Sample preparation

Lecture 7

Outline

Safety

Why thin?

Standard laboratory methods

- Grind / polish / dimple / ion mill
  - Tripod
  - X-sections
- Electropolish & Electrochemical
- Tripod
- Nano-stuff …

Focused ion beam

Safety

- Lots of chemicals used in specimen preparation
- Some are very dangerous
  - Perchloric acid in ethanol or methanol - explosive
    - Good aluminum and stainless steel etch
    - Best if done in a dedicated fume hood
  - Nitric acid in ethanol - explosive
    - Keep cold, dispose of it quickly
  - Hydrofluoric acid
    - Fantastic way to make silicon samples
    - Extremely dangerous, eats bone, absorption can lead to heart failure, doesn’t ‘burn’ so you can miss it
    - Wash for 15 min in cold water, then Calcium Gluconate solution, then hospital. Be explicit and demanding about being seen quickly
  - Organic solvents (Acetone; 1,1,1, Tricholorethane)
    - Use in a fume hood, carcinogenic
- Dispose of chemicals properly
- Read and follow MSDS’s

http://www.adpc.purdue.edu/PhysFac/rem/hmm/hmm.htm

Sample preparation

In nearly all cases, TEM samples are best when very thin

- 100 Å maximum for interpretable HREM image
  - If thicker, scattering is dynamical, image cannot be interpreted
- 100 - 500 Å for good EELS, EFI, quantifiable EDS
  - If thicker, multiple scattering
  - Fluorescence, absorption, difficulties in interpretation
- 300 - 500 nm for diffraction contrast work
  - If much thicker, dynamical scattering and absorption obscure images

Preparation from the bulk

self-supporting discs of brittle materials

“Plan view” samples

e.g. imaging along the growth direction

1. Section your sample into pieces of order 250 μm thick
2. Create a 3 mm disc
   - Ultrasonic cutter - ceramics, semiconductors
   - Drill coring tool - ceramics, semiconductors
   - Or scribe to 2.8 mm x 2.8 mm square (if thin film poorly bonded)
3. Grind to 100 μm or so
4. “Dimple” to 10 - 20 μm
5. Ion mill

Preparation from the bulk

Cross sections of thin films on substrates

“Cross section” samples

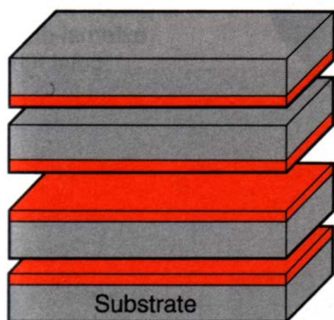
e.g. imaging perpendicular to the growth direction

1. Cleave two 2.5 mm x 5 mm pieces
2. Glue these together with the growth surfaces facing each other (M-Bond or G-1 epoxies)
3. Use a diamond saw to section these into 150 to 200 μm pieces
4. Polish one side first with 320, then 600 grit sandpaper
5. Mount on a Cu 2mm slot grid
6. Polish first with 320, then 600 grit to < 20 μm
7. Ion mill

Preparation from the bulk

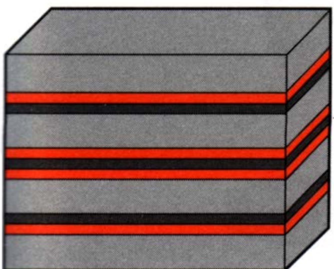
Cross sections of thin films on substrates

(1)



Thin film

Four pieces of a specimen formed from thin film(s) on a substrate

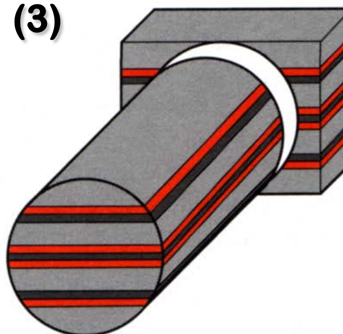


Glue

The four pieces are glued together (*face-to-face* and *face-to-back*) to form a cross-section

(2)

(3)

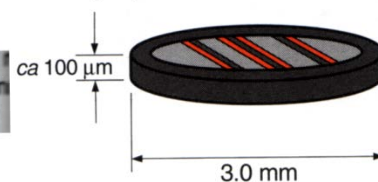


A 2.8 mm diameter piece is drilled from the cross-section

(4)

