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Microstructural Characterization of Materials

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Today's class contents

" TEM Sample preparation "

- Techniques

- Considerations

Useful links for TEM sample prep.

- http://TEM.snu.ac.kr **(김영운 교수님 연구실홈피)**
- http://www.biotech.ufl.edu/EM/tips/tem.html
- www.kaker.com

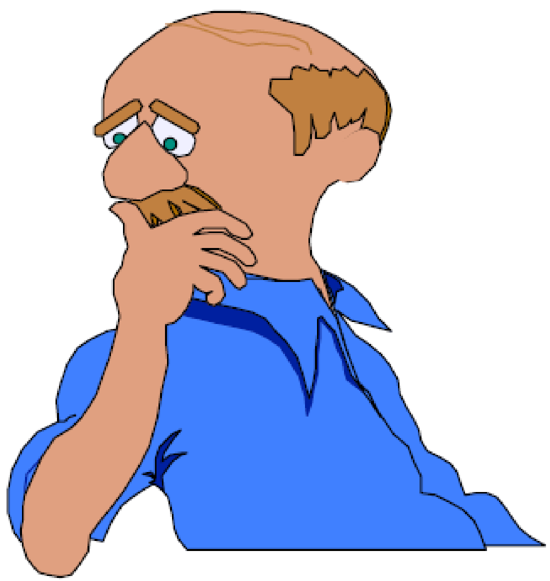
Frequently, ***Sample Preparation 〉 TEM Work***

Purposes of Work

TEM - Microstructure, HREM
SEM - Topography, Microstructure
EPMA - Quantitative, Qualitative
EELS, ...

Materials

Metals
Ceramics
Semiconductors
Powders, Fibers, ...



Preparation Methods

Ion milling, Tripod polishing,
FIB, Ultramicrotomy, ...

TEM specimen must be …

thin : less than ~ 1000 Å **(100 nm)**

shape : 3㎜ disk type

representative of, and unchanged, from bulk

flat, not rugged

amount of transparent area

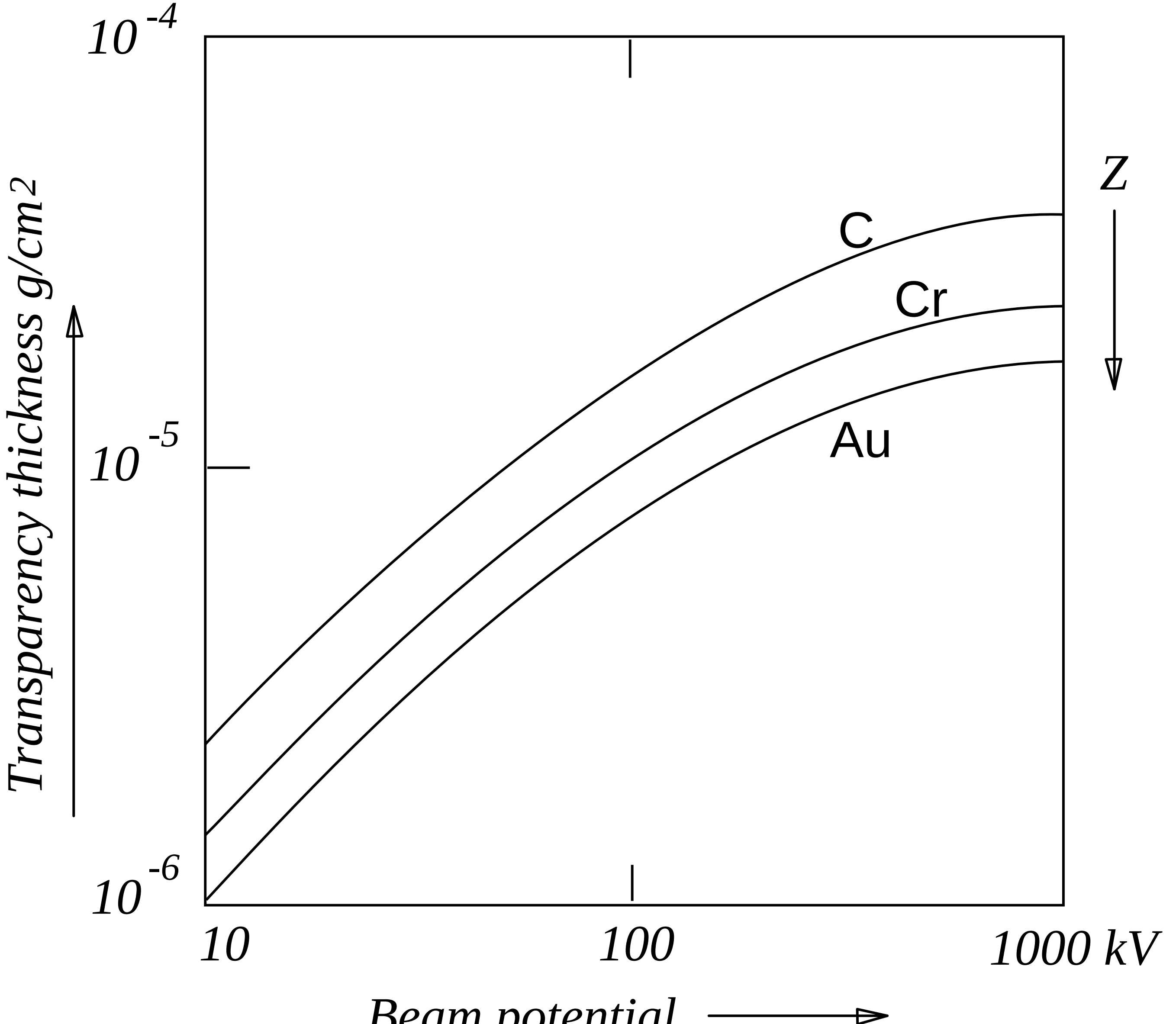
stable in the electron beam

very clean : free from extraneous particles or debris (possibly from specimen preparation)

conductive, non magnetic

Methods: *depend on both the* **type of material** *and* **the information to obtain**.

How thin is thin?

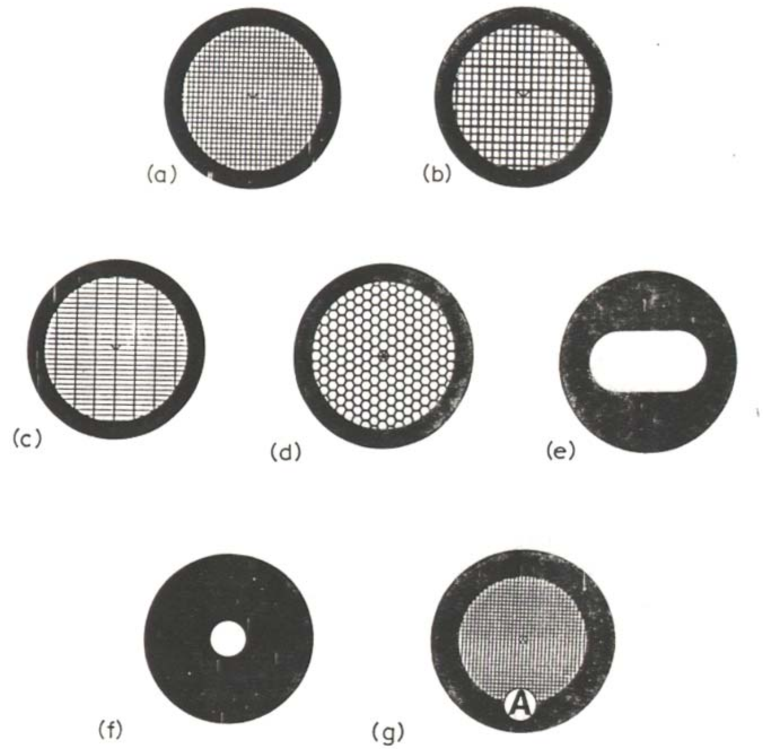


TEM preparation methods

- **Powder material**
- **Plan-view specimens:** **surface study**
- **Cross-section specimens**: **interface study,...**

