

Flat and Cross-Sectional Milling with the New Hitachi IM4000II Ion Mill

Krysten Villalon, PhD, EPIC-SEM Core Scientist

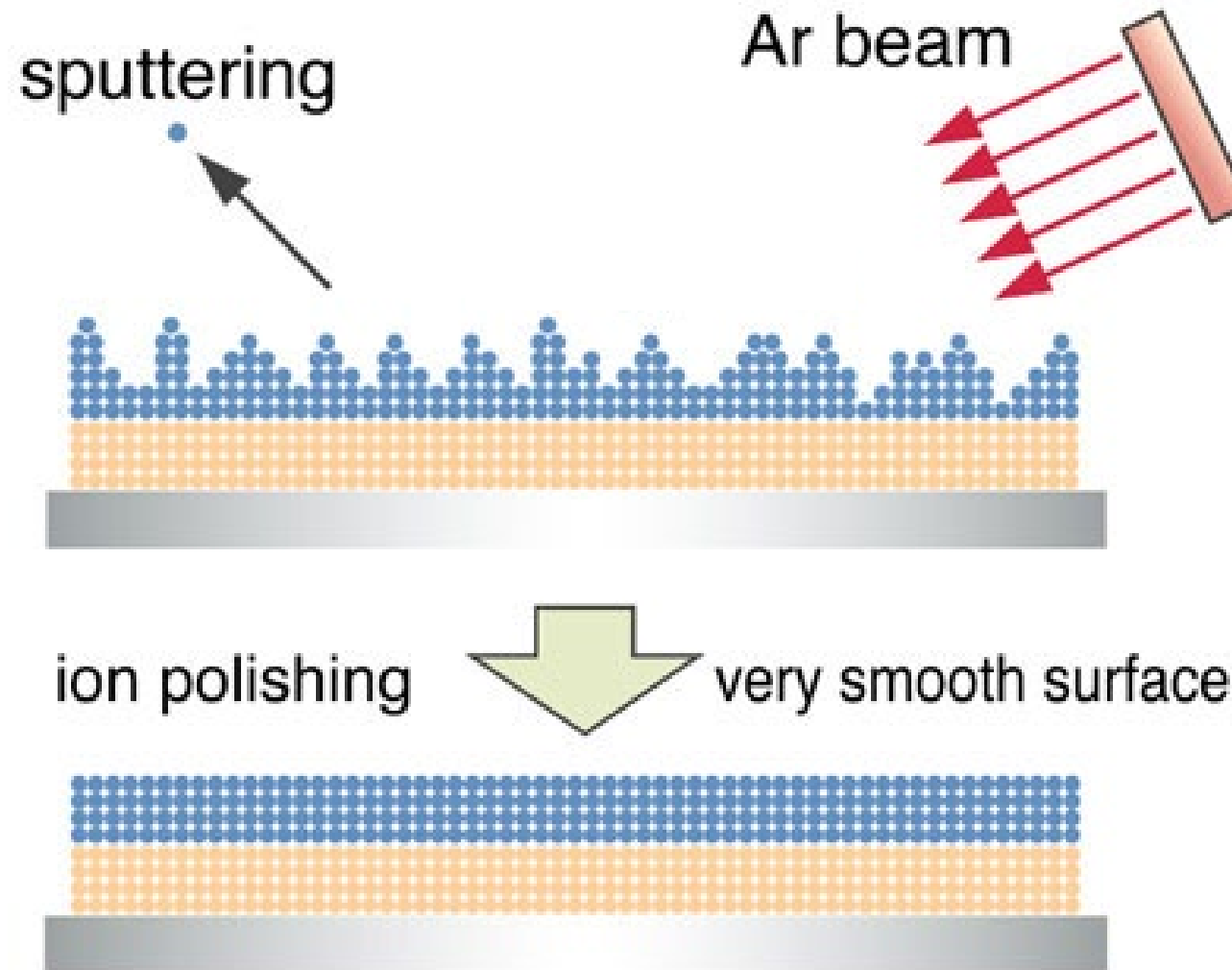
Ion Milling

What is it?

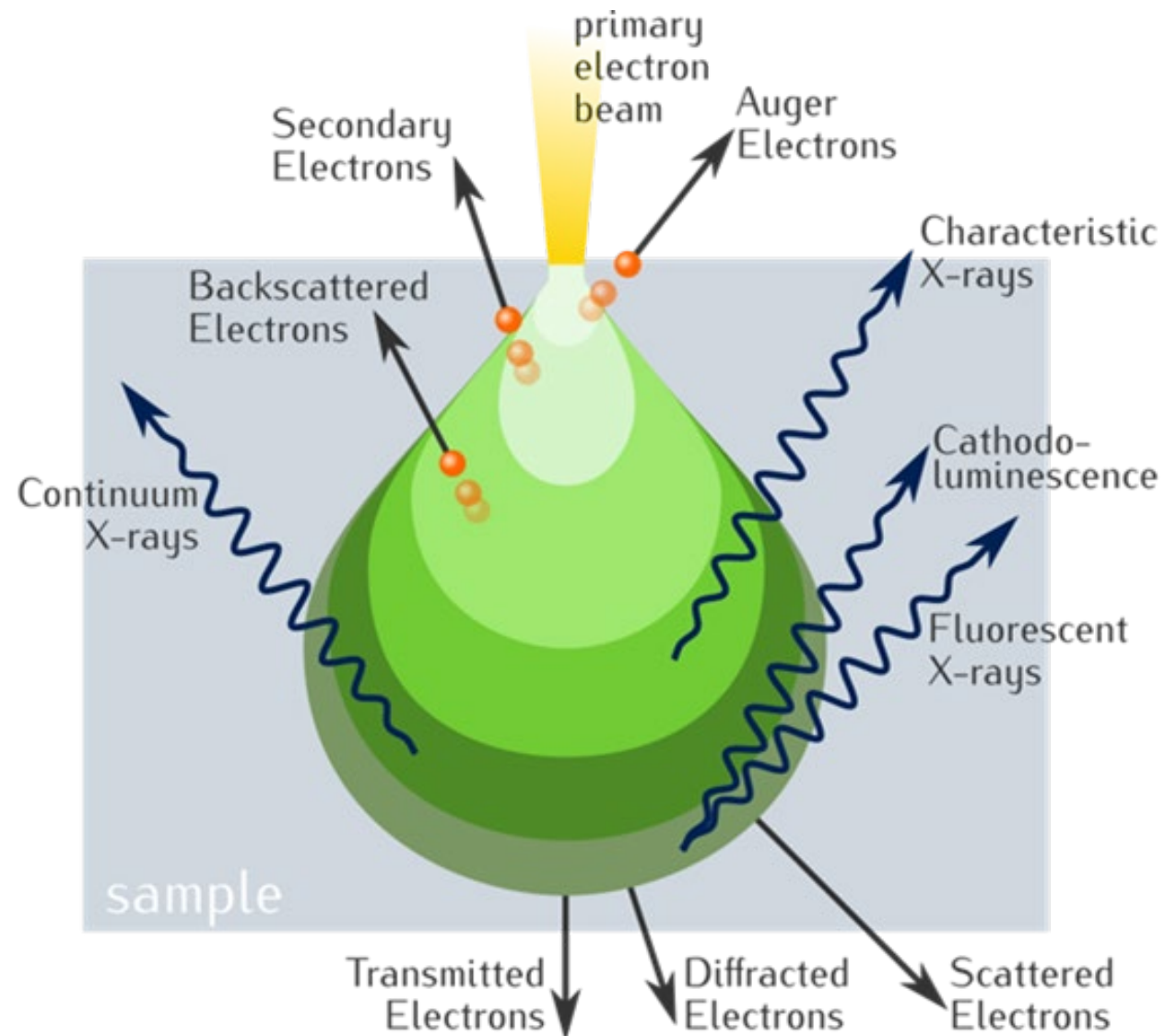
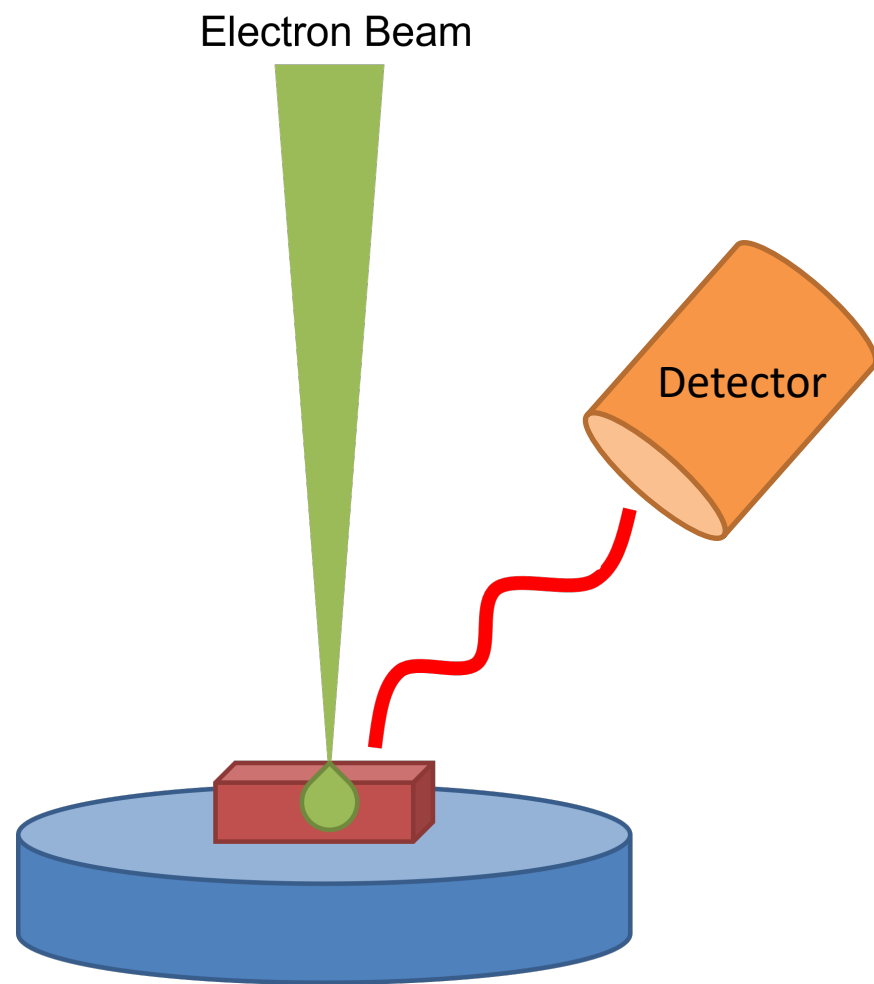
- **Ion milling**: a general term for sample preparation methods that involve removing sample material by bombarding the surface with ions, in a process called **sputtering**.
- An **ion mill** typically refers to instruments that use a **broad** beam of **Argon** ions to gently smooth sample surfaces with minimal defects or artifacts compared to other techniques. Broad Ion Beam (BIB)

