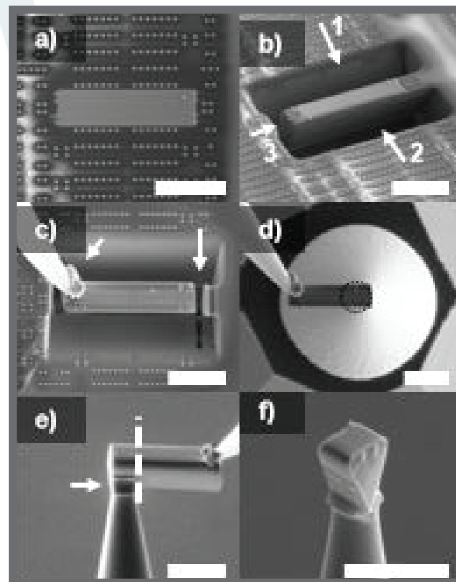

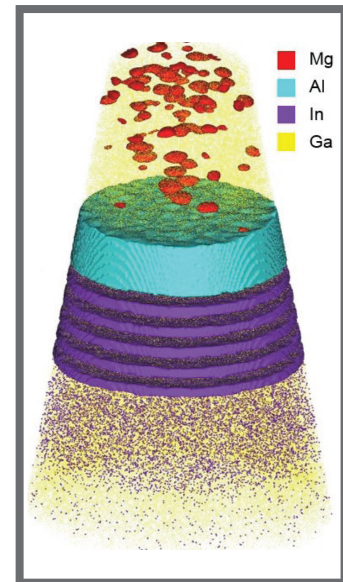

Technical Note TN-01 FIB LO

Standard FIB lift-out sample preparation procedure for APT

Achieving site specific sub-nanometer three-dimensional compositional resolution with a LEAP®, a FIB-SEM, and a microtip sample carrier has been demonstrated to be a straightforward and mature process for a wide variety of materials. This technical note is one of several designed to pass on key information to minimize time-to-knowledge for Atom Probe Tomography (APT) applications. Please contact your CAMECA representative for a list of available technical notes specific to your application.

PROCESS OVERVIEW

Site specific region selection in bulk materials can be isolated through use of a Dual Beam FIB equipped with a micromanipulator and a GIS deposition system. Instructions for standard lift-out (LO) from a silicon substrate follow. These instructions were developed with an FEI Nova and Omniprobe micromanipulator systems, but can be adapted to any other FIB/GIS combined system with the appropriate resolution and stage geometry.

APPARATUS and SETUP

This procedure requires the use of a Dual Beam FIB equipped with a micromanipulator and a Platinum or Tungsten GIS system. A standard SEM stub with carbon tape is not recommended to hold the sample material and the microtip coupon (Fig. 1). To avoid sample drift issues possible with carbon tape, it is strongly suggested to use clips PN 23739 available from CAMECA Instruments Inc., as shown in Figure 2.

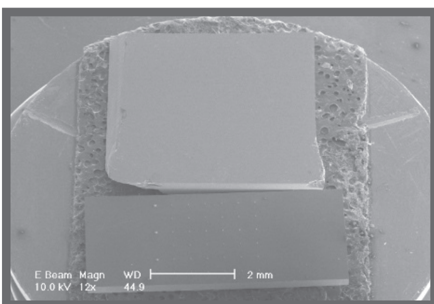

Fig. 1: Sample affixed on SEM stub using carbon tape.

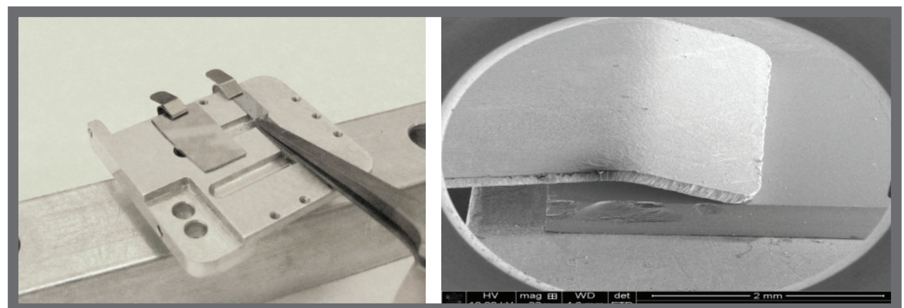

Fig. 2: Corresponding optical and SEM images of a sample clipped onto a stub for LEAP analysis.

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STEP-BY-STEP PROCEDURE

Note on initial capping step prior to FIB specimen preparation

As a prerequisite to FIB specimen preparation, it is advised to use a capping material to ensure that a damage-free volume remains prior to analysis with the atom probe, especially when the surface is part of the region of interest. Ideally, capping materials should exhibit good adhesion to the surface, good ion milling behavior for FIB tip shaping, and good field evaporation properties for atom probe analysis. To maximize coating efficiency, a base vacuum >1e7 torr is required, as well as the ability to tilt and rotate the stage. Magnetron-style "SEM" coating systems are not adequate. For most metallic or semiconductor applications, a 50-nm thick layer of Ni cap is sufficient. Capping considerations are available in Technical Note TN-03 (Doc. #30345).

1 Protection of region of interest (ROI) by Pt or W layer deposition

Deposition of a sacrificial layer is needed since the Ga beam is operated at an accelerating voltage of 30kV until the last sample preparation step performed at 5kV. In order to contain Ga implantation and sample amorphization as illustrated in Figure 4, Pt or W have to be laid to protect the region of interest. Both species have been found to fulfill that function equally well.

Note: Set the scan rotation in the ion image to 180° and always place the mill pattern below the Pt strap to ensure cutting in the proper direction.

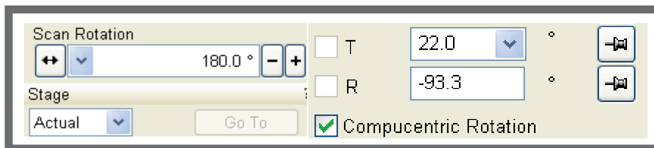

Fig. 3: Scan rotation setup at 180°.

