# Mechanical TEM Sample Preparation


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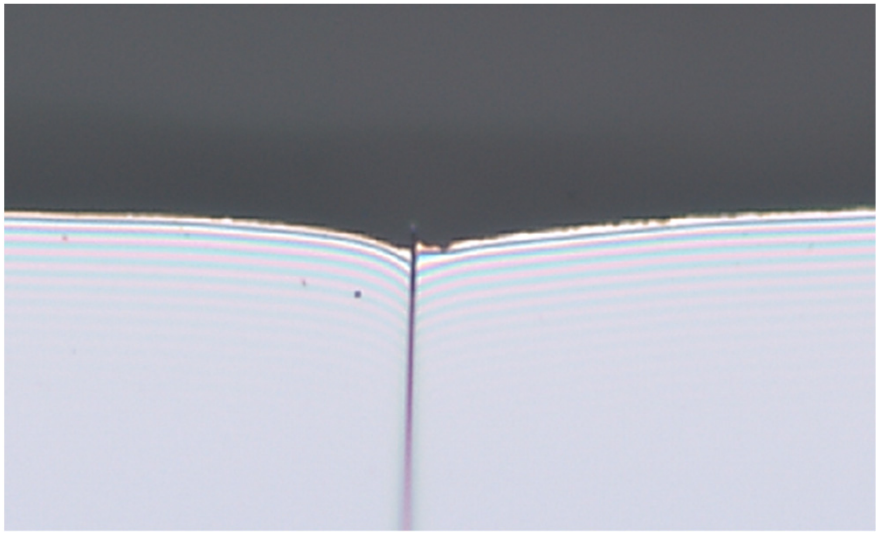
# Overview

- Basics
  - What is a TEM sample?
  - What are the requirements?
  - What are the methods of preparing TEM samples?

- Thin Film Preparation Process



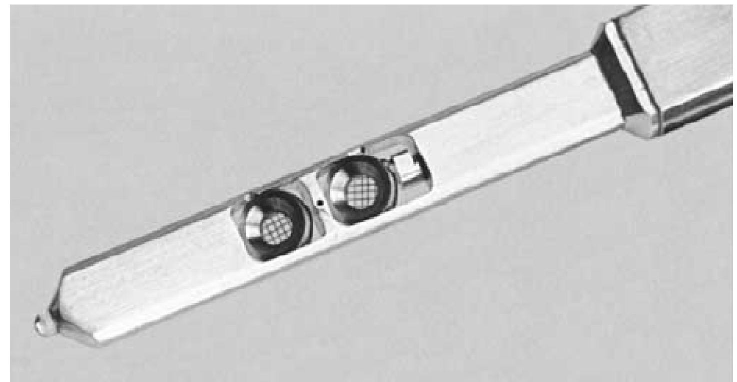
# What is a TEM Sample?

- **TEM**
  - Transmission Electron Microscopy is a microscopy technique in which a beam of electrons is transmitted though a specimen to form an image.
- **TEM Sample**
  - A specimen that is usually ultra thin (<100 nm) so that electrons can be transmitted through it.

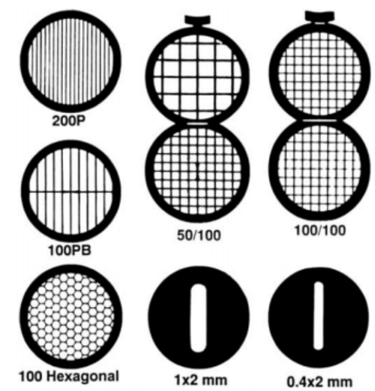
Zeiss HRTEM [3]

# TEM Sample Requirements

- **Must be electron transparent**
- **Must have a submicron thickness**
- **Must have an area of ≤3 mm to fit various TEM grids and holders**
- **Any deformation from previous processing must be removed.**



Two-specimen holder with double-tilt [3]