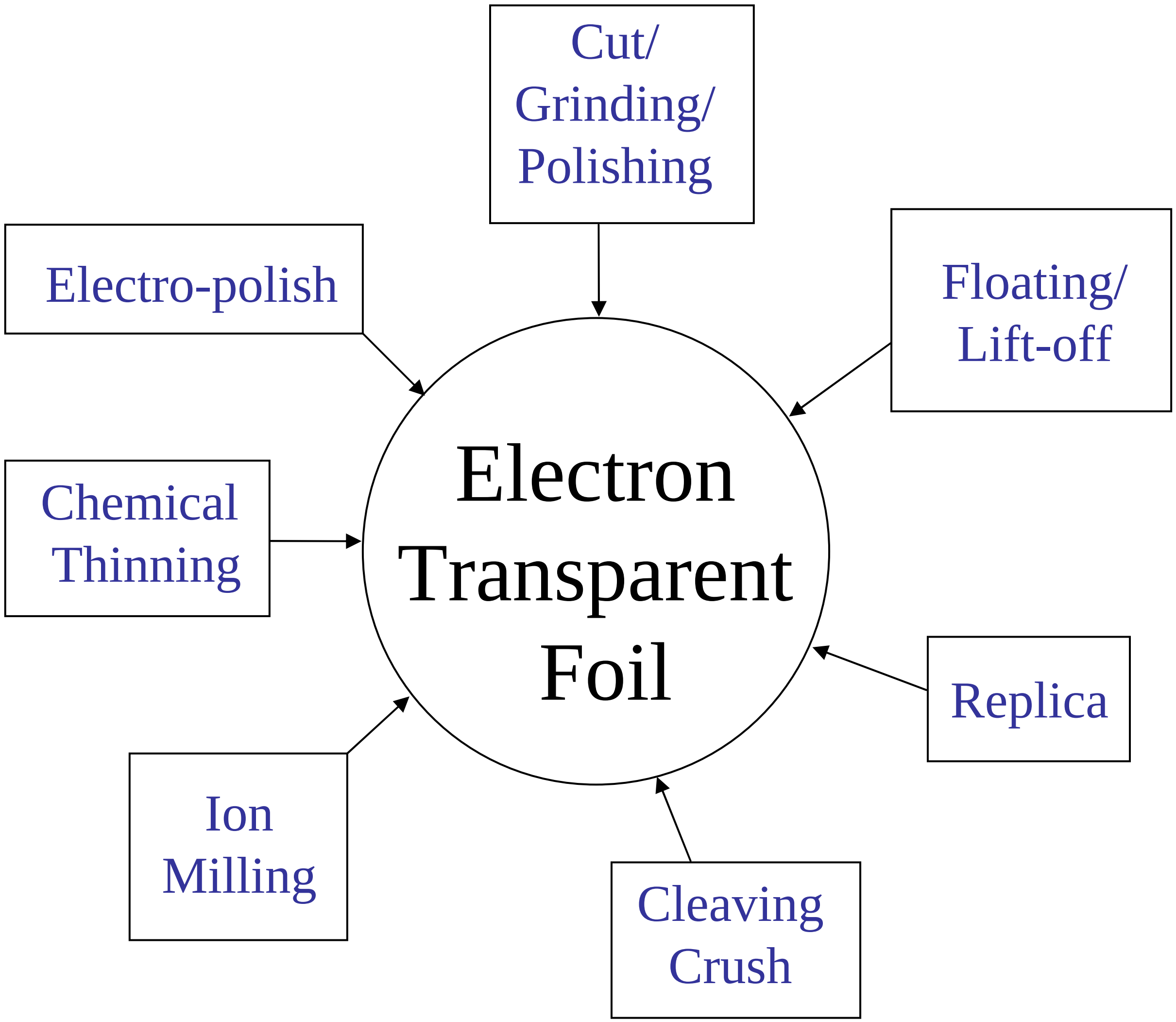
Powder specimen

1. Dissolve the powder into a solution, mix.
2. Splash one drop of solution onto a copper grid with carbon supporting film
3. Wait until the specimen is dry before observing in TEM



- *Grid*
  - 주로 **Cu** 이고 **Ni, Au, CP(Cu/Pd)**도 이용

Techniques for Thin Foil



Cut/Grind/Polish

- Cutting
  - Diamond Saw
  - Cleave
- Grinding/Polishing
  - Sand Papers, Diamond Impregnated Disc
  - Lapping films (SiC, Al_2O_3, Diamond)*
- Lapping
  - Tripod Polisher*
- Ultramicrotomy*

* **Possible to make thin foils for TEM without further treatment**

Cut/Grind/Polish

- Diamond saw

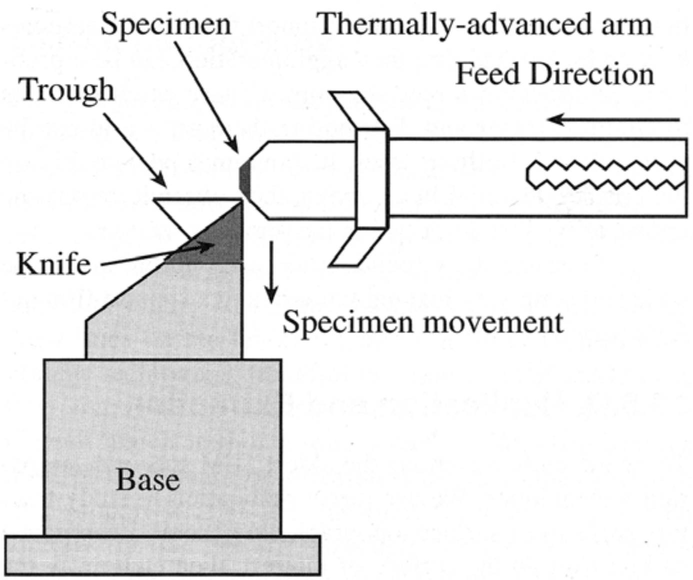
- 3mm disc cutter

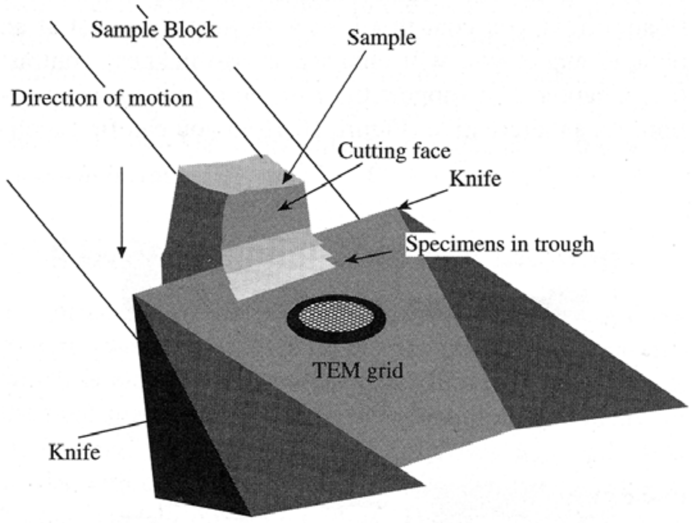
Ultrasonic Cutter

Slurry circular cutter

Physical cutting

- Microtome





Electro-polishing

Basic

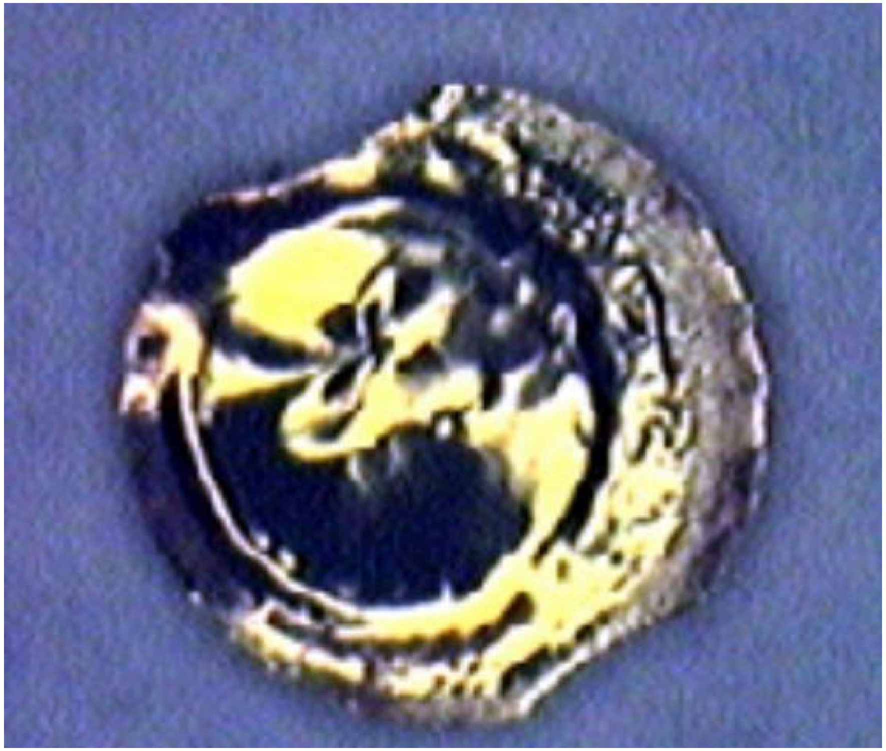
- Electrolytic etching/polishing
- Requirement : the specimen must be conductive

  - this process works like a galvanic cell. The applied current forces the anode(sample) into solution to deposit onto the cathode
- Initial condition of specimen surface influences polishing time

  -better the surface at start the higher the current density, the shorter the polishing time
- 전압, 전해액 조성, 반응 온도, 용액 순환 속도에 따라 시간 결정
- Window technique / Jet polishing