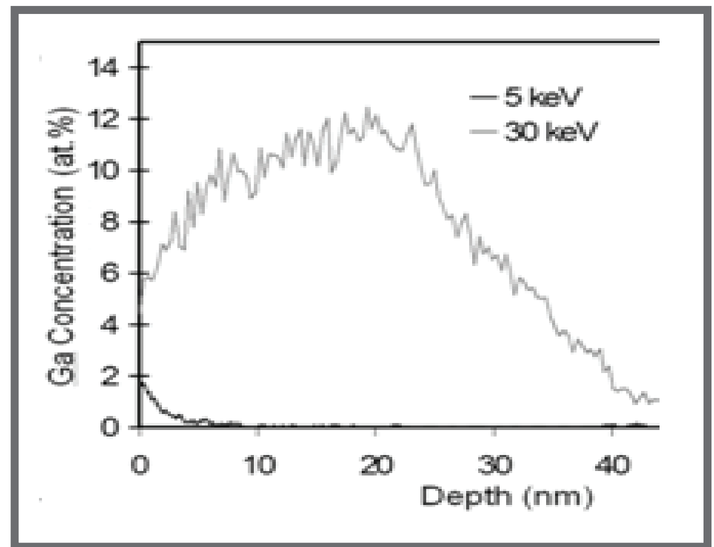

Fig. 4: Ga implantation into Si using 5kV and 30kV acceleration voltage.

Prerequisite: Eucentric, warm GIS, Pt selected

A–Tilt stage to 52 degrees

B–Select beam current of 28 pA

C–Draw a box of the following dimensions after having inserted the GIS needle:

X: 30µm

Y: 2.50µm

Z: 0.15µm (150nm)

Expected time: ~10 minutes

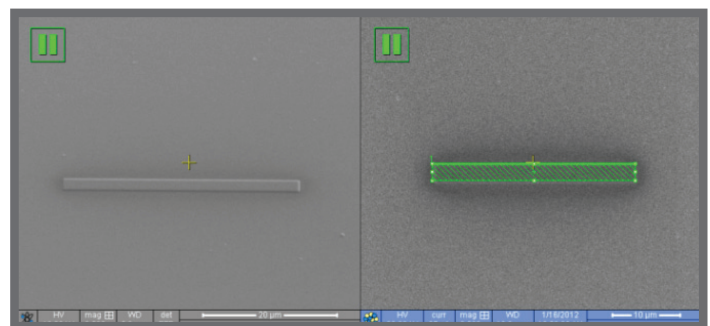

Fig. 5: ROI protected with Pt deposition.

STEP-BY-STEP PROCEDURE

2 Wedge cut #1 (adjacent to the 1st side of the deposition layer)

Prerequisite: GIS removed, Si selected

A–Tilt stage to 22 degrees

B–Select beam current of 6.5 nA

C–Draw a box of the following dimensions:

X: 35μm

Y: 3.50μm

Z: 3.50μm

Expected time: ~5 minutes

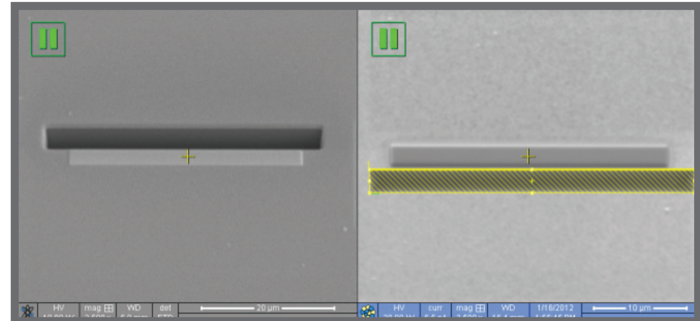

Fig. 6: 1st wedge cut along Pt strap.

3 Wedge cut #2 (adjacent to the 2nd side of the deposition layer)

Prerequisite: Comp-eucentric stage rotation at 180°, stage tilt 22°, Si selected

A–Select beam current of 6.5 nA

B–Draw a box of the following dimensions:

X: 35μm

Y: 3.50μm

Z: 3.50μm

Expected time: ~5 minutes

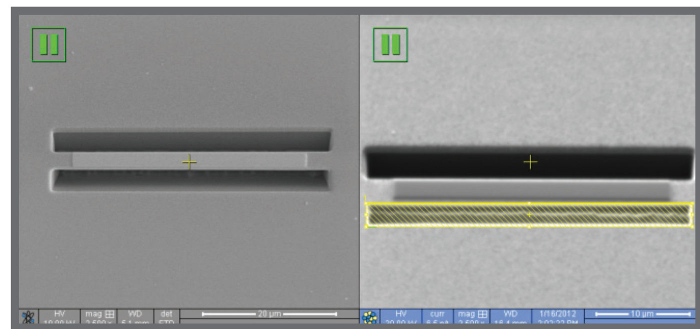

Fig. 7: 2nd wedge cut along Pt strap.

Note: Redeposited materials have poor thermal and electrical properties and therefore need to be removed using clean up cuts.

4 Clean-up cut #1 (adjacent to the 2nd side of the deposition layer)

Prerequisite: Comp-eucentric stage rotation at 180°, stage tilt 22°, Si selected

A–Select beam current of 6.5 nA

B–Draw a box of the following dimensions:

X: 35μm

Y: 1μm

Z: 3.50μm

Expected time: ~30 seconds

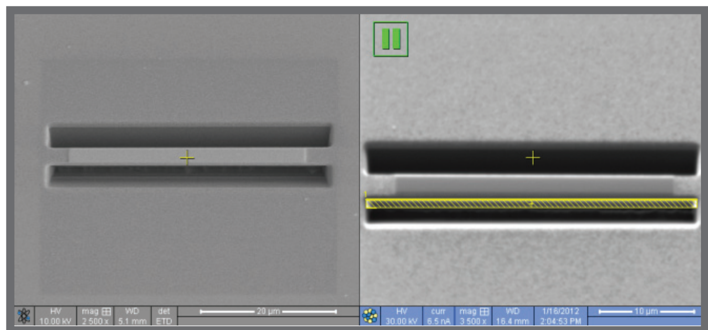

Fig. 8: 1st clean up cut along Pt strap.