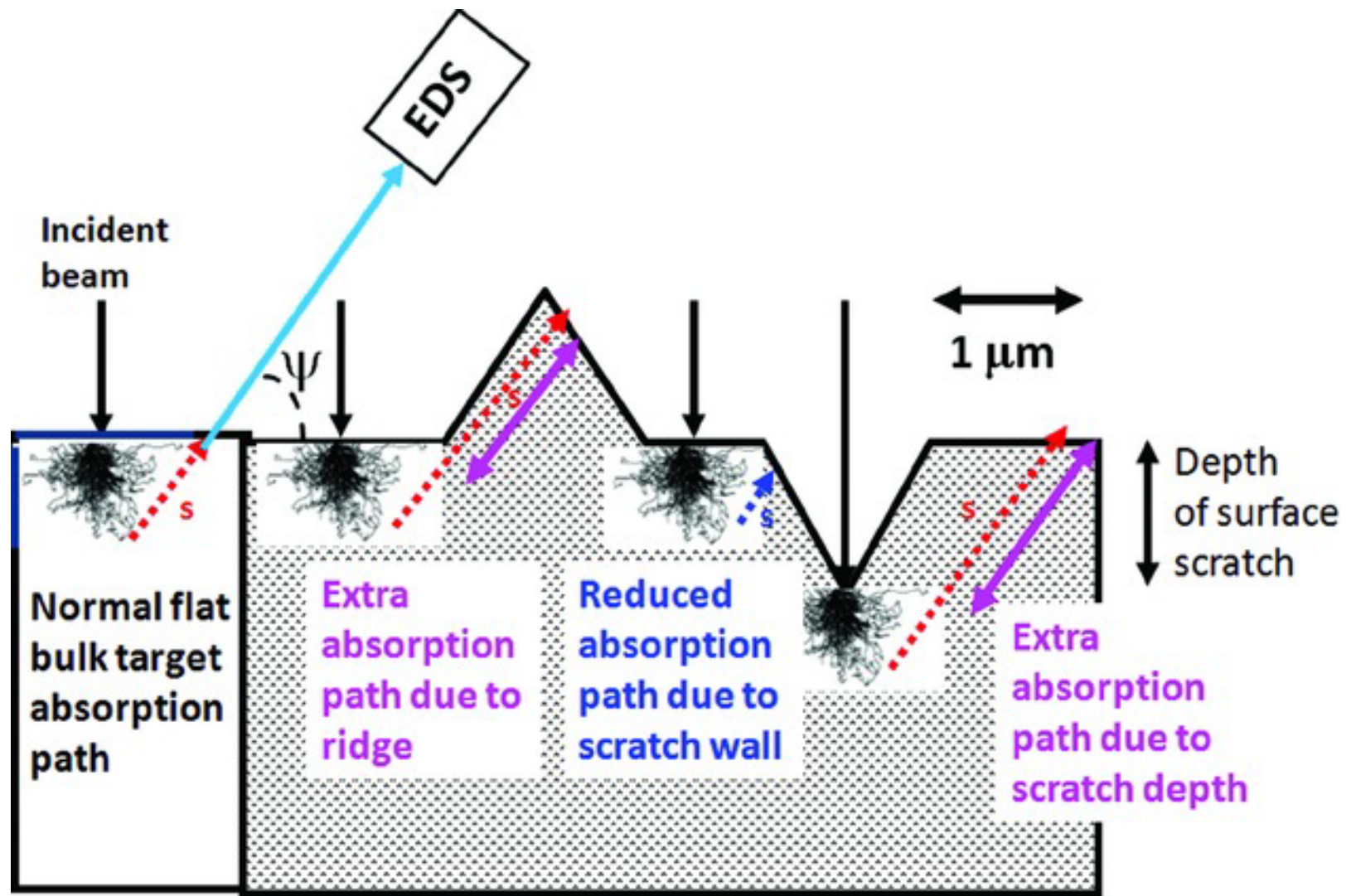


Hitachi IM4000II

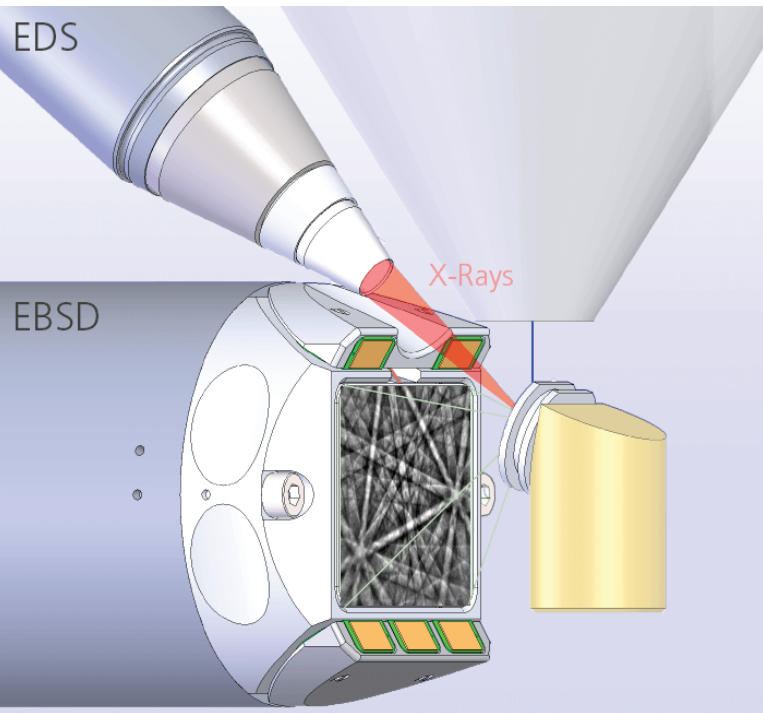


Img: Japan Atomic Energy Research Institute





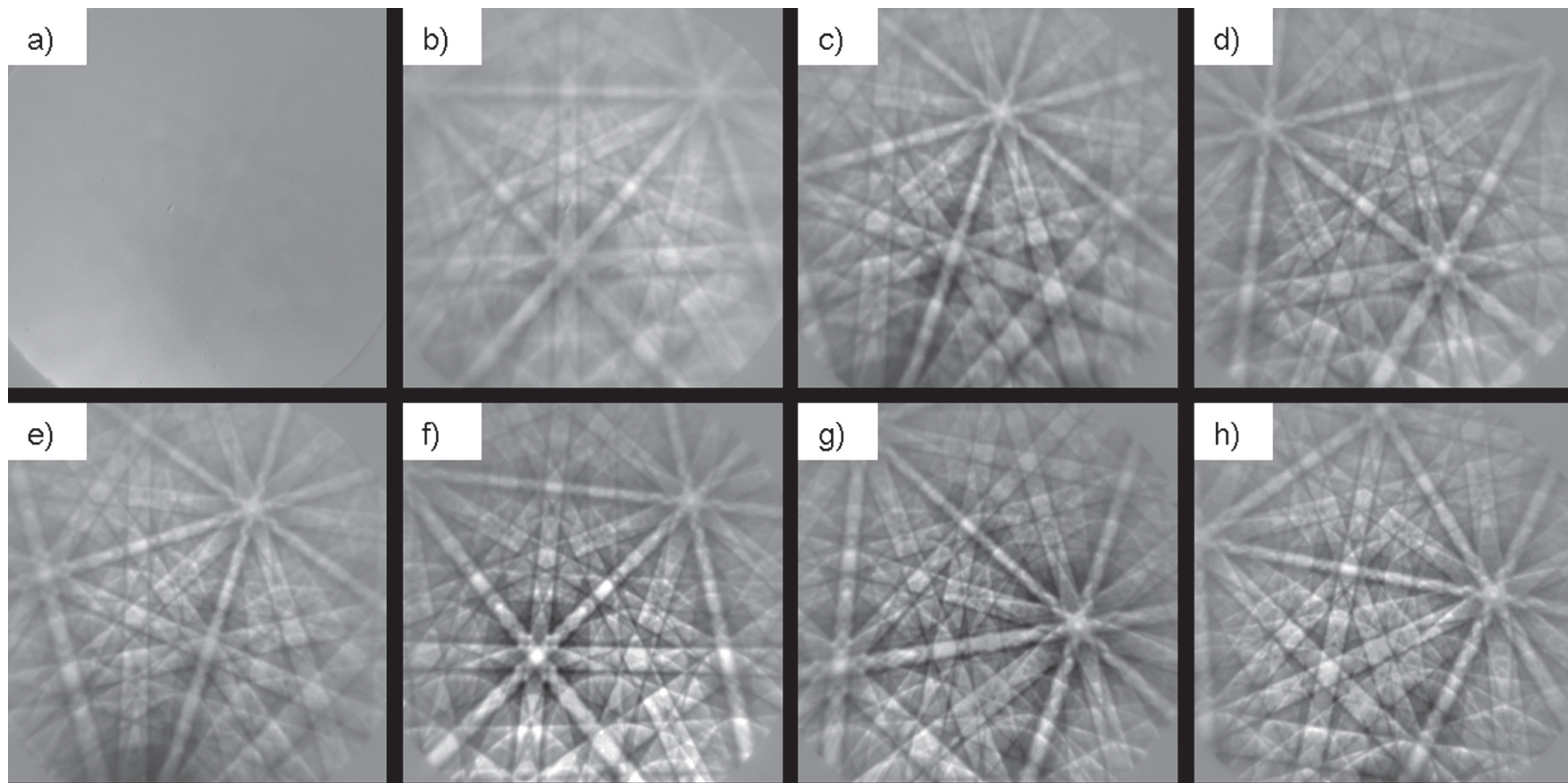
Newbury and Ritchie 2012

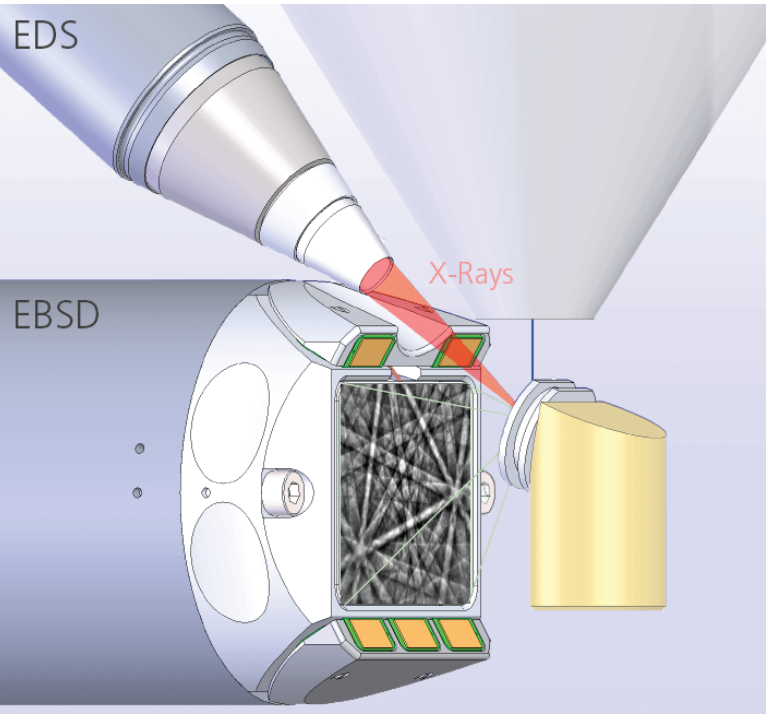


Oxford Instruments

EBSD Pattern Quality vs Surface Quality

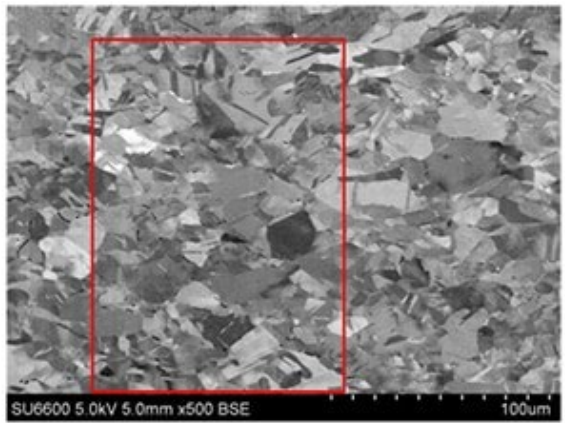
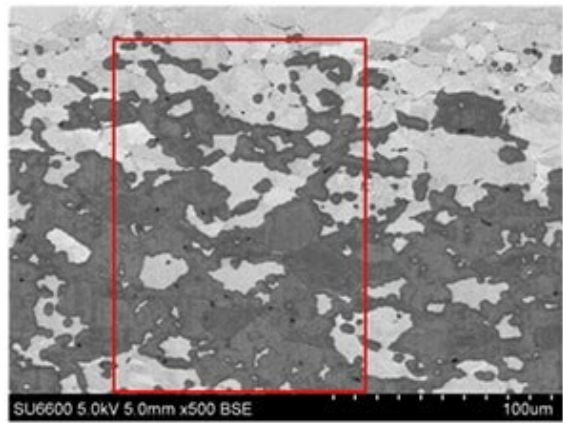
Nowell et al. 2005



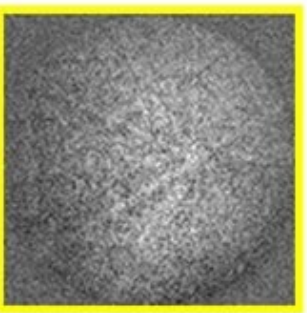


Oxford Instruments