Electro-polishing

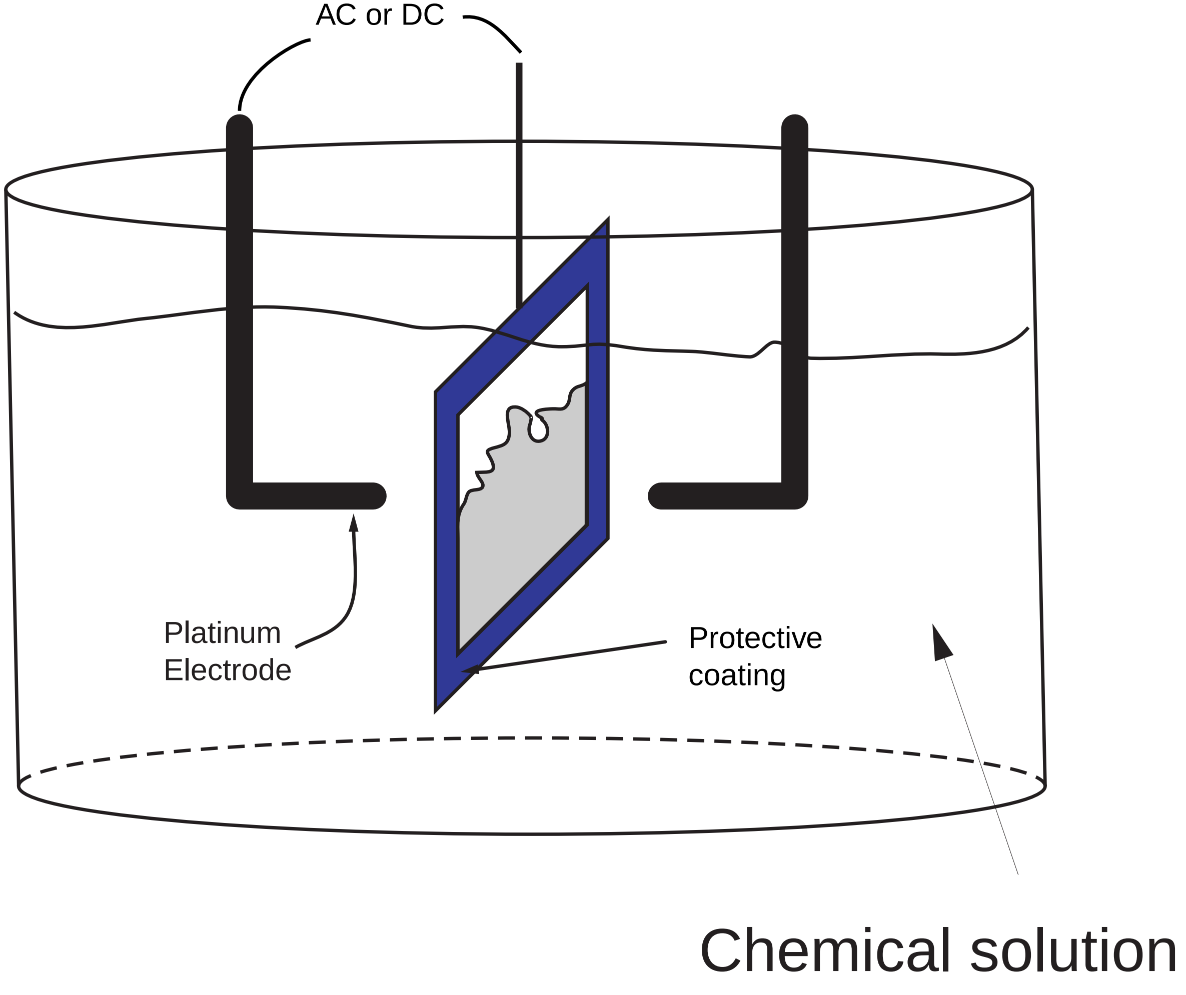
- Jet polisher



* Every lab equipped with Jet polisher has their own know-how solution, but Perchloric acid+Acetic acid+Methanol(or Water) is the most widely used “*universal*” solution. - ***Safety First!***

Electro-polishing

- Ballman method (window technique)



Tripod Polishing

- 정밀 연마용 치구 및 diamond lapping film을 사용하여 전자 빔이 투과 가능한 두께까지 균일하게 연마하는 것
- 원래 반도체 소자 제작공정상에서 주사전자현미경 분석결과 결함이 있다고 여겨지는 수 ㎛크기의 특정 부위를 투과전자현미경으로 정밀 분석하기 위해 고안.
- 저배율로 넓은 부위 동시 관찰에 매우 유리
- 아주 취약한 일부재료를 제외, 적용재료에도 제한이 없다. 비교적 높은 숙련도가 요구
- 시편표면에 최종 연마제 굵기에 해당하는 0.05㎛ 정도의 미세 흡집들이 남기 때문에 고분해상 획득 등 고배율 분석작업에는 부적합
- 시편 표면을 짧은시간 동안 이온빔 polishing 하는 과정 필요 100-500Å정도로 얇은 박막층에 대한 평면 시편제작, ion milling 속도 차이가 매우 큰 충상재료의 단면시편 제작 등에 유용.

◆ Advanced method

* 방 법 : Tripod Polishing + Sector Speed Control

* 장 점
- 연마율 차이 제거
- 넓은 관찰 영역
- 저렴한 가격의 시편제작 방법
- 깨끗한 이미지 관찰 가능 (최소의 이온빔 연마 시간)

* 단 점
- 숙달 과정 요구

◆ Mechanical Polishing

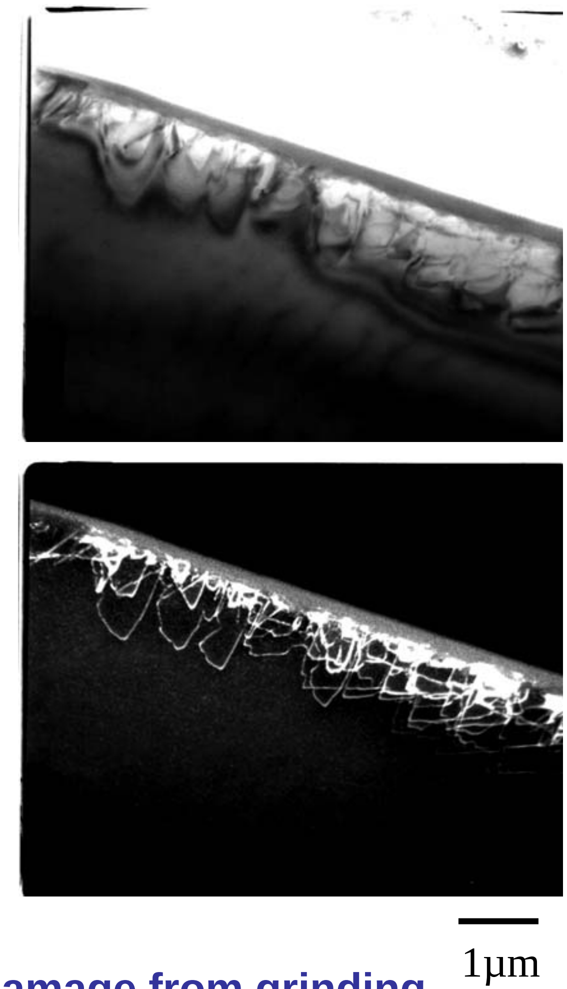
* Tripod Polisher
- Designed to accurately prepare SEM and TEM samples of pre-specified micron-sized regions
- 이온 연마 시간을 획기적으로 단축
- 이온 연마에 의한 단점 제거 (연마속도 차이, radiation damage, 오염, 시편의 가열, 평탄하지 않은 표면 등.)

* 제작 과정
- 시편 절단 및 접착
- wax를 이용하여 시편을 pyrex에 부착
- 마이크로미터 조절
- Diamond abrasive film을 이용하여 wet polishing (순서 : 60, 30, 15, 6, 3, 1 micron)
- 실리카 현탁액을 뿌린 연마천 위에서 미세 연마
- 시편을 아세톤에 담궈 떼어 냄
- 시편을 pyrex에 부착하고 마이크로미터 조절
- wedge 부분이 1micron 이하가 될 때 까지 같은 순서로 연마
- 실리카 현탁액을 뿌린 연마천 위에서 미세 연마
- 도립 현미경으로 시편 상태 수시로 관찰
- 2 x 1 oval grid에 부착, 시편 떼어냄

- **Particle size vs. US Grit**

Micron size	US Mesh size
9µm	1200
12µm	1000
15µm	600
20µm	500
30µm	400
40µm	320
45µm	280[&]
50µm	240
60µm	220
80µm	180
100µm	150

& Typical diamond grinding tool bits.

