TEM Sample Preparation
&
Optical Microscopes

Pablo Mendoza
Allied High Tech Products, Inc.

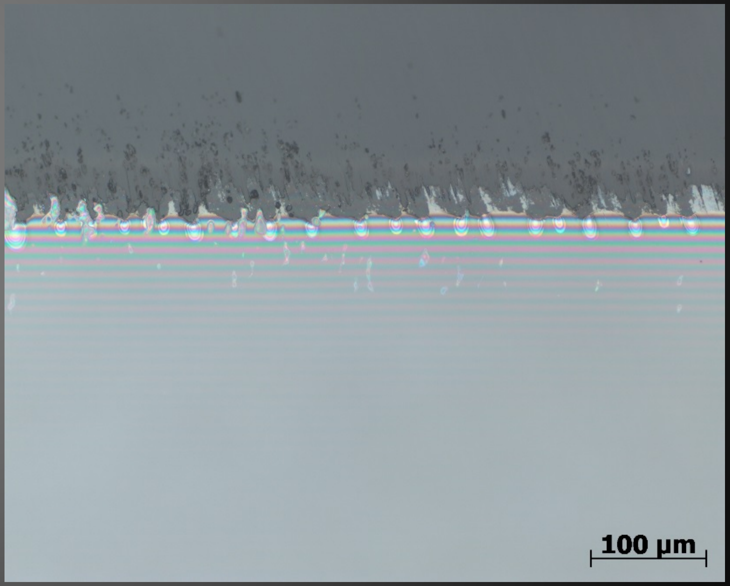
Introduction

- Materials Engineer
- Allied High Tech Products, Inc.
- 4 years of laboratory experience
- Primary responsibilities:
  - Procedure development for various materials
  - Image Analysis
  - Customer support/training

- Allied High Tech Products, Inc.
- Founded in 1983
- An American Manufacturer and distributor of high quality equipment and consumables for metallographic sample preparation and analysis.
- Zeiss Optical Microscope National Dealer
- Mitutoyo Rockwell and MHT Dealer

Topics

- Basics
  - What is a TEM sample?
  - Requirements?
  - Methods of preparing TEM samples?
- Mechanical preparation of a TEM sample
  - Preparation process
  - The role of optical microscopy in the sample preparation process
  - Stopping point: Pre-FIB thinning or TEM Wedge?



What is a TEM Sample?

TEM – Transmission electron microscopy is a microscope technique in which a beam of electrons is transmitted through a specimen to form an image.

TEM Sample – Specimen that is usually ultra thin (<100 nm) to allow electrons to transmit through.

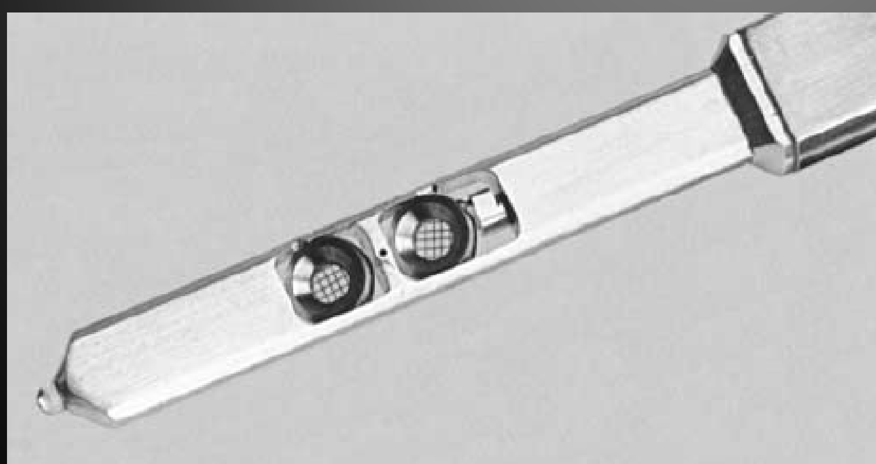
[3] Zeiss HRTEM

TEM Sample Requirements

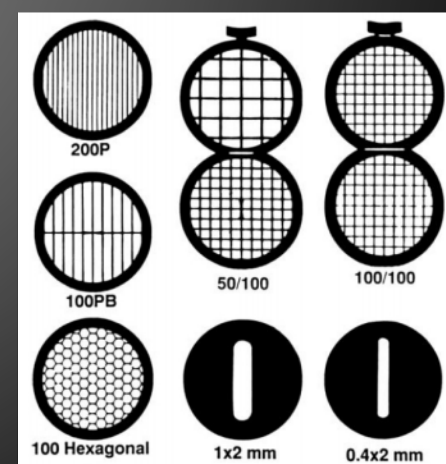
TEM samples must be electron transparent and generally the goal is a sample thickness of <100 nm

Any deformation from thinning process or previous processing must be removed

Small area, < 3 mm or less, to fit various TEM grids and holders



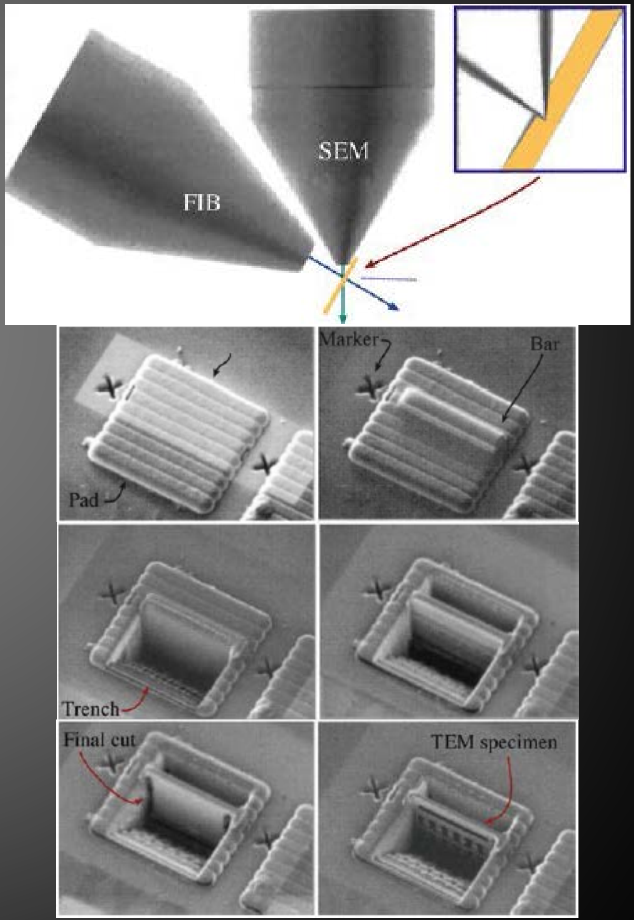
[3] Two-specimen holder with double-tilt



[3] Specimen support grids of different mesh size and shape

Common Methods of Preparation

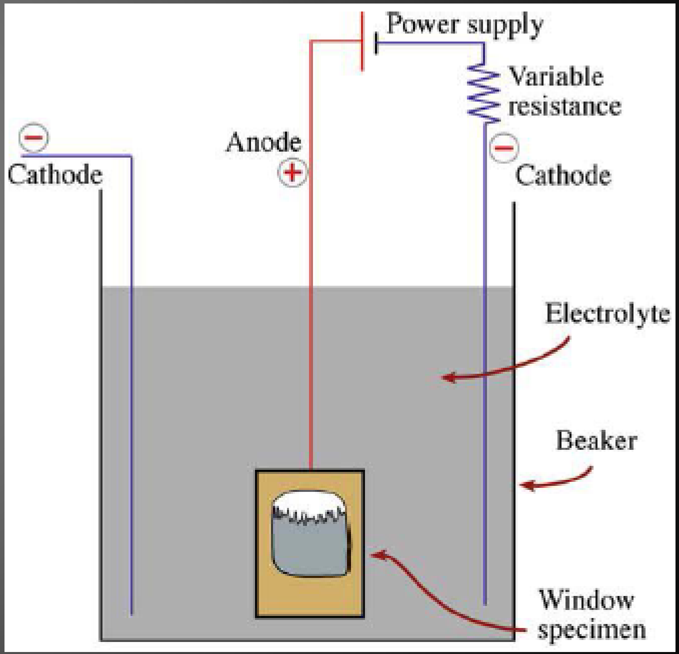
- Ion Milling/FIB – Focused Ion Beam
- Process that involves bombarding a TEM sample with energetic ions or neutral atoms and sputtering material from the sample until it is thin enough to study in the TEM [3].
- The most versatile thinning process that can be used for a wide variety of materials, however very expensive to initially invest in and to run.
- Disadvantage is implantation of source material and amorphous layer is created.
- Most commonly used in semiconductors.