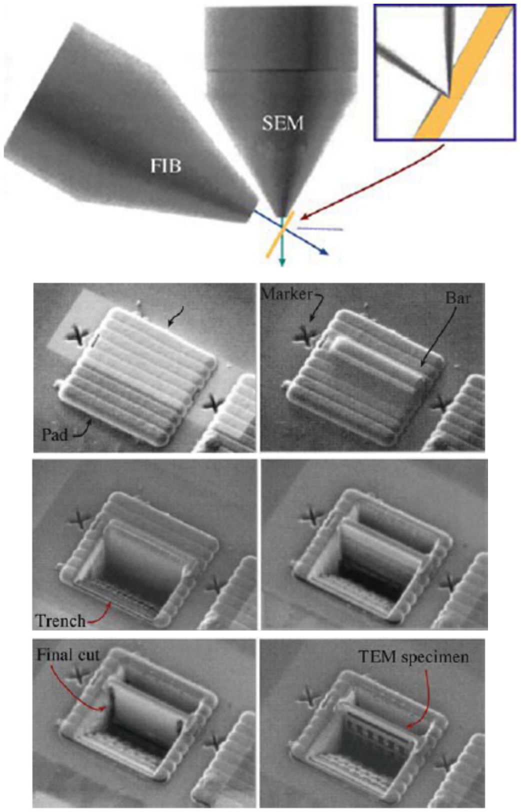

Specimen support grids of different mesh sizes and shapes [3]

# Common Methods of Preparation

- Ion Milling/FIB
- Electropolishing
- Mechanical Preparation
  - Thin Film Preparation

# Ion Milling/FIB

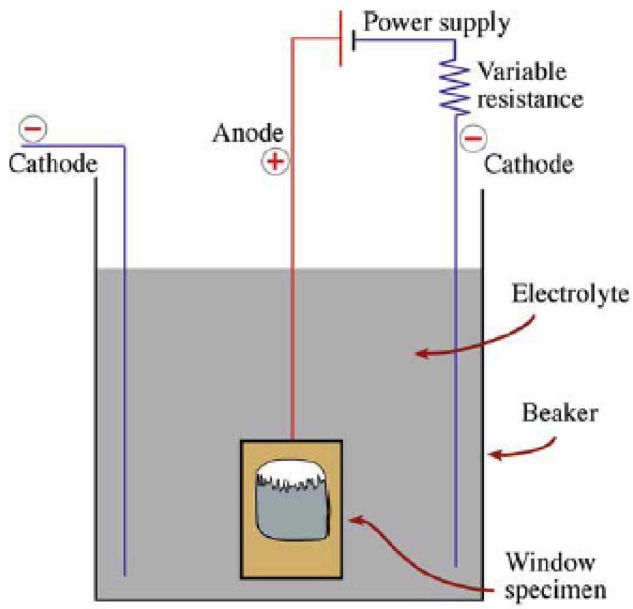
- **Bombarding a TEM sample with energetic ions or neutral atoms and sputtering material from the sample until it is thin enough to study in the TEM [3]**
- **A versatile thinning process that can be used for a wide variety of materials**
- **Is expensive to purchase and run**
- **Implantation of source material and an amorphous layer is created**



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

# Electropolishing

- **Immersing a sample in an electrolyte and subjecting it to a direct electrical current**
  - **Keep the sample anodic with a cathodic connection to a nearby metal conductor [2].**
  - **The anodic dissolution of the sample polishes the surface.**
- **Relatively quick and can produce samples with no mechanical damage**
- **Can only be used for electrically conductive metals and alloys**




The Window Method [3]

# Mechanical Preparation

- **Smoothing the sample surface using abrasives and mechanical tools**
- **Common types:**
  - **Manual: Uses hand tools (tripods) that allow the user to make angle adjustments**
  - **Semi-Automatic: Uses precision polishers with digital indicators and micrometers**
  - **Dimpling: Mechanical dimplers use a small-radius tool to grind and polish samples to a fixed radius of curvature in the center.**


