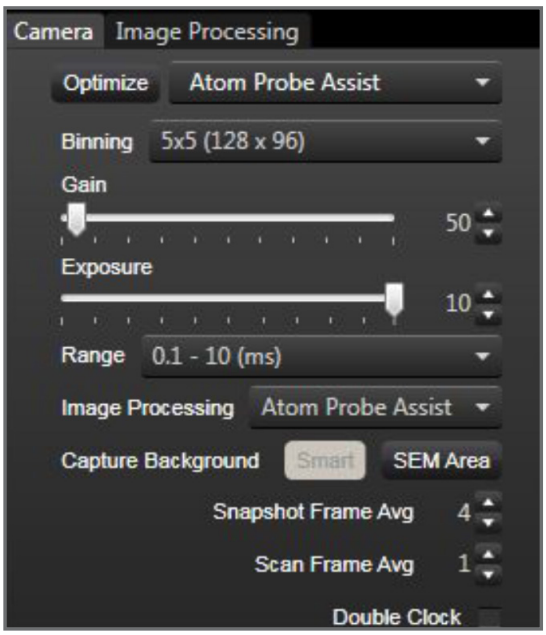

Fig. 12: Camera parameter selection tab.

It may be helpful to increase the passes and balance values to improve pattern contrast, which can be done by selecting Dynamic Background Subtraction and using the slider bars as shown in Fig 13. Fig 14a shows a pattern using 10 passes and a balance value of 10%. Fig 14b is a pattern from the same spot with 40 passes and balance value of 80%. Fig 14a was not able to be automatically indexed, while Fig 14b indexed correctly.

Changing brightness and contrast values can also improve pattern indexing. Select Intensity Histogram Normalization from the recipe list, then adjust the contrast and brightness using the slider bars as shown in Fig. 15.

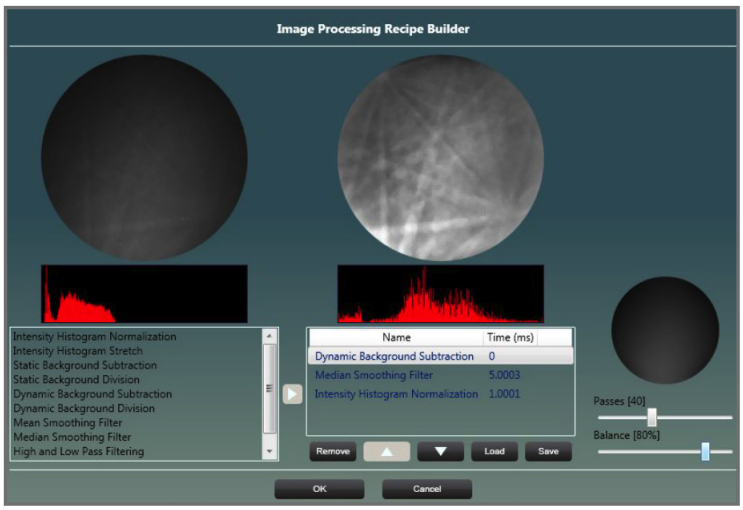

Fig. 13: Image Processing Recipe Builder, passes and balance slider bars.