STEP-BY-STEP PROCEDURE

5 Clean-up cut #2 (adjacent to the 1st side of the deposition layer)

Prerequisite: Comp-eucentric stage rotation at 180°, stage tilt 22°, Si selected

A–Select beam current of 6.5 nA

B–Draw a box of the following dimensions:

X: 35µm

Y: 1µm

Z: 3.50µm

Expected time: ~30 seconds

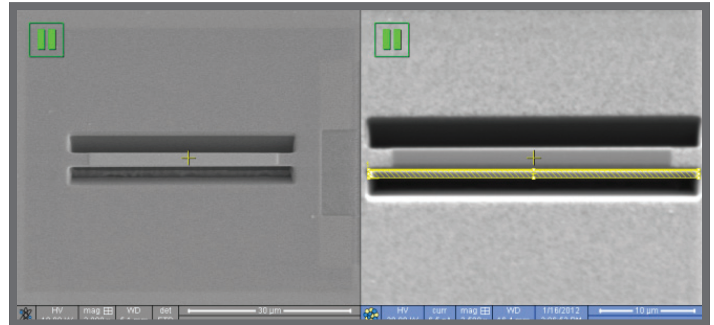

Fig. 9: 2nd clean up cut along Pt strap.

6 Cantilever cut (wedge cut #3 normal to the 2 long wedge cuts)

Prerequisite: Comp-eucentric stage rotation at 180°, stage tilt 22°, Si selected

A–Select beam current of 6.5 nA

B–Draw a box of the following dimensions:

X: 1.5µm

Y: 4µm

Z: 3.50µm

Expected time: Less than 15 seconds

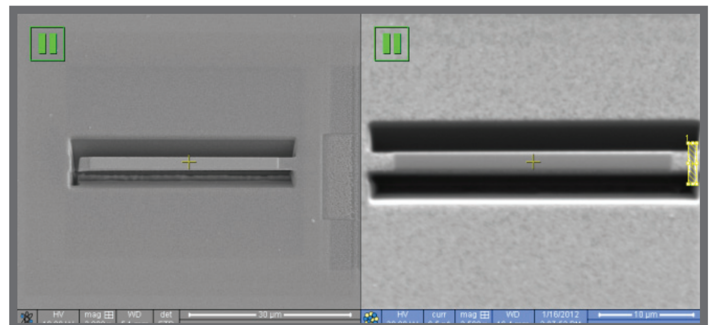

Fig. 10: Wedge cut normal to Pt strap.

7 Probe welding to cantilever cut using Pt or W

Prerequisite: Eucentric, warmed-up GIS, Pt selected

A–Tilt stage to 0 degrees

B–Select beam current of 28 pA

C–Insert GIS and micromanipulator.

D–Make gentle contact to the cantilever free end with the micromanipulator

E–Draw a box and weld the micromanipulator to the cantilever using Pt or W deposition

Expected time: ~1 minute

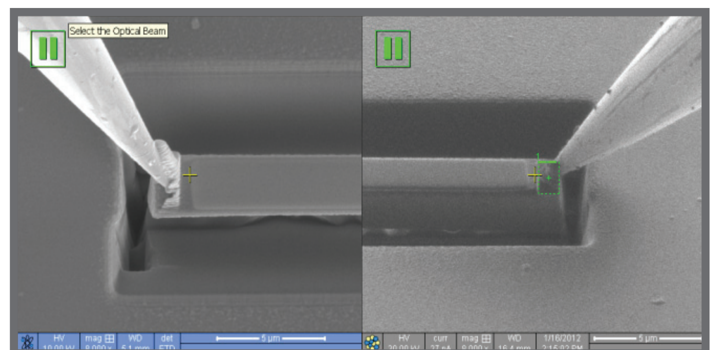

Fig. 11: Probe welded to cantilever end.

STEP-BY-STEP PROCEDURE

8 Wedge cut #4 to free lamella from bulk sample

Prerequisite: Eucentric, Si selected, stage tilt 0°

A–Select beam current of 0.28 nA

B–Draw a box of the following dimensions:

X: 1.5μm

Y: 4μm

Z: 3.50μm

Expected time: Less than 1 minute

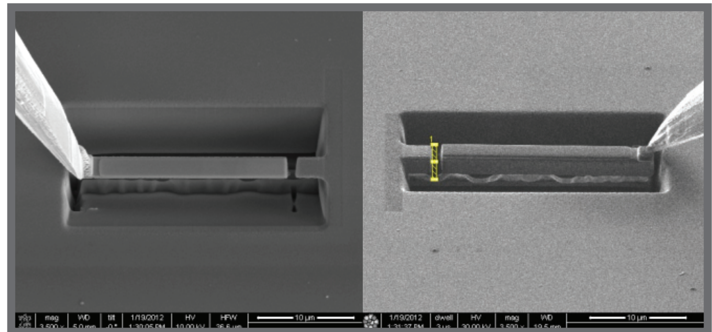

Fig. 12: Lamella cut free from bulk.

Note: Watch for wedge vibration on micromanipulator to be sure it is free and ready to be lifted out.

9 Wedge lifted out from bulk sample using micromanipulator

Prerequisite: Wedge free from bulk sample, stage tilt 0°

A–Lift the probe up with a slow Z movement until the wedge is above bulk surface

B–Cross-check position with electron image

C–Move probe and wedge system to the park position

Expected time: ~1 minute

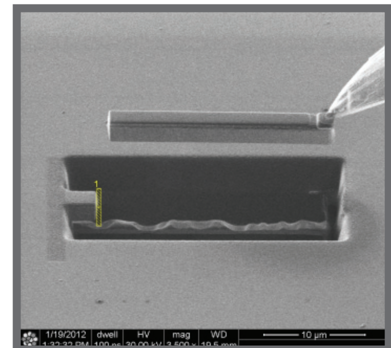

Fig. 13: Lamella lifted out from bulk.

10 Lift-out positioning to microtip coupon

Prerequisite: Stage Z lowered 3mm below eucentric, stage tilt 0°

A–Move to microtip coupon

B–Orient the coupon with fiducials located at top or bottom of the image

C–Re-establish stage eucentric position on 1st microtip post to be mounted

D–Slowly bring the wedge into contact with microtip post

Expected time: ~2 minutes

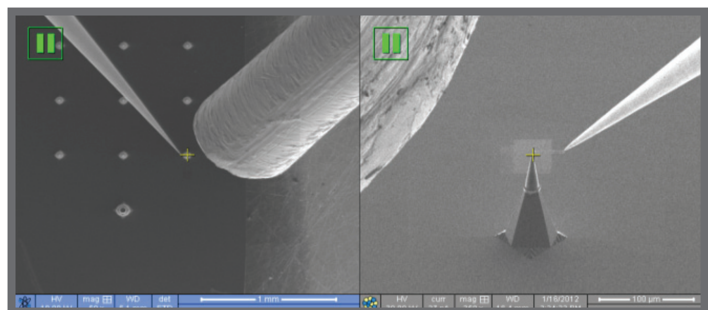

Fig. 14: Positioning on 1st microtip post.

