Sample Repositioning Solutions for in situ Preparation and Analysis

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EXTENDING FIB & SEM CAPABILITIES

OUTLINE

- **TEM sample preparation challenges and solutions**
- **Atom probe challenges and solutions**
- **3D analysis challenges and solutions**

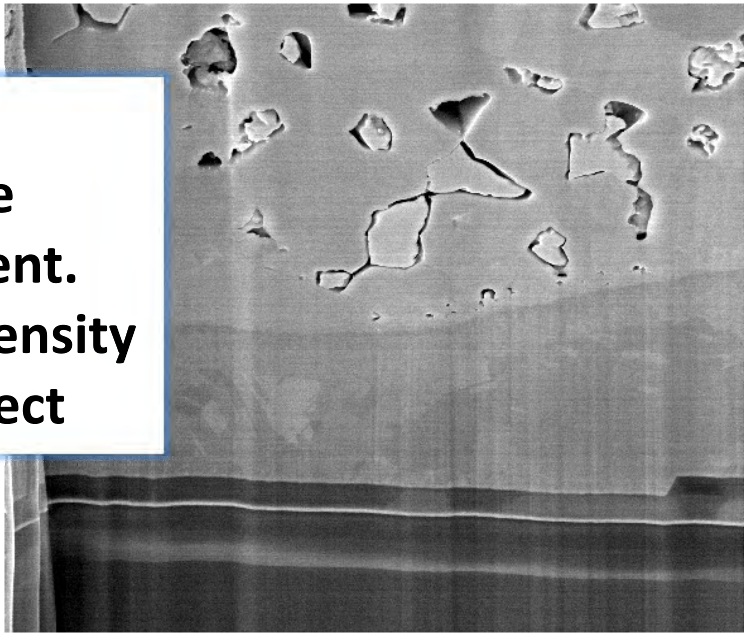
TEM SAMPLE PREPARATION

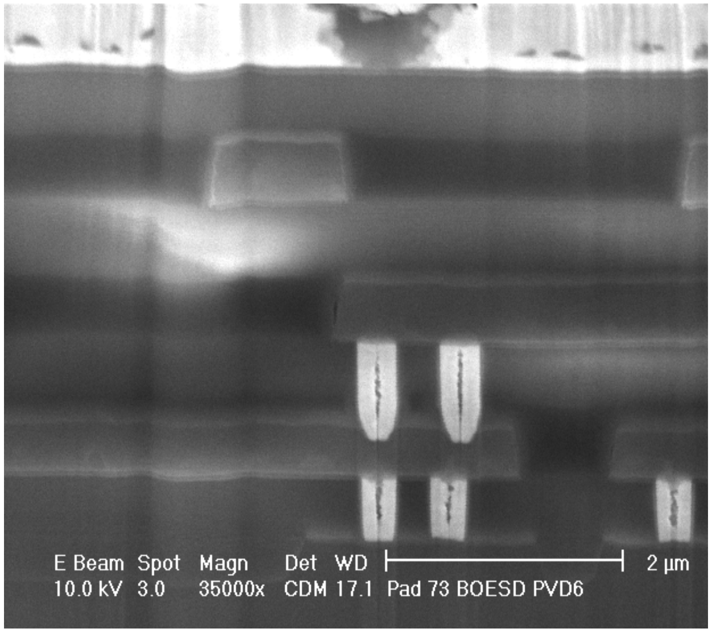
Challenges and Solutions

1. Smooth surfaces

Example: Flip chip bump and Au wire-bonded Al pad

Challenge:
- Milling rate
- Crystal orient.
- Material density
- Curtain Effect





Solution: Backside thinning

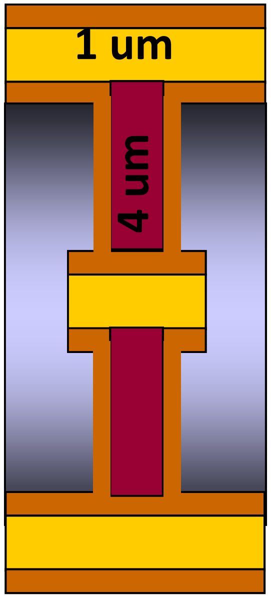
TEM SAMPLE PREPARATION

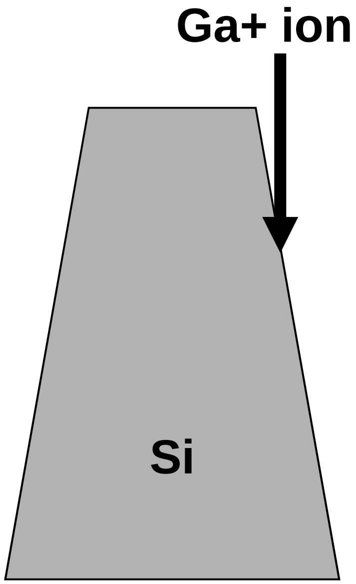
Challenges and Solutions