The 2.8 mm diameter is placed within a 3 m external-diameter metal tube

Dimple, then ion mill from here



Thin slices are cut fr the rod, and mechanic thinned to *ca* 100 µ

(5)

Sample preparation
argon ion milling

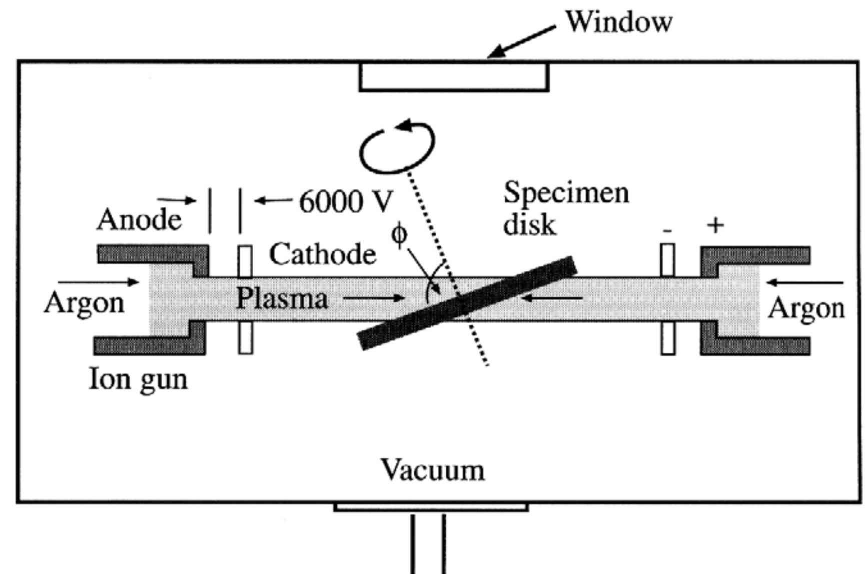
Controlled sputtering using energetic argon ions

Incident at between 100V and 6 kV

Control angle of incidence, current and voltage

Maximum thinning rate at around 14° - 16°

Final thinning done at lower angles, currents and voltages



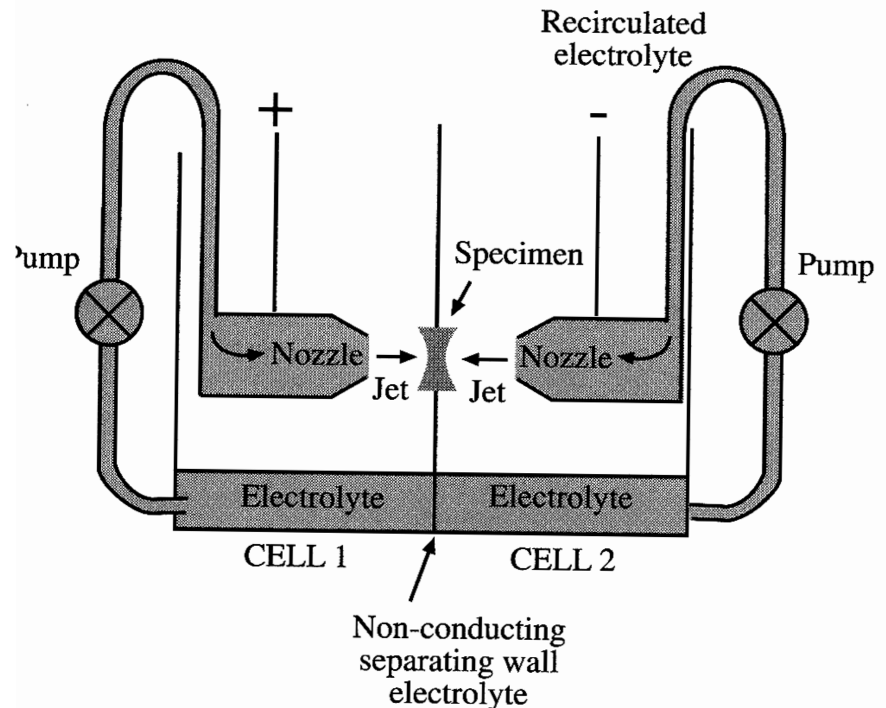
Sample preparation
Electropolishing / Electrochemical

Used to thin metals:

- Does not introduce deformation.
- A electrolytic solution is directed at sample. Voltage across anode / cathode varied.