STEP-BY-STEP PROCEDURE

Note: The SEM image will change slightly in contrast once contact is made between the wedge and the microtip post to be mounted. From a top view, only a slight edge of the microtip post should be seen. The bottom of the edge should be aligned with the diameter of the microtip post (flat top).

11 Wedge mounting onto microtip post

Prerequisite: Warm GIS, Pt selected, stage tilt 0°

A–Select beam current of 28 pA

B–Define a square pattern about the same width of the microtip post, and Z ~0.3 µm

C–Weld the wedge to the microtip post

Expected time: ~ 1 minute

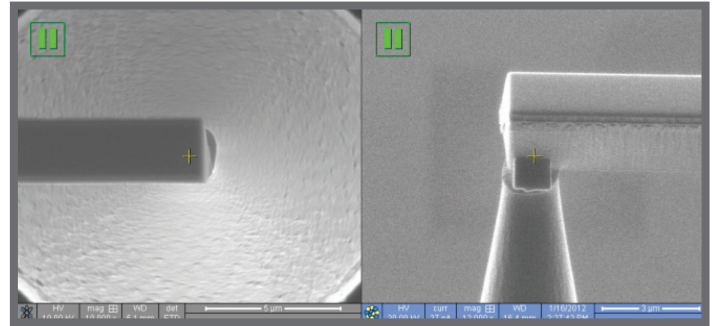

Fig. 15: Wedge welded onto microtip post.

12 Wedge cut into individual mount

Prerequisite: Si selected

A–Select beam current of 0.28 nA

B–Draw a box of the following dimensions:

X: 0.50µm

Y: 3.50µm

Z: 3.50µm

C–Once cut is cleanly through, move the wedge away using XYZ controls of the probe

Expected time: Less than 3 minutes

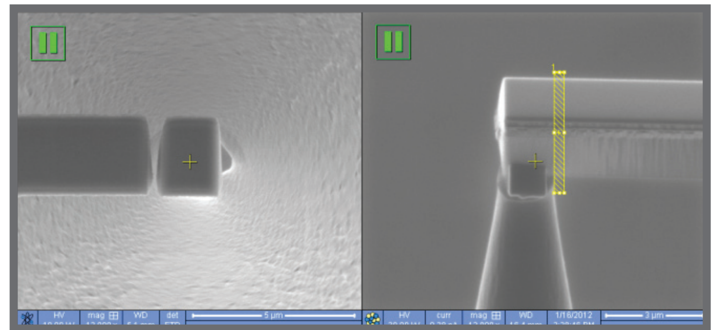

Fig. 16: Individual mount cut free.

Note: The whole initial wedge can be subdivided into 6 to 12 individual mounts successively by repeating steps 10 to 12. This operation is called Propagation.

STEP-BY-STEP PROCEDURE

13 Micromanipulator clean up and reshaping

Prerequisite: Wedge propagated, Si selected

A–Select beam current of 6.50 nA

B–Define a box to cut any remaining material from micromanipulator

C–Define 2 boxes to reshape it sharply

D–Retract micromanipulator as well as GIS needle

Expected time: Less than 3 minutes

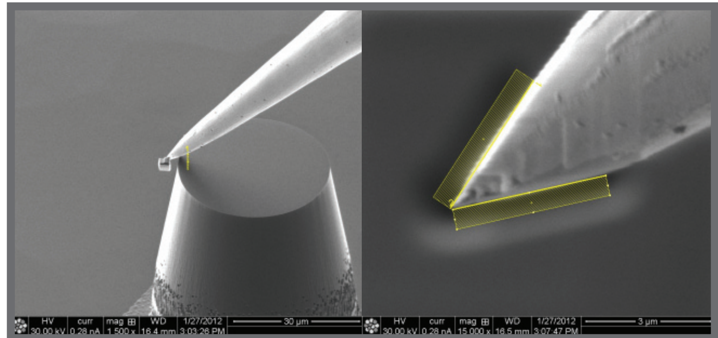

Fig. 17: Micromanipulator clean up.

14 Backside brazing of individual mount

Prerequisite: Eucentric, 180° ion beam scan rotation, stage tilt 0 °, warm GIS, Pt selected

A–Get positioned on 1st mount to be brazed

B–Select beam current of 28 pA

C–Insert GIS needle

D–Define a square pattern about the same width of the microtip post, and Z ~0.3 µm

E–Weld the backside of the wedge to the microtip post using Pt deposition

Expected time: less than 1 minute for the tack

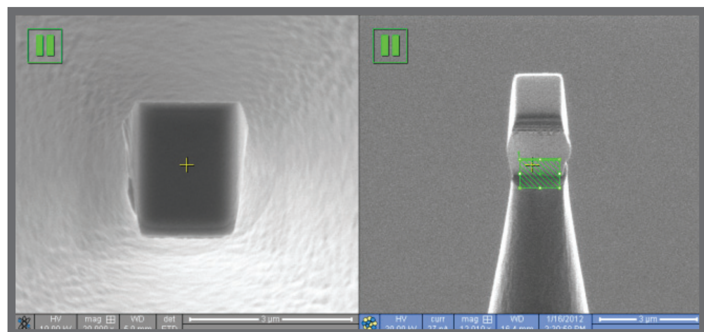

Fig. 18: Backside brazing of the individual mount on microtip post.

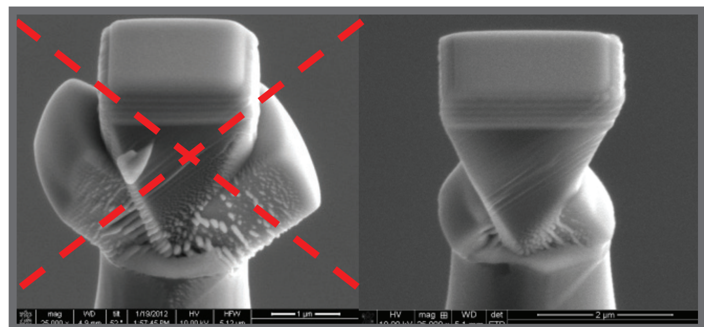

Fig. 19: Bad vs. good side brazing.

Note: Use a minimal amount of Pt/W for side tacking in order to reduce the processing time in both deposition and sharpening steps.

At that point, individual mounts are ready to be sharpened and transformed into needle-shaped specimens.

The sharpening step-by-step procedure is covered in the Technical Note TN-02 (Document Number 30444).