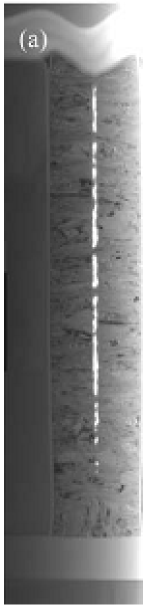
2. Constant lamellae thickness

Example: Tall and narrow semiconductor structures

Challenge:
- Curtain Effect
- Tilt Correction







Solution: Sideways thinning

R. B. Irwin *et al., JVST A* ***27, 1352 (2009)***

TEM SAMPLE PREPARATION

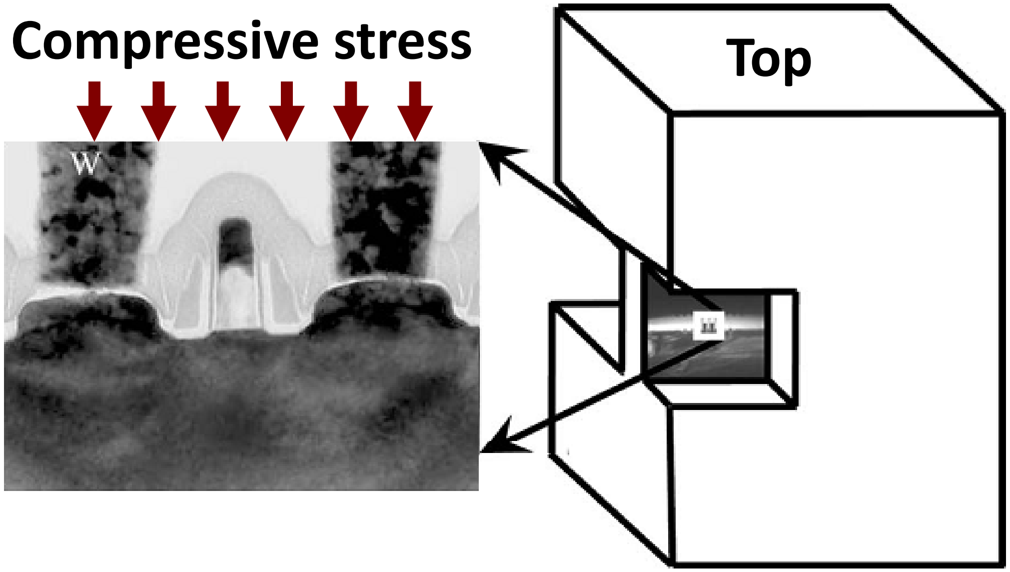
Challenges and Solutions

3. Conservation of stress

Example: PMOS transistor with epitaxial SiGe in the source/drain regions

Challenge:

- **TEM sample reflects native strain condition**



Solution: Sideways thinning

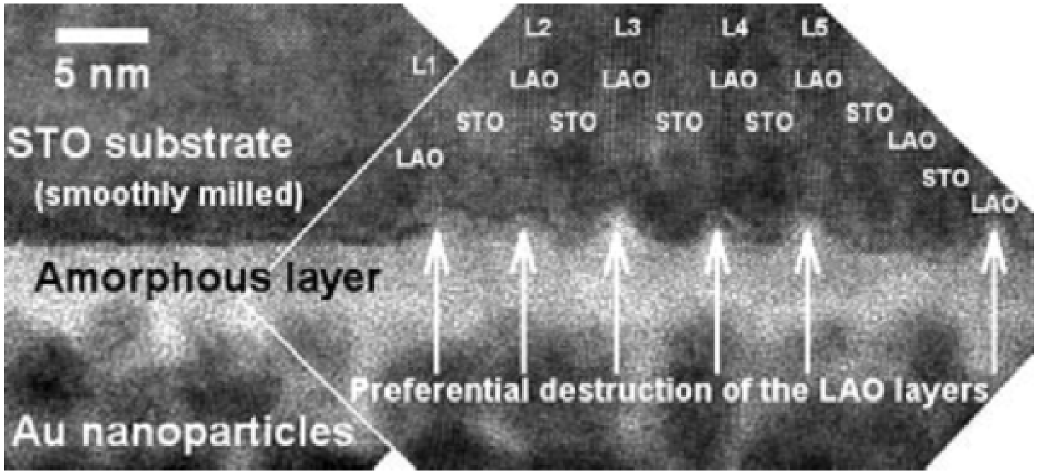
R. B. Irwin *et al., JVST A* ***27, 1352 (2009)***

4. FIB damage during cleaning

Example: Atomic stacking analysis of wide band gap perovskite insulators

Challenge:

- **5kV FIB cleaning**
- **Amorphization**
- **Curtain effect**
- **Available area for TEM**



LaAlO3/SrTiO3 (LAO/STO) multilayers

Solution: Angled thinning

E. Montoya et al., *Microsc. Res. Tech.* ***70, 1060 (2007)***

TEM SAMPLE PREPARATION

Challenges and Solutions

Results of FIB cleaning orientation on LAO/STO multilayer “performance”

Cleaning Orientation	Size of HRTEM Area	HRTEM	HAADF-STEM	Curtain effect	Preferential LaO destruction
Top	Extended	Good	Good	Strong	Observed
Top-angle	Extended	Good	Good	Weak	Observed
90° degree	Small	Good	Good	None	Observed
180°-angle	Small	Good	Good	None but uneven milling	Not observed

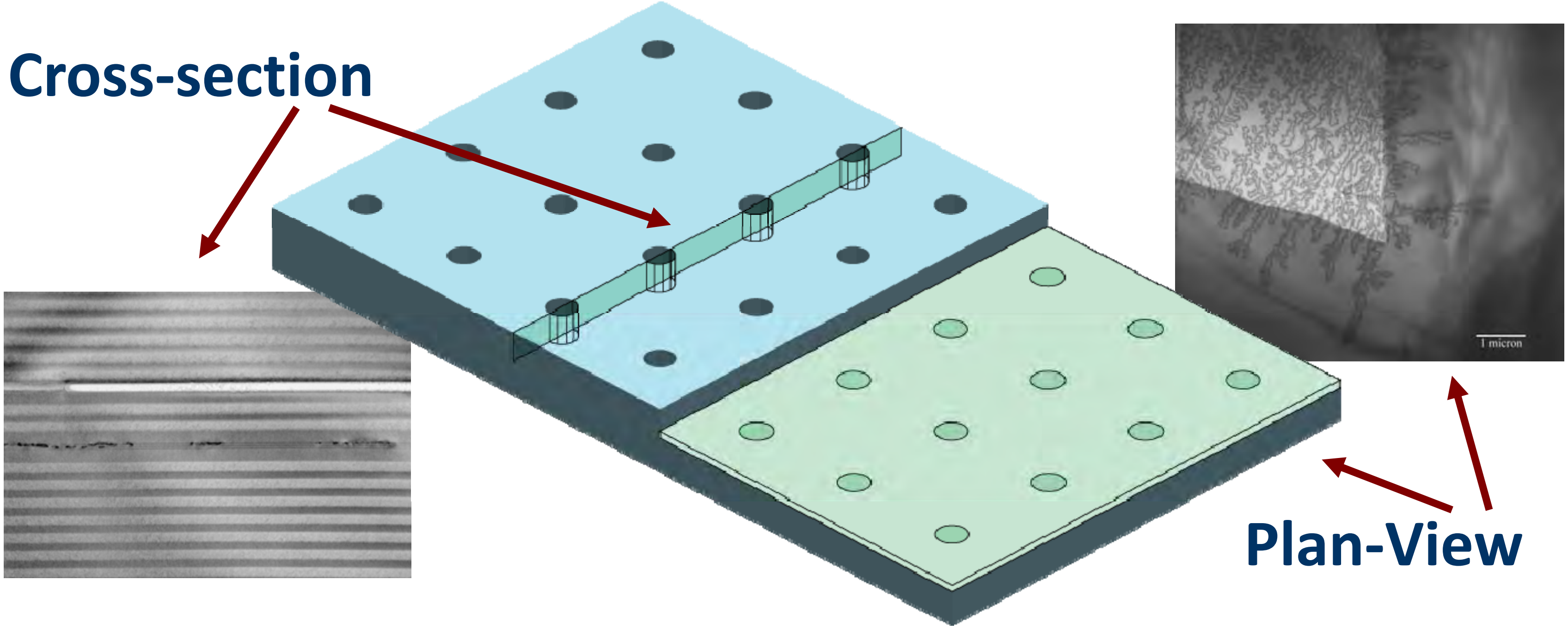
Solution: Depends on desired outcome

E. Montoya et al., *Microsc. Res. Tech.* ***70, 1060 (2007)***

TEM SAMPLE PREPARATION
Challenges and Solutions

5. Cross-section vs. Plan View

Example: Vertical Cavity Surface Emitting Lasers (VCSELS)



