The Focused Ion Beam – Scanning Electron Microscope

A tool for sample preparation, two and three dimensional imaging

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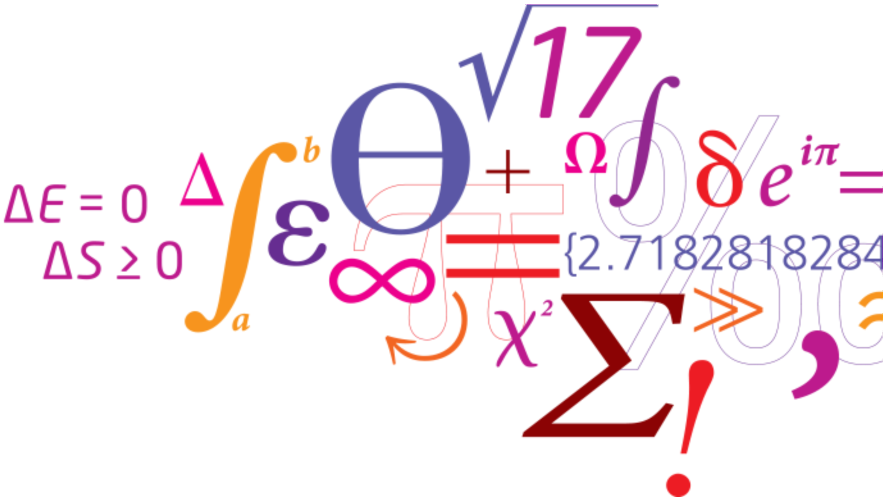
The Focused Ion Beam – Scanning Electron Microscope:

A tool for sample preparation, two and three dimensional imaging

Jacob R. Bowen



DTU Energy Conversion

Department of Energy Conversion and Storage

Contents

- Components of a FIB-SEM
- Ion interactions
- Deposition & patterns
- Probes and alignment
- TEM lamella preparation
- Some examples of investigations on FIB prepared samples
- Serial sectioning and 3D microscopy
- 3D-EBSD
- Summary

- Questions…

Take minute to discuss with your neighbour

- What differences are there between electrons and ions and their interactions with matter?

- Size
- Charge
- Penetration depth / stopping power
- Generation of secondary electrons
- Generation of X-rays
- Damage

