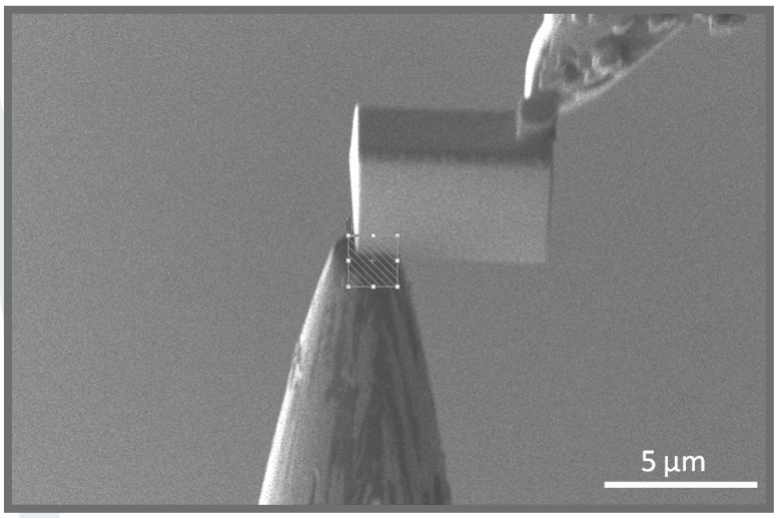


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Technical Note TN-08 Specimen Preparation on Wires

FIB Mounting to wire substrates

Specimens can be mounted to thin metal wires (typically tungsten, but other materials can be used) for correlative microscopy purposes or to be used in either the LEAP™ or EIKOS™ atom probe platforms. This technical note reviews considerations for preparing wires for FIB liftout and mounting specimens to wire substrates in a focused ion beam (FIB) microscope. The procedures for liftout and sharpening using FIB methods are covered in detail in TN-01 and TN-02 and are not included in this technical note.

STEP-BY-STEP PROCEDURE

1 Substrate Preparation: Crimping into copper stubs

Wires can be crimped in copper sleeves (stubs) to provide easier handing and facilitate transfer into atom probe specimen pucks for analysis. Crimping a specimen wire into a stub can be accomplished with the Cameca crimping station (PN: 25742) or with pliers. Tungsten wires provide a substrate that is robust to handing, so it is recommended that they are used for crimping into the copper stubs for subsequent FIB liftout mounting. If using the Cameca crimp station, place copper stub in the crimping hole as shown in Fig 1. Introduce wire into the stub as in Fig 2 and pull lever to crimp evenly. Trim any excess material at the bottom of the stub with a W-capable cutting tool as shown in Fig 3.

Note: crimped tungsten wires ready for mounting can be purchased from Cameca PN 24649

Note: Wear eye protection at all times during this step.



Fig. 1: Copper stub introduced into Cameca crimping station

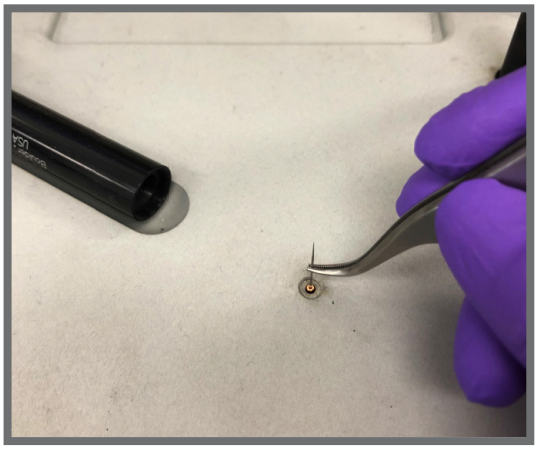


Fig. 2: Wire introduced into copper sleeve

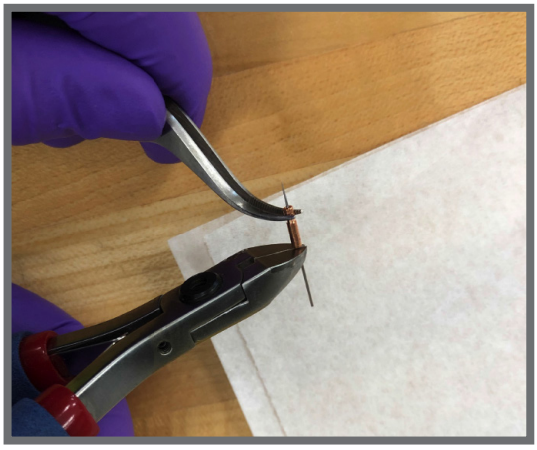


Fig. 3: Excess wire trimmed after crimping

STEP-BY-STEP PROCEDURE

2.1 Method 1: Mounting wires horizontally

After crimping, position wires in a fixture that holds them horizontally. Figure 1 shows a compatible holder made by Cameca (PN: 25100) but any method that securely holds the wires horizontally is acceptable.