# Mechanical Preparation

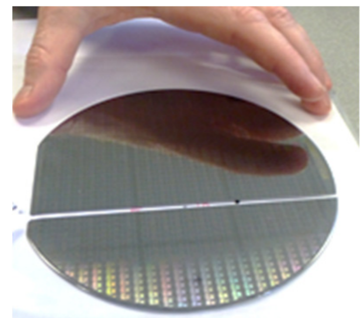
- Advantages
  - TEM Wedge Capabilities
  - All damage removed using proper material removal procedures
  - No foreign material implanted
  - Repeatable and fast with experience
  - Lower cost of required equipment
  - Decreases milling time and costs
- Disadvantages
  - Requires careful handling throughout the process
  - Greater learning curve than other techniques
  - Some samples may require additional processing using one of the other TEM preparation techniques

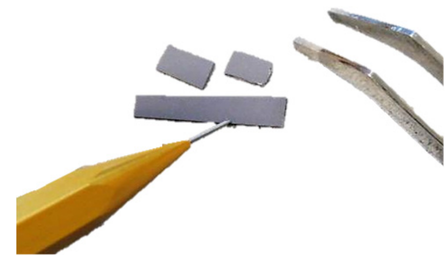
# Thin Film Preparation Process

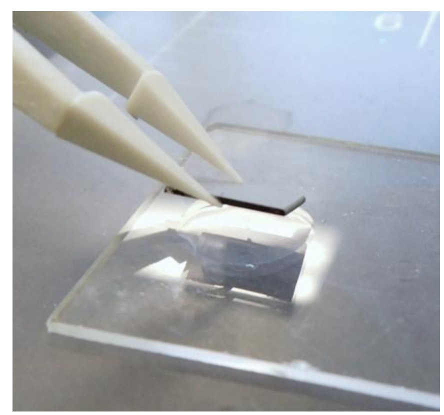
- Thin film samples are commonly prepared using mechanical methods. The process includes:
  - Preparing the Sample
  - Fixturing
  - Grinding the Pyrex®
  - Thinning the First Side
  - Flipping the Sample
  - Stopping Point
  - Inducing a Wedge
  - Color and Fringes

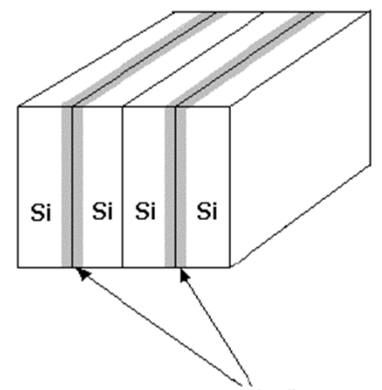
# Preparing the Sample

- **Wafers are scribed down to a manageable size.**
- **Using an appropriate adhesive, such as M-Bond 610, the thin film surfaces are "sandwiched" together.**



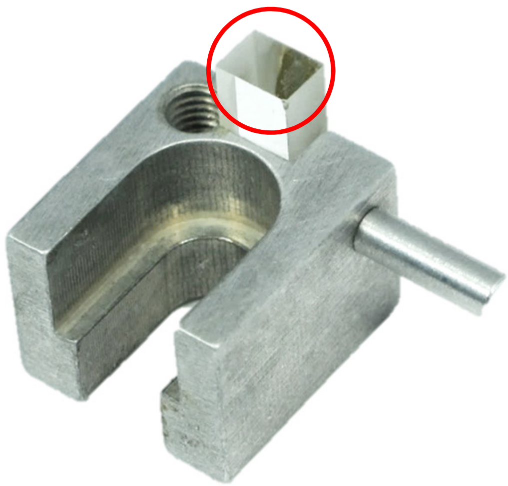






# Grinding the Pyrex®

- The surface of the Pyrex® piece on a thinning fixture requires grinding to ensure it is parallel to the platen, or the grinding plane.
- Pyrex® can be ground using diamond lapping films.
  - A 15-9 µm finish is acceptable.
  - Do not polish the surface; having some scratches will help with adhesion.