A FIB-SEM

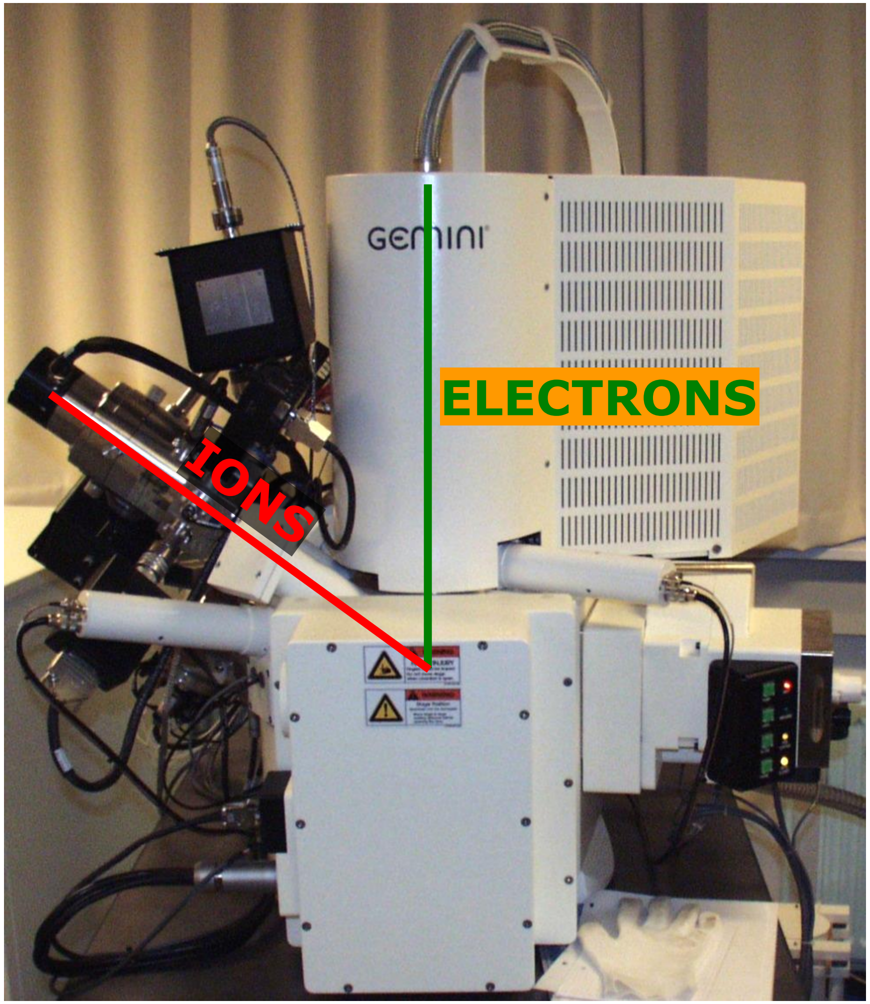
Two more FIB-SEMs

Very brief evolution of commercially available FIB systems

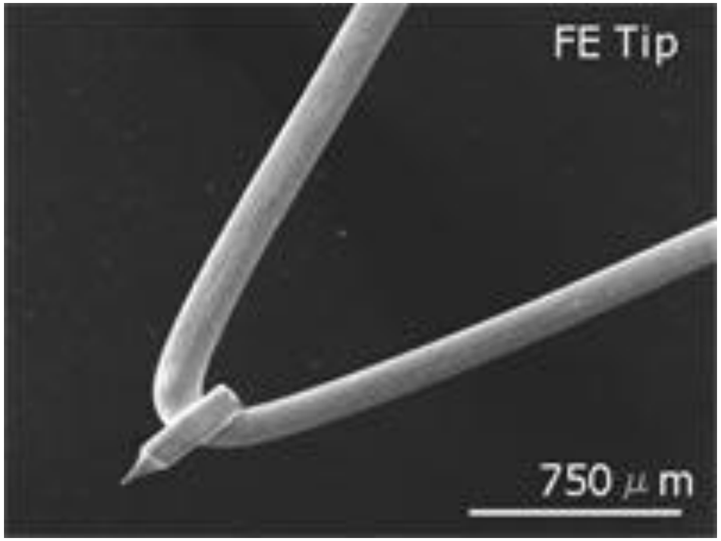
- Single beam FIBs
- “Dual” beam FIB-SEMs
- Lithographic systems
- Semiconductor industry automated systems for FAB assistance
- Multiple ion source FIBs
- Plasma FIB (> 1µA probe current!)
- Laser assisted

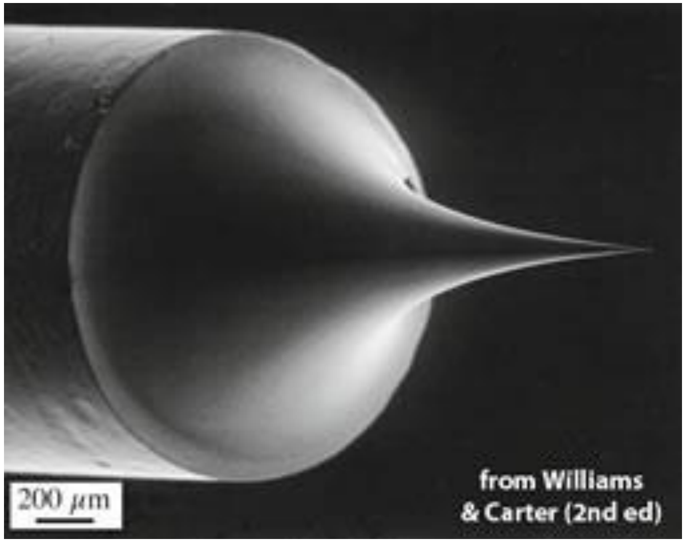
Components of a FIB-SEM

Dual Beam FIB Basics



Schottky field emission electron gun





from Williams & Carter (2nd ed)