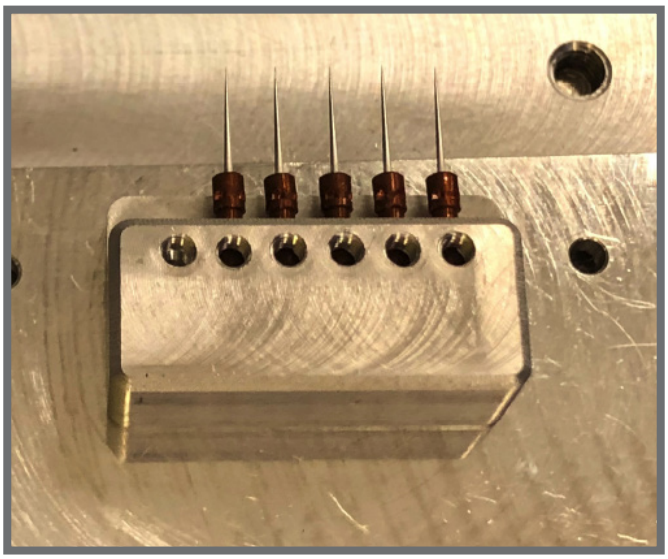


Fig. 4: Horizontal wire holder for FIB

2.2 Method 1: Horizontal milling

With wires held horizontally, Align specimen to eucentric height, tilt stage such that the wire is normal to the ion beam (Fig 5a). Use a rectangular mill pattern with approximate dimensions of 1.5 x 1.5 microns (Fig 5b) to cut the rounded tip of the wire with the ion beam to produce a flat plateau for mounting (Fig 5c). Typical ion beam currents used are between 0.28-2 nA. After all wires are milled, transfer them to a vertical holder as shown in Fig 6.