A variant is used to prepare semiconductors

- No voltage drop used, just an acid mixture.
- Excellent methods exist for Si, SiGe, GaAs, other III-V's
  - Not III-N's, though



Sample preparation

other techniques / terms

Tripod polishing:

- Very controlled method of grinding a sample to close electron transparency or close, followed by ion mill

Nanoparticles / Nanotubes / Nanowires:

- Disperse in methanol, drop onto a thin carbon web.
- Can also embed particulates in harder medium, and grind / dimple / ion mill

Cleavage:

- Controlled fracture across a known cleavage plane.
- Only effective in certain cases

Replica:

- Old technique
- Coat surface with carbon, sputter heavey metal on to this at oblique angle, remove carbon
- Fracture surfaces, particle counts

Focused ion beam microscopy

Liquid metal ion source (LMIS)

Ga^+ ions incident on sample

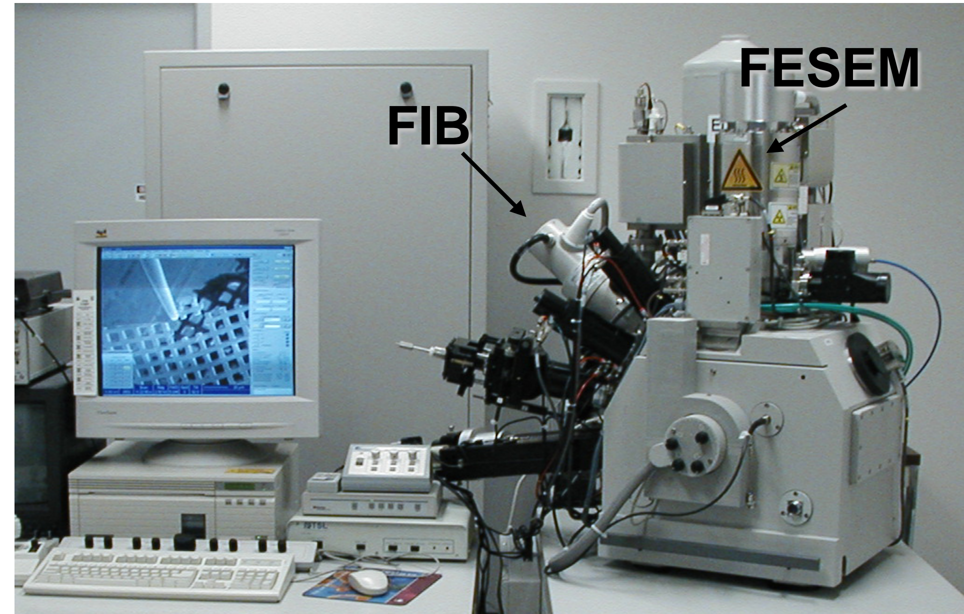
- Causes sputtering
- Causes, SE, BSE, SI emission

Rate of sputter proportional to current

- Ranges from 1 pA to 20 nA

Superb for:

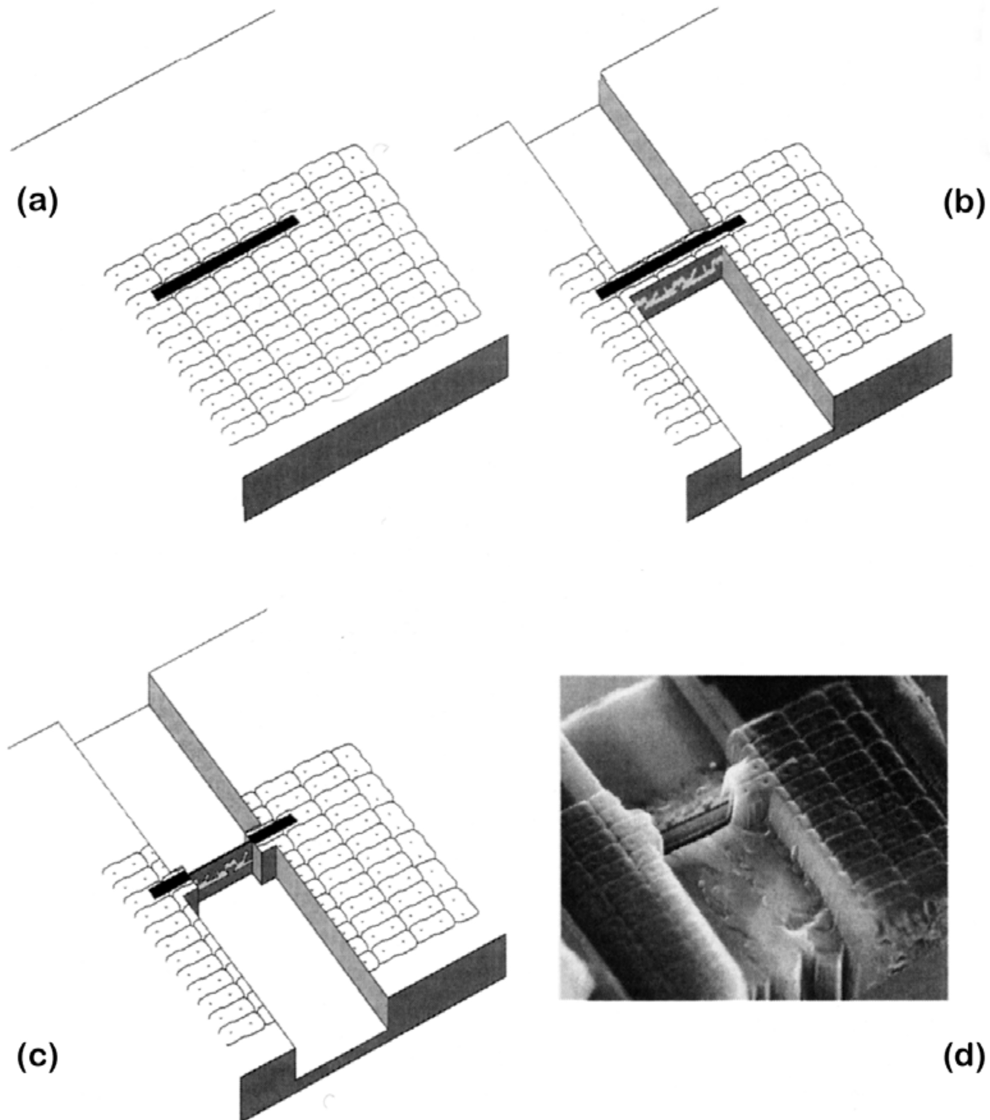
- Site specific preparation
- Cross sections & plan views of 'difficult' samples
- Cross sections samples with highly different hardness / ion sputtering rate