Solution: Frontside and planar thinning

TEM photos courtesy Phil Russell

TEM SAMPLE PREPARATION

Summary: Challenges and Solutions

CHALLENGE

1. Smooth Surfaces
2. Thickness Consistency
3. Conservation of Stress
4. FIB Cleaning Damage
5. Different Views

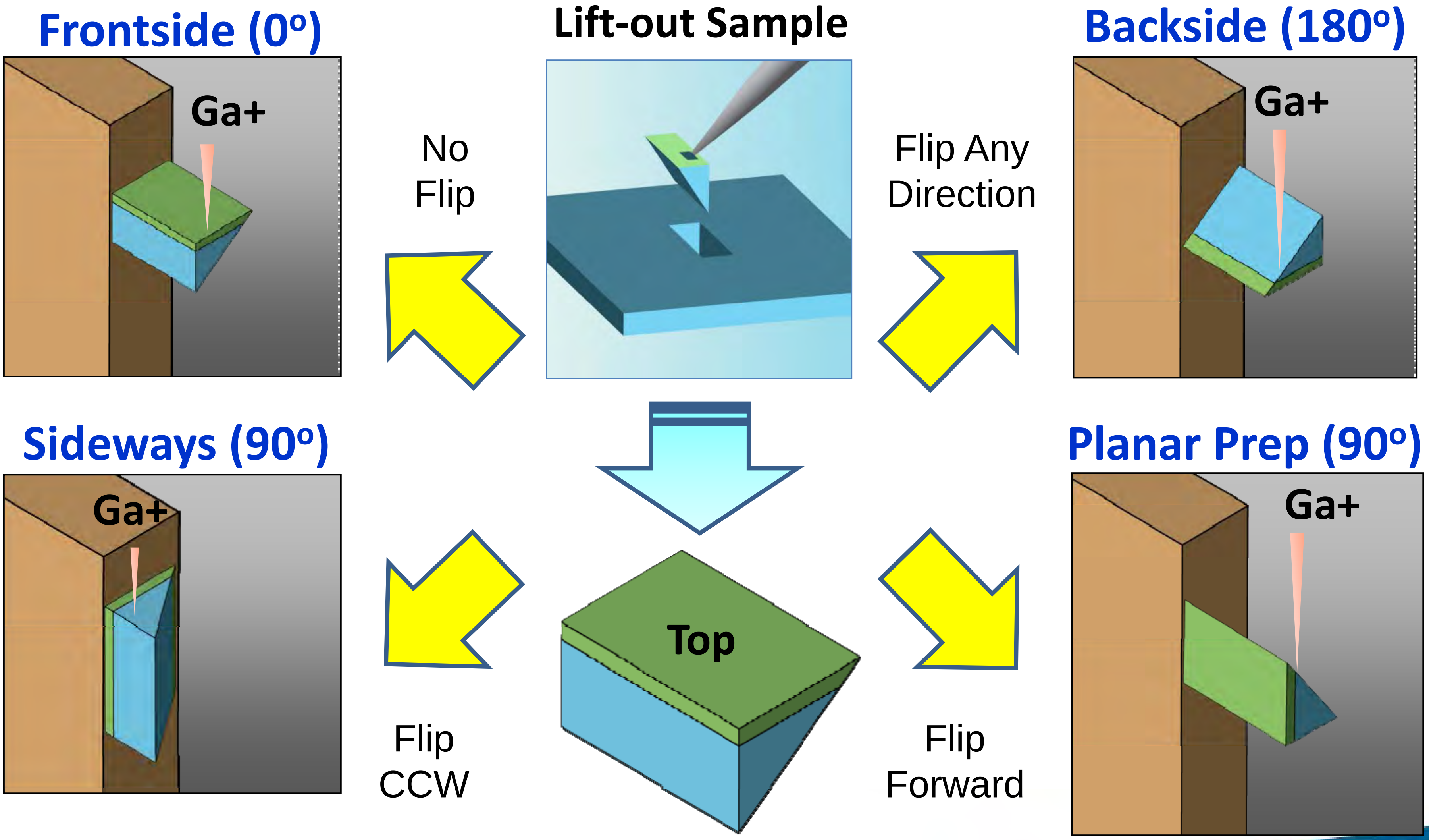
SOLUTION

Sample Repositioning

CHALLENGE:
How to reorient efficiently?

REPOSITIONING SOLUTIONS

INLO Sample Orientations



Frontside (0°)

Lift-out Sample

Backside (180°)

No Flip

Flip Any Direction

Sideways (90°)

Planar Prep (90°)

Flip CCW

Flip Forward

Movie

- Rotation alone does not provide desired repositioning

REPOSITIONING SOLUTIONS

Planar Preparation Strategies

Double Lift

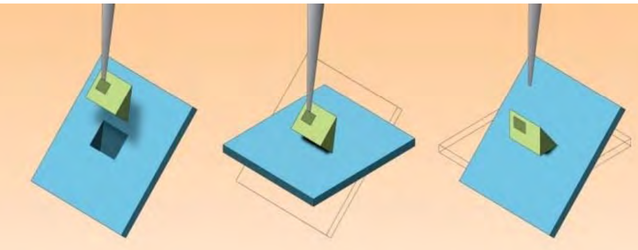
Lift sample at 45° tilt

Attach to substrate (0° tilt)