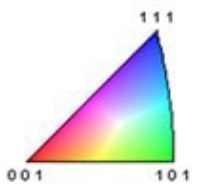
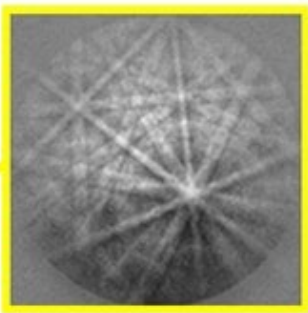
EBSD Pattern Quality vs Surface Quality



Before ion milling



After ion milling



Hitachi High Tech

Sample preparation methods

Cutting/Slicing

Cleaving

Grinding / Polishing

Microtomy

Focused Ion Beam
(FIB)

Broad Ion Beam
(BIB)

Sample preparation methods

Cutting/Slicing

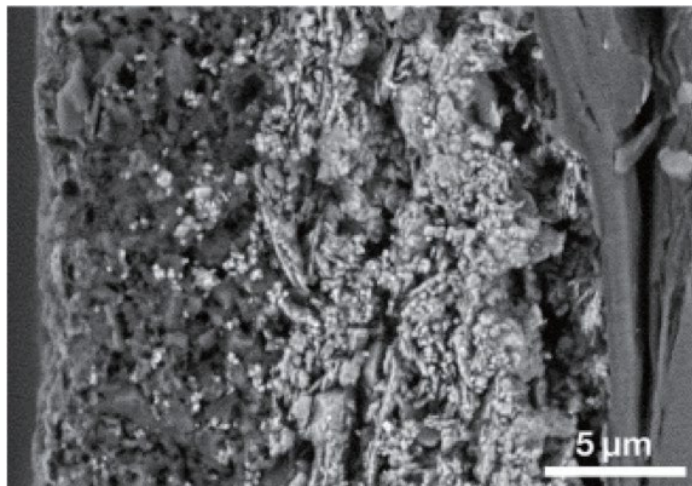
Cleaving

Grinding / Polishing

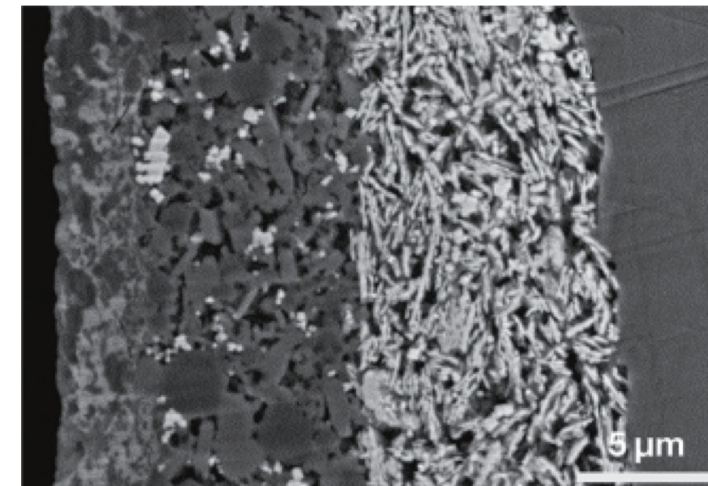
Microtomy

Focused Ion Beam
(FIB)