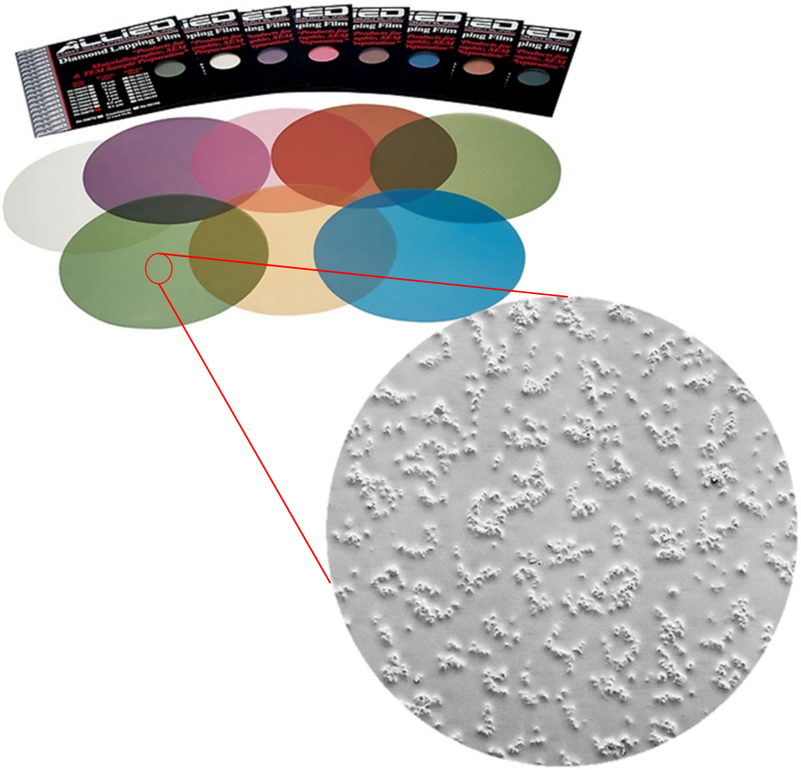


# Fixturing

- **Secure the sample on a thinning fixture with mounting wax.**
- **Place the fixture on a hot plate to melt the wax.**
- **Apply light pressure using a cotton-tipped applicator to assist in parallel registration of the sample to the fixture.**

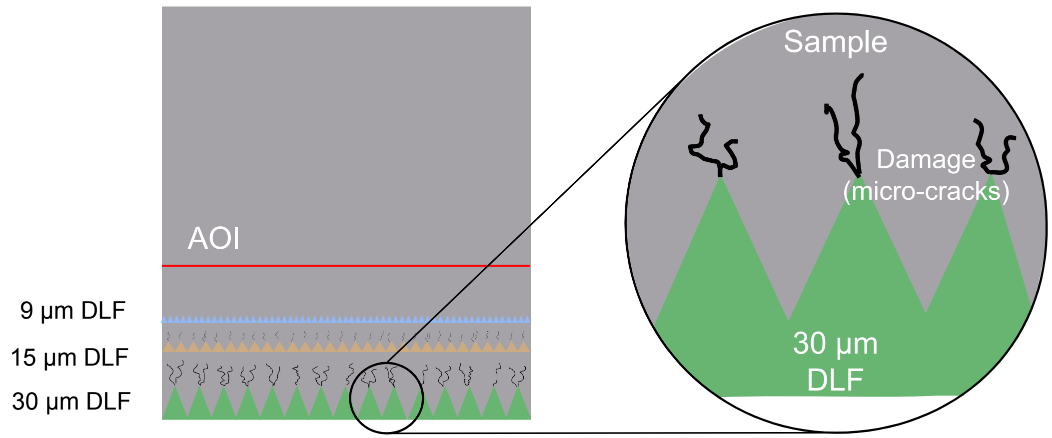
# Thinning the First Side

- **Abrasive: Diamond Lapping Films**
  - **Provide excellent edge retention and maintain coplanarity**
  - **Typically used for unencapsulated cross-sectioning, TEM preparation, backside polishing, etc.**