Re-lift sample at 45° tilt

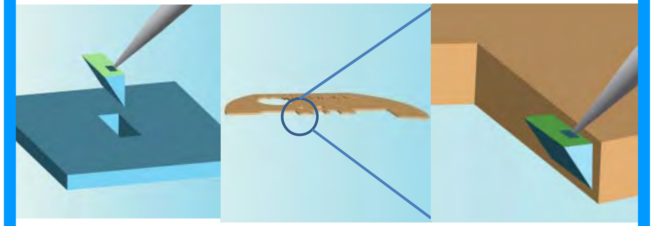
Attach to grid/holder

Final thinning

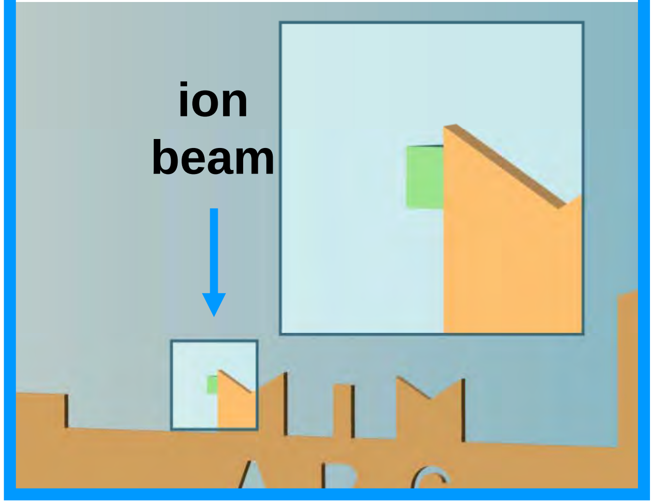


Grid Rotation

Attach cut face to flat grid



Rotate grid 90 degrees



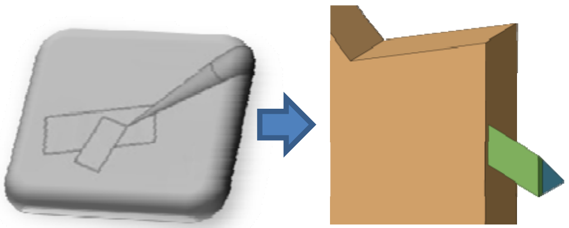
Stage-Shaft Rotation

Lift sample at specific stage tilt and rotation

Rotate probe shaft

Attach to aligned (rotated) grid

Final thinning



patent pending

Uses mathematical transformations based on conversion from angle-axis to rotation-matrix representation

REPOSITIONING SOLUTIONS

Planar Preparation Strategies

Shaft-Stage Rotation Method, Example

FIB 1, 50° port elevation over door

Lift sample at **52°** tilt and **17.11° CW** stage rotation

⇩

Rotate probe shaft **66° CCW**

⇩

Attach to grid rotated **47.2° CCW**, zero tilt

⇩

Final thinning

Movie

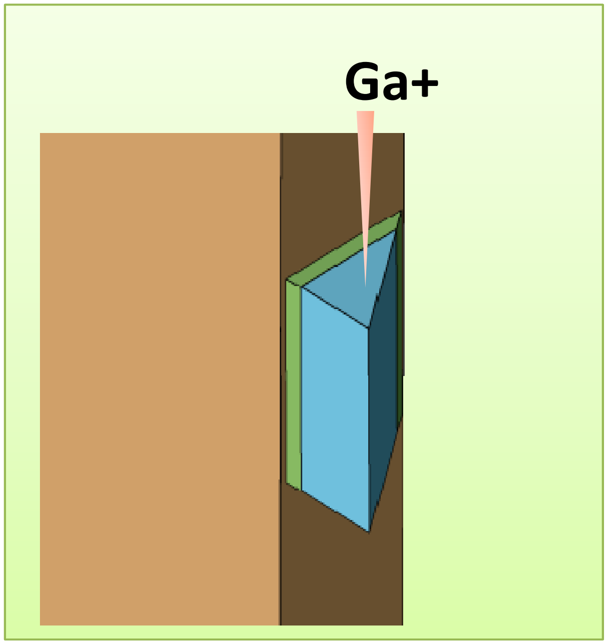
- Planar preparation using the stage-shaft rotation method