Broad Ion Beam
(BIB)



Razor blade



Ion Mill

Hitachi High Tech

Sample preparation methods

Cutting/Slicing

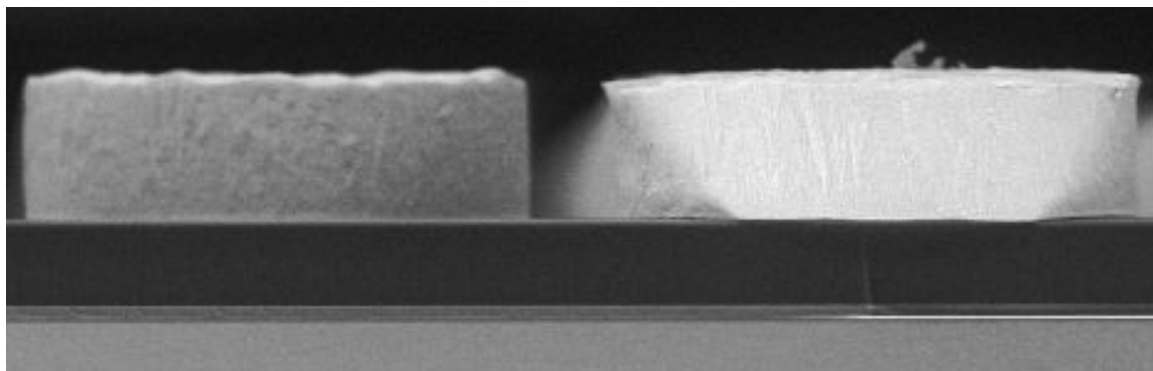
Cleaving

Grinding / Polishing

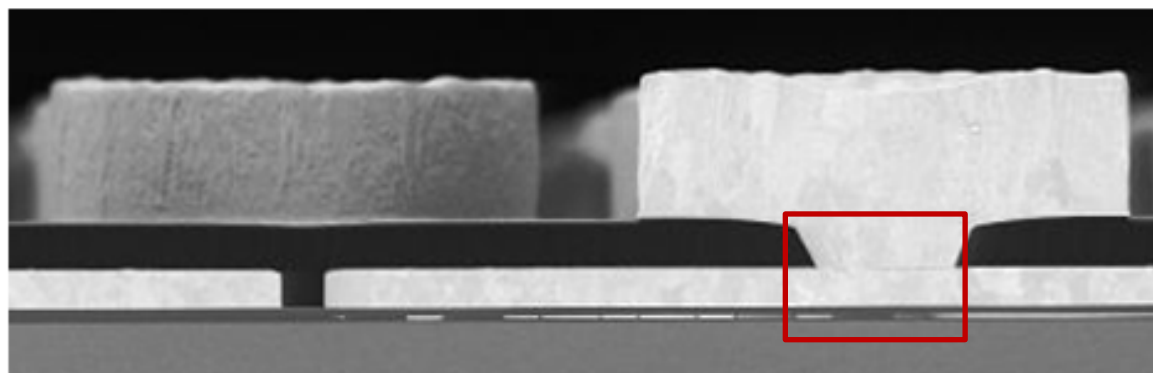
Microtomy

Focused Ion Beam
(FIB)

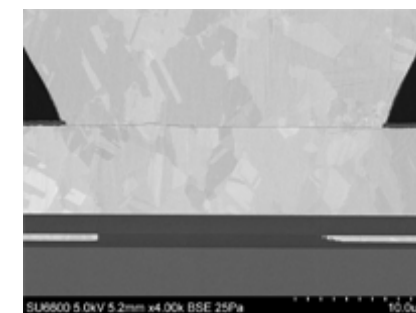
Broad Ion Beam
(BIB)



Cleaved



Ion Mill



<https://www.latticegear.com>

Sample preparation methods

Cutting/Slicing

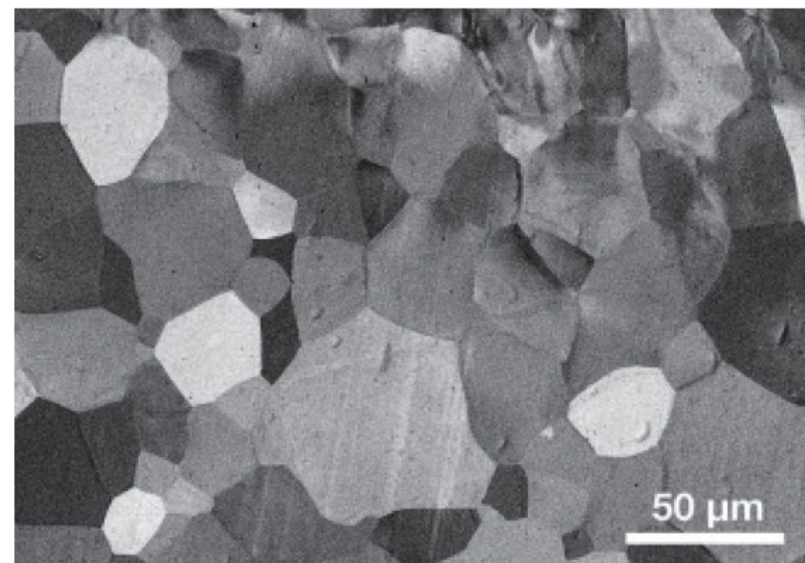
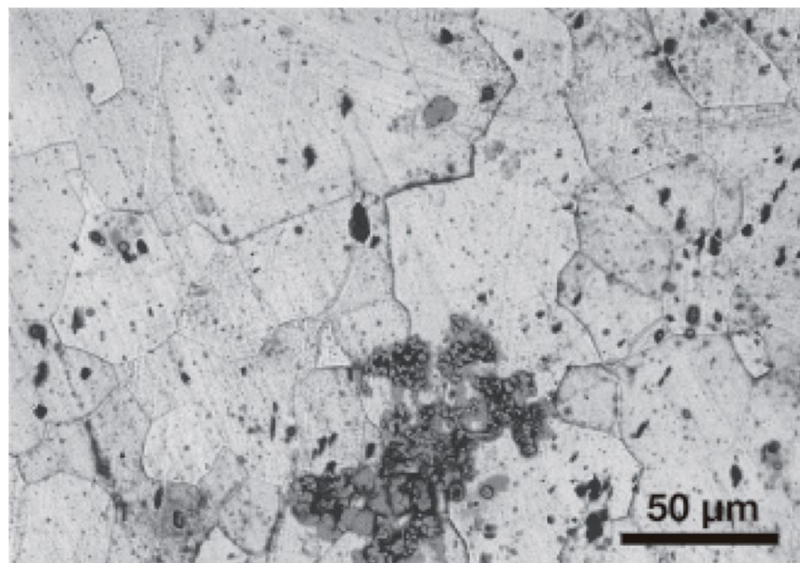
Cleaving

Grinding / Polishing

Microtomy

Focused Ion Beam
(FIB)

Broad Ion Beam
(BIB)