# Thinning the First Side

- **The sample surface is damaged throughout the grinding process.**
  - **Scratch patterns**
  - **Micro-cracks that can propagate further into the sample**



# 3X Rule

- **Remove 3 times the previous abrasive to completely remove any damage.**
  - **Ex: If a 30 µm lapping film is followed by a 15 µm lapping film, the 15 µm film must remove at least 90 µm (3 x 30 µm) to completely remove any damage.**

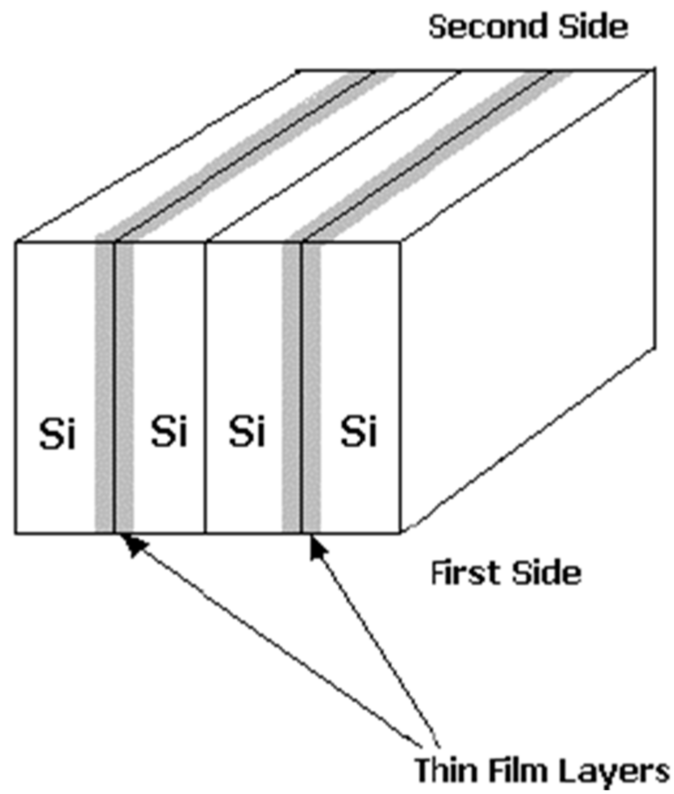
| Current Step (DLF) | Current Distance from Target | Previous Step | Remove At Least 3x Prev. Step Size | Distance to Target After Current Step |
|---|---|---|---|---|
| 30 µm | Varies | N/A | N/A | 192 µm |
| 15 µm | 192 µm | 30 µm | 90 µm | 102 µm |
| 9 µm | 102 µm | 15 µm | 45 µm | 57 µm |
| 6 µm | 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

- **Certain materials, such as ductile steels, may only require a "2X Rule."**
- **Fragile, brittle materials, such as ceramics, may require a "4X Rule" since cracks can propagate further into the sample.**
- **Main Concept: No matter what steps follow, all damage introduced by the previous abrasive must be removed to obtain an accurate representation of the microstructure.**

# Flipping the Sample

- Reheat the fixture on a hot plate.
- Carefully remove and clean the sample, and then place it back on the fixture with the polished side face down.
- Thin the sample according to the 3X rule.