REPOSITIONING SOLUTIONS

90º (sideways) preparation

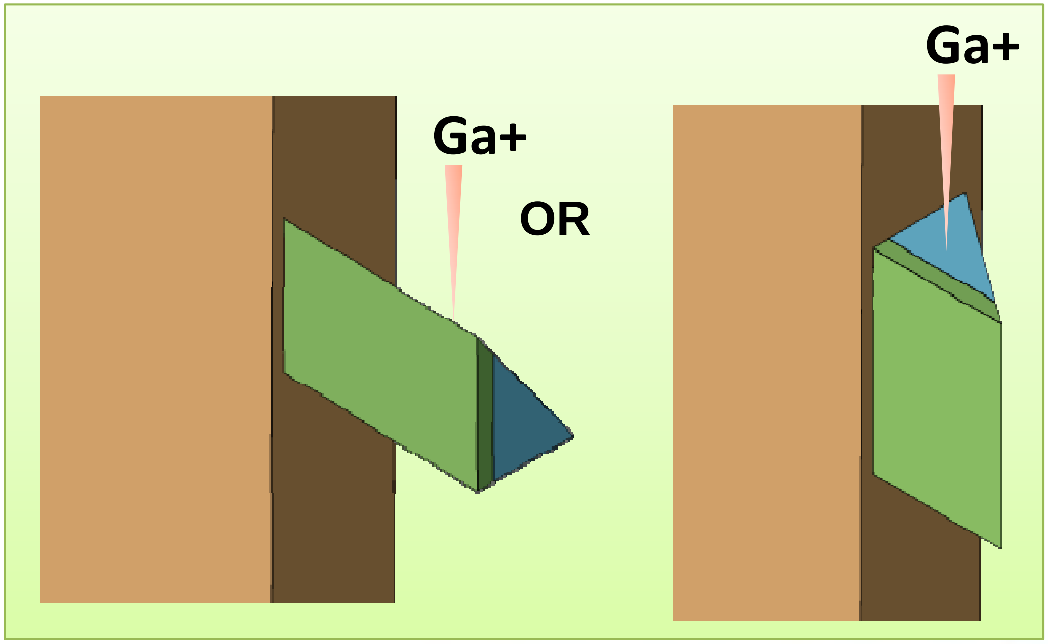
- The lift-out process is identical to planar prep
- Orientation when welded to grid is different

90º Preparation



VS

Planar Preparation



Movie

- Sideways (90^{o}) preparation using the stage-shaft rotation method

REPOSITIONING SOLUTIONS

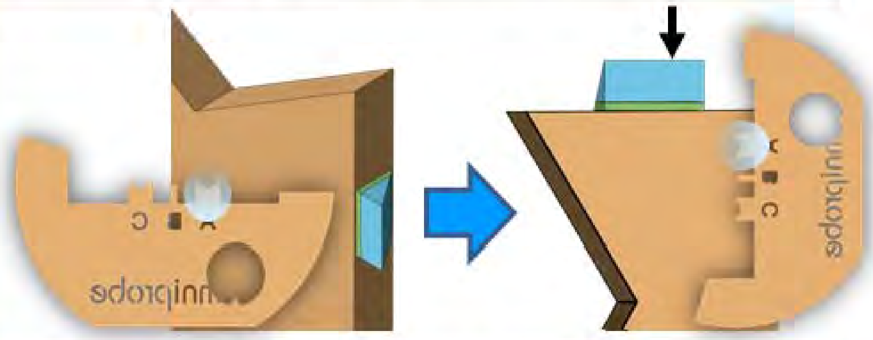
180° (backside) preparation

90° + 90° Grid Rotation

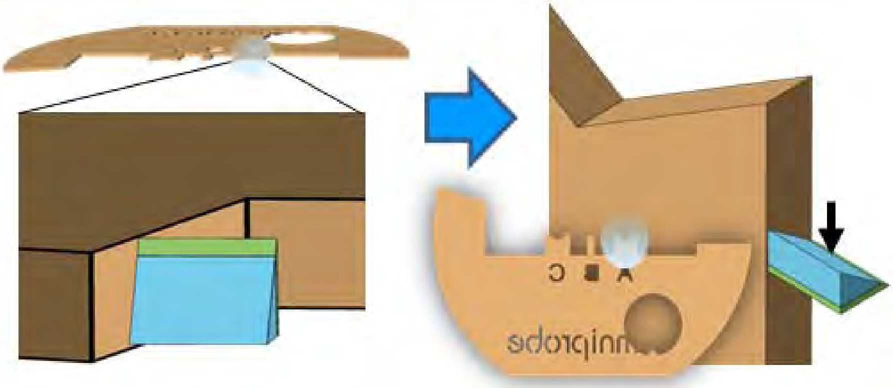
Attach using 90° solution of choice; rotate grid 90°