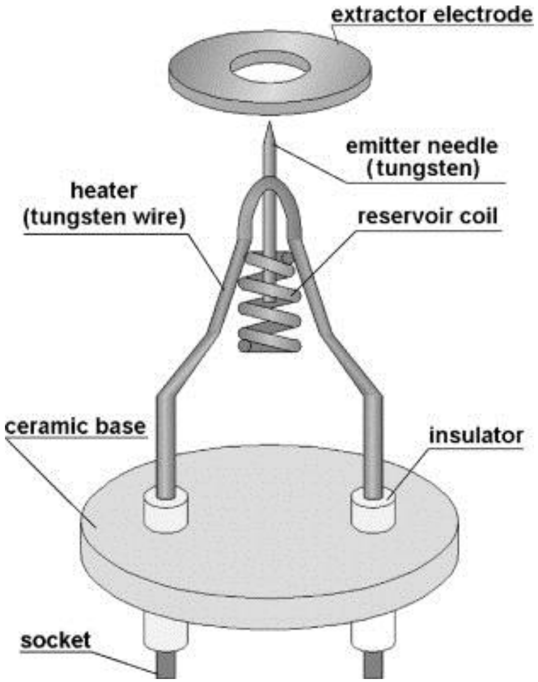
Take minute to discuss with your neighbour

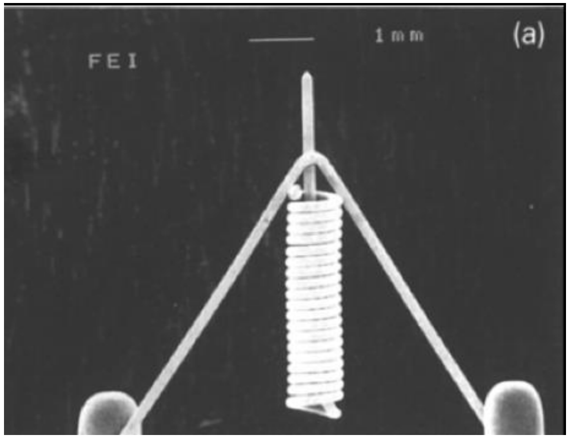
- If you had an ion gun what ammunition would you choose?

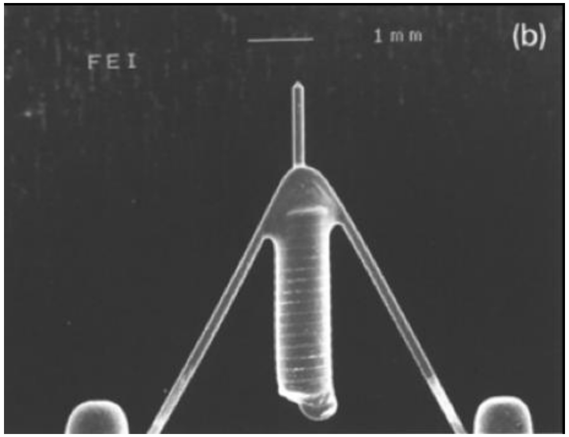
  - Which ion & why?

- Ga^+
- Ease of gun design – liquid metal ion source (LMIS)
- Low melting point
- Atomic weight & size
- Little EDS overlap with other elements

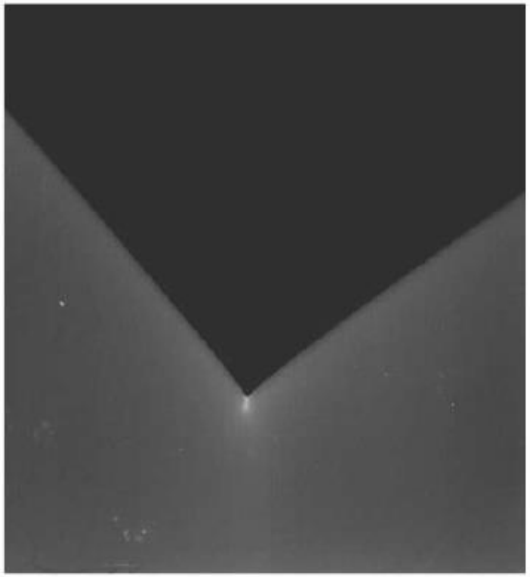
Liquid metal ion source (LMIS)