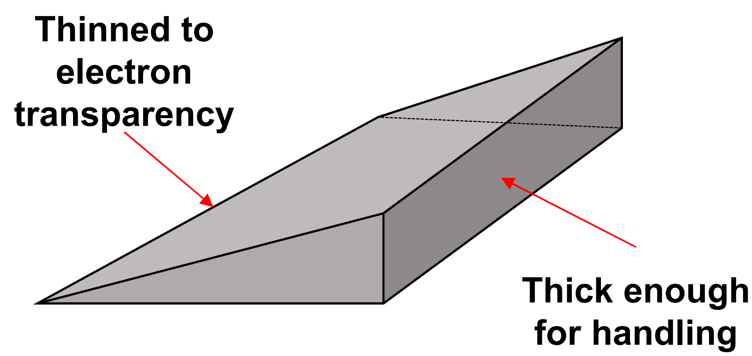
# Goal: Pre-FIB or TEM Wedge?

## Pre-FIB Thinning

- Remove the sample to prepare for FIB thinning by placing the paddle into a piece of filter paper, and then into a container of acetone to remove the wax.

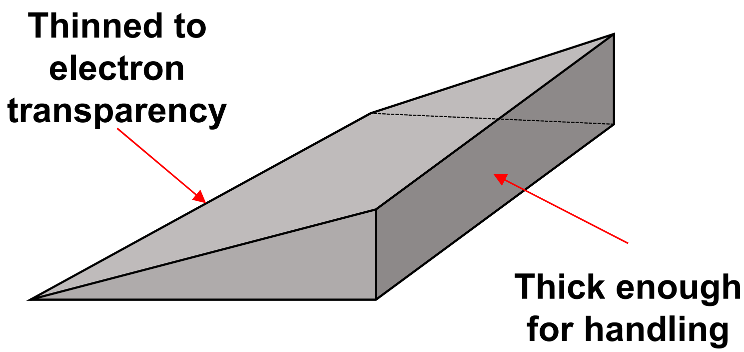
## TEM Wedge

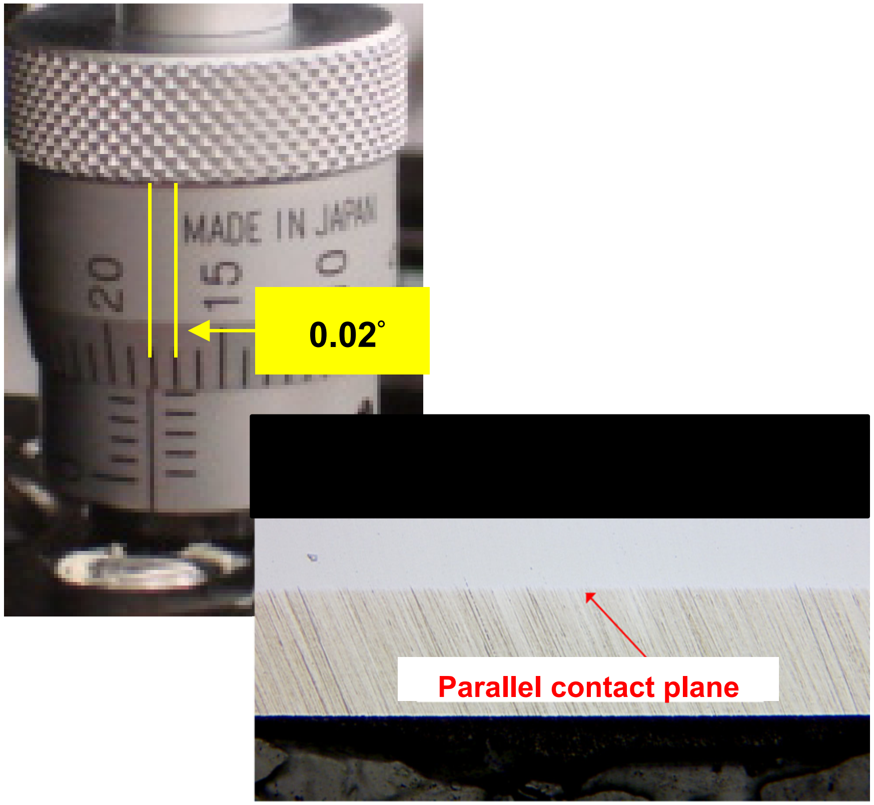
- Induce an angle to create a wedge sample.
- The degree of the angle depends on the material being prepared.