GRD-0001.07.01

1. "Sideways" attach vertically

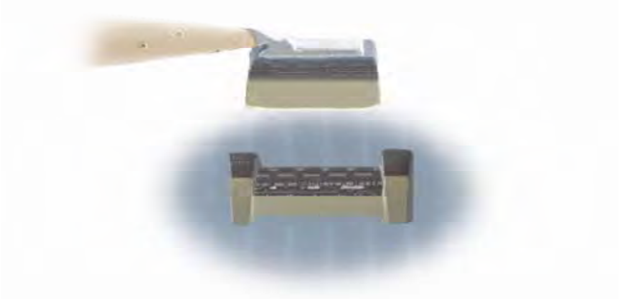


2. "Planar" attach to flat grid

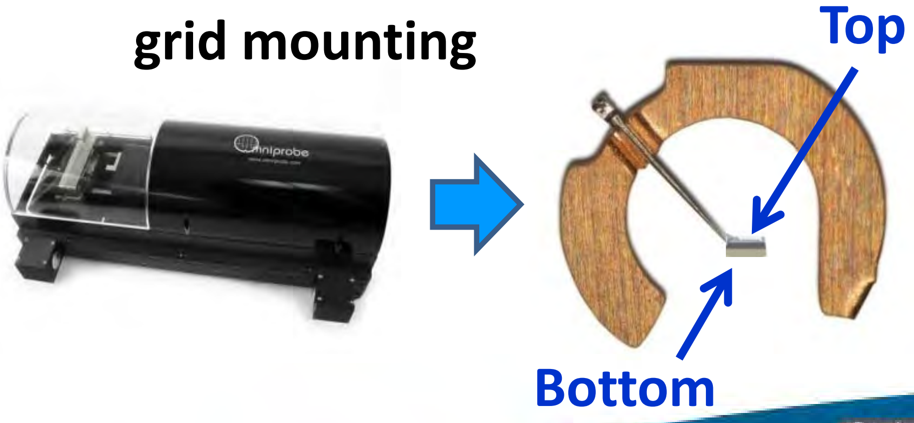


Short-Cut™

Perform standard lift-out.
Do NOT mount to support.



Short-Cut™ product for ex situ grid mounting

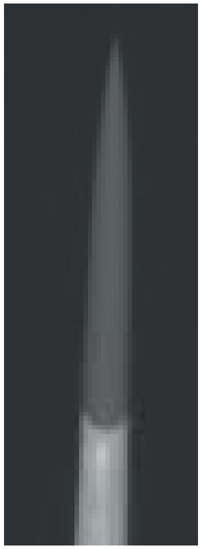


ATOM PROBE SAMPLE PREPARATION

Challenges and Solutions

Atom probe specimen geometry:

- Sharp needle
- Tip radius 50-150nm
- Modest taper <10deg semi-angle



Requirements and needs:

- Multiple samples for one result*
- Cross-correlation with TEM
- Backside preparation
- Minute samples: nanowires, particles
- Optimized sample prep*
    - Cap adhesion
    - Min. amorphization
    - No oxidation

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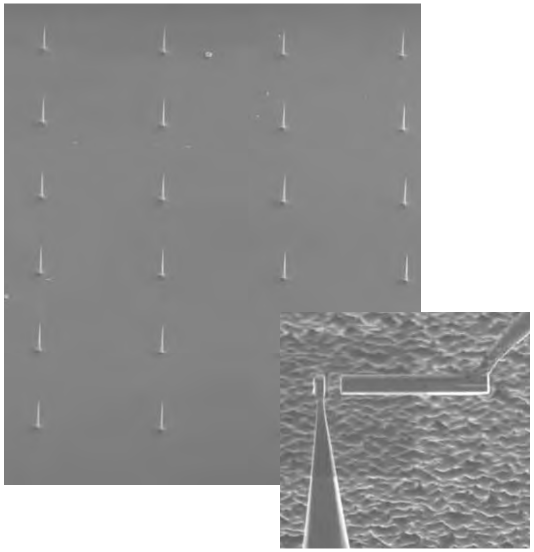
*Könnig et al., Ultramicroscopy 109 (2009) 486–491

Standard preparation methods:

- The needle IS the sample (metal electropolishing)
- Placement on post arrays and FIB shaping



Photos courtesy Brian Gorman/David Diercks, University of North Texas

ATOM PROBE SAMPLE PREPARATION

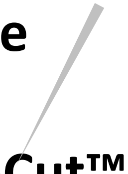
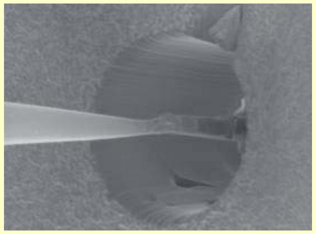
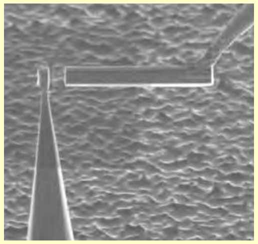
Correlative TEM & Atom Probe Analysis