[3] Schematic of a two-beam FIB and stages in making TEM samples using a FIB

Common Methods of Preparation

- Electropolishing
- Sample is immersed in an electrolyte and then subjected to a direct electrical current. The sample is maintained anodic, with the cathodic connection being made to a nearby metal conductor. [2]
- Anodic dissolution of sample creates polished surface.
- Relatively quick and can produce samples with no mechanical damage, however can only be used for electrically conducting metals and alloys.



[3] The Window Method

Common Methods of Preparation

- Mechanical Preparation: Can be performed by hand or with the aid of a machine
- Manual: Utilizes hand tools (Tripods) that allow the user to make angle adjustments
- Semi-Automatic: Precision polishers, such as the MultiPrep™ System, with digital indicators and dual micrometers allow:
  - Monitor amount of material removed in real time in 1-micron increments
  - Precise sample tilt adjustments relative to the abrasive plane. Alignment ensures controlled angles.

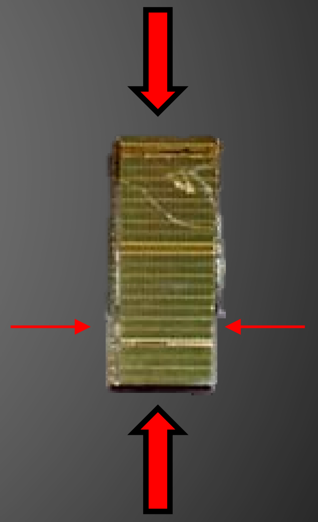
Mechanical Preparation

- Advantages
  - TEM Wedge capabilities
  - All damage removed using the 3x rule
  - No foreign material implanted
  - With experience it is repeatable and fast
  - Lower cost of required equipment
  - Decrease milling time and costs
- Disadvantages
  - Requires careful handling throughout process
  - Greater learning curve than others techniques
  - Some samples may require one of the other TEM preparation methods such as metals due to internal strain

Preparation

- Area of interest needs to be identified; site-specific or bulk?
- Once determined, sample needs to be prepared
- Preparing TEM samples is essentially performing two cross-sections

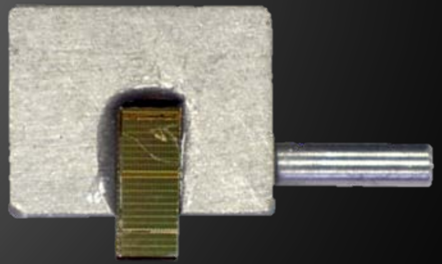
Second side
cross-section



First side
cross-section

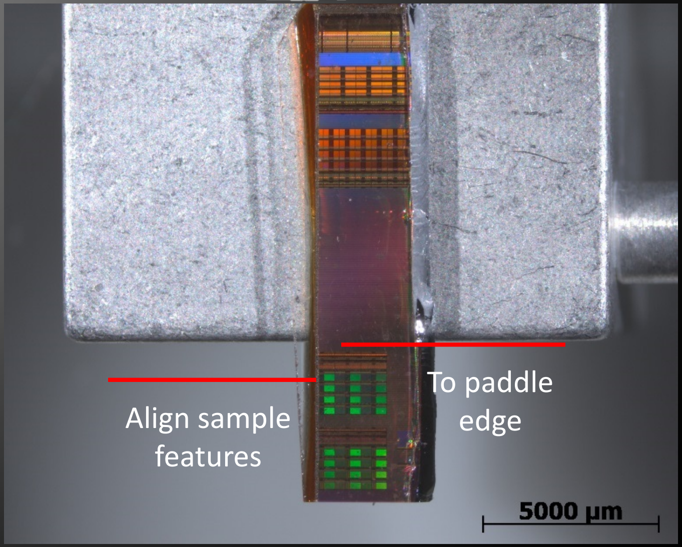
Thinning First Side

- Sample placed onto a cross-sectioning paddle using mounting wax
- Paddle placed on hot plate to melt wax
- Light pressure can be applied using a cotton-tipped applicator to assist in parallel registration of the sample to the fixture.



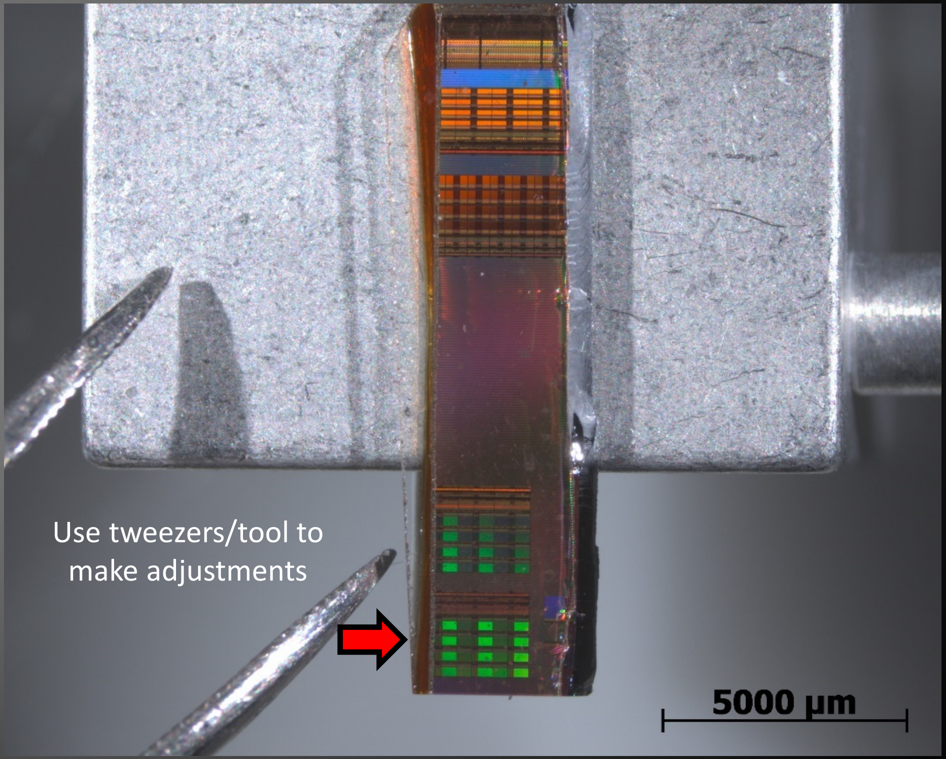
Thinning First Side

- Stereomicroscope can greatly assist in aligning the sample to the fixture
- Tools such as the MultiPrep™ may have methods of correcting for the angle; however, using a stereomicroscope can minimize the amount of corrections required



Thinning First Side

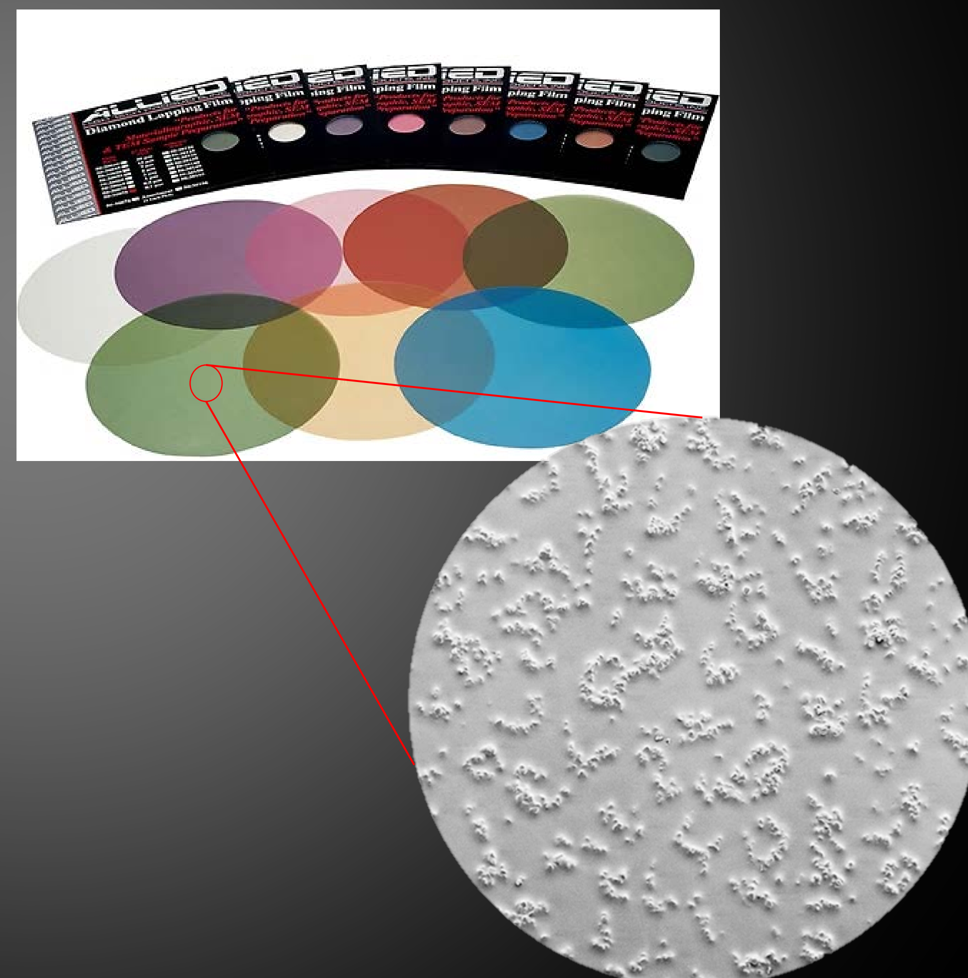
- While the paddle is hot, it can be taken to a stereomicroscope
- This allows for sample adjustments before the wax cools and hardens