REFERENCES

This reference list is not an exhaustive list. However, it contains the most useful and practical reviews and publications used for the purpose of preparing specimens for atom probe tomography.

- D. J. Larson, T. J. Prosa, R. M. Ulfig, B. P. Geiser, T. F. Kelly, Local Electrode Atom Probe Tomography—A User's Guide, Springer Characterization & Evaluation of Materials, New York: Springer, (2013). ISBN 978-1-4614-8721-0.
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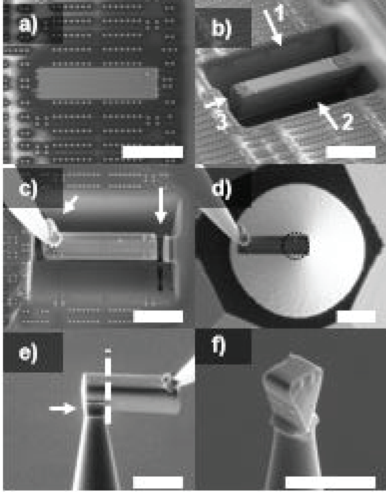
- Giddings A. D., Prosa T.J., Olson D., Clifton P.H. and Larson D.J., Reverse Engineering At The Atomic Scale: Competitive Analysis Of A Gallium-Nitride-Based Commercial Light-Emitting Diode, Microscopy Today, Volume 22, Number 5 (2014).

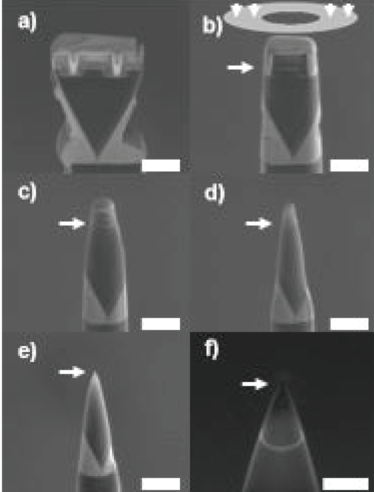
CAMECA FIB/SEM Preparation Technical Notes

Lift-out and Mount



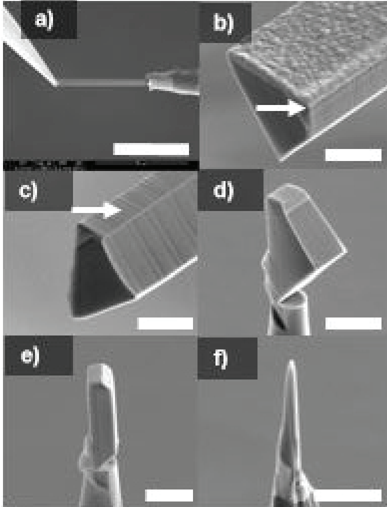
All scale bars are 5µm

Sharpen



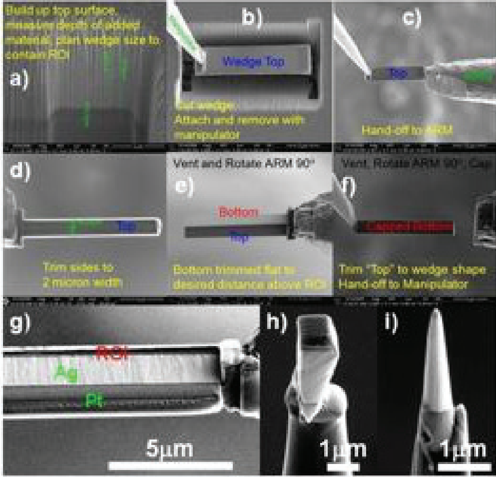
All scale bars are 1µm except f) at 200nm

Cross Section



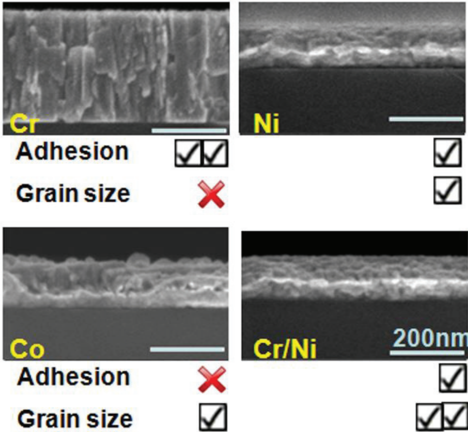
All scale bars are 3µm except a) at 25µm

Backside

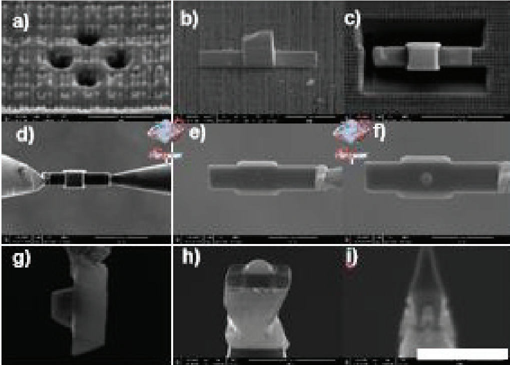


Last scale bar represents 300nm

Capping



Single Device Analysis



Last scale bar represents 300nm

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Fax: 608-442-0622

Technical Note TN-01 FIB LO
Standard FIB lift-out sample preparation procedure for APT
Document Number 30443
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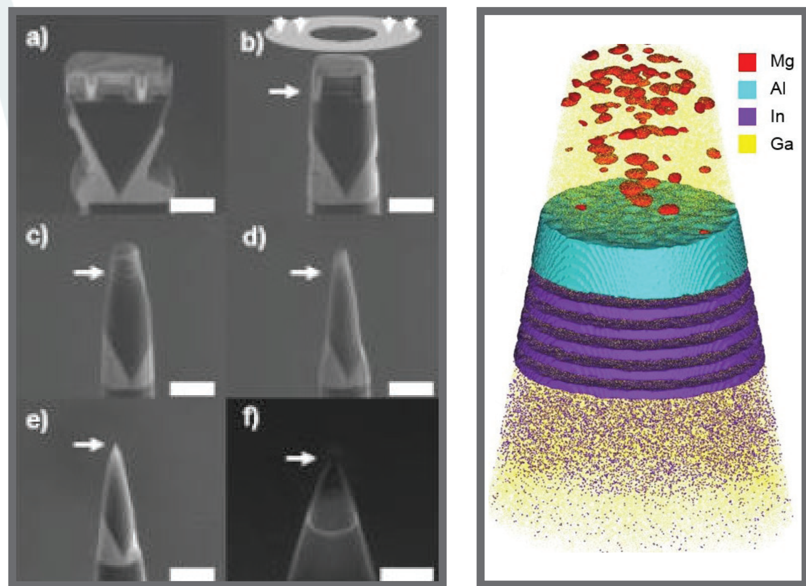

Technical Note TN-02 FIB Sharpening

Standard FIB sharpening procedure for APT

This technical note on FIB specimen sharpening focuses on completing the procedure required to obtain suitable specimens for atom probe analysis. As a prerequisite, materials should already be mounted on flat-top microtip posts (Fig. 1). The steps for mounting materials are described in TN-01 (Document Number 30443).