# Inducing a Wedge

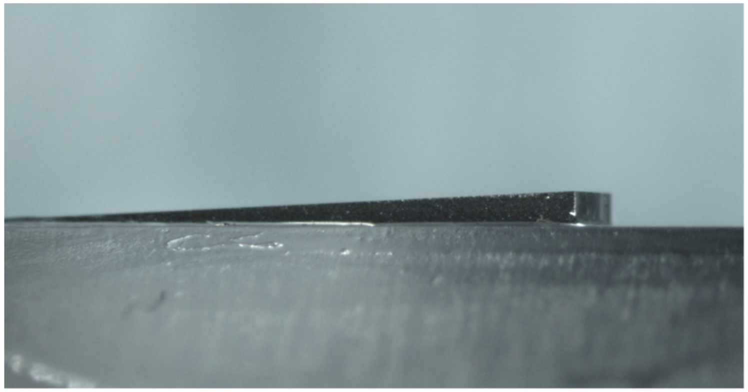
- **Micrometers on precision polishers are used to induce an angle on the sample and create a wedge.**



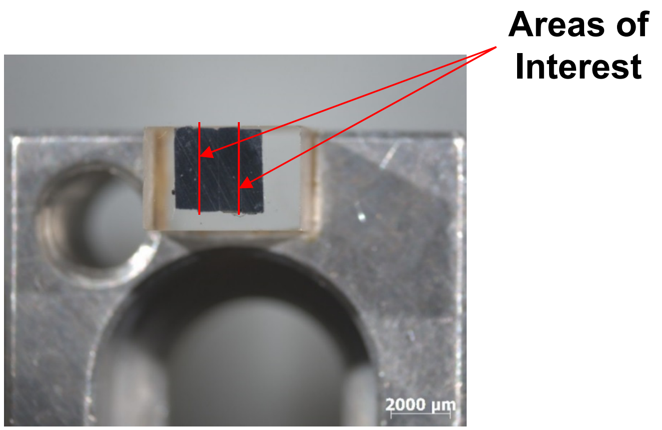



# When is the Sample Complete?

- **Bulk**
  - Homogeneous material
  - Can stop anywhere; only a useable area is needed



- **Thin Film**
  - Film on material deposited on surface
  - Prepared properly, can stop anywhere



# Color and Fringes

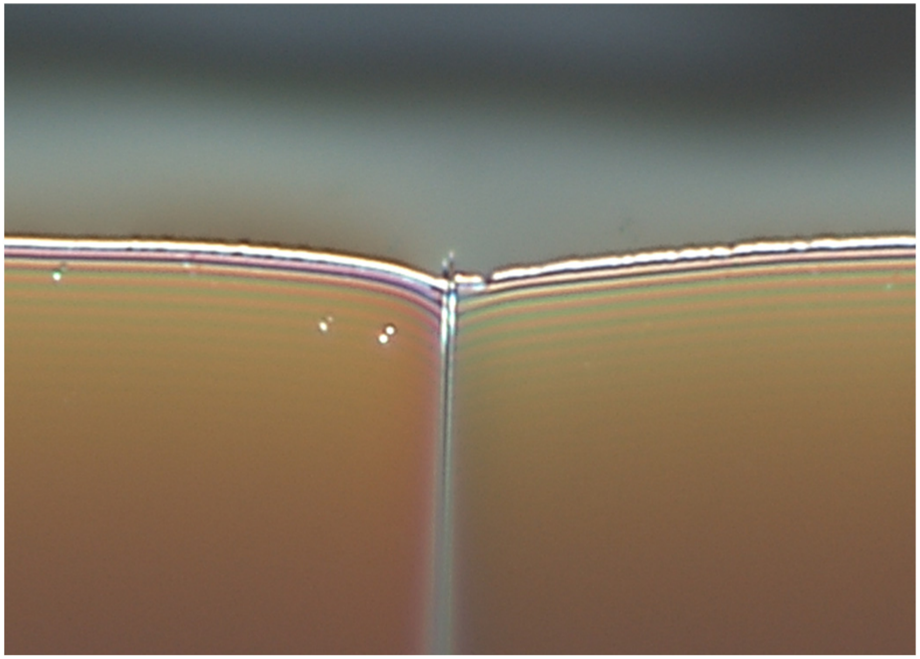
- Certain TEM samples can display a series of colors in regions <10 μm thick with a transmitted light microscope.
- Fringes can also occur in regions <2 μm thick.
- Colors that correspond with different thicknesses vary based on materials; however, some are well documented, such as silicon.