Initial adjustments under microscope

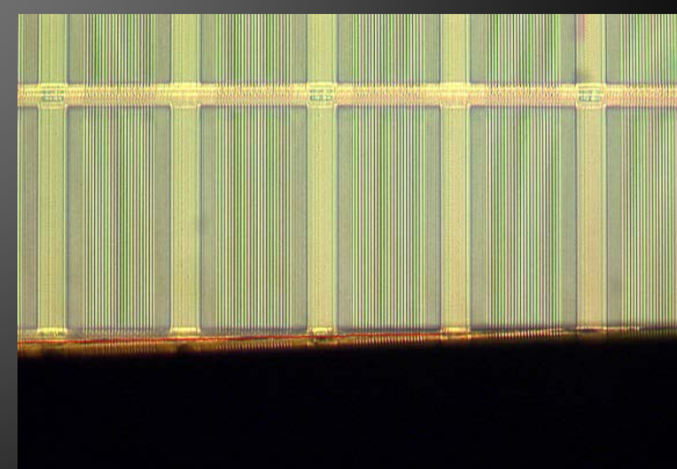
Thinning First Side

- Lapping films used as abrasive
  - Provide excellent edge retention and maintain coplanarity
  - Typically used for unencapsulated cross-sectioning, TEM preparation, backside polishing, etc.



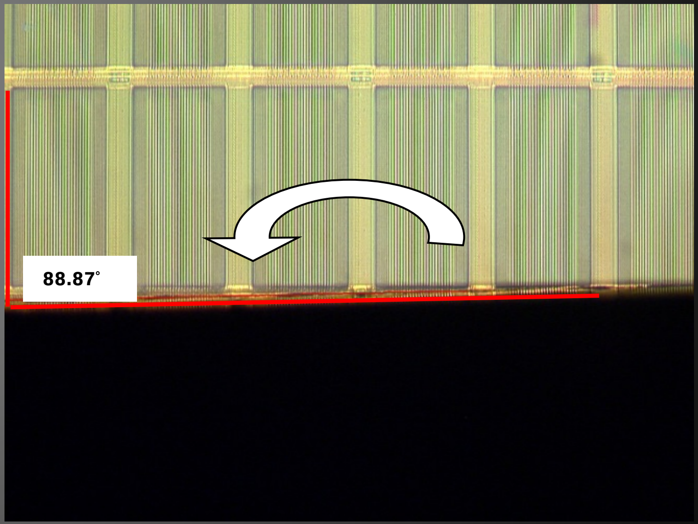
Thinning First Side

- Initial grinding with a coarse lapping film, such as 30 µm, will give a ground edge
- This ground edge can be used to obtain angle information using optical microscopy



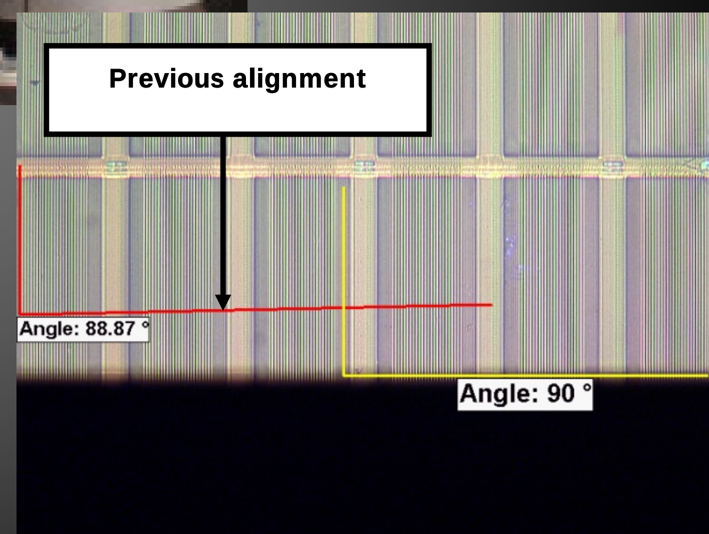
Thinning First Side

- Compound light microscopes will give the resolution and details needed to perform corrections
- Imaging software can be used to obtain angle measurements



Thinning First Side

- Angle corrections can be made using the micrometers
- Each tick mark is 0.02°
- More material can be removed to obtain a new ground edge
- With the new ground edge, the process can be repeated until an angle correction is no longer necessary



Corrected alignment

Target: Site-Specific or Bulk?

Site-Specific

- Particular feature on sample
- Failure Analysis

Must stop at target, without damage from previous steps

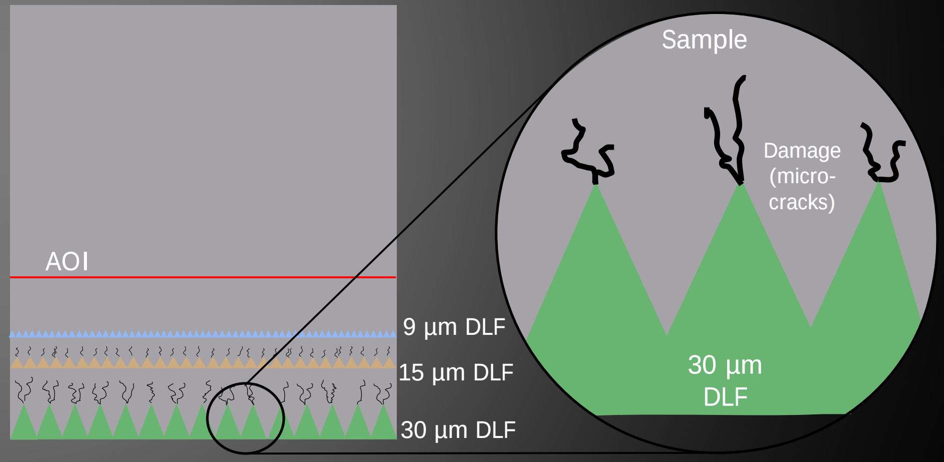
Bulk

- Homogeneous material

Can stop anywhere, however ensure no damage from previous steps

3X Rule

- Damage is introduced into a sample throughout the preparation process.
- This damage is not only represented by a scratch pattern on the surface, but also by smaller micro-cracks that can propagate further into the sample that may not be seen by eye or optical microscopes (A 30 µm lapping film will **not** cause **only** 30 µm deep scratches).