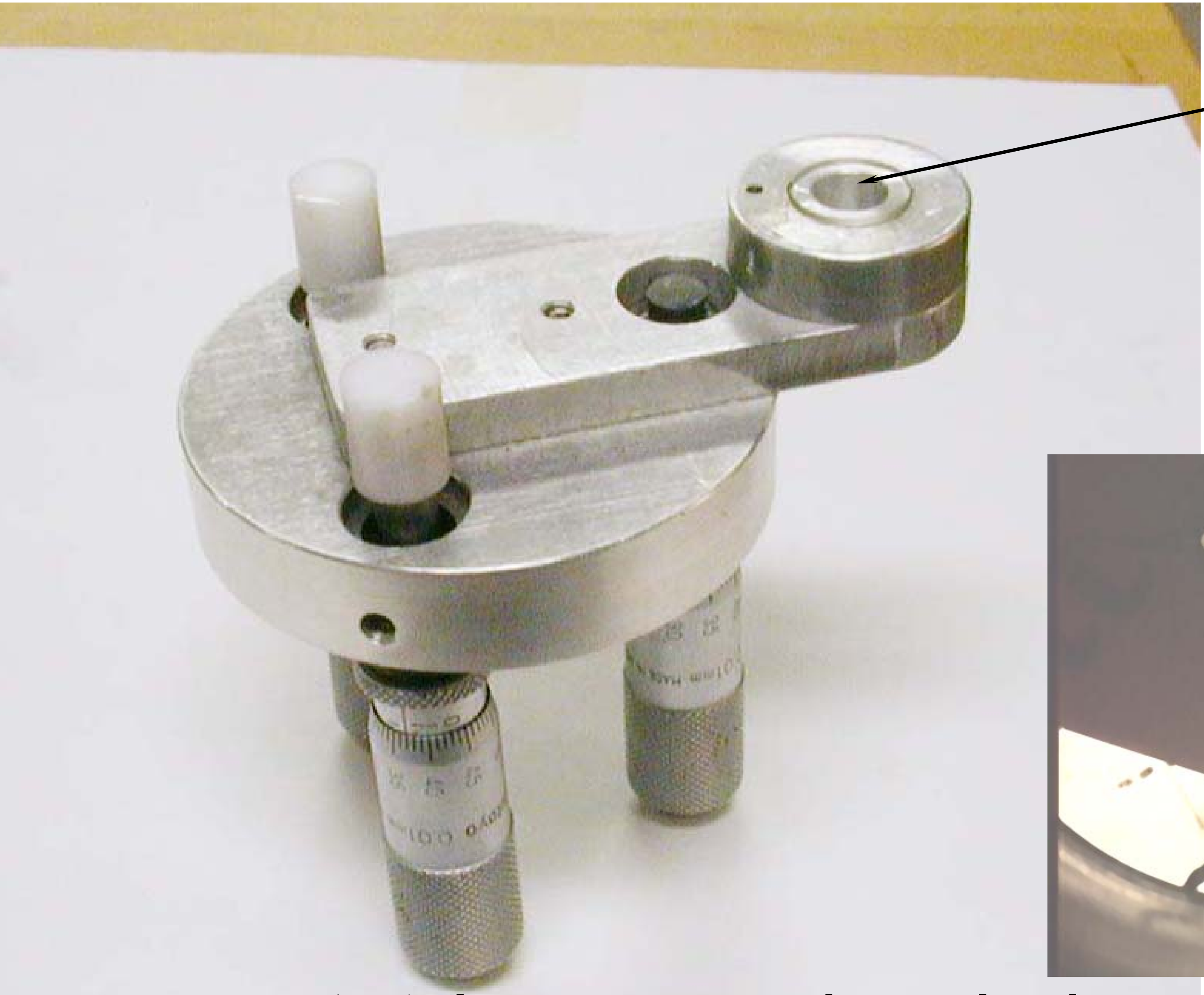


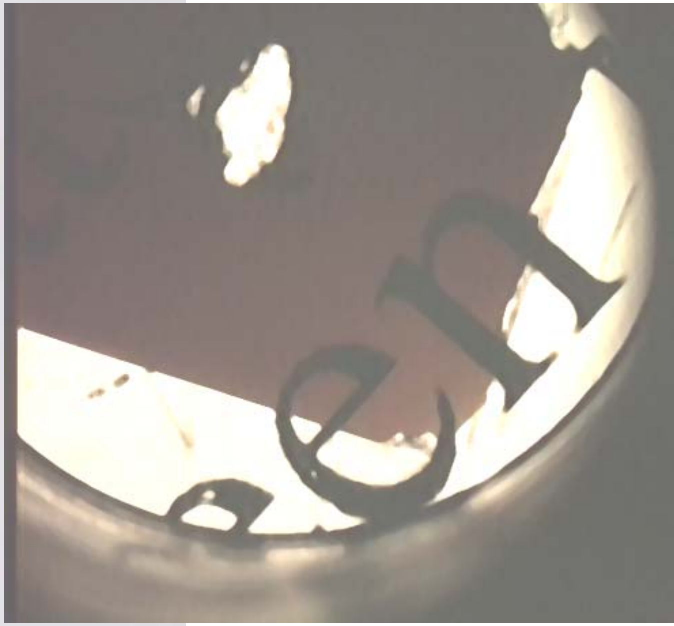
Better sample can be obtained when finer grid was used for surface finish (typically, ~ 1 micron before ion milling for Si).

- **Damage from grinding**

[Si surface was ground with 1200 grit]