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Sample preparation methods

Cutting/Slicing

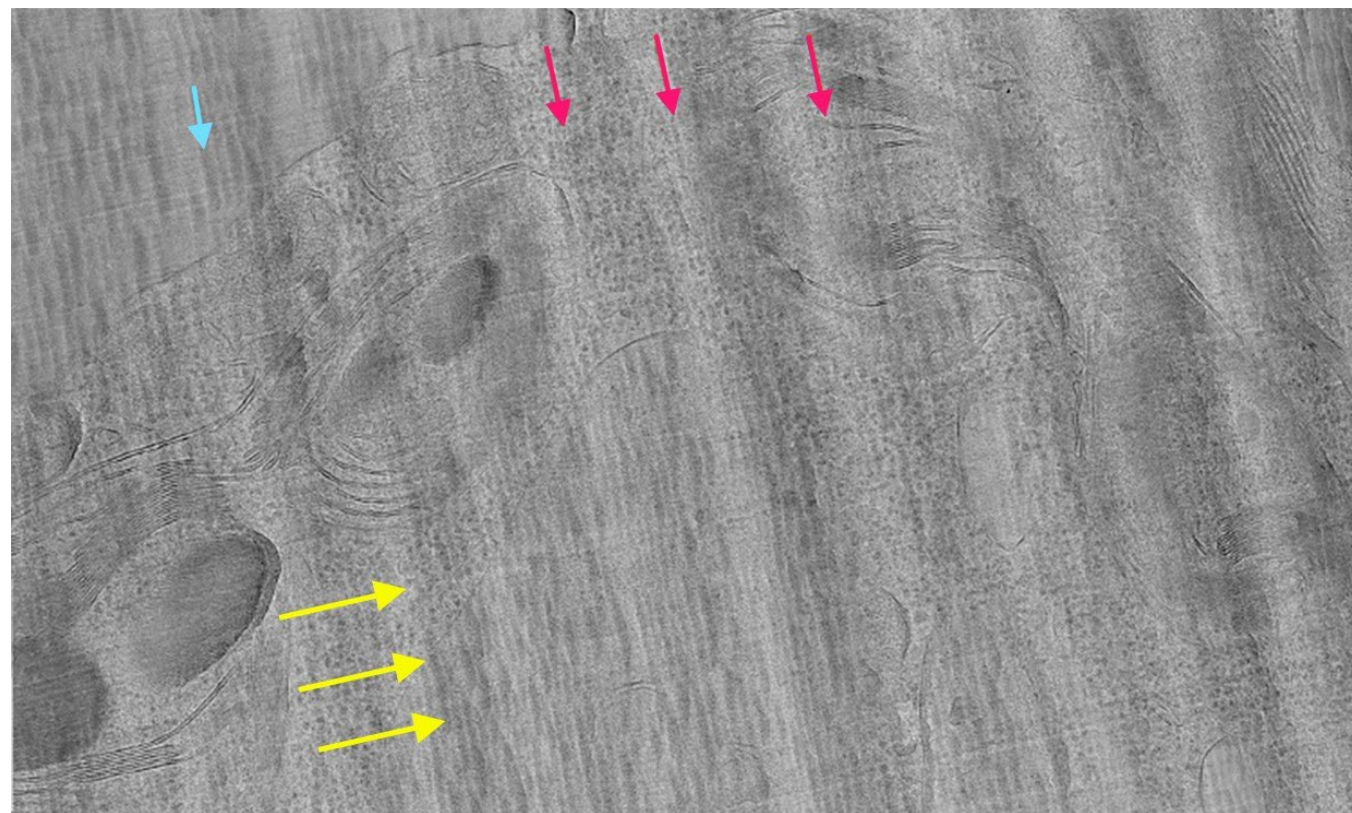
Cleaving

Grinding / Polishing

Microtomy

Focused Ion Beam
(FIB)

Broad Ion Beam
(BIB)



Microscopy Australia, Myscope.Training

Sample preparation methods

Cutting/Slicing

Cleaving

Grinding / Polishing

Microtomy

**Focused Ion Beam
(FIB)**

**Broad Ion Beam
(BIB)**

Sample preparation methods

Focused Ion Beam (FIB)



Helios 5 Hydra CX PFIB

Fischione 1040 NanoMill



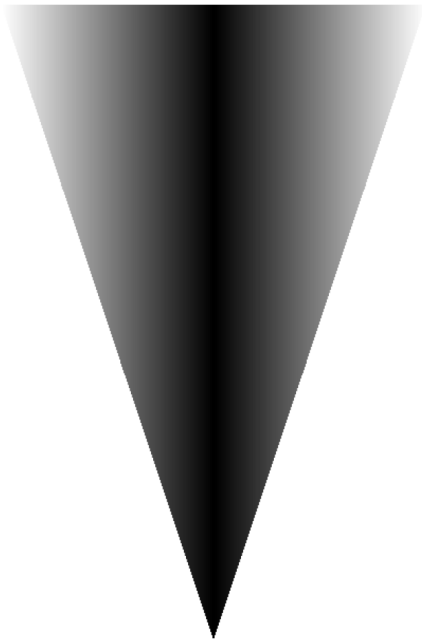
Broad Ion Beam (BIB)



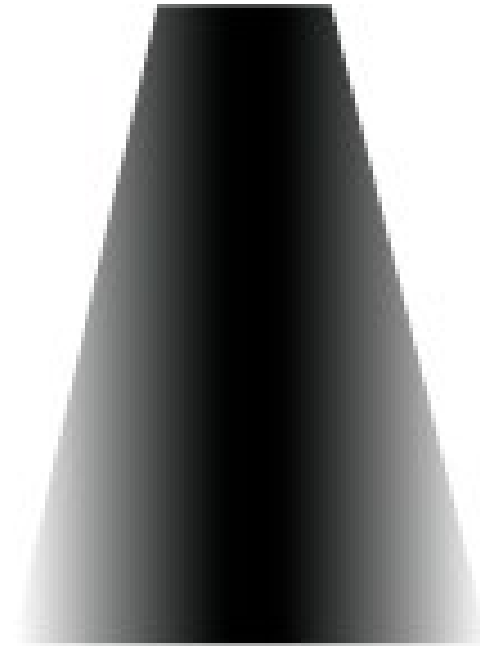
Hitachi IM4000II

Sample preparation methods

Focused Ion Beam
(FIB)



Broad Ion Beam
(BIB)



Sample preparation methods

**Focused Ion Beam
(FIB)**

**Broad Ion Beam
(BIB)**

Spot sizes	nm-regime, converged beam	mm regime, broad, near parallel beam.
Current	lower current/ higher current density	higher current, lower current density
Acc. Voltage	Usually 30kV, lower for cleaning	Usually < 10kV

Sample preparation methods

Focused Ion Beam (FIB)

- High site specificity
- Relatively slow milling rates
- Implantation damage, amorphization
 - low kV clean up important! (Nanomill)
- Usually Ga, also Xe, O, N, and Ar options and more.
- **Great for:**
 - TEM liftouts
 - Atom probe prep
 - nanomachining,
 - 3D tomography/EBSD
 - Cross-sections (tens of μm^2 , site specific)

Broad Ion Beam (BIB)

- Low site specificity.
- Relatively fast milling rates,
- Minimal damage
- Usually Ar ions, others less common.
- **Great for:**
 - Polishing for EDS/WDS/EBSD
 - Damage/contamination removal
 - Cross-sections ($\sim 10\text{-}20\text{ mm}^2$)