3X Rule

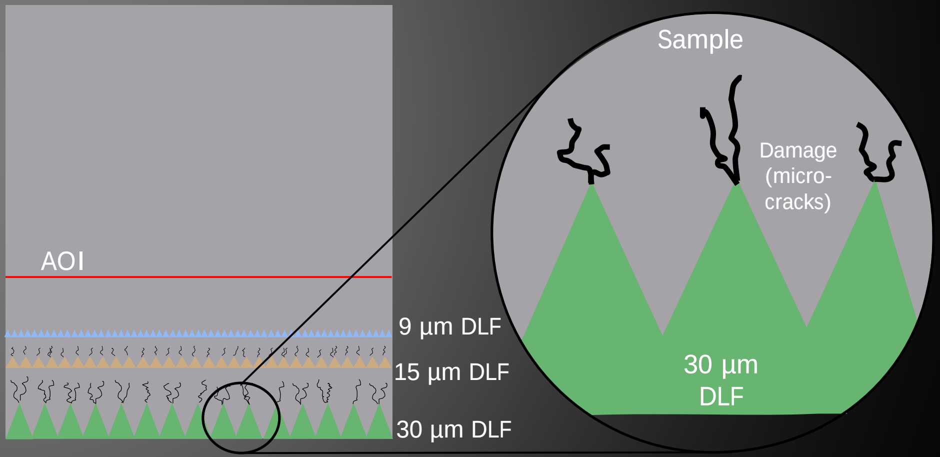
- If a 30 µm lapping film is used and it is followed by a 15 µm lapping film step, the 15 µm DLF must remove at least 90 µm of material to completely remove the damage from the 30 µm DLF. Similarly, if a 30 µm DLF is used and it is followed by a 6 µm DLF, then the 6 µm DLF must remove at least 90 µm of material to completely remove the remaining damage.

3X Rule				
Current Step (DLF)	Current Distance from Target	Previous Step	Remove At Least 3x Prev. Step Size	Distance to Target After Current Step
30 µm	Vary	N/A	N/A	192 µm
15 µm	192 µm	30 µm	90 µm	102 µm
9 µm	102 µm	15 µm	45 µm	57 µm
6 µm	57 µm	9 µm	27 µm	30 µm
3 µm	30 µm	6 µm	18 µm	12 µm
1 µm	12 µm	3 µm	9 µm	3µm
0.5 µm	3 µm	1 µm	3 µm	At Target

3X Rule

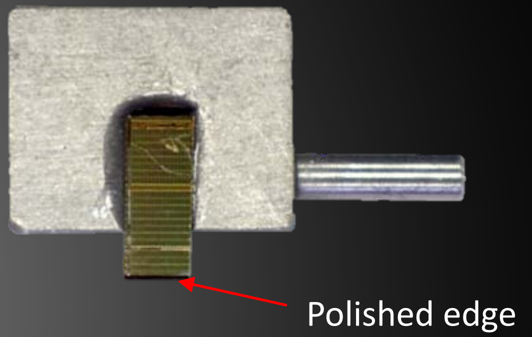
- Other materials, such as ductile steels, may only require a "2X Rule", while fragile, brittle ceramics, where cracks can propagate further into the sample, may require a "4X Rule".

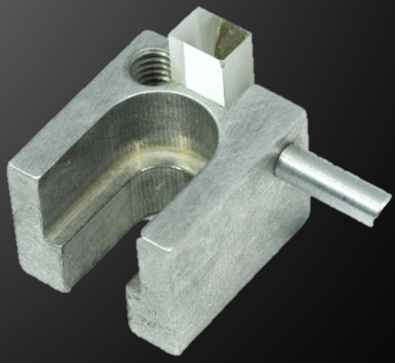
- The main concept to understand is that no matter what steps follow, all damage introduced by the previous abrasive must be removed to get an accurate representation on the microstructure.



Flipping Sample

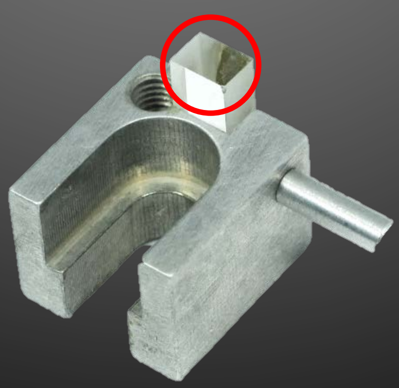
- 2nd side needs to be prepared to thin sample
- Previous method using cross-sectioning paddle can't be used; can't overhang sample properly
- TEM paddles with Pyrex® inserts are used to continue with the 2nd side



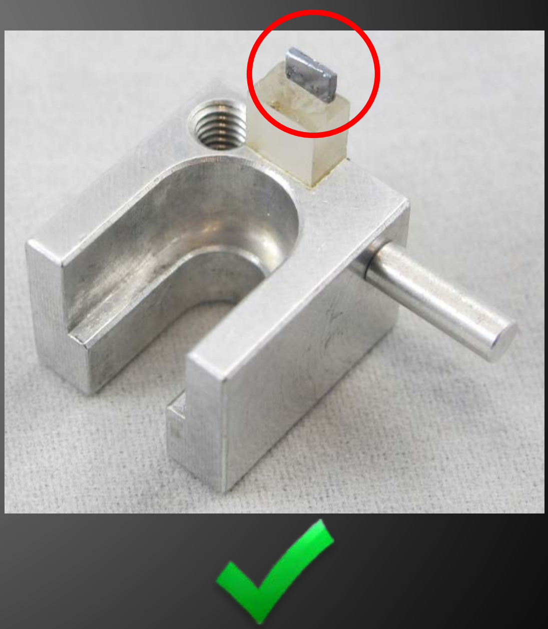


Flipping Sample

- Surface of Pyrex® requires grinding
  - Grinding the surface of the Pyrex® will align it with that of the platen, ensuring parallelism
- Pyrex® can be ground using diamond lapping films
  - 6-3 µm finish is good enough
  - Do not want to polish the surface as having some scratches will help with adhesion