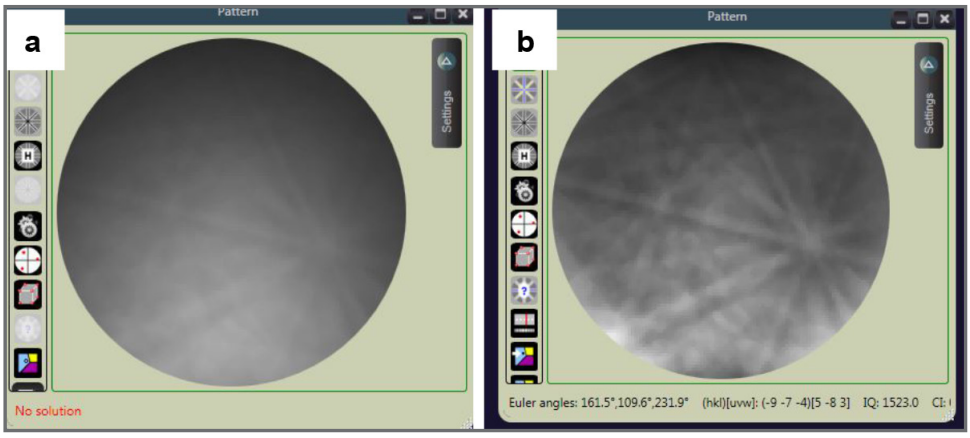

Fig. 14: (a) low passes and balance, (b) increased passes and balance values.

The goals of adjusting the camera and image processing parameters are to take maps as quickly as possible with the greatest indexing accuracy. Mapping times will depend on camera settings as well as the chosen step size. Recommended map times are under 5 minutes to avoid contamination from the chamber. For the settings shown in Fig 13, a 900 point map with an exposure time of 190 ms was collected in approximately 2 minutes.

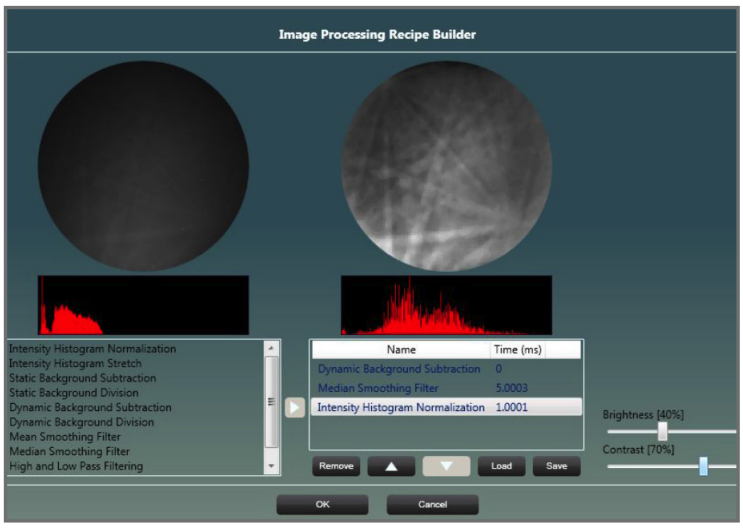

Fig. 15: Image Processing Recipe Builder, brightness and contrast slider bars.

STEP-BY-STEP PROCEDURE

6 Collecting Sequential Maps

One goal of tEBSD mapping of an APT specimen is to take maps at points in the milling process to assist in positioning the grain boundary of known type or grain of interest near the apex of the specimen. The ion-milling area can be adjusted based on feedback from the tEBSD map that will give the highest chance of preserving the ROI. First, set the material phase in the map menu. Then, select the mapping menu and indicate trapezoidal map shape. Trapezoidal mapping allows for time to be saved by only mapping over the conically-shaped specimen. Initially, the specimen may be too thick to get clear patterns throughout the entire mapped region of the conical APT specimen.[7] Patterns will first appear through the thinner regions, around the side and near-apex first as shown in Fig. 16a. Non-indexed points will be seen in thicker regions and those points mapped over vacuum, as seen on the sides of each mapped region

Figs. 16a-f show a tEBSD mapping series for successive milling steps. As the sample becomes thinner, the central blue grain becomes visible, after being initially obscured by diffraction from the pink grain. The mapping step size was progressively decreased as the ROI became smaller.

Unnecessary beam exposure should be avoided to prevent possible carbon contamination of the specimen, particularly when the specimen reaches it's final shape. When mapping APT specimens, the largest map step-size that enables an accurate ROI identification should be used to decrease mapping time. A survey-mode scan of the specimen may be enough to identify the grains, avoiding the need for a full map. Exposure time can also be decreased to improve mapping speed. Before mapping, check the save patterns box under "EBSD Mapping" to allow easy post-processing of the Hough Transform.