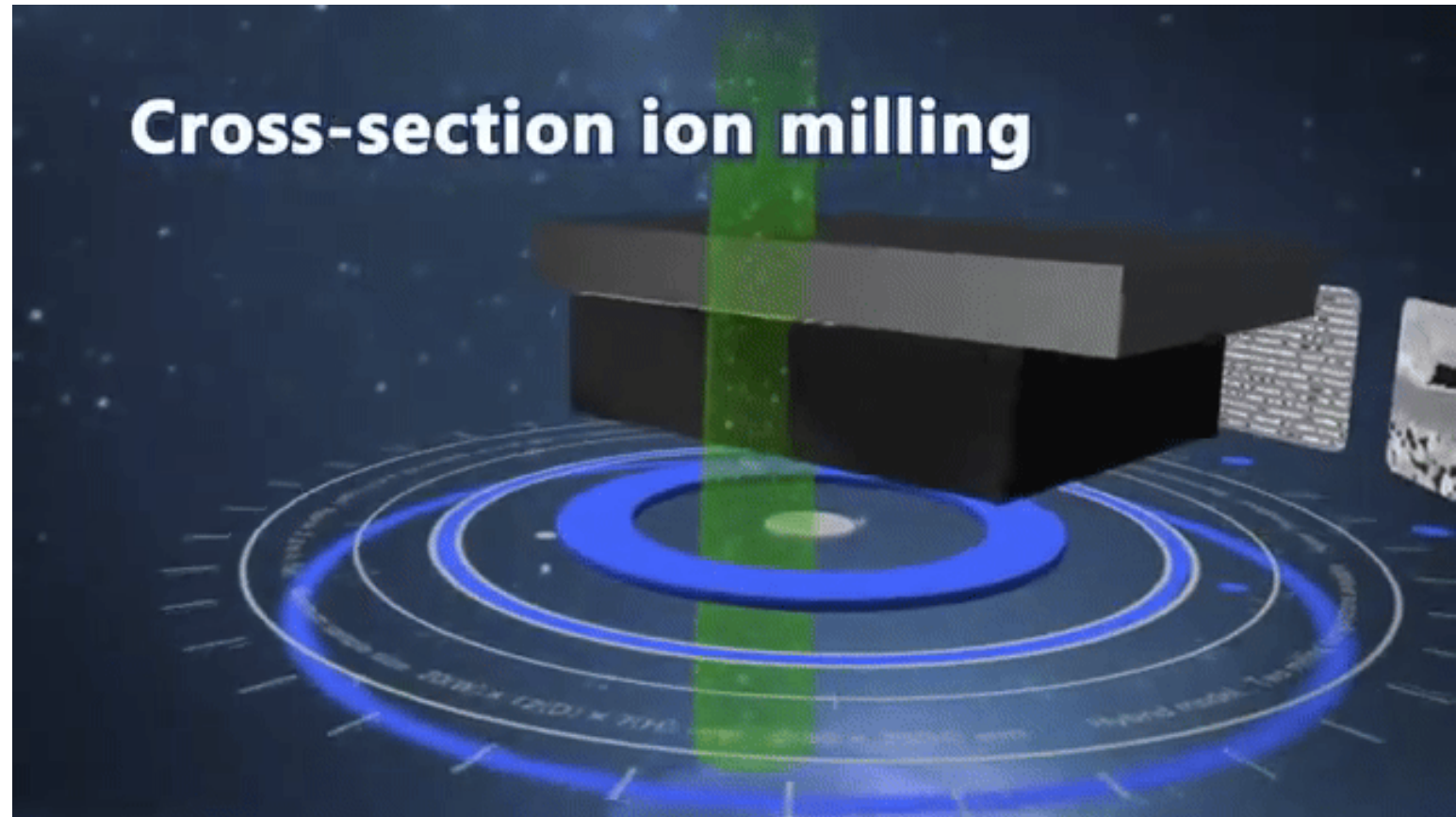
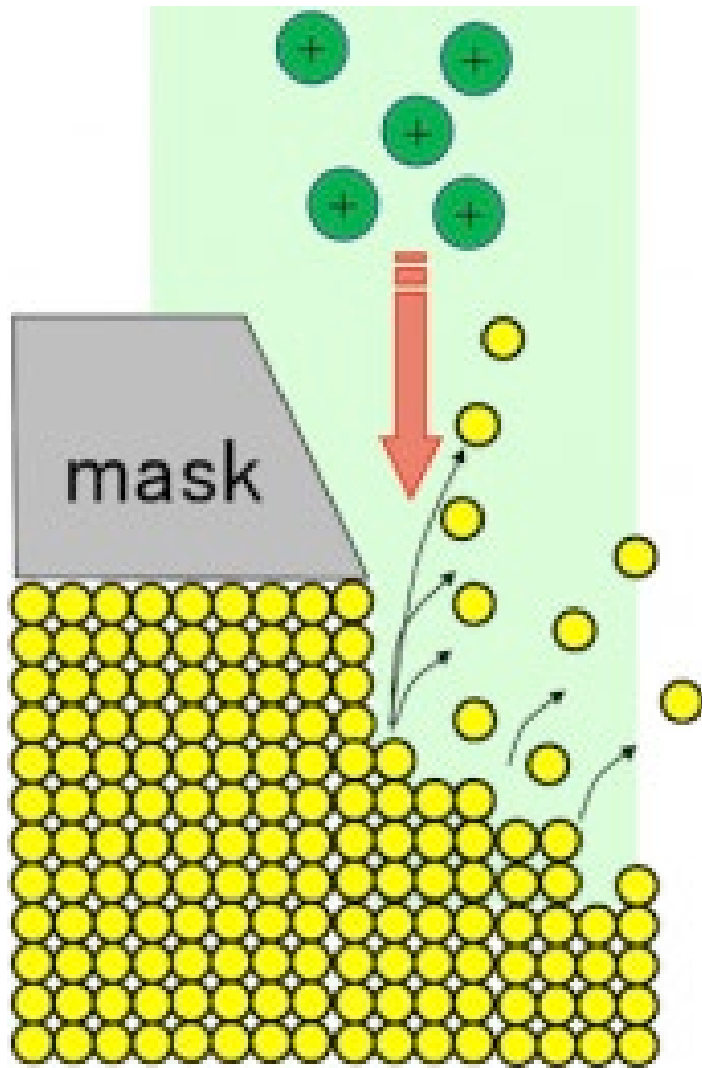
Hybrid Milling in the Hitachi IM4000II

Cross-section



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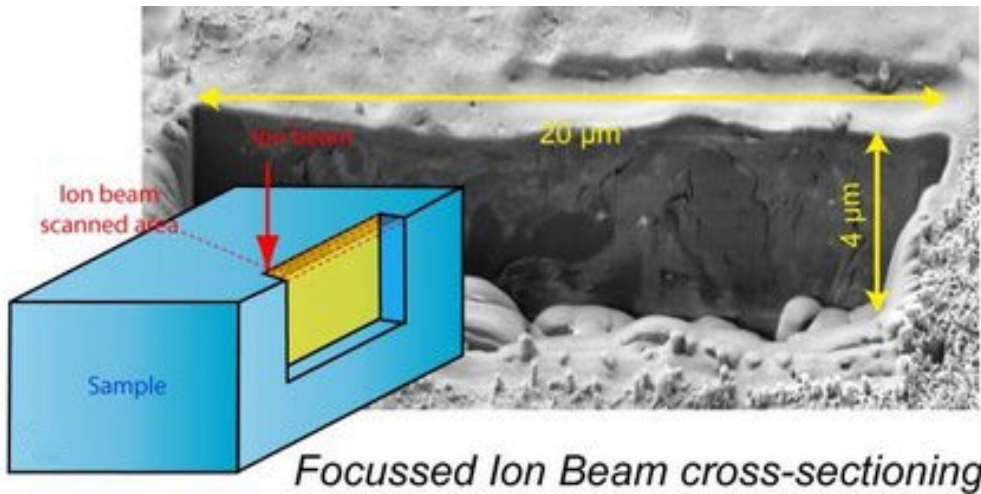




- ± 30 degree swing angle
- 500 $\mu\text{m}/\text{h}$ or greater
- 20(W) $\times$ 12(D) $\times$ 7(H) mm

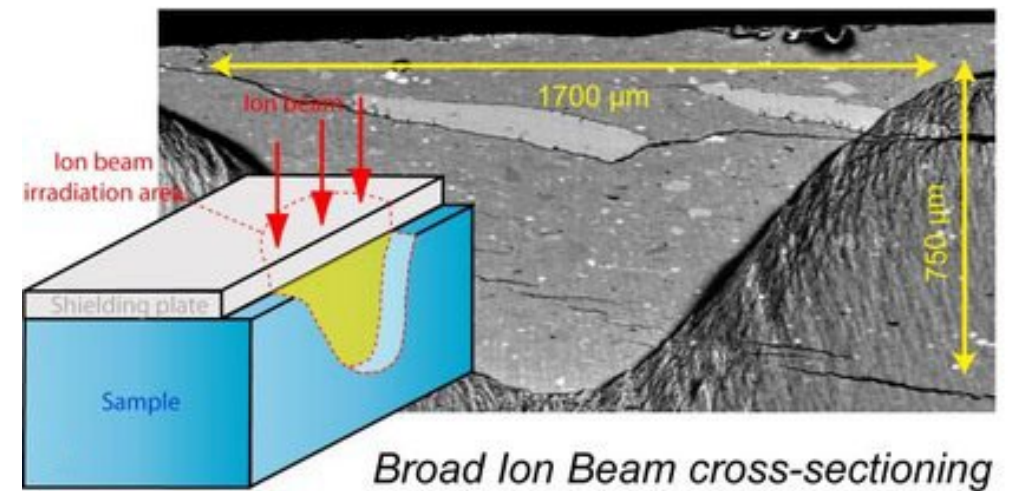
Cross Sections

Focused Ion Beam (FIB)



Desbois et al 2010

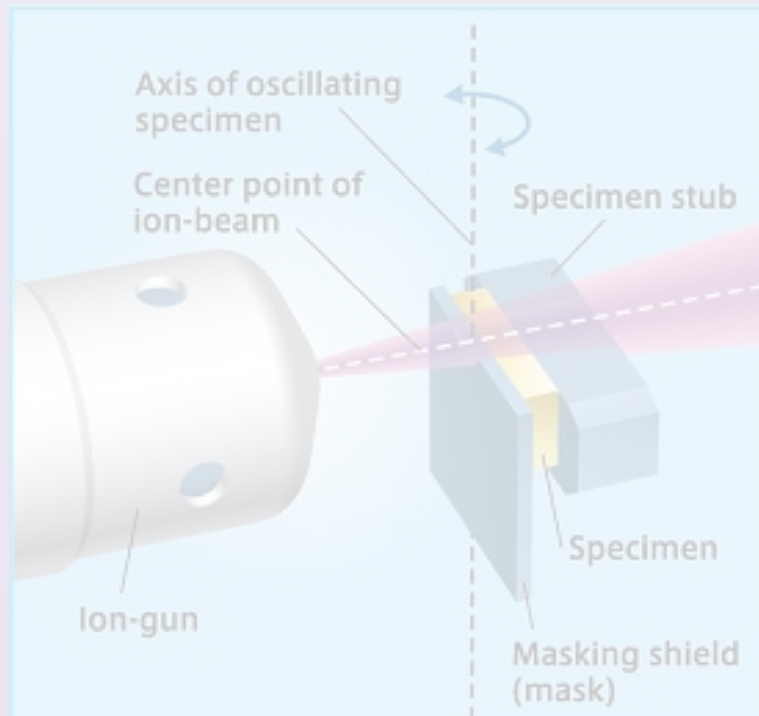
Broad Ion Beam (BIB)