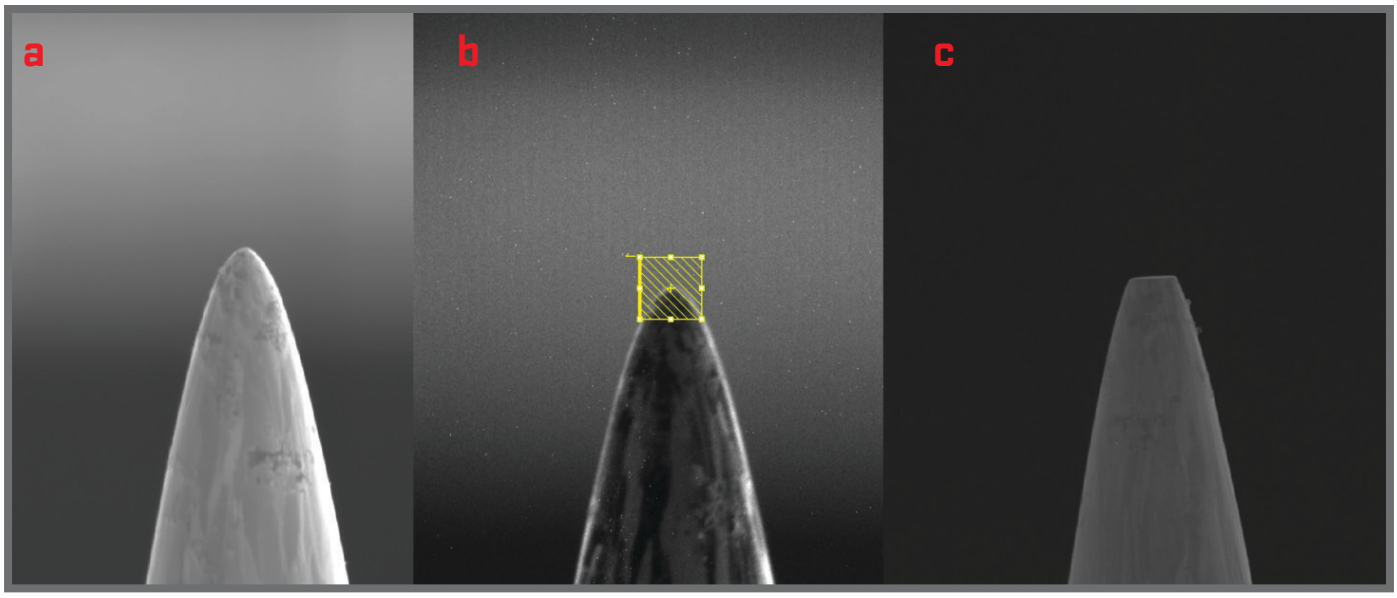


Fig. 5: Milling flat surface into sharp wire specimen for mounting

STEP-BY-STEP PROCEDURE

3 Method 2: Vertical milling

Another option is to cut wires held vertically. Fig 6 shows a compatible vertical holder made by Cameca (PN: 24673) This should provide a suitable surface for mounting a FIB liftout, but the angle of the ion beam will result in an angled surface rather than a flat surface with respect to the tip axis. With the wires held vertically, align the wire apex to eucentric height at 0° stage tilt (Fig 7a). Use a mill pattern rectangle with approximate dimensions of 1.5 x 1.5 microns to cut the rounded tip of the wire with the ion beam to produce a flat plateau for mounting (Fig 7b). Typical ion beam currents used are between 0.28-2 nA. Fig 7c is rotated 90 degrees from the cutting position to demonstrate angled surface that results. After all wires are milled, proceed directly to the liftout process.

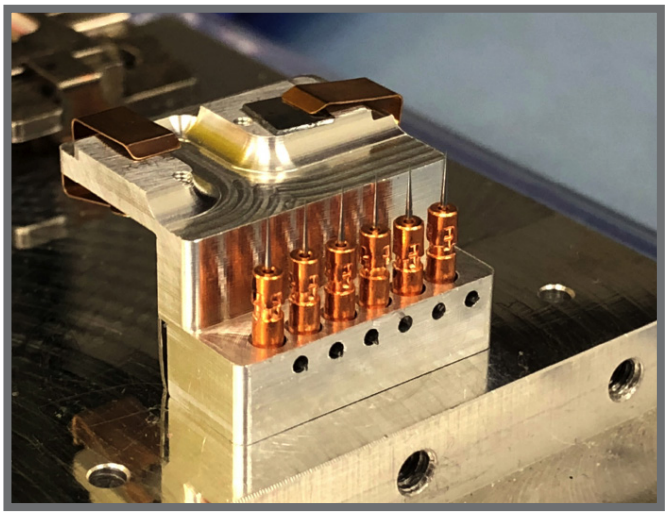


Fig.6: Vertical wire holder for FIB

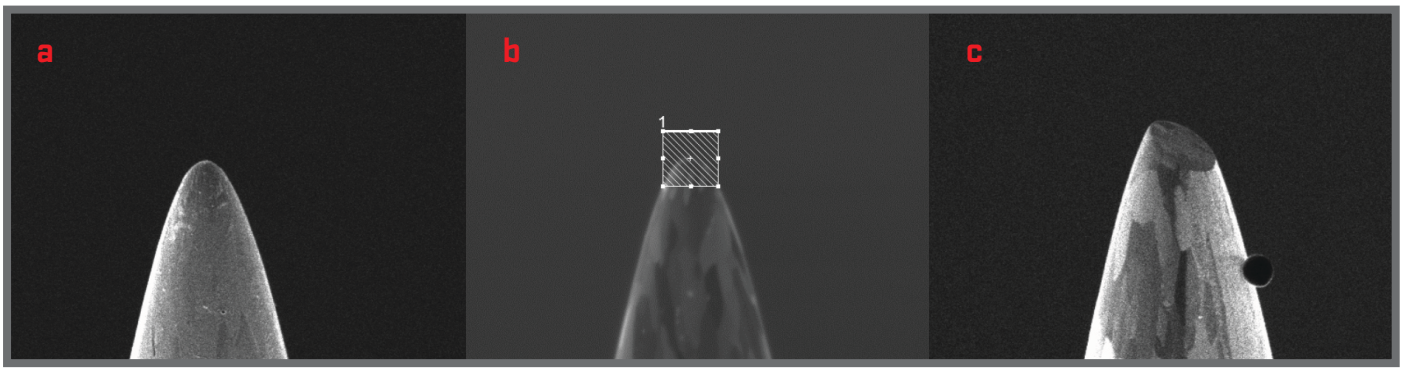


Fig. 7: (a),(b) wire specimen and cutting pattern at 0° stage tilt (c) wire rotated 90° to show angled mounting surface