FEI DualBeam FIB / SEM

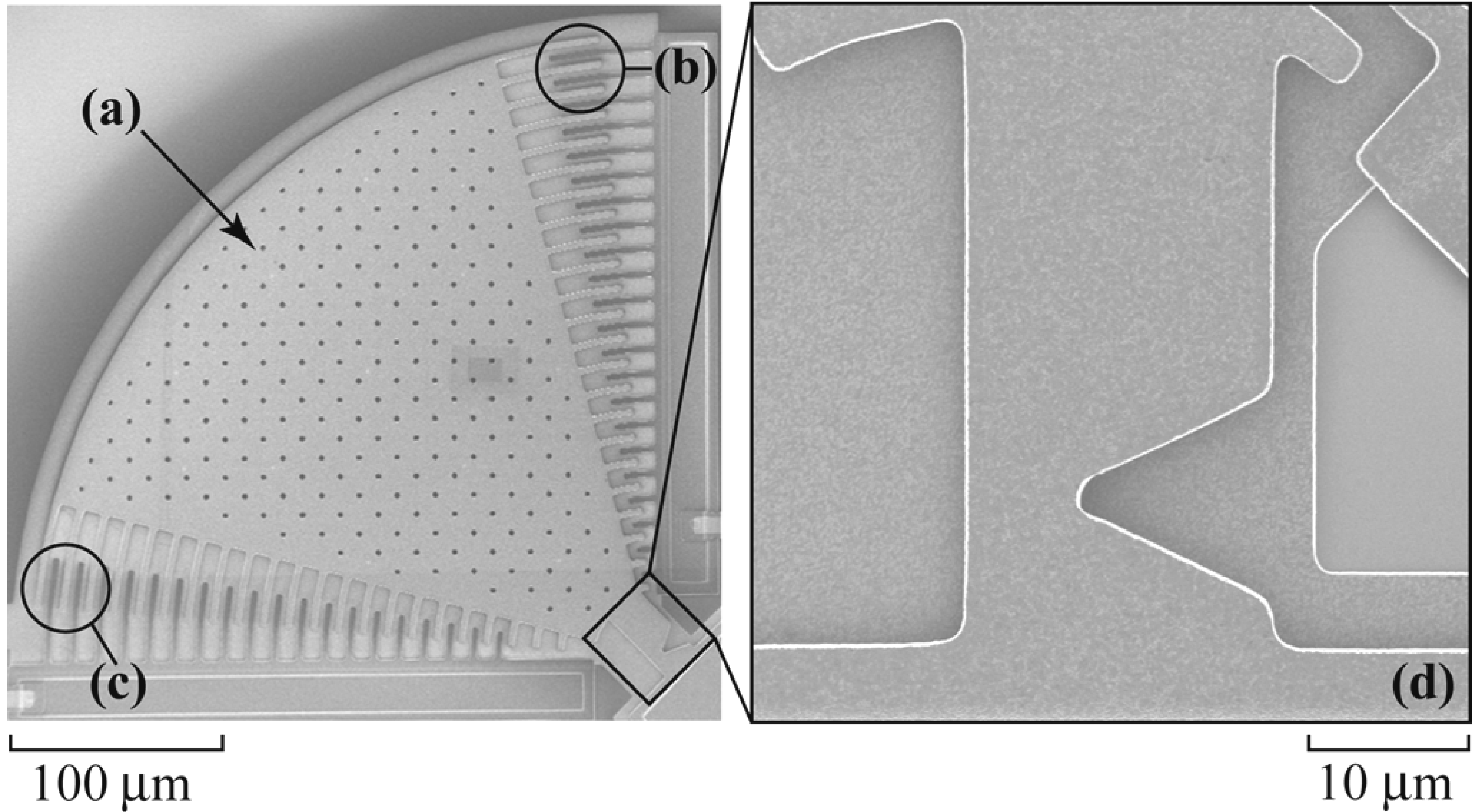
Focused ion beam

“H bar” method



Focused ion beam

site specific prep