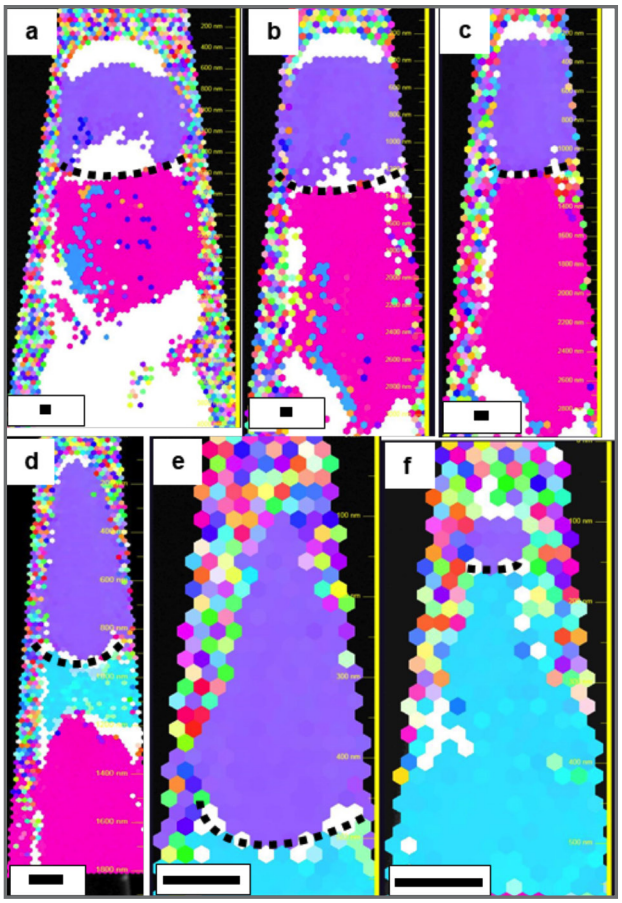

Fig. 16: a-f: Sequential, raw tEBSD maps taken during the FIB-milling process. The black dashed-line shows the approximate position of the grain boundary being targeted. All scale bars are 100 nm.

STEP-BY-STEP PROCEDURE

7 Additional Comments on Resolution

The absolute resolution of tEBSD depends on sample type and thickness. The exposure times and step size for mapping may vary considerably with sample type, based on the diffraction characteristics of the material being mapped. Additionally, when camera parameters are set for mapping, these will be used for the entire map. Monte Carlo simulations[7] can provide insight about how the incident beam might be expected to interact with the material and what kind of resolution might be practical for different thicknesses. Fig 17 a and b show simulations of a 30 kV incident electron beam scattering on a conically-shaped Ni APT specimen. Each dot represents an electron, colored by the amount of incident energy it has retained when exiting the specimen. Figure 17c shows experimental data revealing a breakdown of indexing in the thicker central section of an APT specimen, which can be attributed in part to beam broadening.

Figure 18 a and b show a histogram of counts per pixel when the beam is at (a) 100 nm away from the apex and (b) 1000 nm from the specimen apex. Figs 17 and 18 both show that mapping 100 nm away from the apex provides a compact beam at the exit surface, making high resolution measurements possible. When the beam is 1000 nm from the apex, internal scattering of the electrons within the material severely degrades the beam resolution. In this case, the user may want to select different camera parameters for different map sections, which must be done by manually creating separate maps.