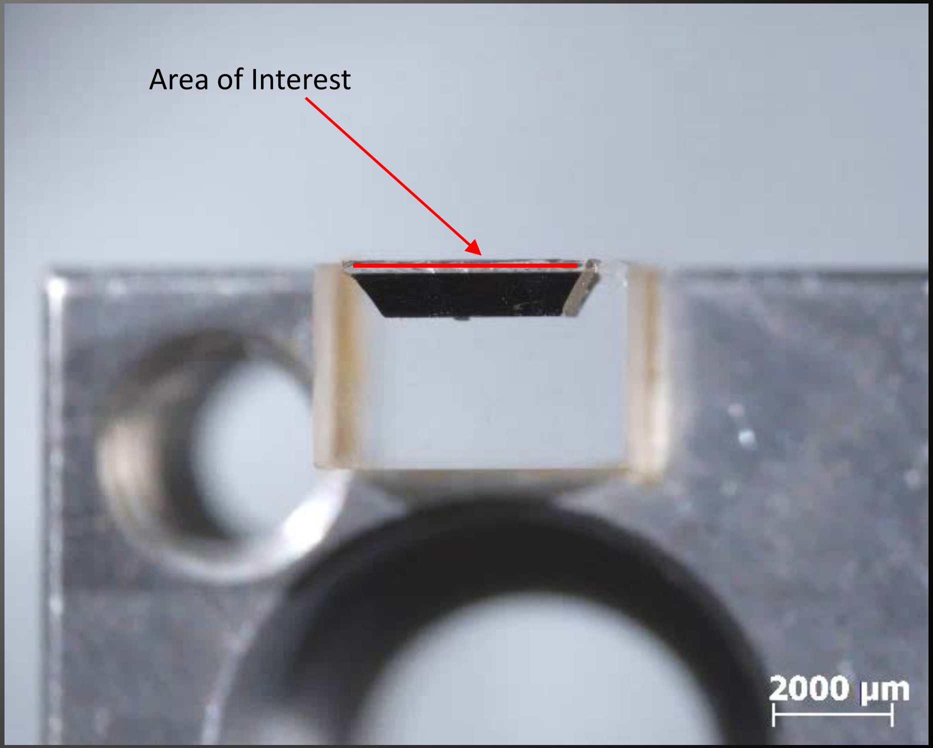


Site-Specific Orientation

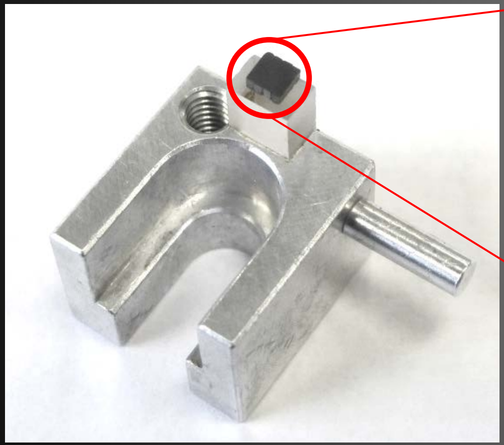


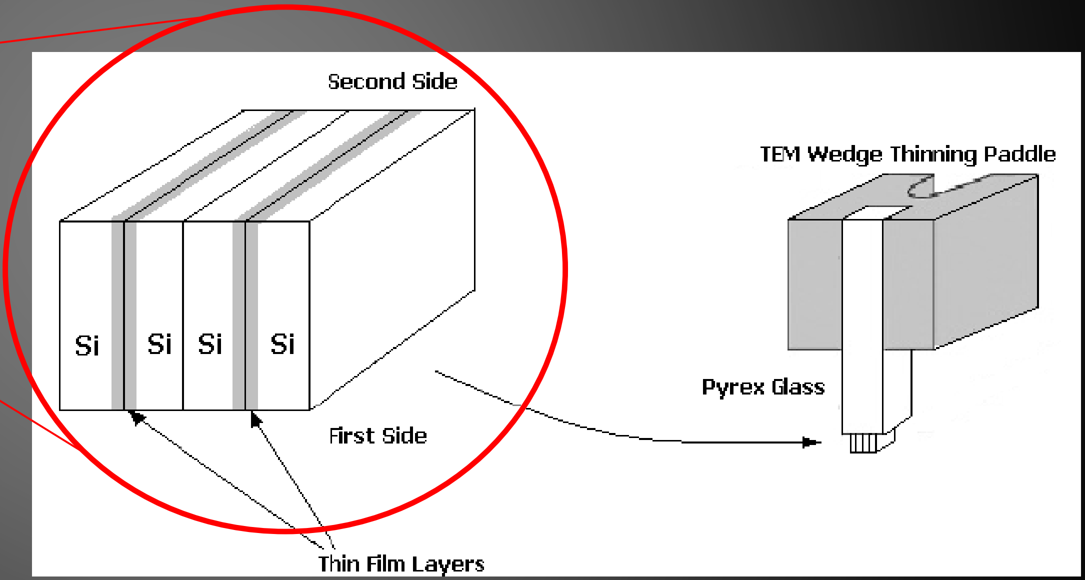
Site-Specific Orientation

- With site-specific orientation, the area of interest is parallel to the Pyrex® edge



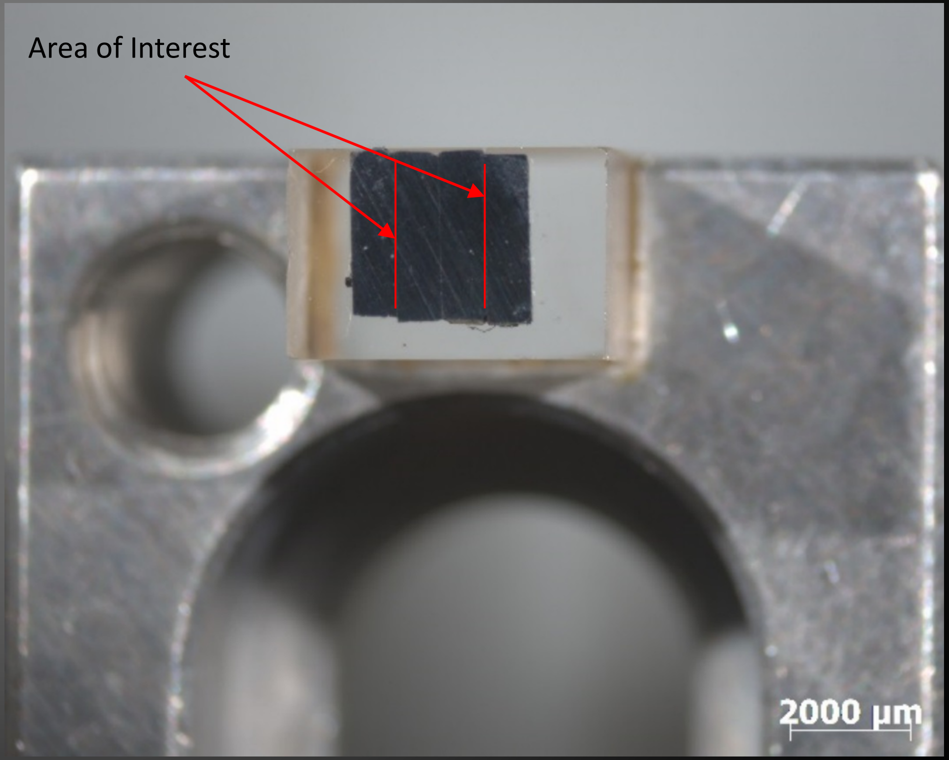
Bulk Orientation





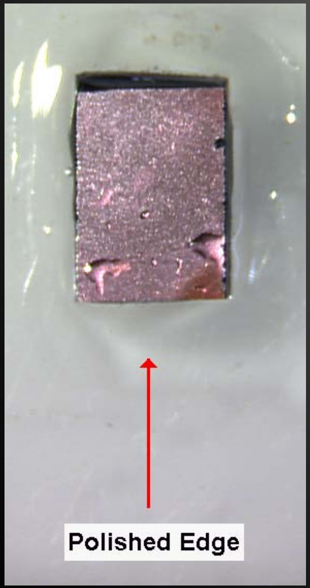
Bulk Orientation

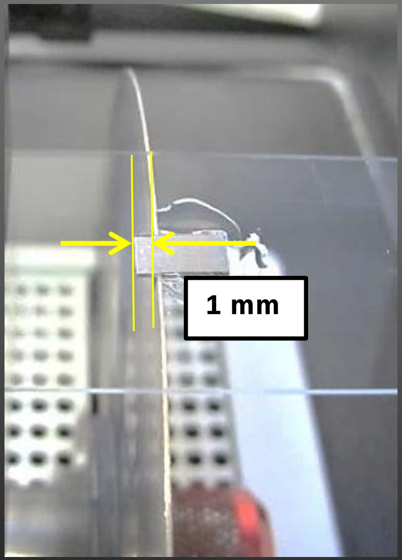
- With bulk orientation, the area of interest is perpendicular to the Pyrex® edge

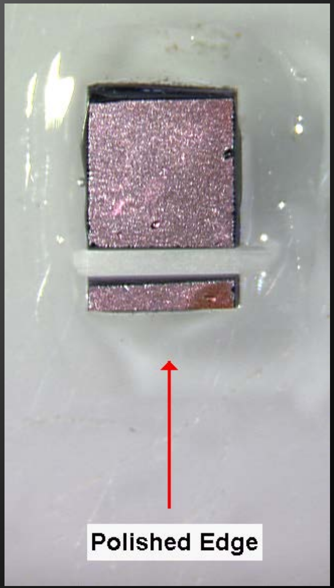


Flipping Sample

- Sample waxed onto a glass slide
- ~1 mm thick piece sectioned from sample
  - This allows placement onto TEM paddle without risk of the sample breaking off due to being too tall
- Sample should be cleaned to remove abrasive particulates, excess wax, etc.

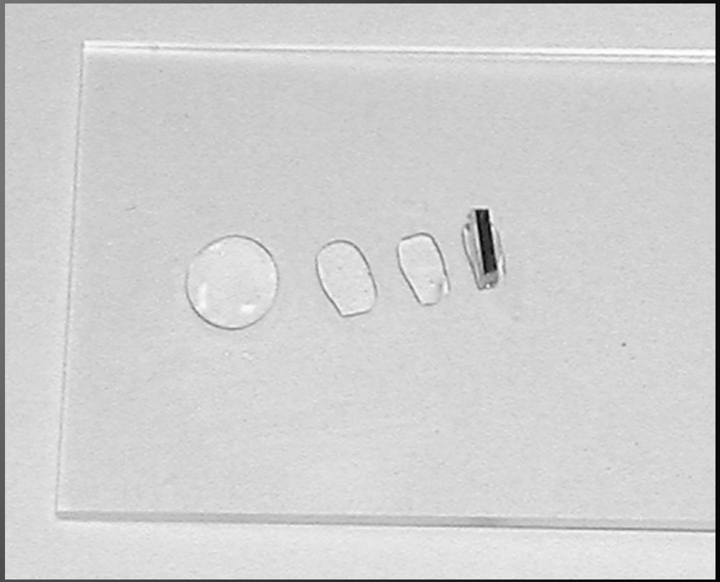






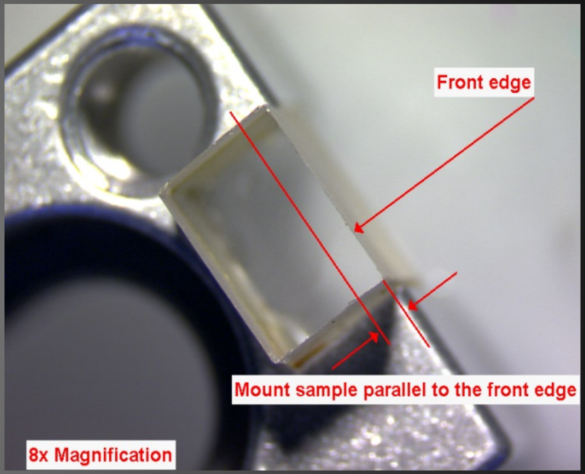
Flipping Sample

- To ensure stability, a thin, uniform layer of wax needs to be used.
- Wax Bead Method
  - Place sample, polished side down, into wax bead
  - Pick up sample and move over to empty part of glass slide
  - Allow wax to wick away
  - Repeat process 2-3 more times to end up with thin wax layer