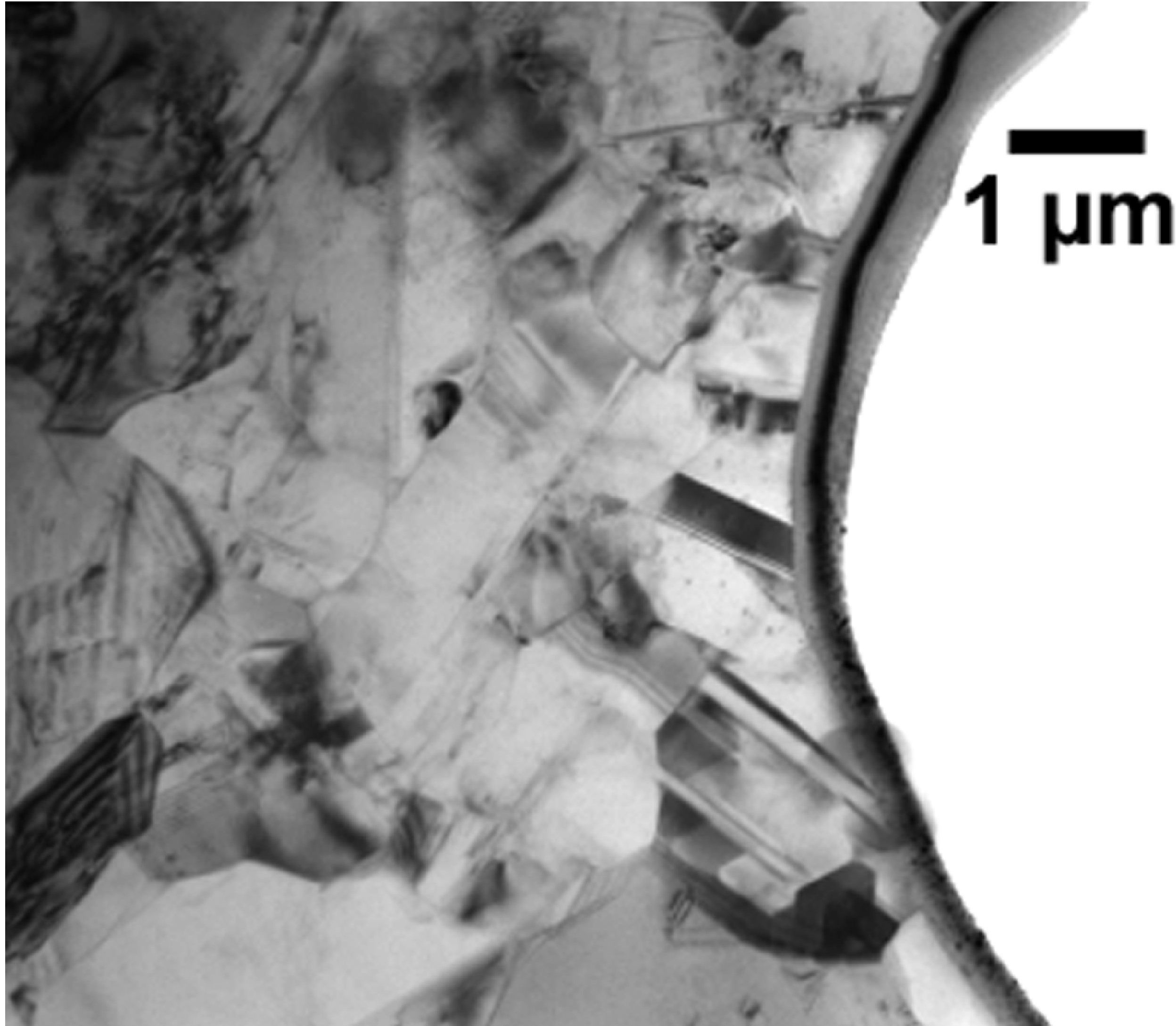
Focused ion beam

site specific prep

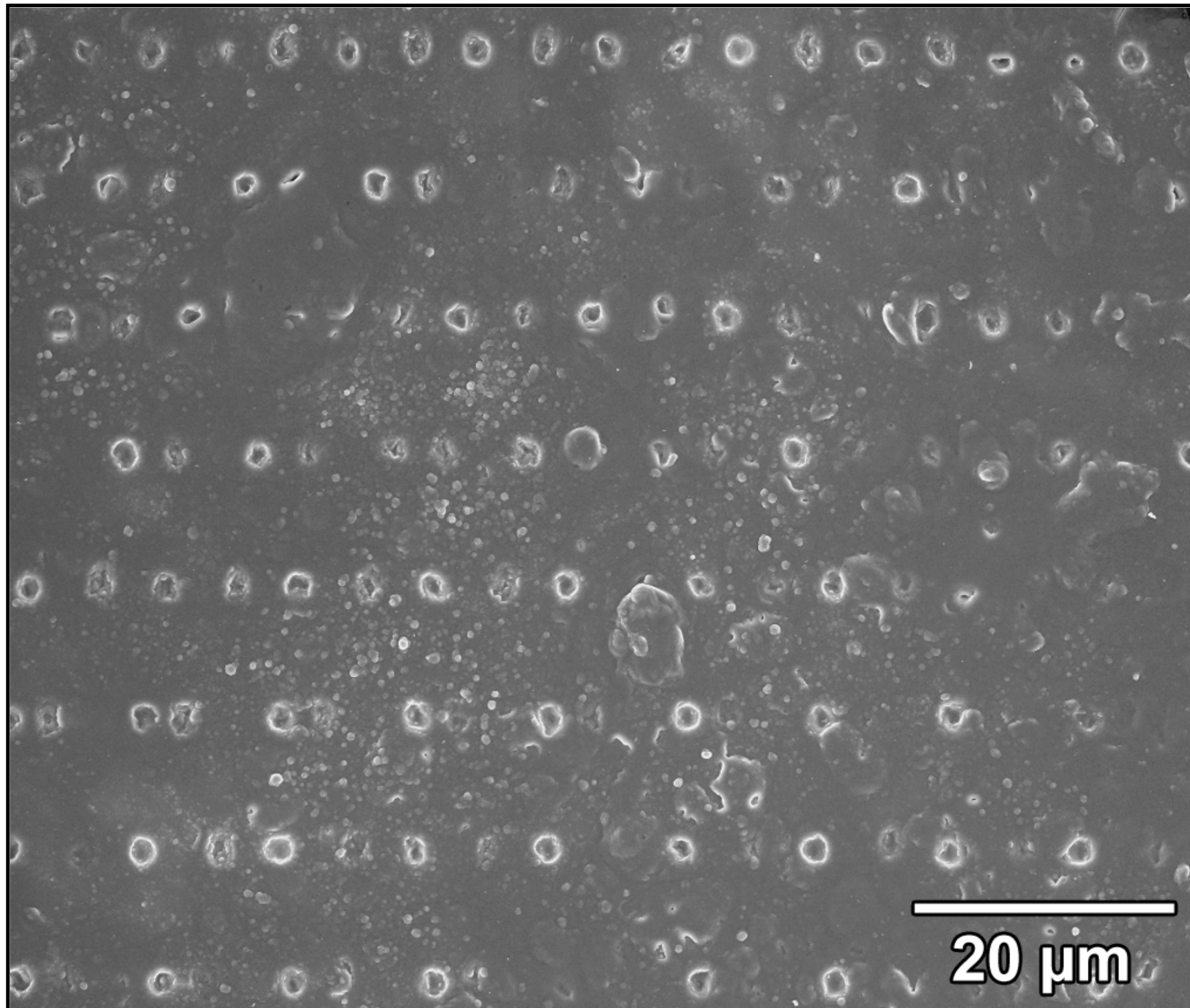
QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