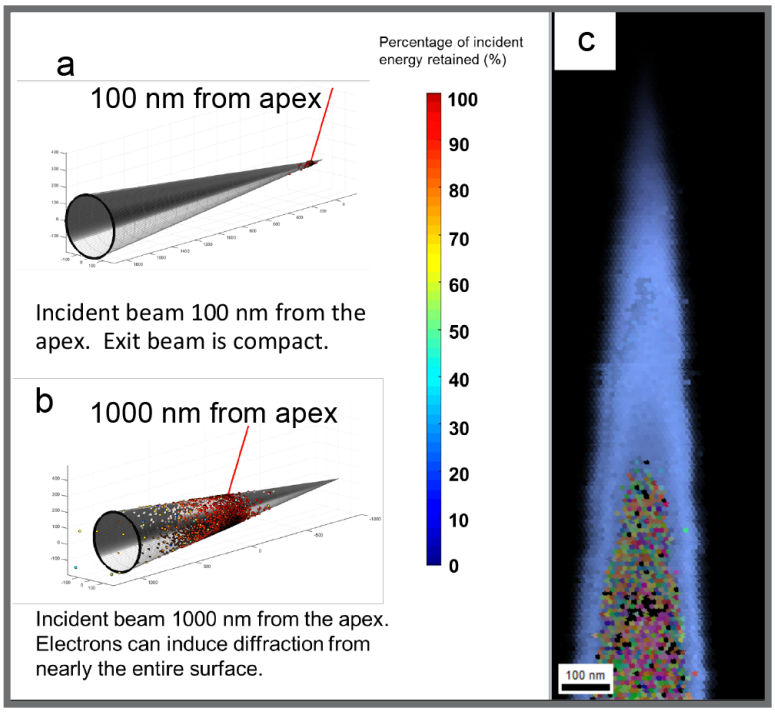

Fig. 17: a-b) Monte Carlo simulations of electron exit position and energy, (c), mapping a single crystalline specimen showing non-indexed points in thicker regions.

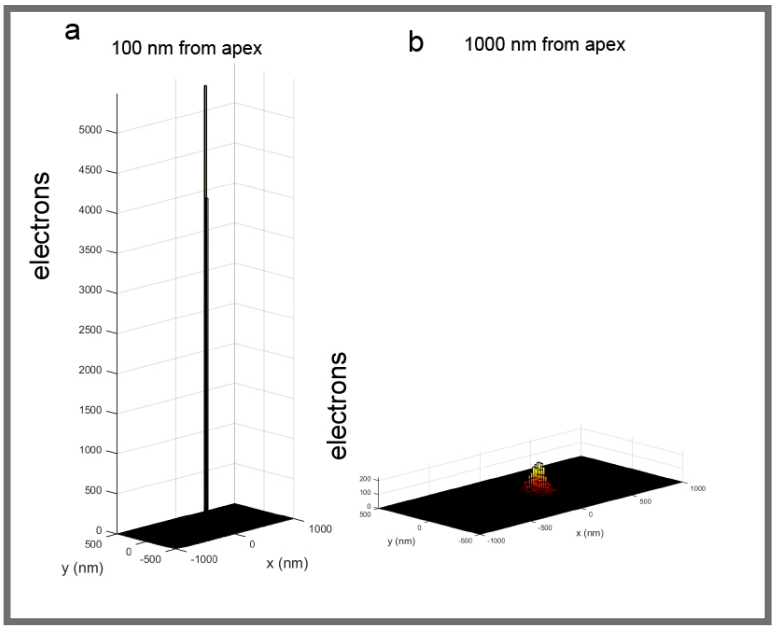

Fig. 18: Electrons per 20x20 nm exit surface pixel at a) 100 nm from the specimen apex, b) 1000 nm from the specimen apex.

STEP-BY-STEP PROCEDURE

8 Data Processing

The OIM Analysis™ (v8) software has many options available for data processing and analysis, so the reader is referred to the EDAX OIM™ manual for detailed descriptions of available features. Here, two useful features are described for APT specimens.

Many times, the final map contains a mixture of points mapped over open area in the chamber, as well as the area occupied by the specimen, which leads to non-indexed points over the open area. Creating a partition by video signal will filter out map points that were mapped over dark areas in the image (i.e. vacuum).