- **Example of lapping Si(001)**



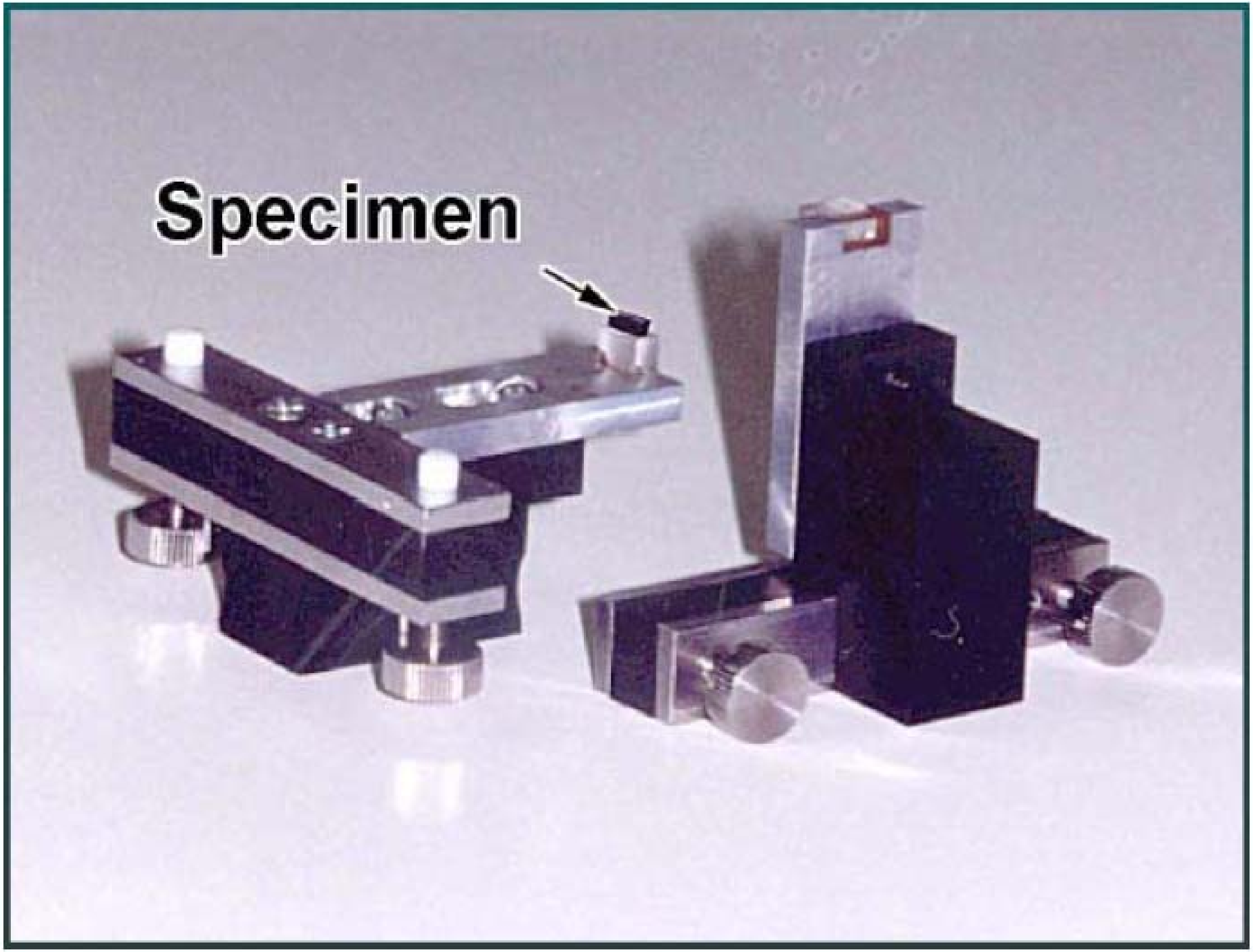


Si(001) plane is transparent when it is less than 10μm thick

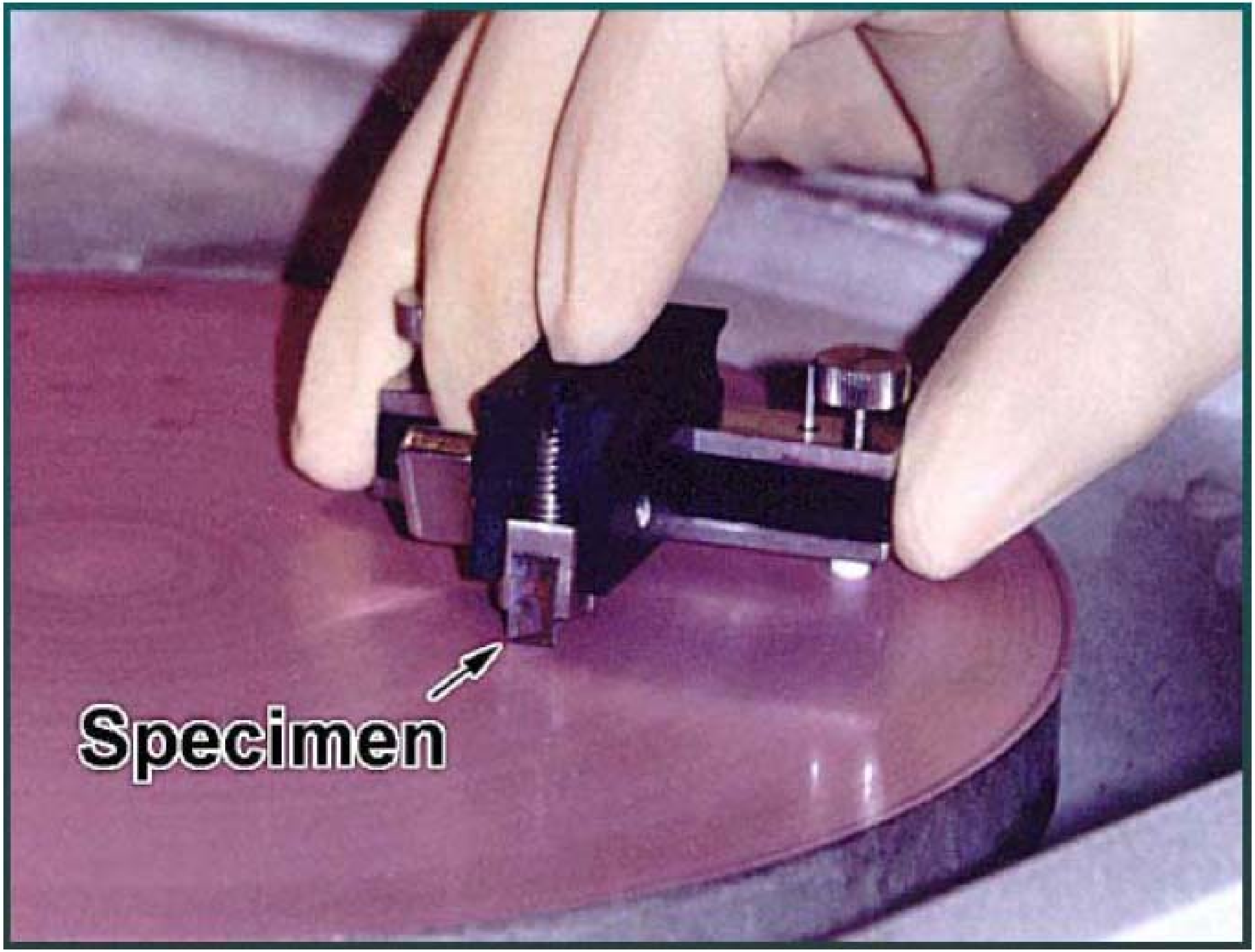
Wedge Polishing Technique

- Very flexible with great potential
- Needs extensive training and results vary
- Original tripod polisher idea Proposed by Ron Anderson et al at IBM (EMSA Bulletin, Fall 1989)
- A special mechanical fixture holds the specimen for polishing
  - Micrometer screws introduce a shallow angle to the sample
  - Shallow polishing angle produces a thin (leading) edge <1um thick
  - Thicker edge of the specimen remains strong for handling
- Hong Zhang at Applied Material introduced the T-Tool
  - Improved design permits sample viewing on optical microscope
  - Same accuracy and precise control of wedge angle as the tripod
  - Light weight reduces the chance of specimen damage.
  - Small size makes it comfortable to hold and use.