PROCESS OVERVIEW

The main goal is to prepare a tip that is sharp enough to field emit during APT analysis. The region of interest (ROI) should be near the end of the tip (less than 100nm deep) and undamaged by Ga ions. Although specific magnifications, beam currents, and milling times are used in this technical note, a wide variety of operations can produce specimens that are of high quality.

APPARATUS and SETUP

This procedure requires the use of a Dual Beam FIB. These instructions were developed using a FEI Nova 600 system, but can be adapted to any other FIB with the appropriate resolution and stage geometry. As mentioned in TN-01, it is strongly suggested to use clips PN 23739 available from CAMECA Instruments, Inc. for holding the microtip coupon. Avoid using too much Pt for side tacking (Fig. 2) and/or mounting off-center (Fig. 3) in order to reduce the processing time in both deposition and sharpening.

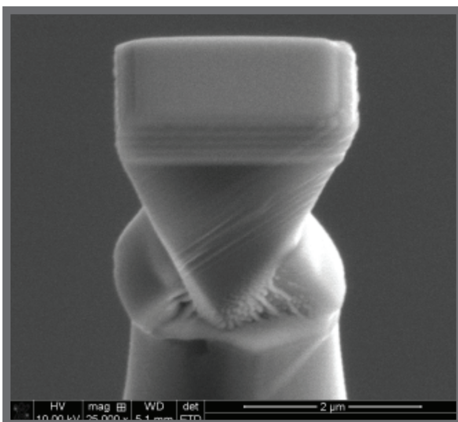

Fig. 1: Mount properly set for the FIB sharpening process.

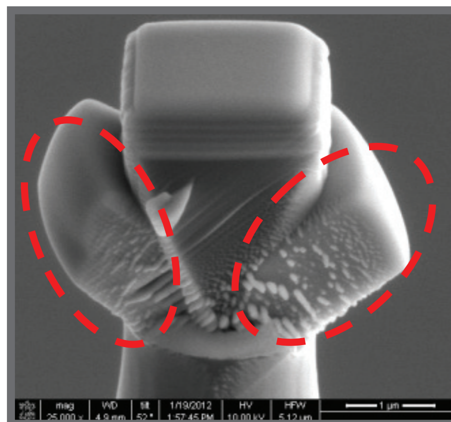

Fig. 2: Excessive Pt side tacking.

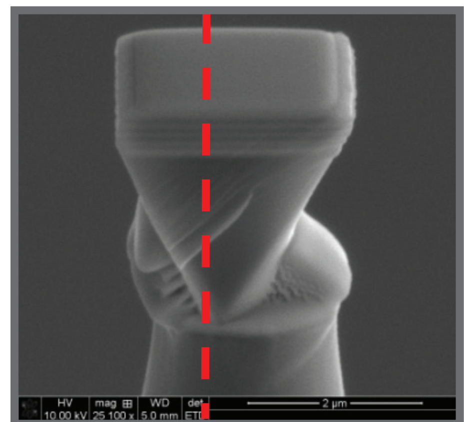

Fig. 3: Off-centered positioning on microtip post.

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STEP-BY-STEP PROCEDURE

Notes on annular milling pattern files, stream files, and bitmaps

Depending on the FIB system used, sharpening can be completed using annular milling patterns defined either by stream files or bitmaps. Due to the differences in file formats, it is important to note the correlation between inner/outer diameters and magnification (Table 1). This table will be particularly useful when approaching the critical final sharpening steps.

As a general rule, annular milling patterns should follow a motion from outside to inside with respect to the tip in order to limit any undesired redeposition. Figure 4 shows the two patterns used in this sharpening procedure to obtain a final tip diameter of 50 to 200nm.

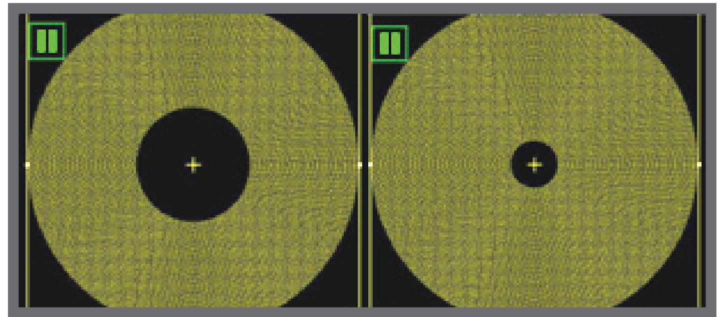

Fig. 4: Annular milling patterns (1 & 2) defining inner and outer diameters.

Magn. (kX)	10	20	30	50	65	80
ID#2 (nm)	4000	2000	1100	800	600	500
ID#1 (nm)	1700	850	550	330	250	200
OD (nm)	7900	5900	3900	2400	1800	1500

Table 1: Patterns for inner and outer diameter dimensions with respect to magnification level.

1 First milling pass: Cylindrical push down

This milling step is used to remove the bulk of the material and to push it down far enough to prevent parasitic spikes from forming on the shank of the microtip. Parasitic spikes should be at least 10µm away from the region of interest in the tip to avoid interfering with the desired area of evaporation.

Prerequisite: Comp-eucentric, stage tilt 52°, Si selected, 30kV Ga beam.