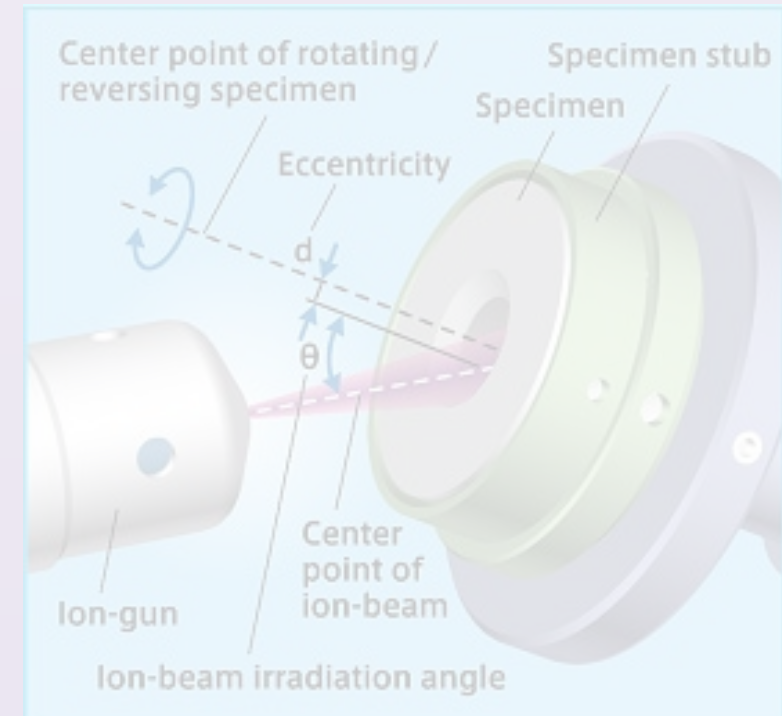
Notice the scale!

Hybrid Milling in the Hitachi IM4000II

Cross-section milling

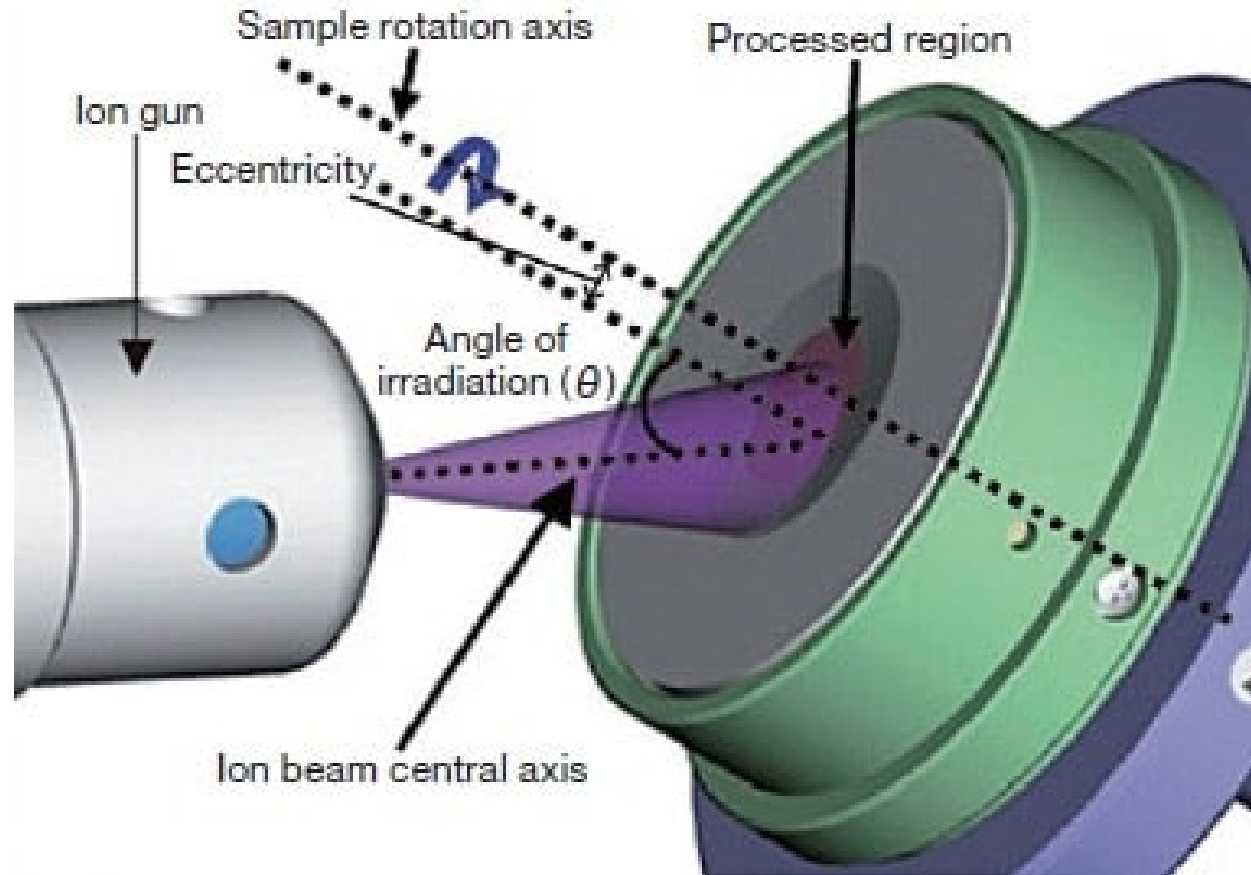
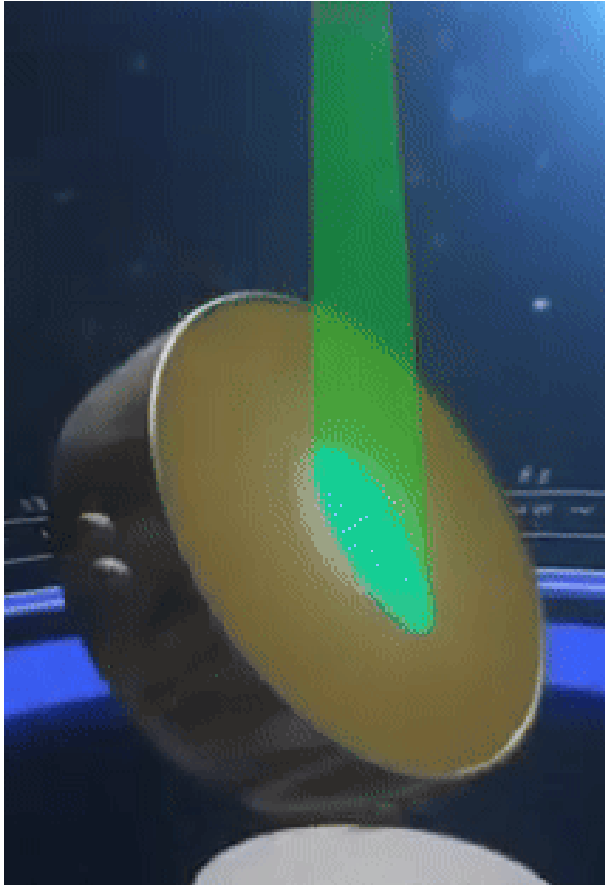