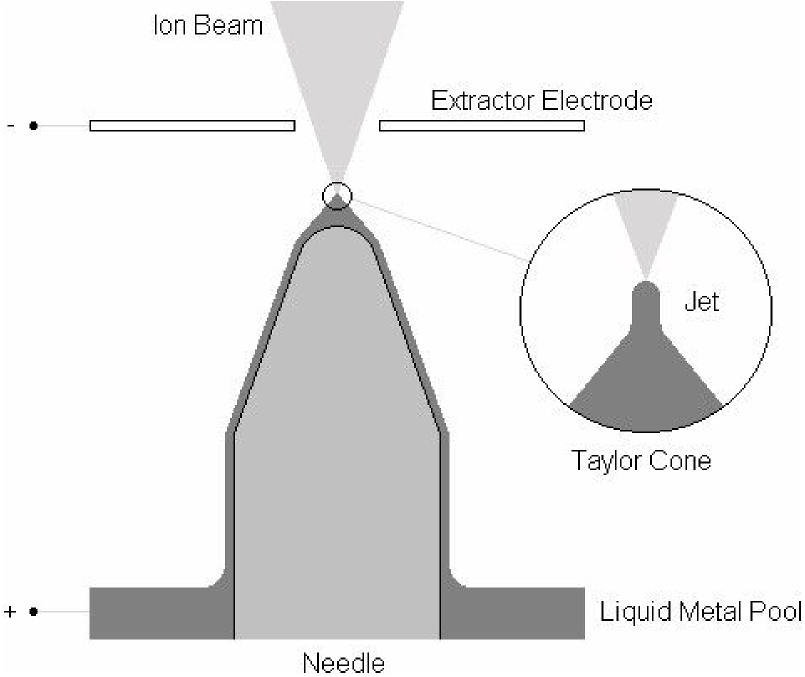


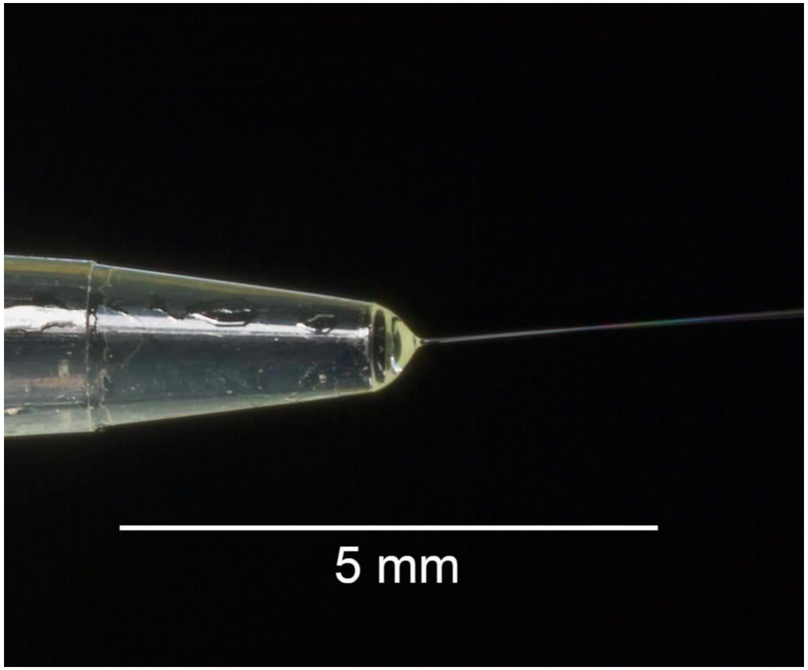




Taylor cone

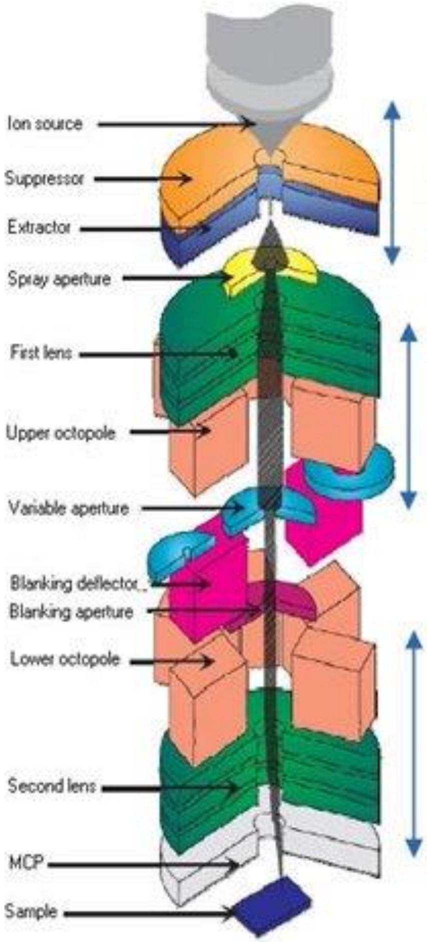






LMIS literature:
10.1016/j.mee.2004.02.029
10.1016/S0042-207X(96)00227-8
10.1016/0169-4332(94)90327-1