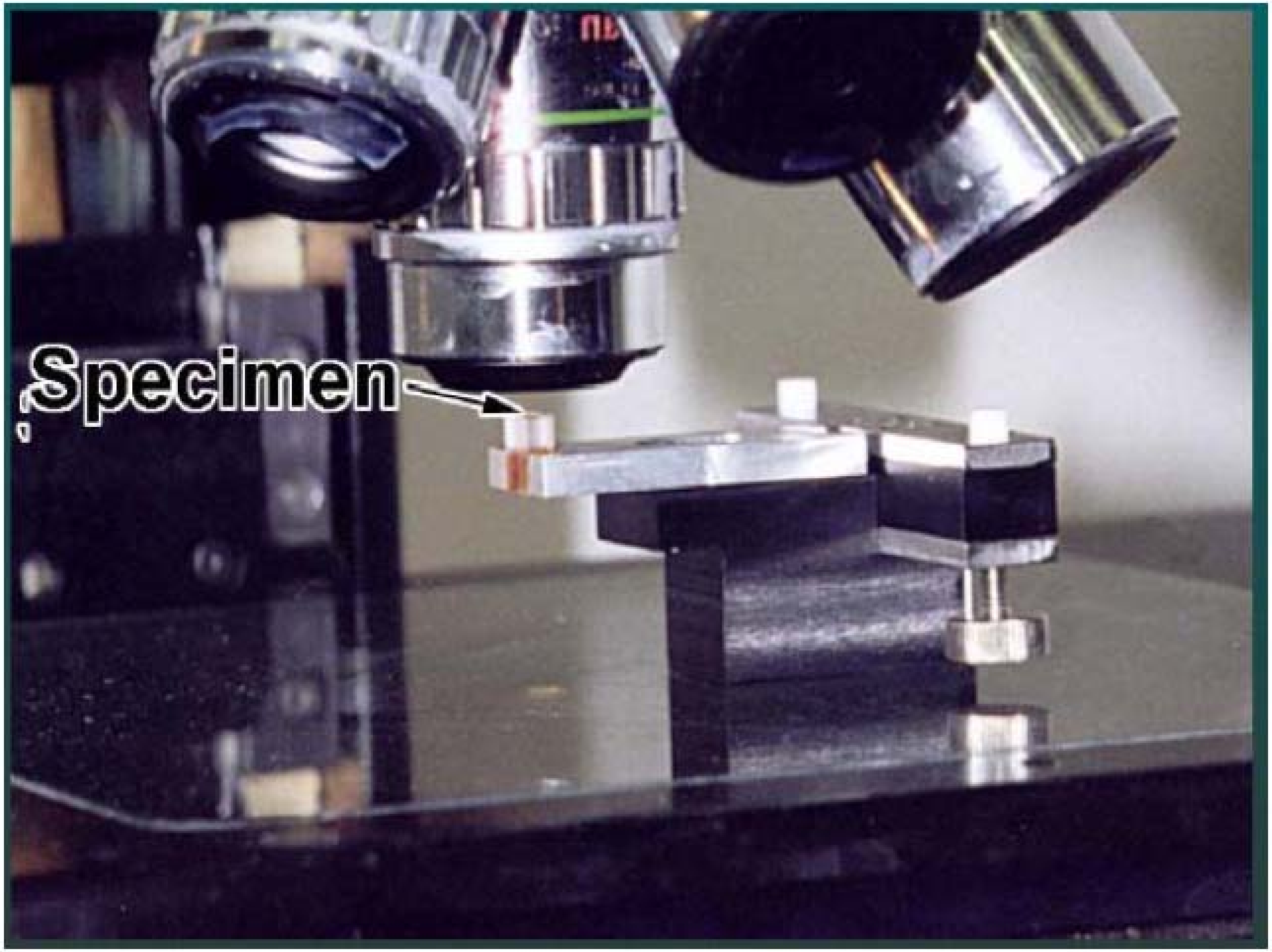
T-Tool for Wedge Polishing



T-Tool for Wedge Polishing



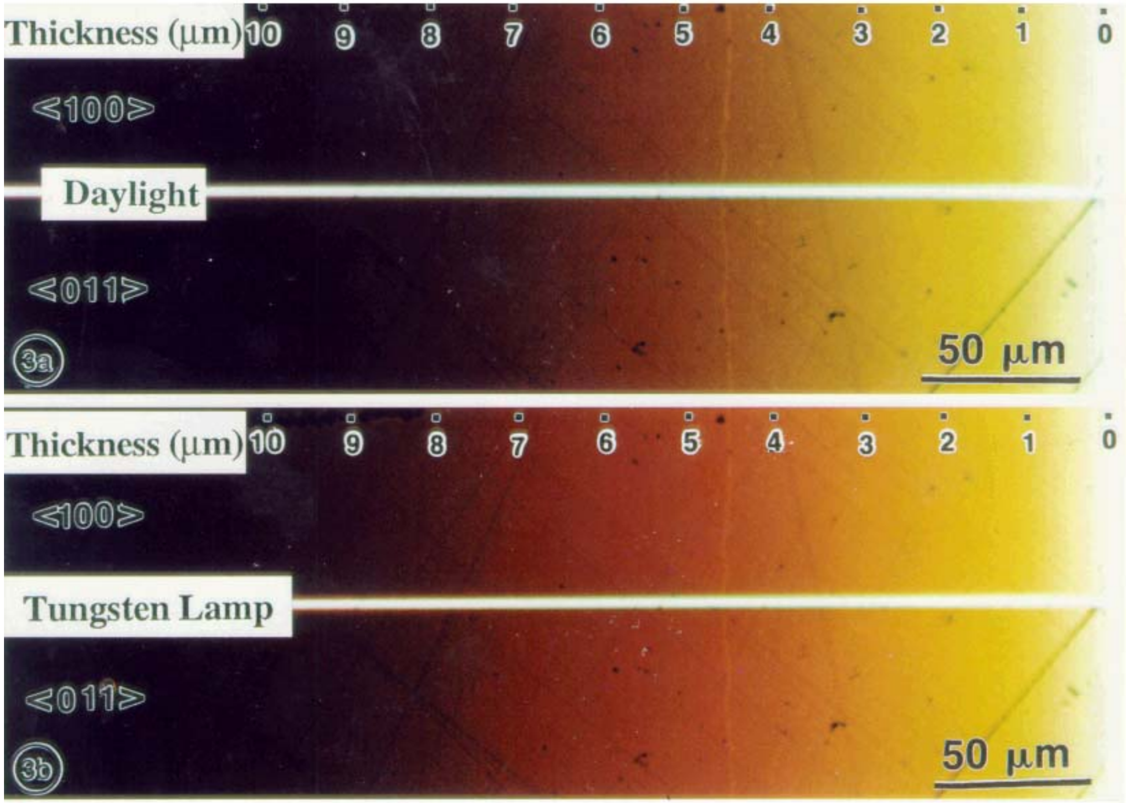
T-Tool for Wedge Polishing



Monitoring the specimen thickness until it becomes light transparent

Sample preparation

– Si thickness color chart



J.P. McCaffrey, Micron 29, 139 (1998)

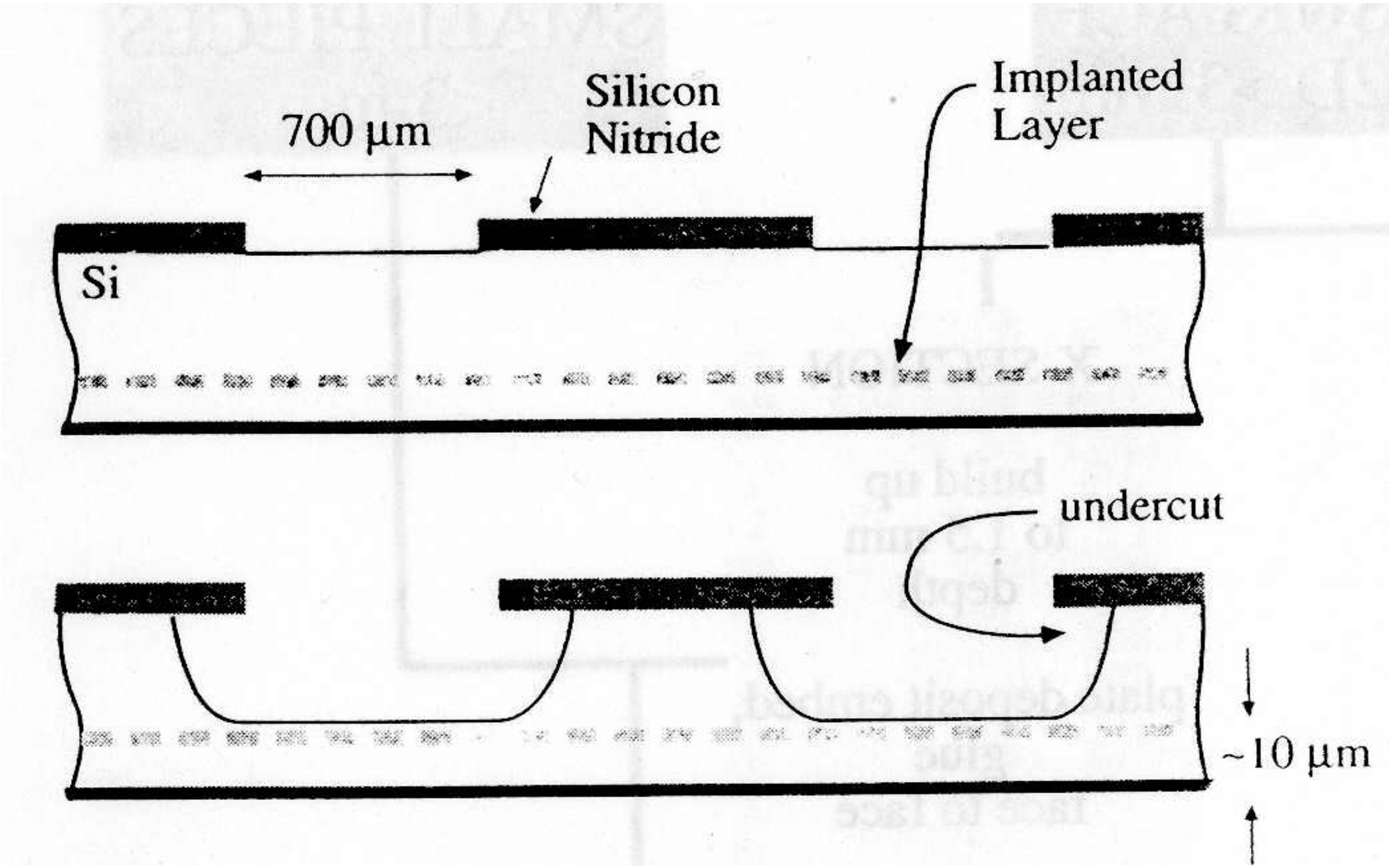
Chemical Thinning

- Choice of chemical
  - **Should not have preferential etching of grain boundaries, embedded second phase, and lattice defects.**
  - **Can prepare damage-free surface in short time.**
  - **Time consuming to find literature related to the specific materials or trial-and-error.**
  - **Good accumulation of materials for Silicon* and Germanium*.**

*** For example, see Thin Film Process First edition (Not that extensive in second edition) by John Vossen, page 438. Academic Press.**

Preferential chemical etching

- Remove part of the sample by chemical etching to leave an area which is electron transparent

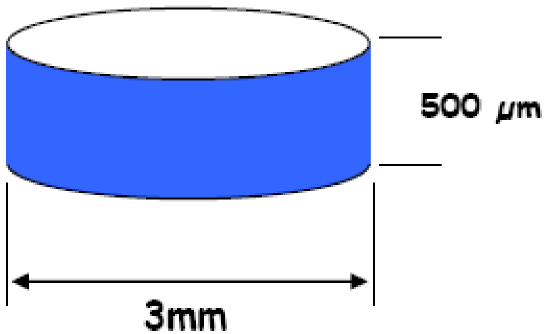


Ion Beam Sample Preparation

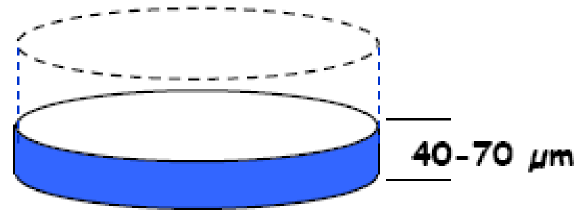
- Two primary ion beam technologies
  - Broad ion beam
  - Focused Ion Beam (FIB)
- Broad Ion Beam
  - Most accepted technique in use today
  - Suitable for all types of sample materials
    - Semiconductors, Ceramics, Metals and Geological materials
  - Requires mechanical pre-preparation of samples
    - Dimpling technique
    - Wedge technique (Tri-Pod / T-Tool) Semiconductors only
- Focused ion beam (FIB) technique
  - Expensive, but advantage is precision
  - Suited primarily to semiconductors for precision cross sections
  - Produces considerable ion beam damage
  - Samples not suitable for HREM without additional cleanup

Dimpling Technique

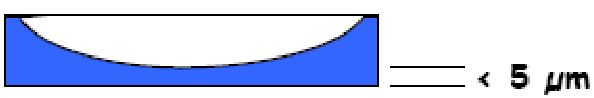
Step 1 Disc Cutting



Step 2 Disc Grinding

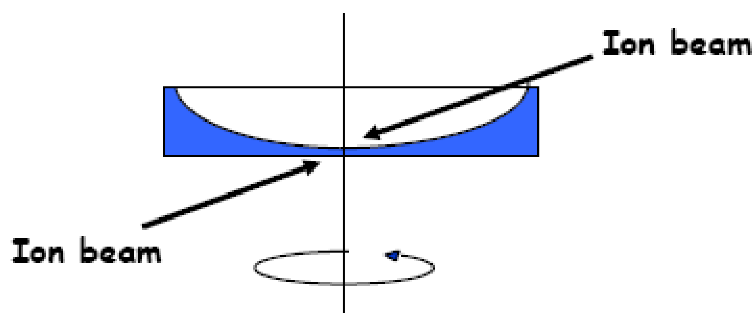


Step 3 Dimple Grinding



Drawings Not to Scale

Step 4 Ion Milling



Step 1 Preparing Discs

- Starting material must be polished, sliced or cleaved to obtain a slab about 500μm in thickness.
- Ultrasonic Cutter
  - For coring or cutting TEM discs from brittle materials (Ceramics and semiconductors)
- Disc Punch
  - Used to punch TEM discs from metals