Flat Milling



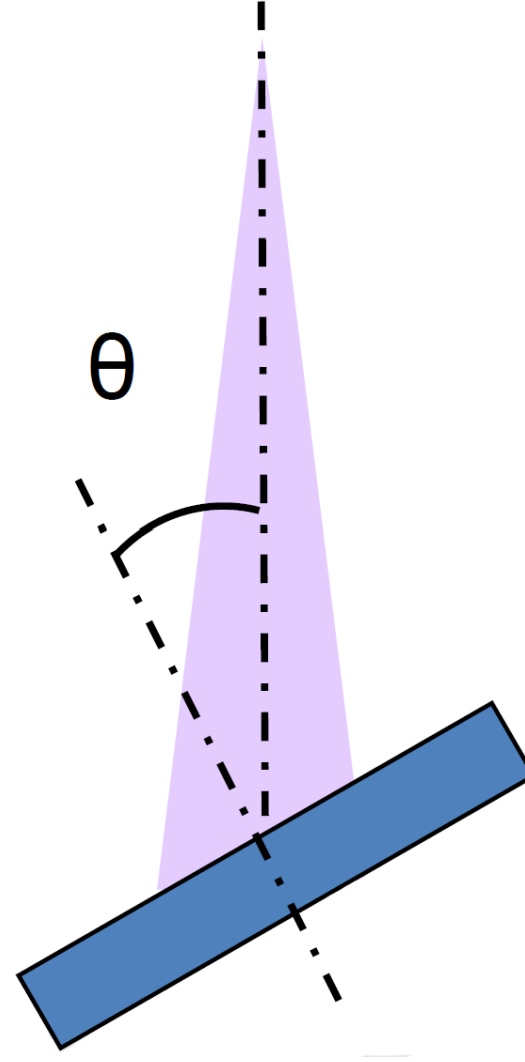
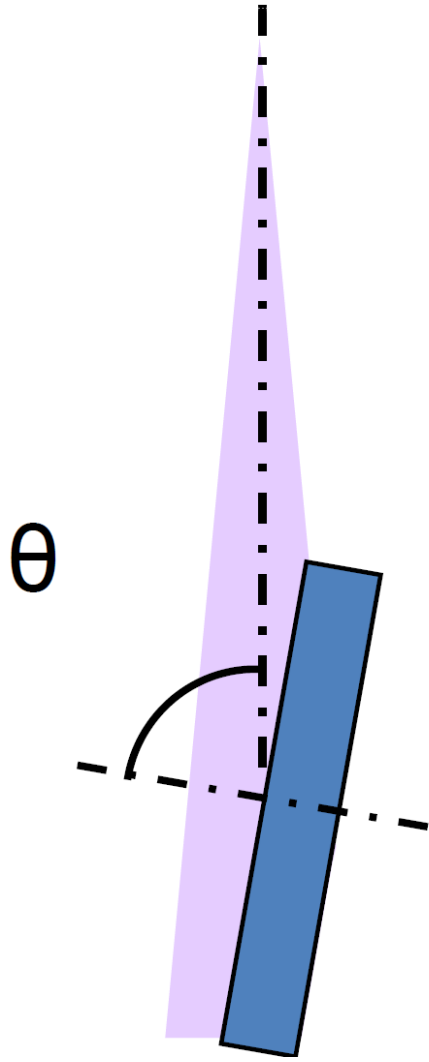
Flat Milling

Hitachi High Tech

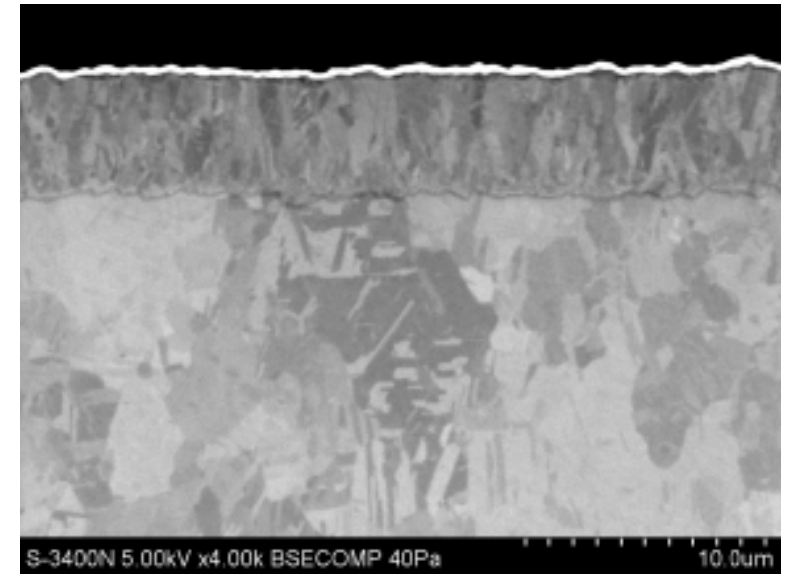
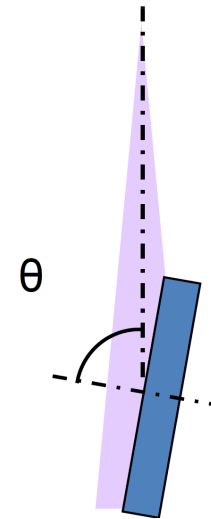
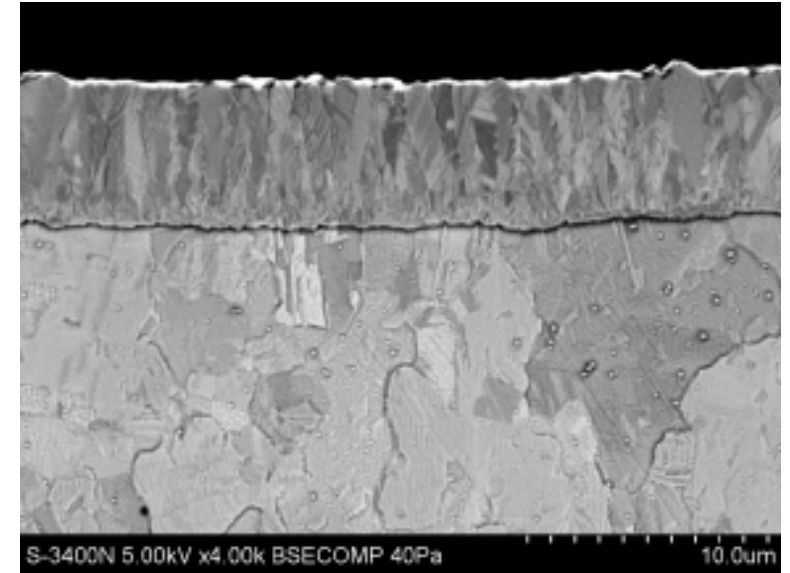
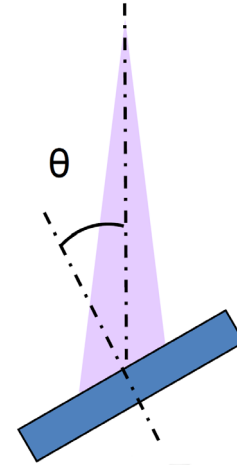
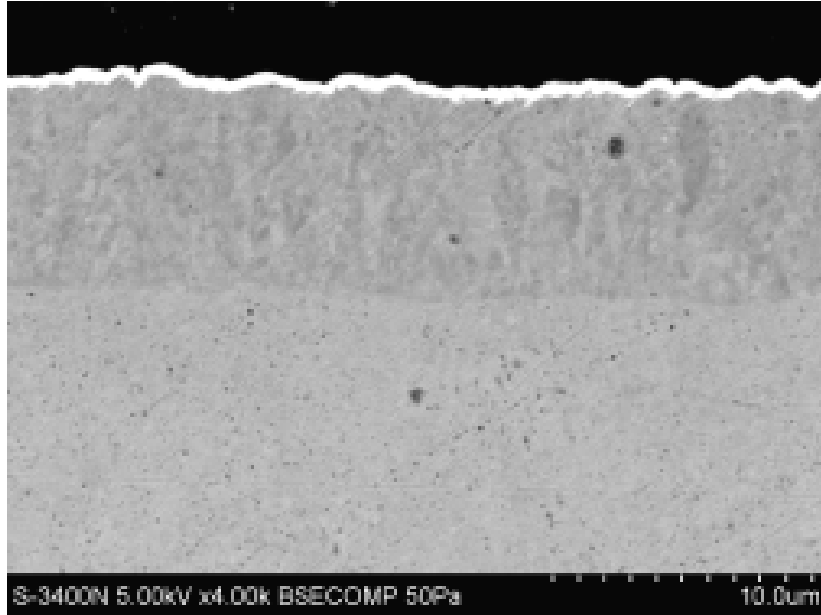


- 50 (W) x 25 (H) specimens
- 32 mm max diameter

Flat/Relief Milling



Flat/Relief Milling



Hitachi High Tech

Additional capabilities

Intermittent Settings

- ON/OFF settings from 1sec to ~1 hour
- Good for heat sensitive samples

Auto Shut Off

- Energy conservation!

Cross-section adapter

- Special holder compatible with the Hitachi 3400 for direct transfer.

Possible future upgrades

- Cryo temp control unit for heat sensitive samples (down to -100 deg C)
- Air protection unit for air sensitive samples

Thank you!

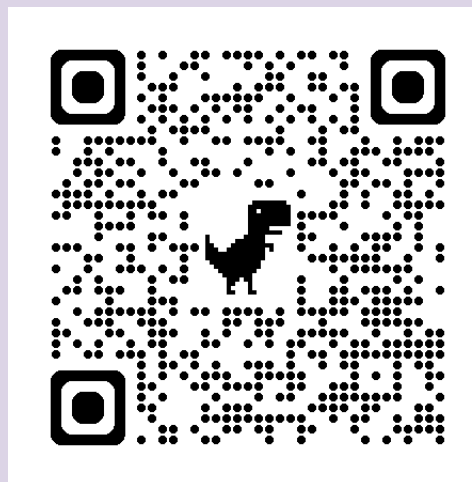


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**Register for Ion Mill
Training!**