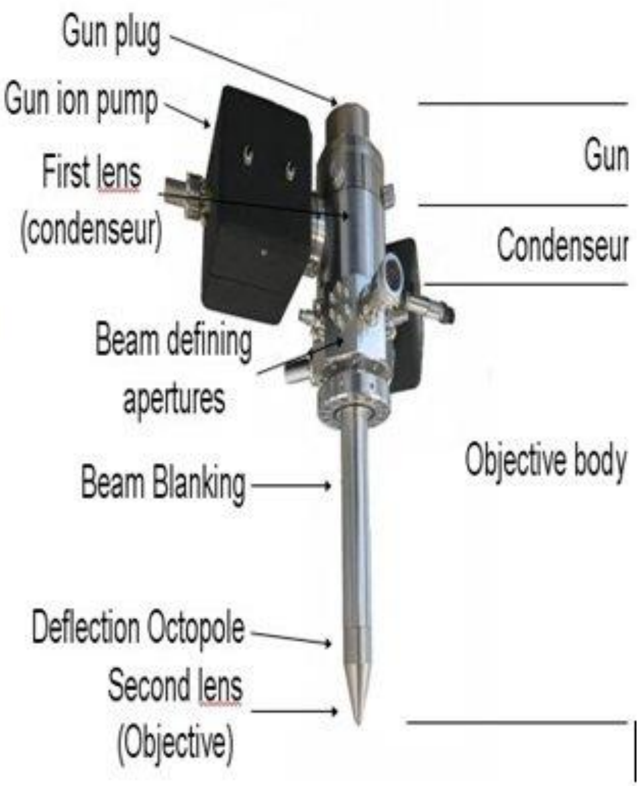
FIB column



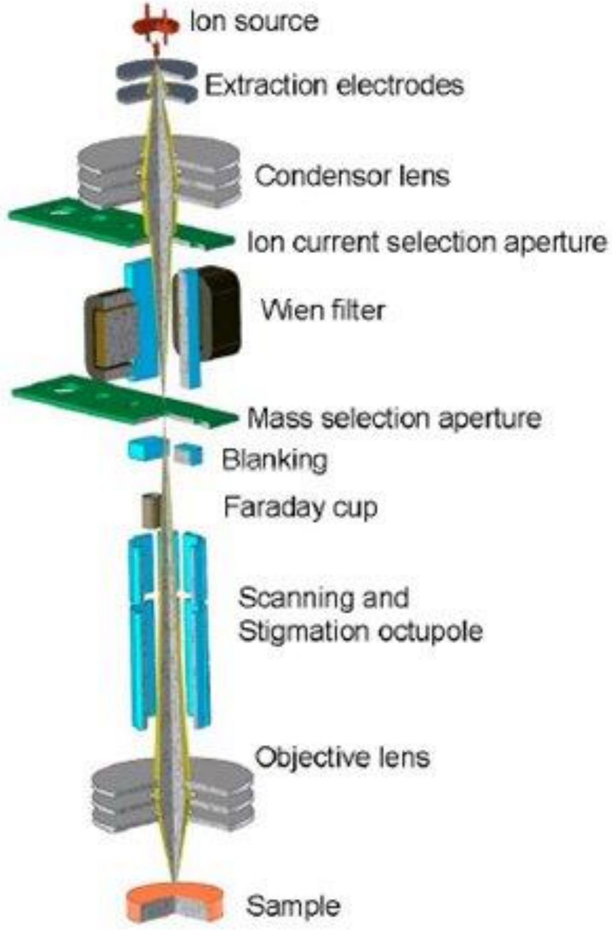
generation of beam (2 µA)