To open the collected map in the analysis software, click Export to OIM in the Review Data tab of the TEAM™ software (Fig. 19). In the Project Tree, right click on the map (e.g. map20180907141155304 in Fig. 20). Choose Partition, and a new Partition Properties window will pop up as shown in Fig. 20. Select Video Signal, and change any default options, which are based on background image from the SEM. The values may need to be adjusted to optimally filter the image. Raw and video signal filtered maps are shown in Figs 21 a and b.

To quickly find the misorientation between two grains visible in the map, click Plots: Misorientation or Vector:Line functions as shown in Fig. 22. Select the first grain, followed by the second grain, or draw a vector between the two grains. The misorientation values will be given in the Interactive Tab or as a separate graph. Complete analysis options are given in the OIM Analysis™ manual.

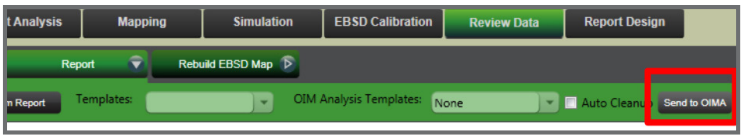

Fig. 19: Exporting data to OIM Analysis™ from TEAM.

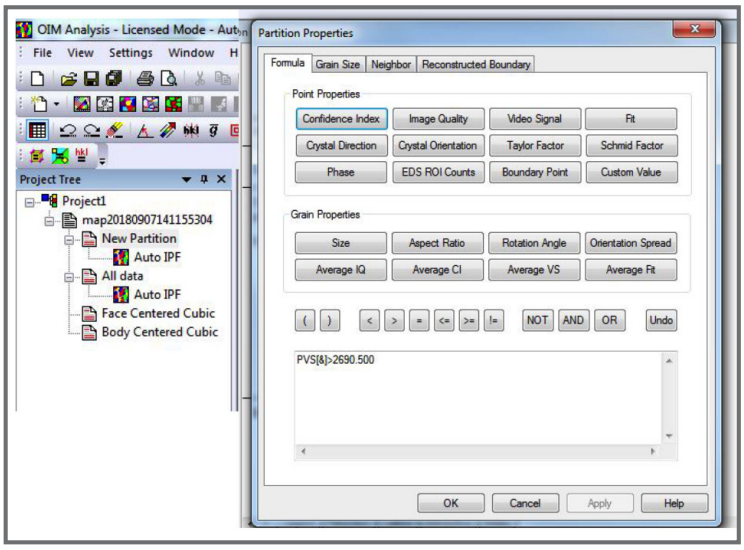

Fig. 20: Video signal partition of the Partition Option window.

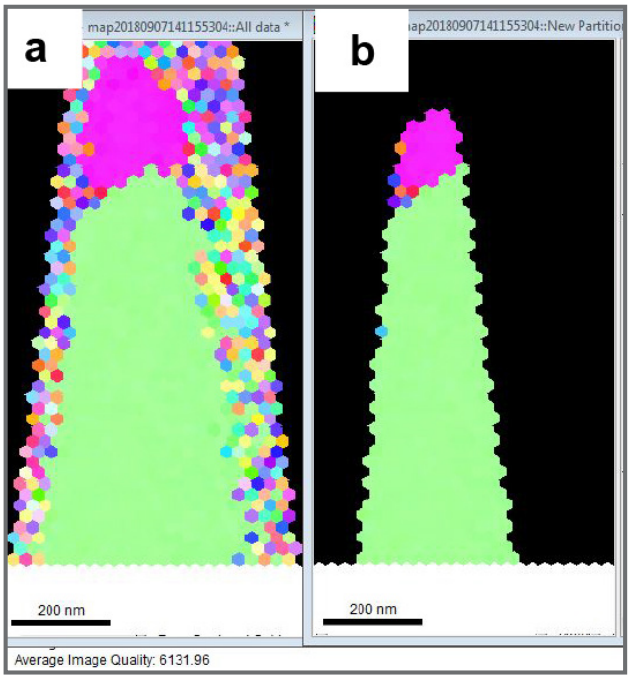

Fig. 21: (a) Raw and (b) partitioned maps by video signal.