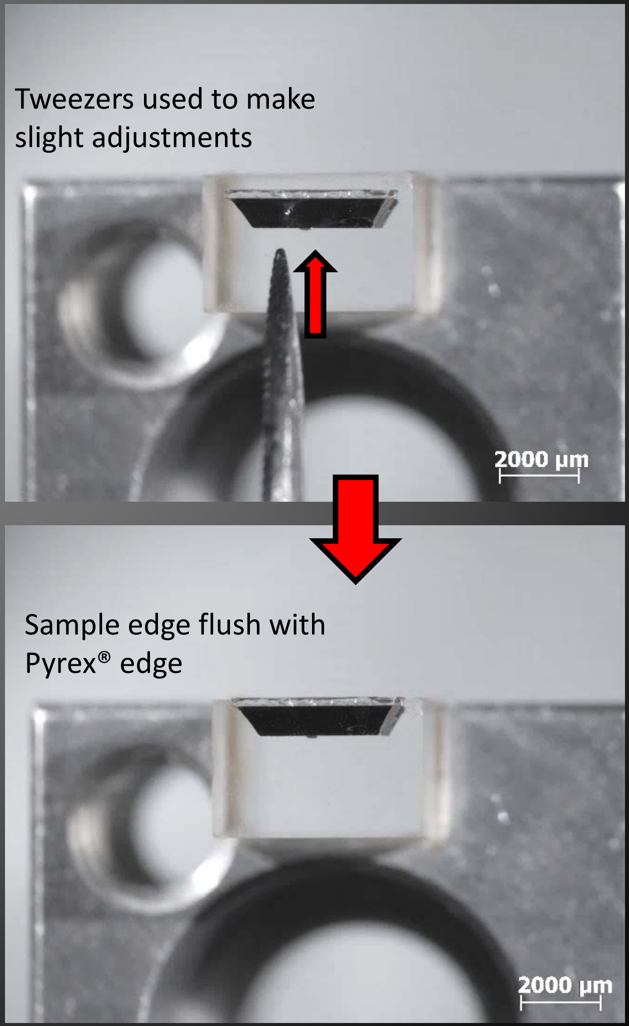
Flipping Sample

- Polished side placed face down onto the Pyrex®
- Positioned near the edge to allow easier thickness measurements later



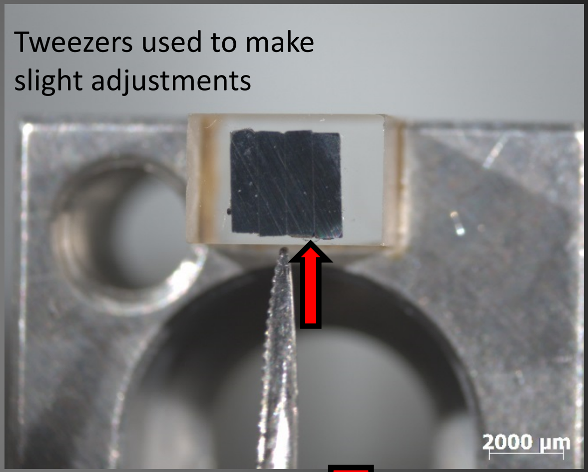
Flipping Sample

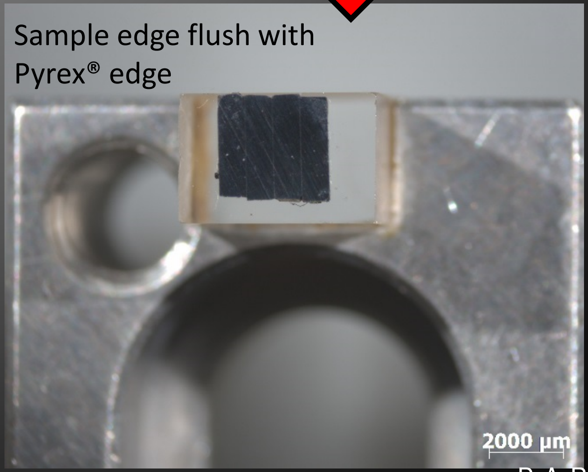
- Stereomicroscope is great for aligning to the edge of the Pyrex®



Flipping Sample

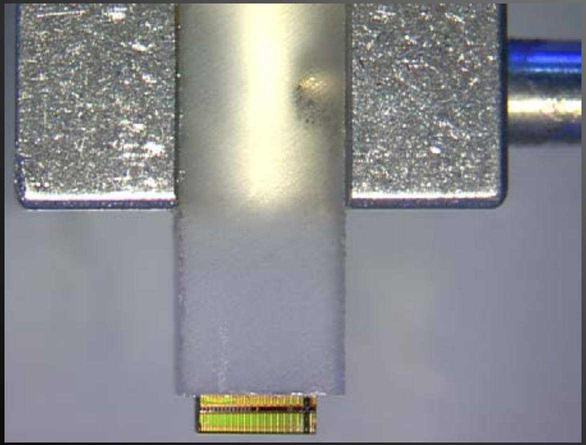
- Stereomicroscope is great for aligning to the edge of the Pyrex®

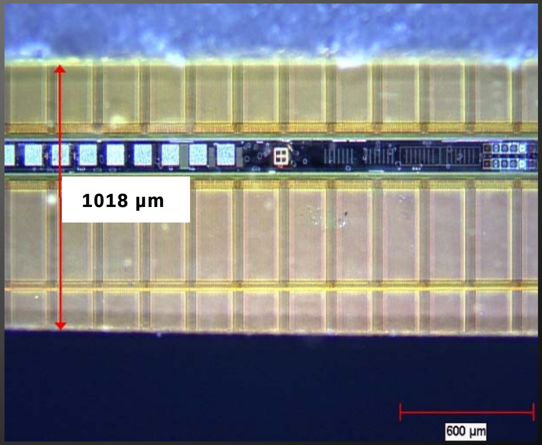




Second Side

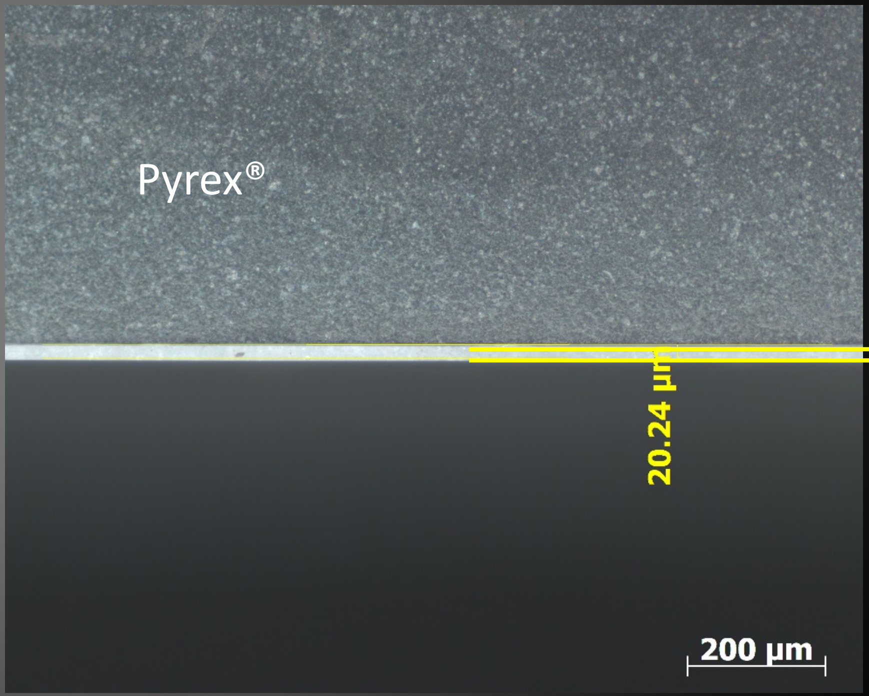
- Second side, whether site specific or not, now has a final target
  - Can lose sample if thin/polish for too long
- Checking sample thickness becomes extremely important if you want to remove all the damage from previous steps
- Certain machines, such as the MultiPrep™ System, may have indicators or other methods to track material removal
  - However, checking progress frequently with an optical microscope is a good way to verify material being removed: trust but verify