Ultrasonic Cutter

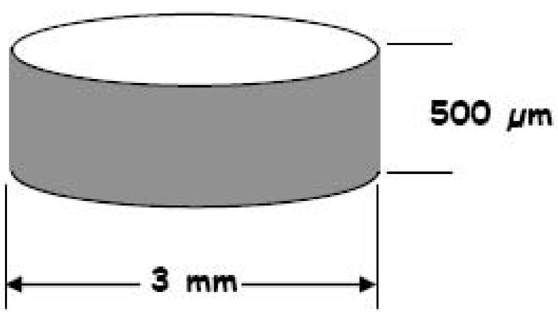
Diamond saw

Disc Punch

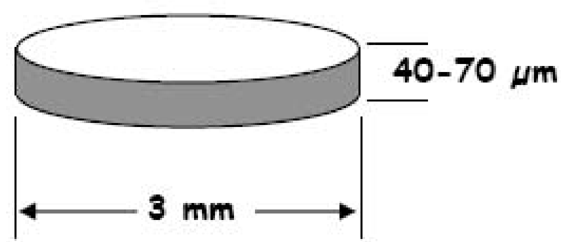
Step 2 Mechanical Pre-thinning

- Disc Grinder
  - Mechanically thins TEM samples prior to dimple grinding, ion milling or electropolishing.
  - Produces high quality parallel-sided thin samples while reducing the chance of sample damage
  - Micrometer dial displays thickness in microns

Starting Disc

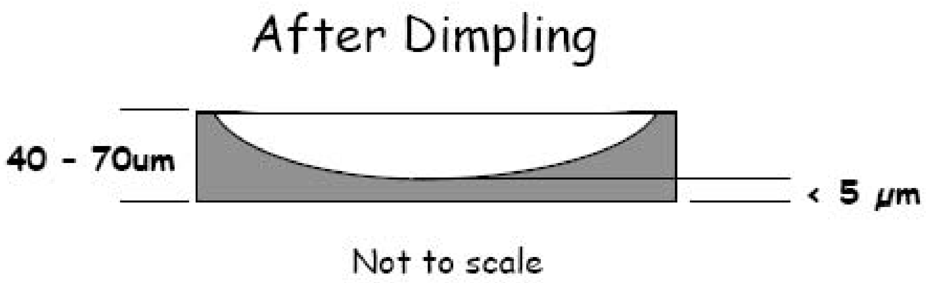


Finished Disc

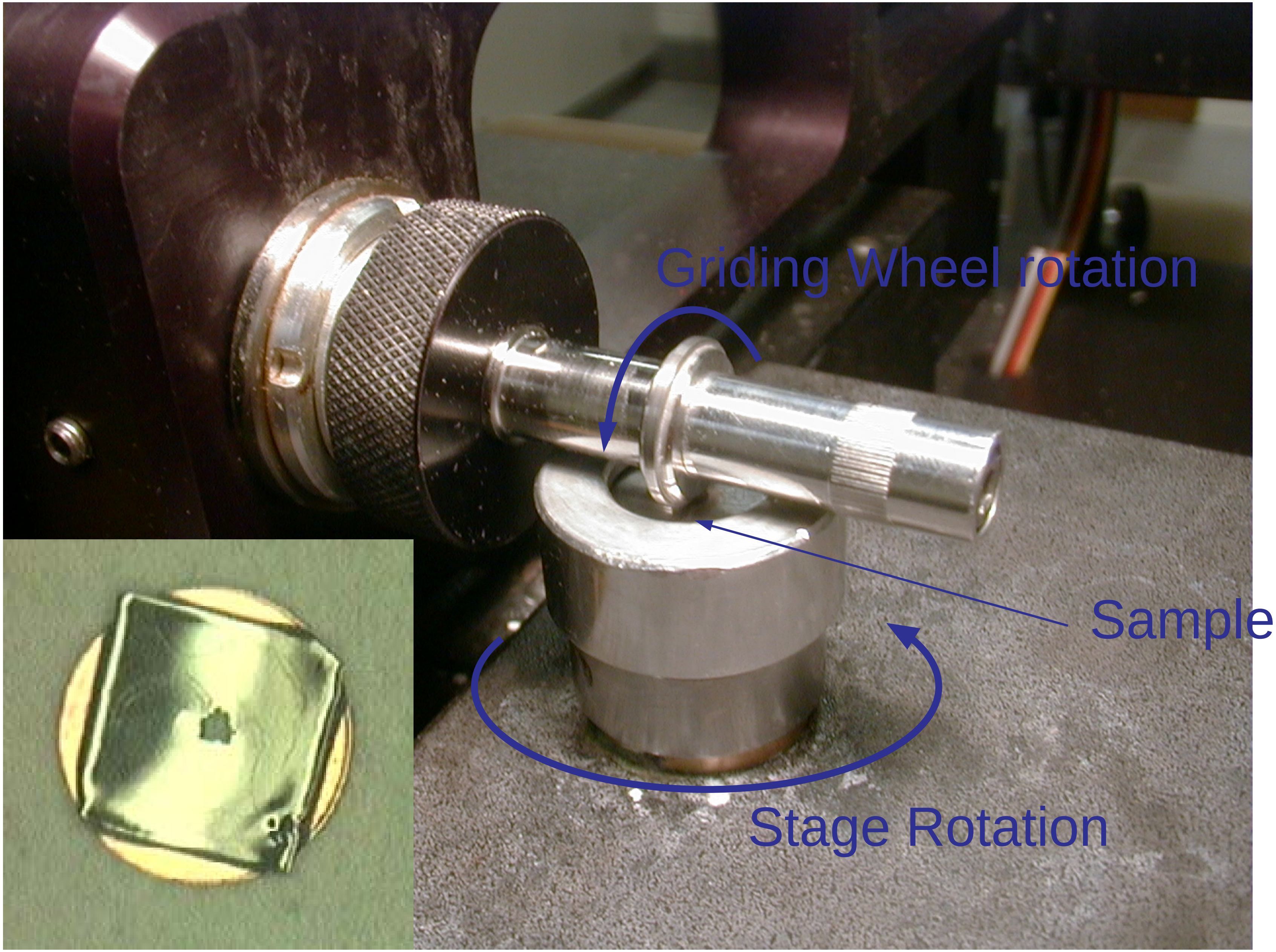


Drawings Not to Scale

Step 3 Dimple Grinding



- Produces large thin area with thick supporting rim
- Reduces ion milling times
- Locates the region of interest to be thinned
- Large thin area in the center surrounded by thick rim helps in handling fragile specimens
- Direct preparation of TEM specimens