Fig. 22: Highlighted Vector: Line and Plot: Misorientation functions in the OIMA toolbar.

STEP-BY-STEP PROCEDURE

9 Additional Tips, Tricks, and Troubleshooting

Mounting specimens for tEBSD

- Use a short stage pedestal with a 45° pre-tilted holder to allow for tilting the tip specimen flat.
- Do not secure APT specimens with carbon tape whenever possible! The heating/relaxation of the tape under the electron beam will cause movement of the coupon/specimen and cause the map to drift during mapping
- When using microtip coupons, make sure the rows of tips you want to map are closest to the camera (lower on the pre-tilted holder).
- When mapping the second row of tips, use a higher tilt angle to minimize reflections from the substrate.
- When mounting to wires, the nominal stage tilt will not be exact due to crimping/bending. Adjust the tilt of the wire such that the mounted wedge is exactly normal to the ion beam for annular milling. That provides an actual tilt of 38° for tEBSD for ion columns oriented at 52°.

Collecting patterns and maps

- Use a beam acceleration voltage of 30 kV in most cases. For lighter elements or oxides, or as the sample gets thinner, 20 kV may be a better choice based on smaller interaction volumes. When using lower currents and voltages, longer camera exposure times may be needed to produce good patterns.
- For wire-mounted specimens, rotate the specimen in the holder to reveal different/better grain boundary views. Multiple grains in the beam direction (z) are challenging as Kikuchi diffraction patterns are formed from the exit surface of the specimen.
- Use the dynamic background subtraction algorithm, intensity histogram normalization, and median smoothing filter for the best results. Atom Probe Assist Mode contains this as a preset, but it can be added also be adjusted under Custom. The order of items in the Image Processing Recipe matters.
- Default contrast of the Kikuchi patterns may not be optimal for indexing. Options to change contrast may need to be explored (see Intensity Normalization option in the Image Processing tab).
- Use good vacuum practices to prevent carbon contamination. Use of a cryogenic anticontamination device is recommended.
- Use the largest step size and fastest exposure settings that still give you the pattern quality and resolution that you need (i.e. grain boundary position).
- Use trapezoidal mapping shape to reduce mapping time.
- Save EBSD positions and FIB positions as presets. This will make it easy to switch between the milling position and mapping position. Retract the EBSD detector before any stage movement.
- Save patterns. This will allow you to adjust the Hough settings after map collection to prevent taking repeated maps and contaminating your sample.
- Use the auto-beam-off setting so that the beam does not contaminate the sample after map collection.
- When setting exposure to prevent beam damage or contamination, use survey mode.
- When using automated background optimization Atom Probe Assist, set the beam in the middle of both x and y regions that you would like to map to minimize contrast variations due to thickness.
- Use low energy ion-beam final milling (low KV cleanup) with a circular milling pattern to remove Ga ion damaged layer and carbon contamination to improve pattern quality. When struggling to collect good patterns, this procedure may need to be used before each sequential map collection. For samples especially sensitive to Ga+ implantation, a 1 kV cleanup may be used.
- A final low-kV cleanup step can be added after the last map to remove any additional carbon contamination before APT.
- Remember, the direction for crystallographic orientation references the normal direction relative to the exit surface.

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Acknowledgements: CAMECA thanks Dr. Mark Stoykovich, Dr. Austin Akey, and Dr. David Dierks for helpful data and comments in preparing this technical note.

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Technical Note TN-09
Transmission EBSD of APT Specimens
Document Number 30876
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