Focused ion beam

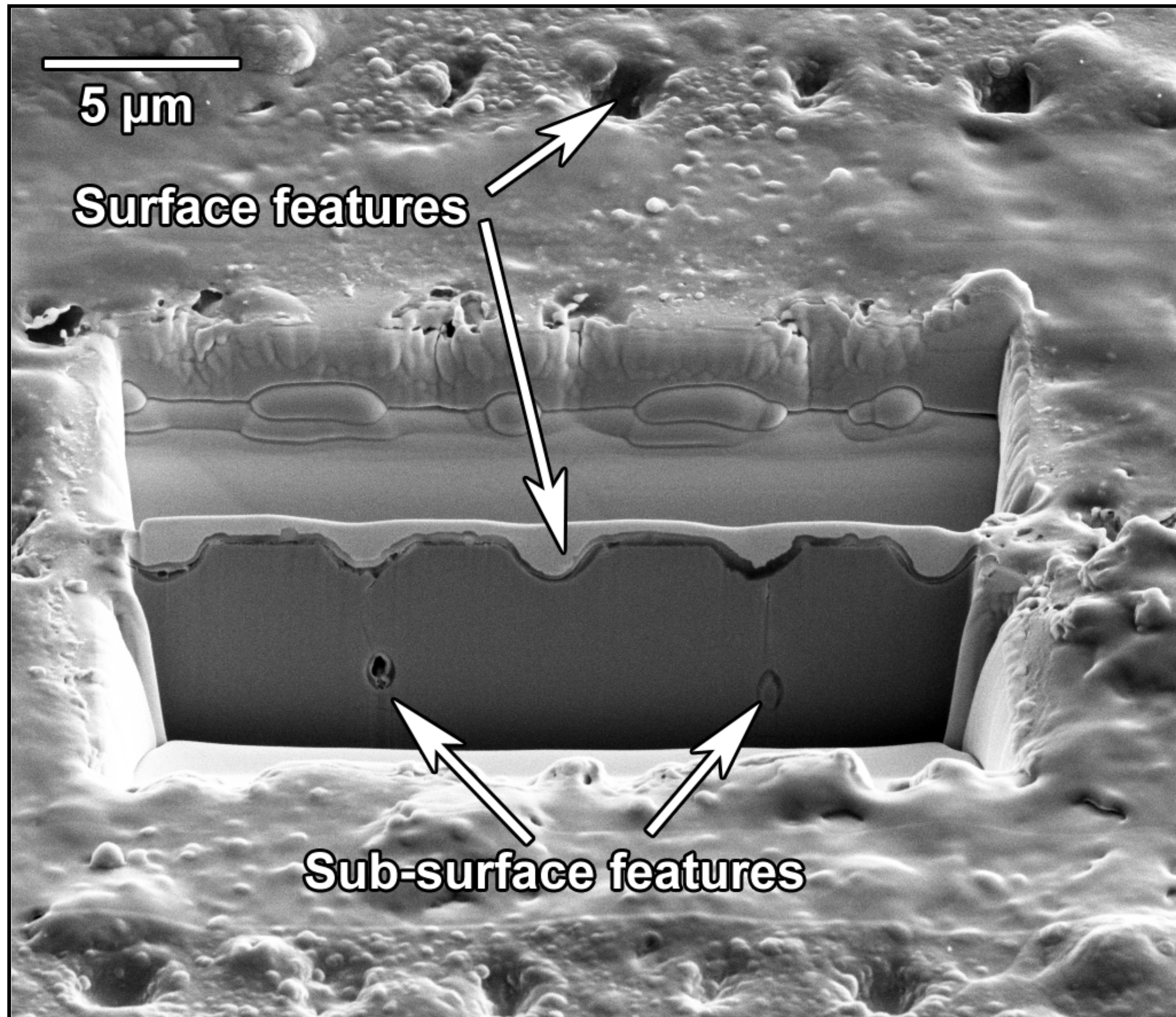
site specific prep