Second Side

- Mounting the sample close to the edge allows for proper focusing on the sample to measure side 1 edge
- Compound scope can give required resolution to view sample thickness and make measurements



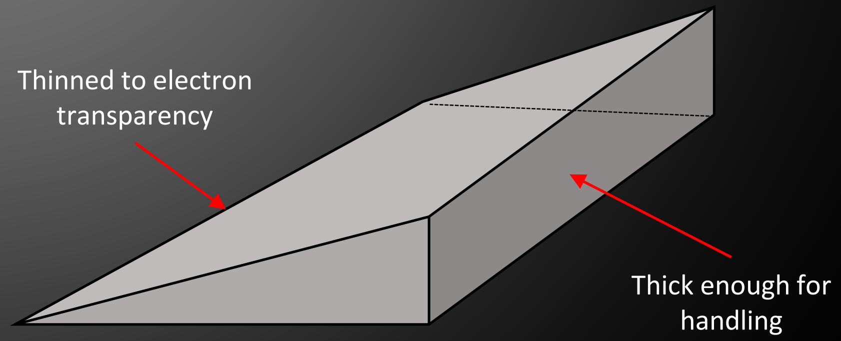
Goal: Pre-FIB or TEM Wedge?

Pre-FIB Thinning

- Site-Specific Orientation
- Remove the sample to prepare for FIB thinning
- Sample can be removed by placing paddle into container with acetone (wax dissolves in acetone)
- Filter paper placed around paddle so sample will eventually pop-off and land on paper

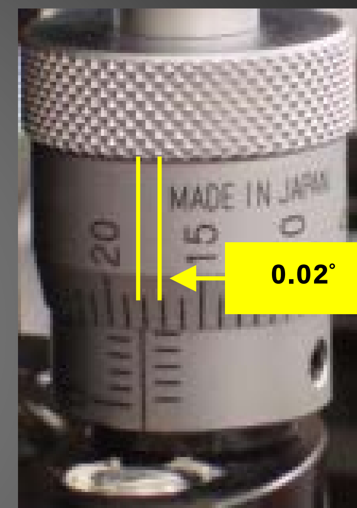
TEM Wedge

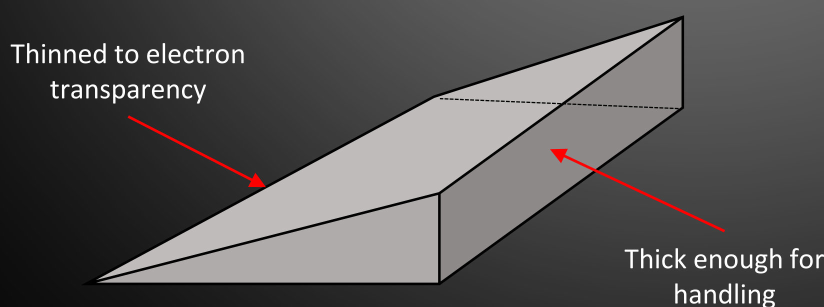
- Bulk Orientation
- Induce an angle to create a wedge sample
- Angle induced will vary depending on the material being prepared

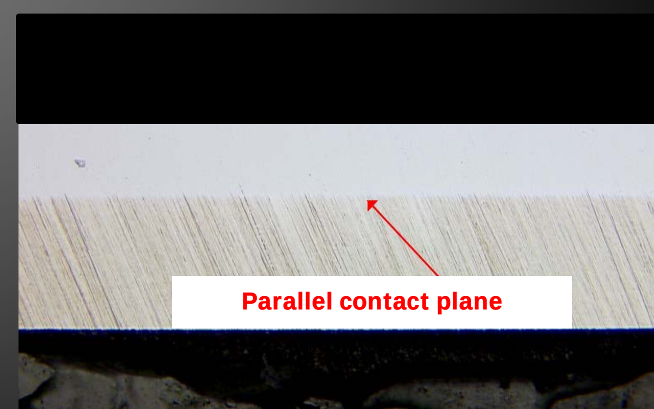


Inducing Wedge

- Micrometers on MultiPrep™ used to induce an angle on the sample to create a wedge
- Wedge allows for a sample thinned on one edge, with a thick enough edge for handling





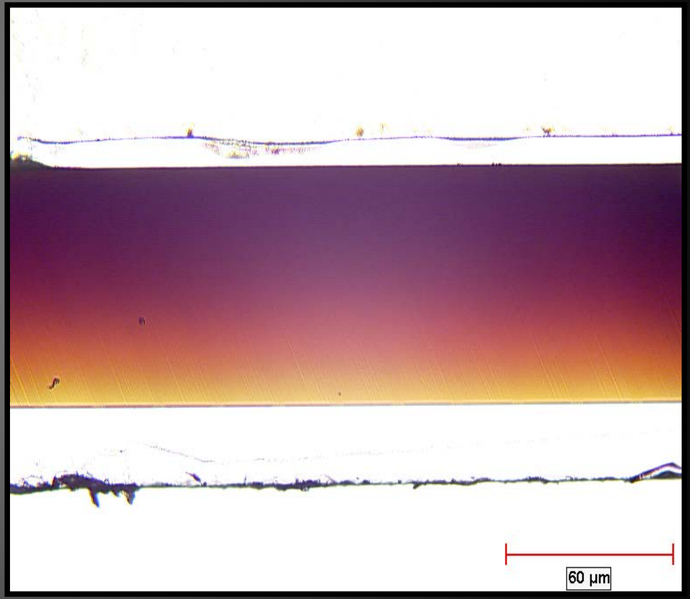


Wedge Polishing

- Certain TEM samples, along with the use of a transmitted light microscope, can display a series of colors in regions less than 10 µm thick
- Fringes can also occur in regions less than 2 µm thick
- The thickness that color corresponds to will vary depending on materials; however, some are well documented, such as silicon

Wedge Polishing

Color and fringe information can be used to assist in the preparation of TEM samples, as they are a guide to overall progress during a TEM wedge procedure

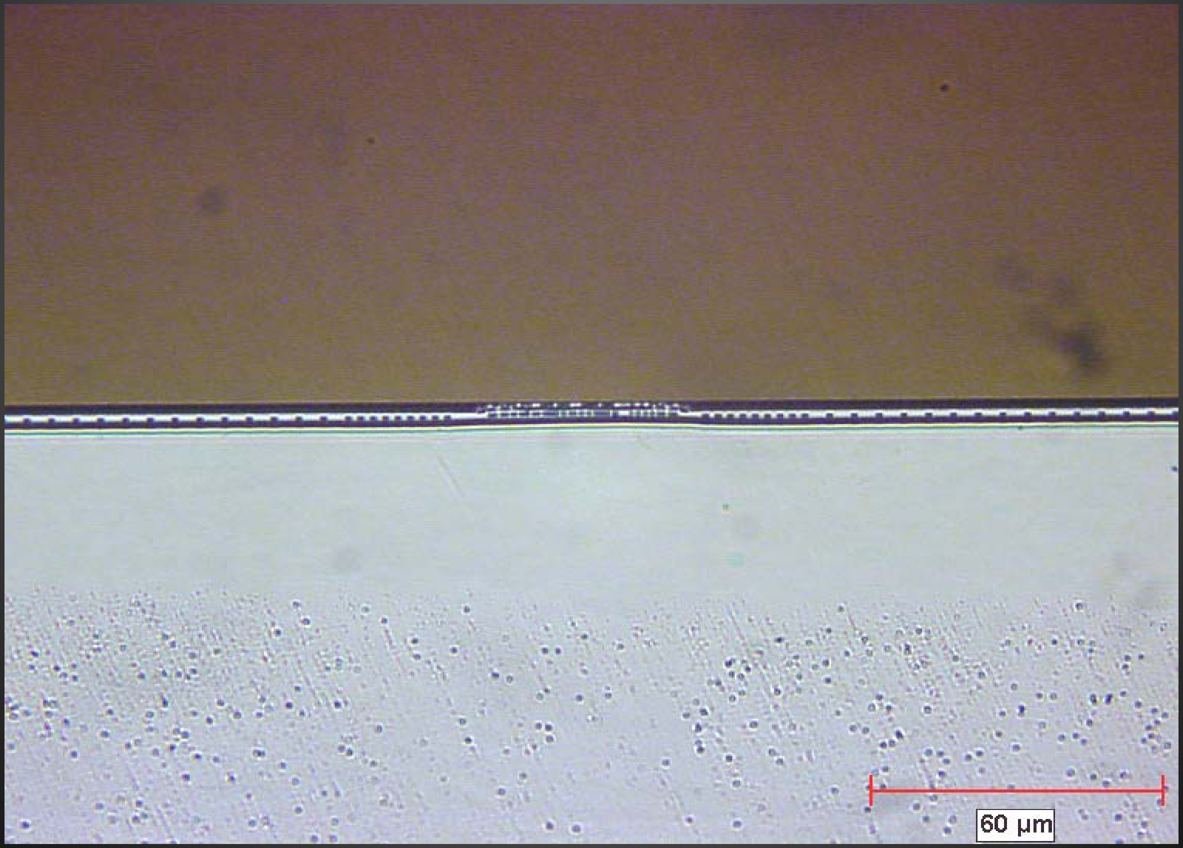


Si thickness [µm]	5	4	3	2	1
Color	Red	Reddish-orange	Orange-red	Orange	Light orange