Milling parameters:

A–Annular milling pattern #1

B–Magnification: 15kX to 25kX

C–Beam current: 0.28nA

Expected milling time: 2 to 6 minutes

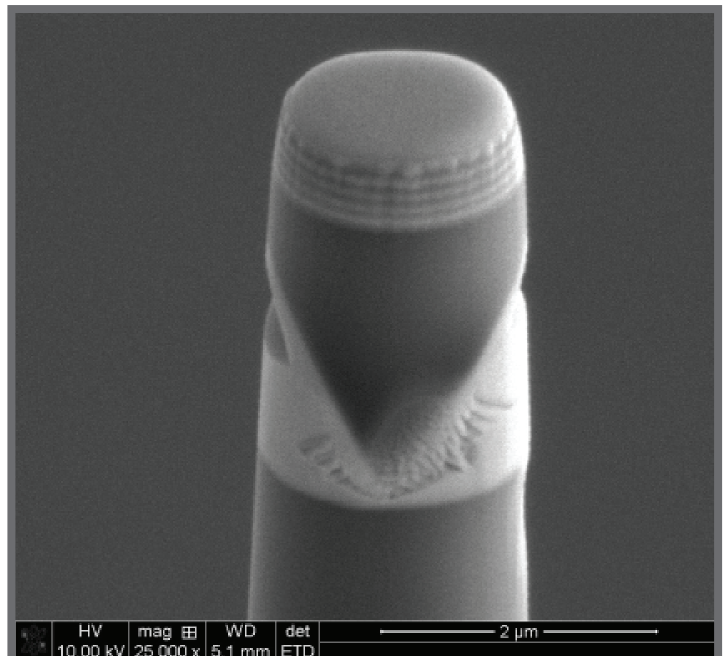

Fig. 5: Cylindrical push down at 25kX. Specimen sides are parallel.

STEP-BY-STEP PROCEDURE

2 Second milling pass: Initial tip shaping

This milling step marks the beginning of the tip shaping.

Milling parameters:

A–Annular milling pattern #2

B–Magnification: 15kX to 25kX

C–Beam current: 0.28nA

Expected milling time: 1 to 4 minutes

In this example, the alternating bright and dark layers close to the apex of the tip constitute the region of interest.

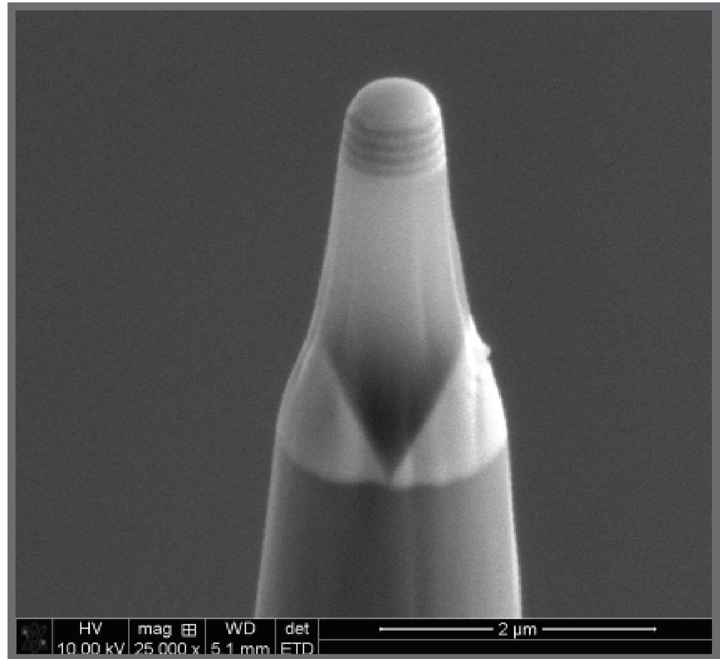

Fig.6: Initial tip shaping at 25kX.

Notes on the importance of specimen tip half shank angle

A typical specimen for APT has a hemispherical cap on a truncated cone with a half shank angle value between 6 and 10 degrees, as illustrated in Fig. 7.

This small angle, also referred to as a taper angle, may help dissipate heat accumulated upon laser impact on the material.

Too long of a second milling pass will result in a tip shape exhibiting a half shank angle close to 0, i.e., a specimen with almost parallel sides (see Fig. 8). This configuration is not desirable as it prevents achieving optimal mass resolving power in APT.

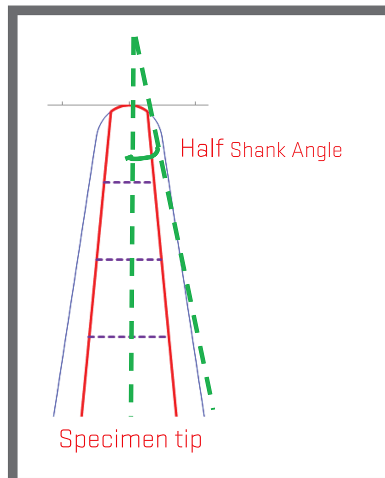

Fig. 7: Schematic of specimen tip and associated half shank angle.

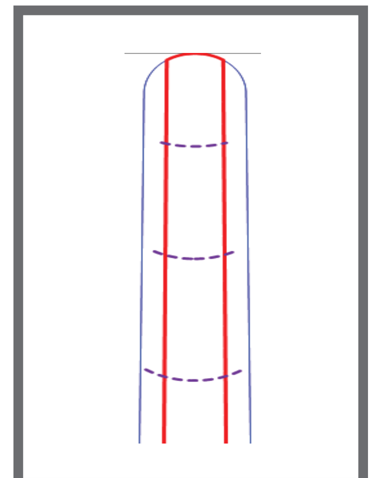

Fig. 8: Schematic of specimen tip with parallel sides (null angle).

STEP-BY-STEP PROCEDURE

3 Third milling pass: Final tip shaping

This milling step is used to define the final tip shape as well as the final tip radius.

Precise centering at the apex of the tip is required to produce a uniform shape.

Milling parameters:

A–Annular milling pattern #2

B–Magnification: 50kX to 70kX

C–Beam current: 28pA

Expected milling time: 30 seconds to 3 minutes

Note: Ultra High Resolution is suitable at this step but should be done quickly as the image will shift substantially from the field of view.