STEP-BY-STEP PROCEDURE

4 Proceed with standard liftout process

After the wires are prepared by using method 1 or method 2, they are ready to be used as substrates for a standard FIB liftout. The detailed procedures for liftout and sharpening into an atom probe ready specimen are covered in TN-01 and TN-02. Use caution when moving the stage from wire to wire as they may be different heights. It is wise to retract both the micromanipulator needle and GIS before moving the stage and make sure you are at a safe eucentric position prior to reinsterting them. During the milling procedure, a particular consideration for wire substrates is to use a large enough outer diameter circle in the annular milling pattern to avoid extra peaks (Fig 10) that may emit and introduce noise during acquisition.

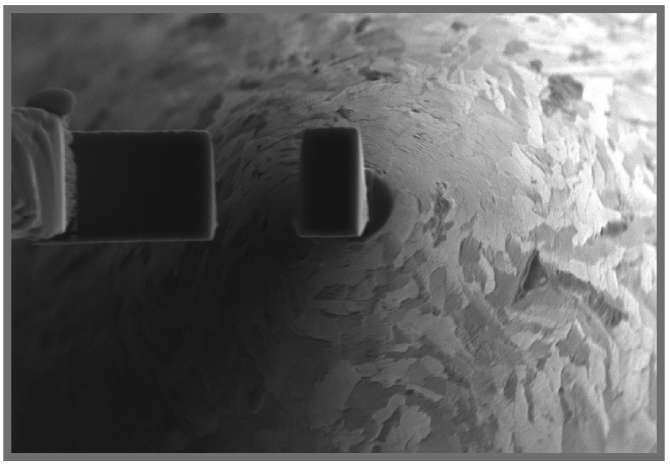


Fig.8: Sample mounting to prepared wire substrate

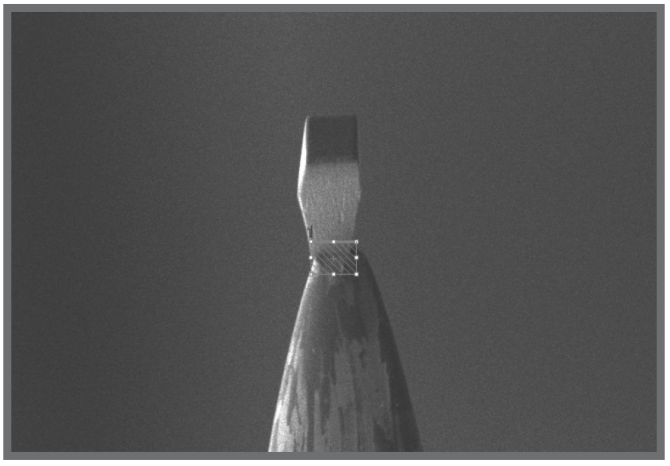


Fig.9: Sample mounting to prepared wire substrate

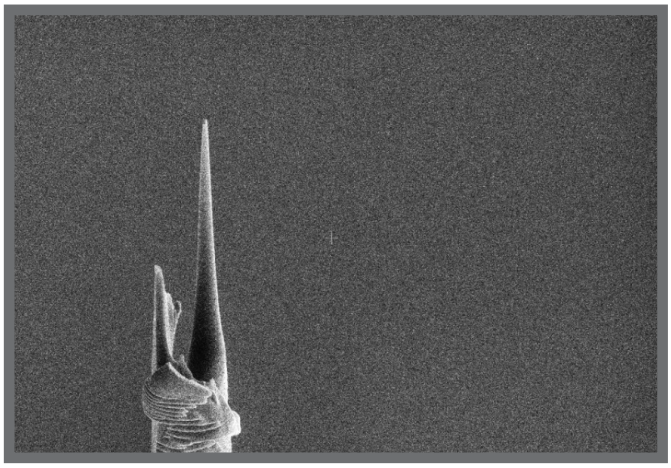


Fig. 10: Extra peak from FIB milling





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