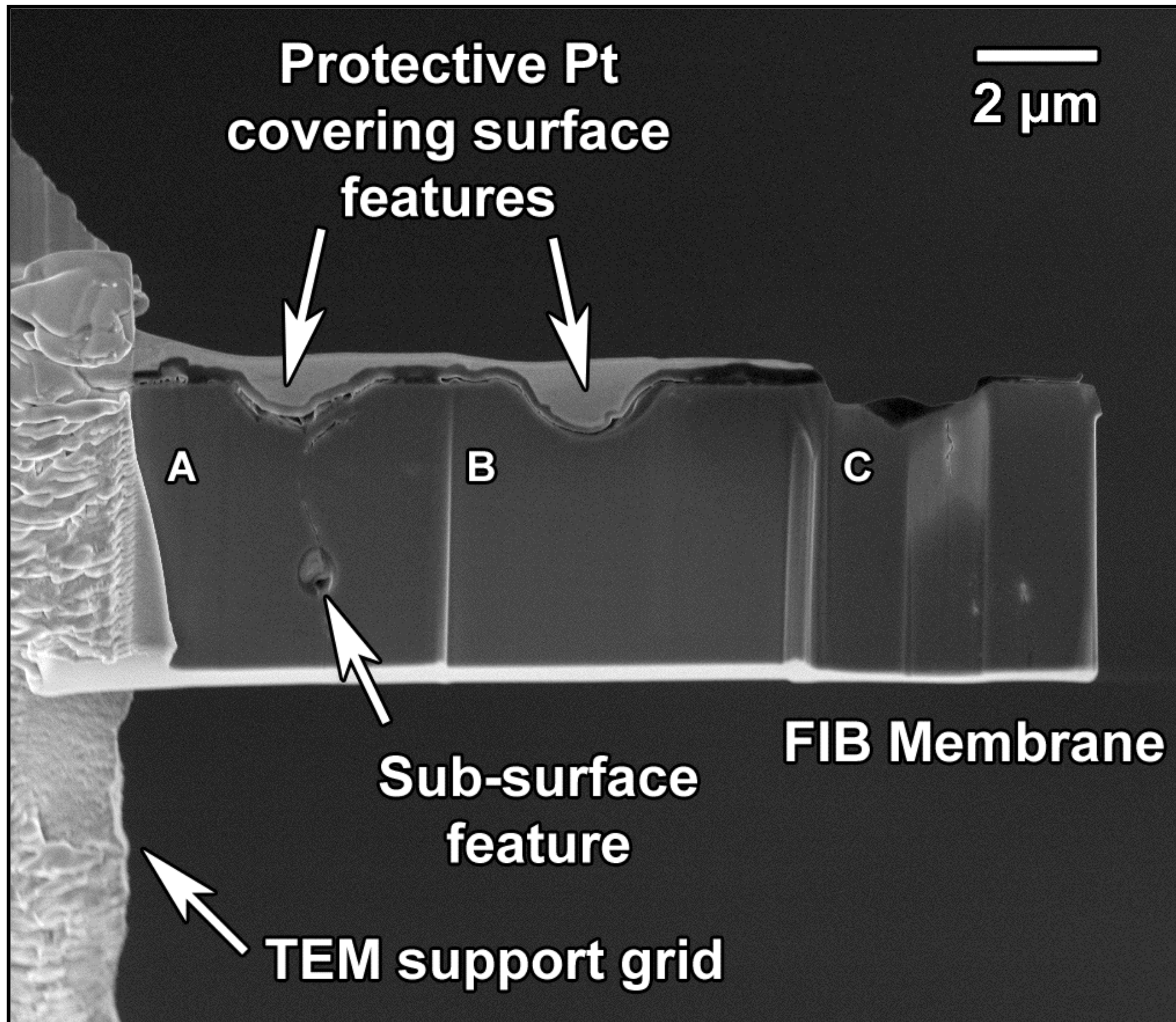
Surface features



Surface features



Surface features



Surface features