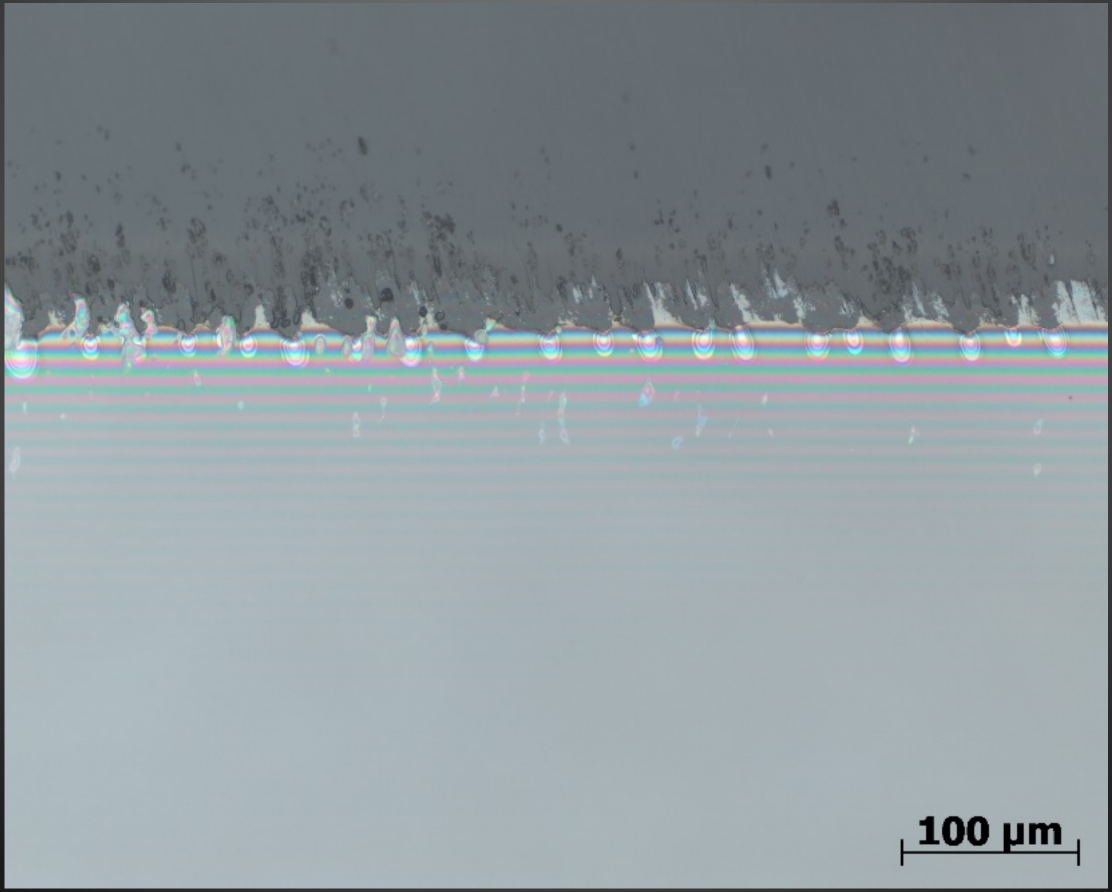
[4] Correspondence between Si wafer thickness and color of transmitted light

Wedge Polishing

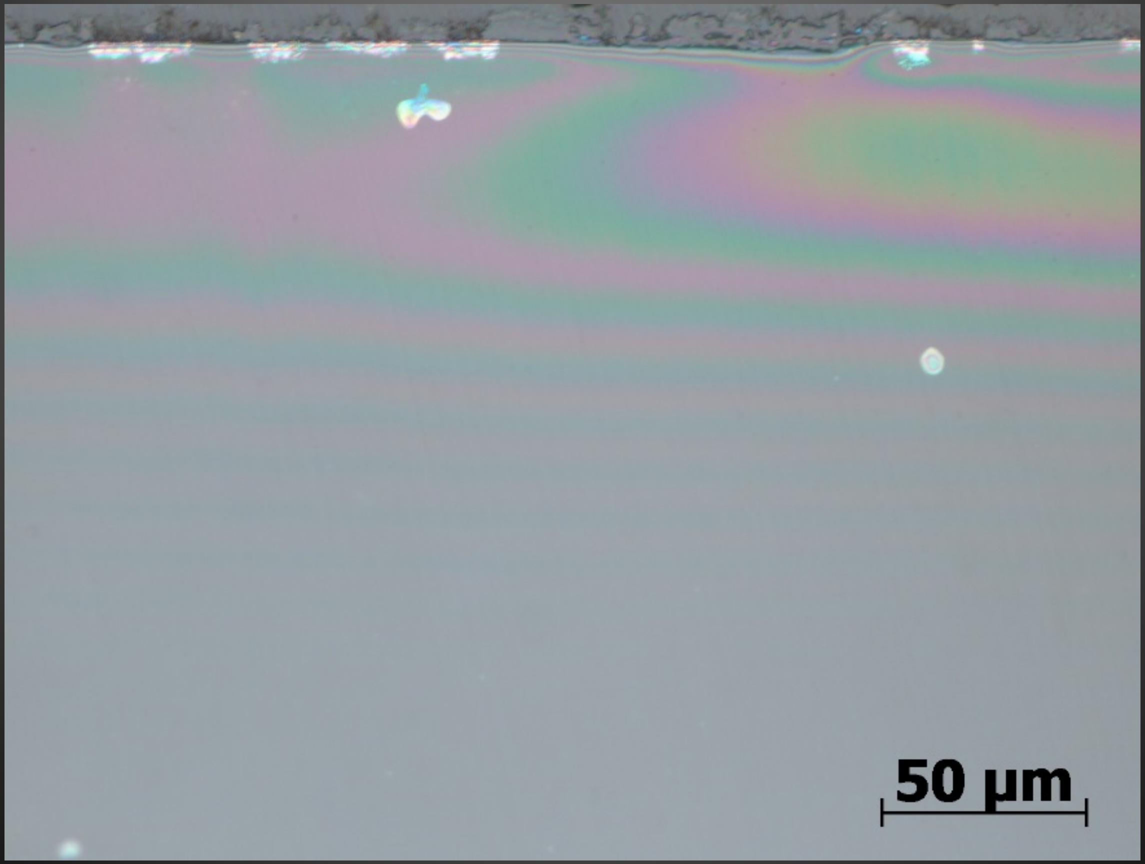
Wedge Polishing



Wedge Polishing



Wedge Polishing



References

- *1.* Allied High Tech Products, Inc., http://www.alliedhightech.com/. Accessed 23 May 2018.
- 2. Delstar Metal Finishing, Inc., *Electropolishing A User's Guide to Applications, Quality Standards and Specifications*, https://www.delstar.com/assets/pdf/epusersguide.pdf. Accessed 23 May 2018.
- 3. Williams, David B, and C B. Carter. *Transmission Electron Microscopy: A Textbook for Materials Science*. New York: Plenum Press, 1996. Print.
- 4. Yougui Liao. Practical Electron Microscopy and Database. URL: http://www.globalsino.com/EM/page2805.html. GlobalSino 2007.

Questions?

Pablo Mendoza
Materials Engineer, Technical Services
Allied High Tech Products, Inc.

pmendoza@alliedhightech.com