Short-Cut™ Prep

Frontside Thinning

Backside Thinning

Tomography (LEAP, TEM)

Approach 1	Approach 2	Approach 3
1. In situ lift-out 	1. Short-Cut™ (blank tips) 	1. Needle IS the sample 
2. Short-Cut™ 	2. Lift-out + in situ attach 	2. Short-Cut™ 
3. FIB shaping	3. FIB shaping	
4. TEM - AP	4. TEM - AP	3. TEM - AP
 Site-specific Backside	 Many at once Back or frontside	No FIB

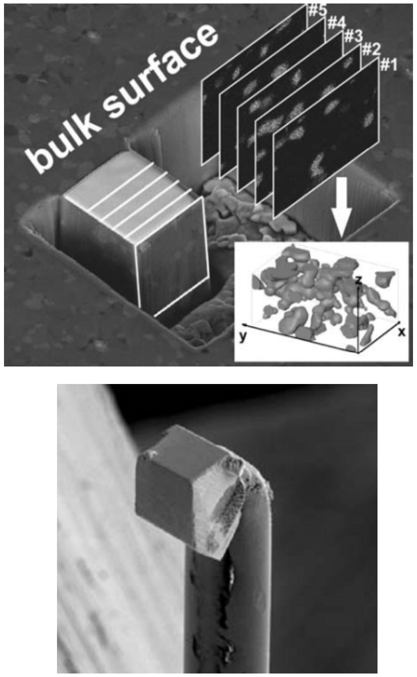
3D ANALYSIS

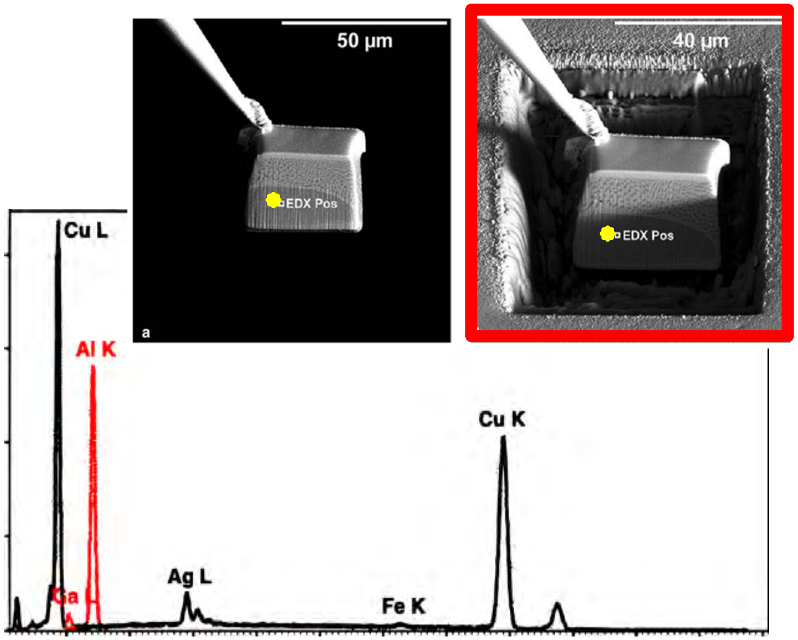
Challenges and Solutions

1. Automated EDX tomography

Example: Ferromagnetic specimen Cu/Ag solder joint

Challenge:
- Bulk effects
  - “shadow”
  - background
- Redeposition
- Detection efficiency





Solution: Block lift-out provides “substantial improvement”

M. Schaffer, J. Wagner, *Microchimica Acta* ***161, 421 (2008)***

CONCLUSIONS

Sample repositioning solves analysis challenges in beam-based microscopy, improving data and results quality.

Efficient solutions for sample repositioning have been presented.

Backup Slides

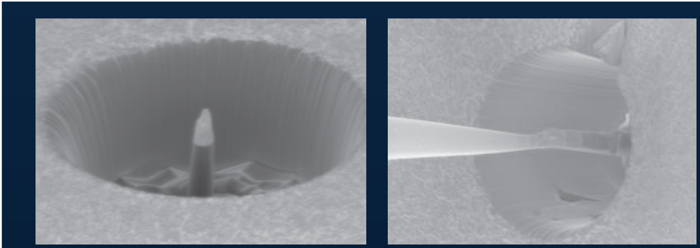
THE SHORT-CUT™: WHAT IS SPECIAL?

Rapid sample reorientation for FIB processing and conversion of unique samples to TEM compatibility

Frontside thinning

Backside thinning

Tomography
(LEAP, TEM)



A cylindrical sample is tilted and attached to a probe tip

Sample is cut free from substrate

Sample completes Short-Cut™ process

Final FIB milling from preferred direction

Tomography Sample

Gorman et al., Microscopy Today, Vol. 16, No. 4, July 2008, p. 42-47