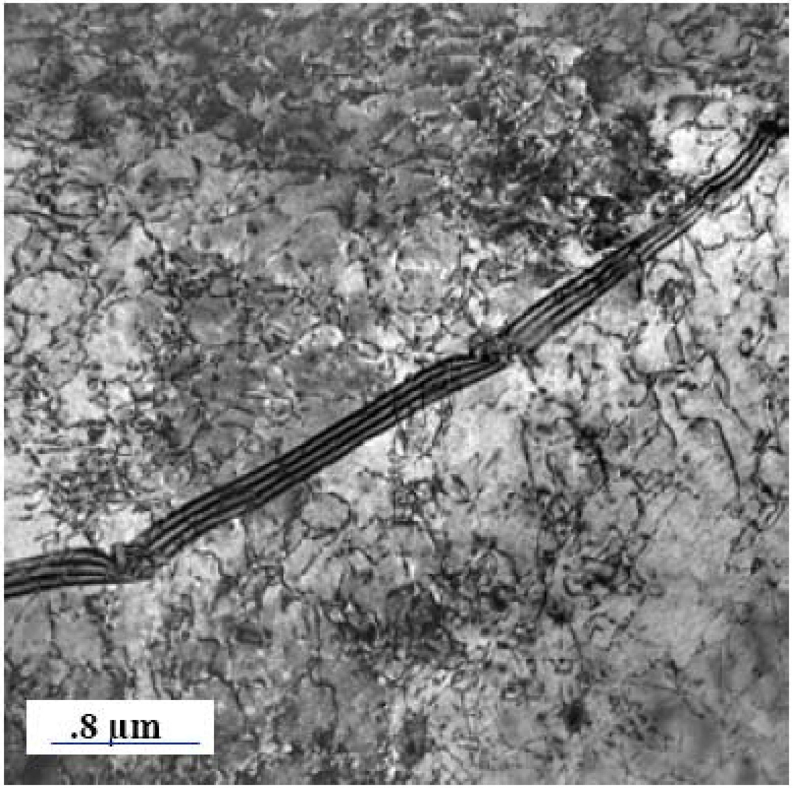

Griding Wheel rotation
Sample
Stage Rotation

Dimple Grinder Application

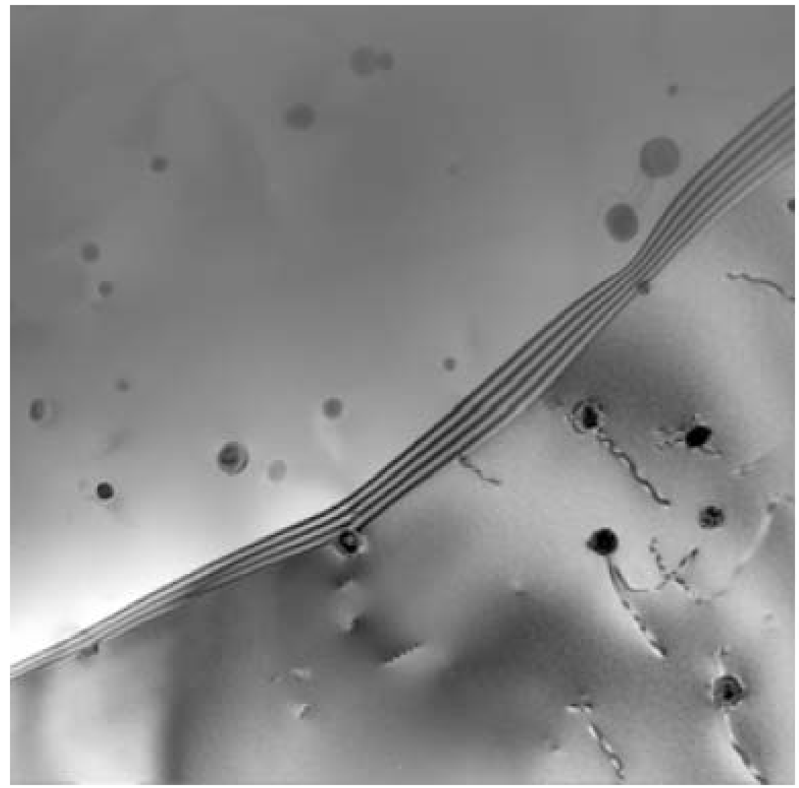
- Material removal rate
  - Typical thinking is always speed or "throughput"
    - Increase the load, increase speed or both
  - Fast removal rate is not always the best solution
    - Increasing load or speed can produce a damaged layer
    - Some hard metals may actually work harden
- The better solution
  - Parallel polish sample to about 60um
  - Course dimple grind to a thickness of 15 - 20um
    - 2 - 4um diamond paste, 20gm load, medium speed
  - Fine dimple grind to a thickness of 5 - 10um
    - 0 - 2um diamond paste, 20gm load, medium speed
  - Fine dimple polish with FELT to a thickness of 5 - 8um
    - 0 - 2um diamond paste, 20gm load, medium speed

Dimple Grinder Applications

Comparison of commercial Al alloy



Over-dimpled

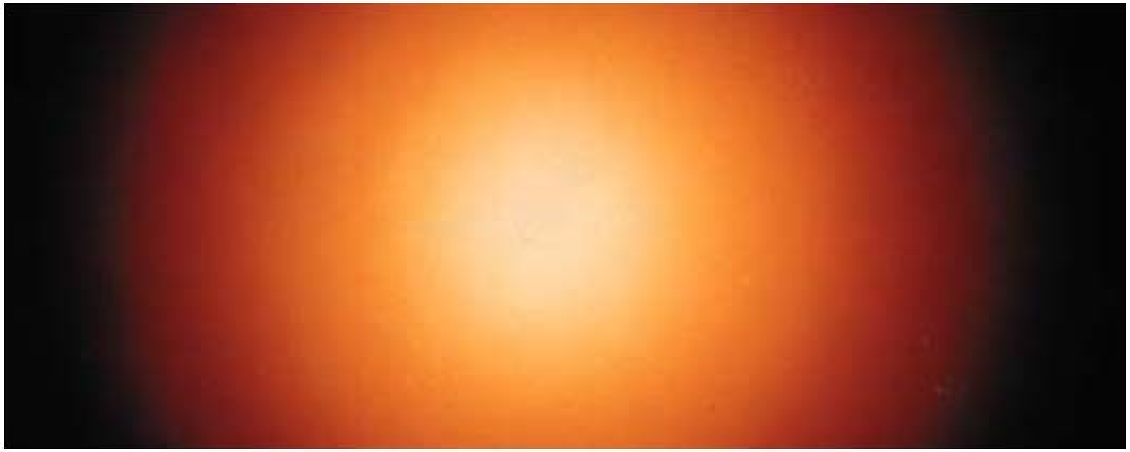


Properly-dimpled

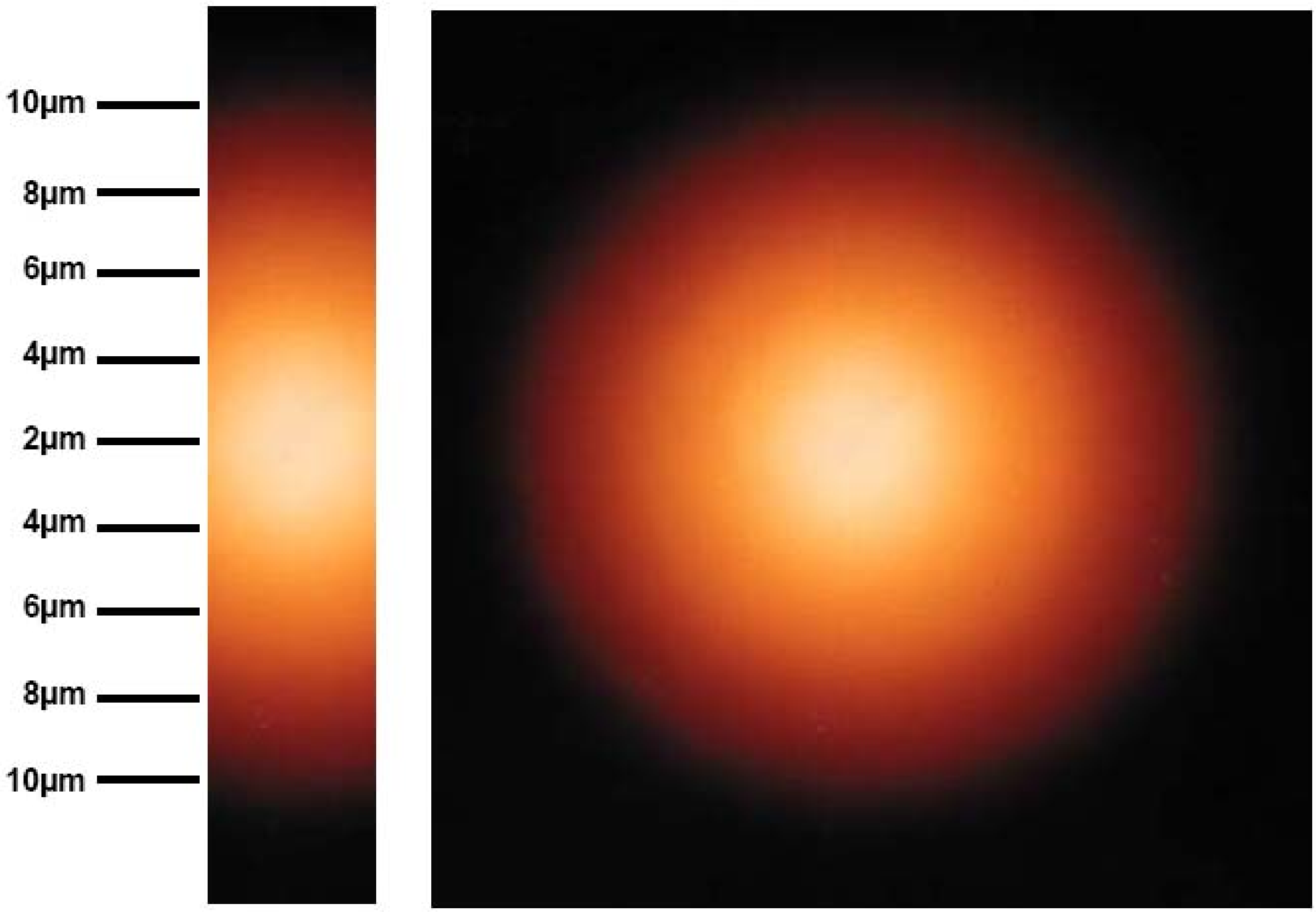
The over-dimpled image shows dislocations
Properly-dimpled image is absent of dislocations
Dislocations caused by excessive load or high speed

Dimple Grinder Application

- Light transparency colors of materials (semiconductors and ceramics) can be used for thickness control
- When Si is used as supporting material for cross-sectioning, the transparency color can conveniently be used for thickness control
- Interference fringes can also be used for thickness control



Dimple Grinder Application



Transparency colors vs. thickness in dimpled Si (100) single crystal