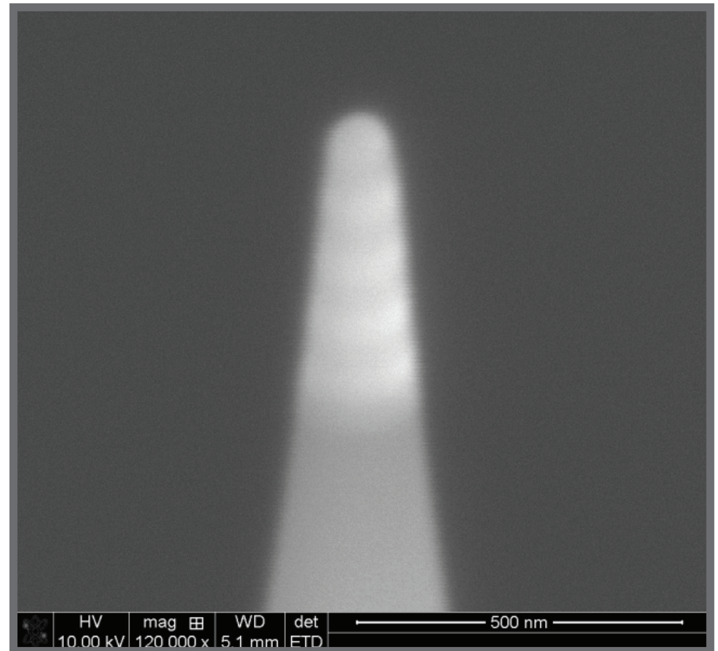

Fig. 9: Final tip shaping was made at 70kX and imaged at 120kX.

4 Low kV Clean-up

This step not only cleans up the Ga damage caused by the 30kV milling steps, but also slightly refines the tip shape.

Milling parameters:

A–Set the acceleration voltage to 5kV

B–Set the beam current to 47pA

C–Acquire an ion beam image to set the circular pattern perfectly over the desired area to be cleaned (Fig. 10)

Expected milling time: 3 to 15 seconds.

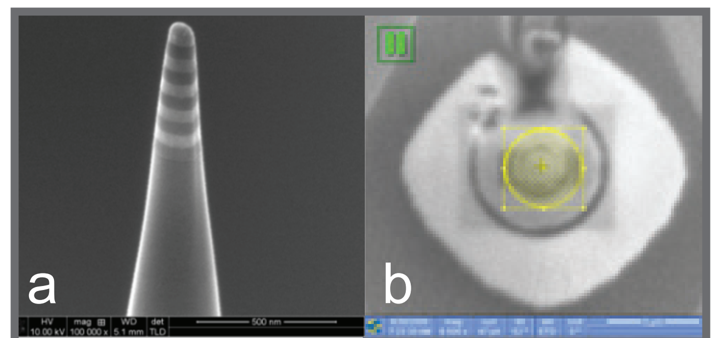

Fig. 10: Final tip shaping was made at 70kX and imaged at 120kX.
a) Cross-section view (Ultra High Resolution imaging)
b) Top view (ion beam)

STEP-BY-STEP PROCEDURE

Notes on capping materials and final imaging steps

The duration of the low-kV step strongly depends on the capping material used as well as the location of the region of interest.

If the capping material runs well during APT analysis (i.e., exhibits good adhesion, maintains suitable electrical and thermal properties, and has an evaporation field close to the material in the tip) it is recommended to leave some cap on the top of the tip. Additionally, if the region of interest is directly below the cap, then a few nanometers of the cap material could be used to fine-tune the LEAP analytical parameters. Use the low-kV step to completely remove capping materials, such as Pt, that produce tip fractures during LEAP analysis. Technical note TN-03 (Document Number 30445) may guide FIB users on the most adequate choice for capping materials.

Upon completion of the low-kV clean-up, it is advised to image the final tip at high, medium and low magnifications for storage positioning as well as reconstruction purposes (average shank angle and tip-profile methods).

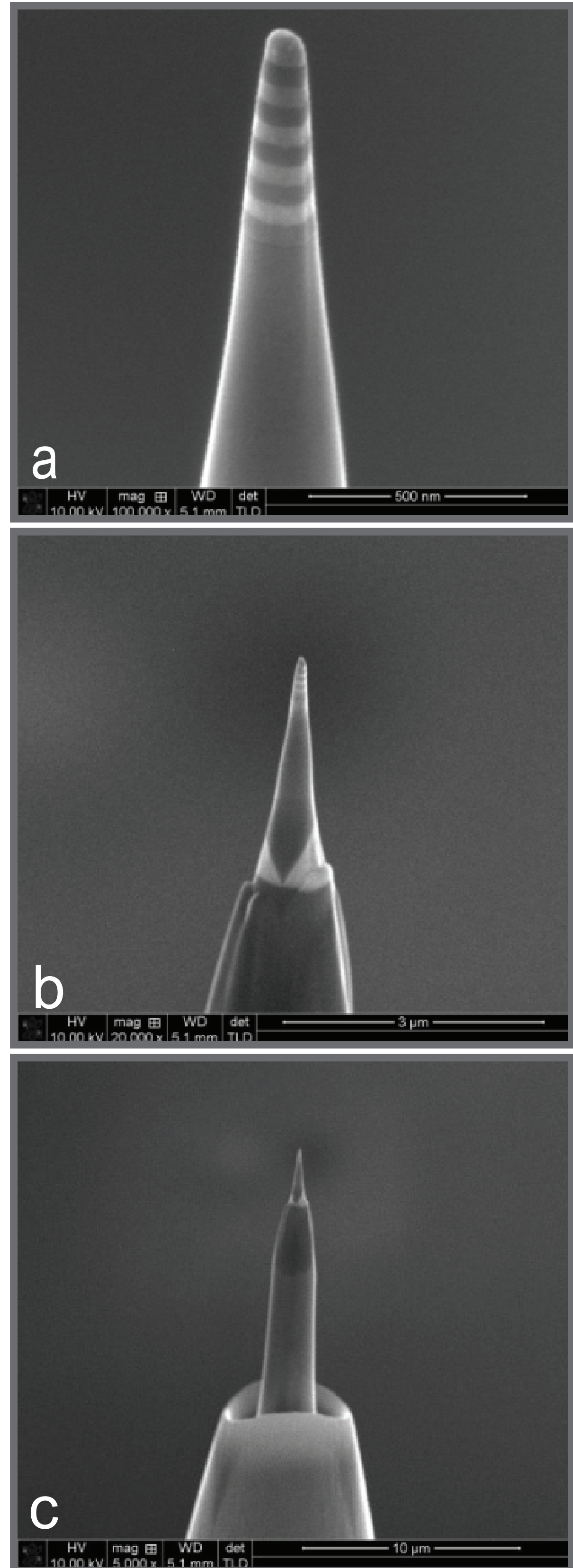

Fig. 11: High (a), medium (b), and low (c) magnification of a finished tip.

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This reference list is not an exhaustive list. However, it contains the most useful and practical reviews and publications used for the purpose of preparing specimens for atom probe tomography.

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