# Color and Fringes

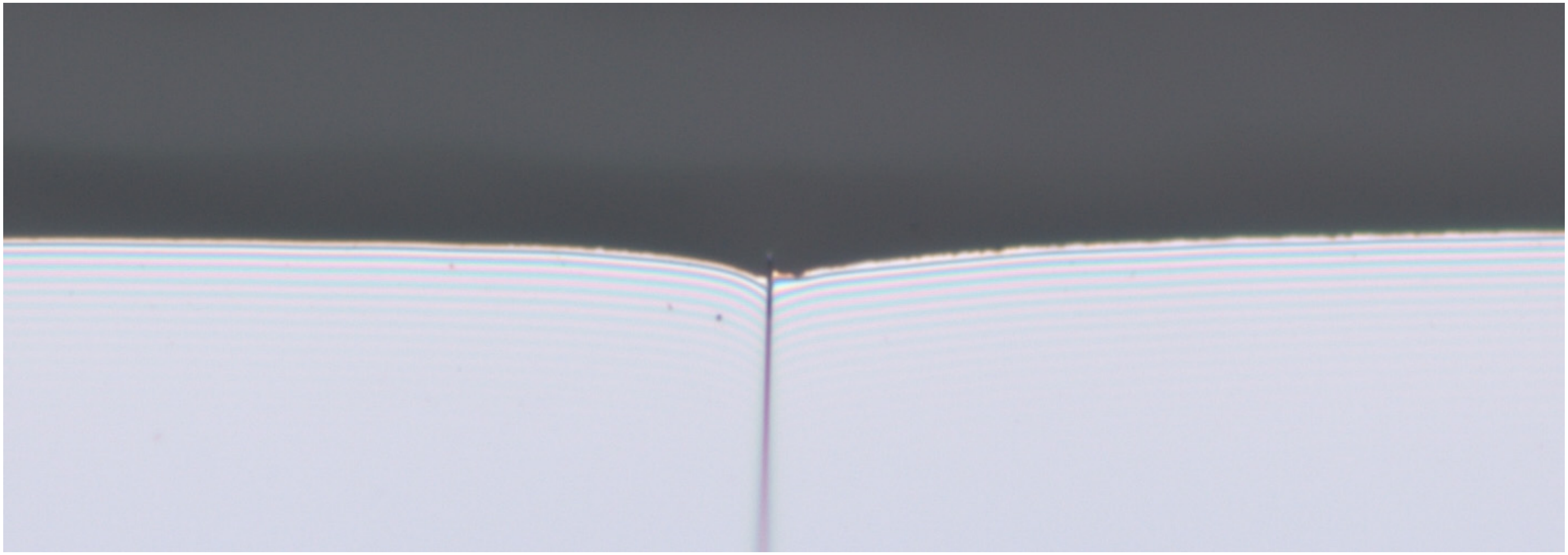
- Color and fringe information can be used to assist in the preparation of TEM samples, as they are a guide to overall progress during a TEM wedge procedure.

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

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




100 µm



# References


- 1. Allied High Tech Products, Inc., http://www.alliedhightech.com/. Accessed 16 September 2019.
- 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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