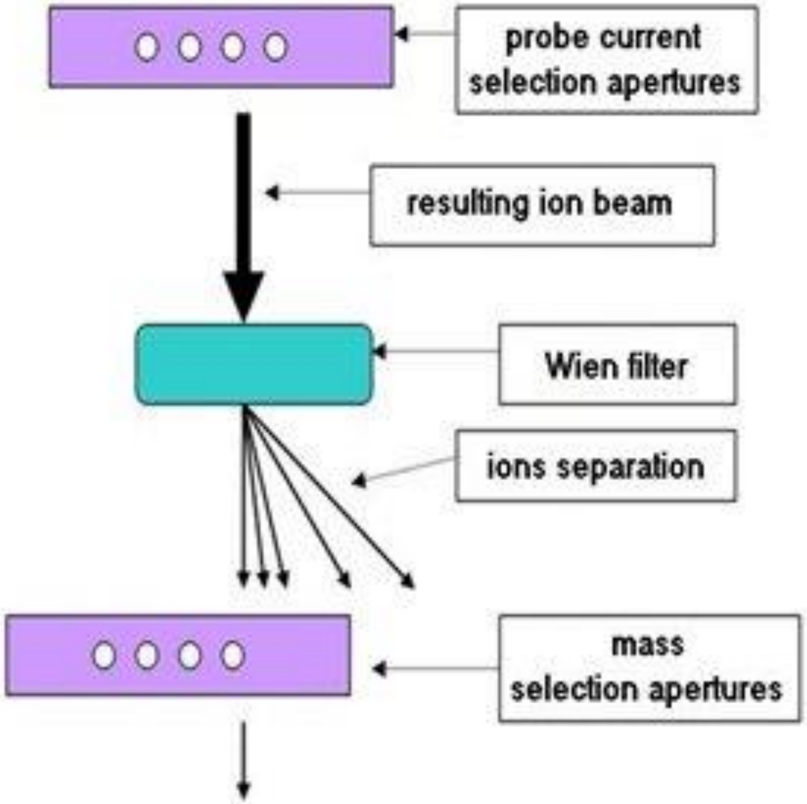
adjusting the current (typically pA to nA)

scanning the beam



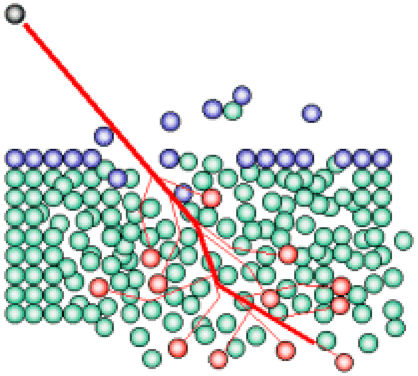
Atomic mass selection FIB column



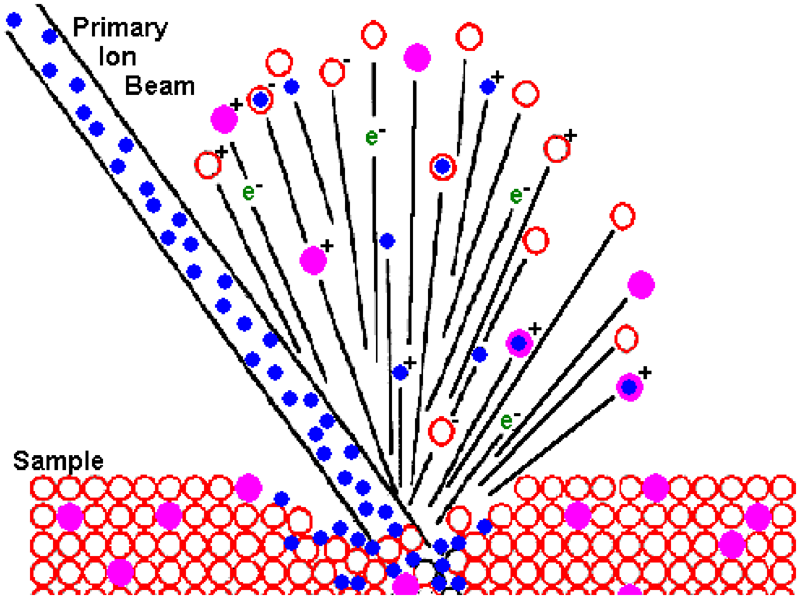


Ion interactions

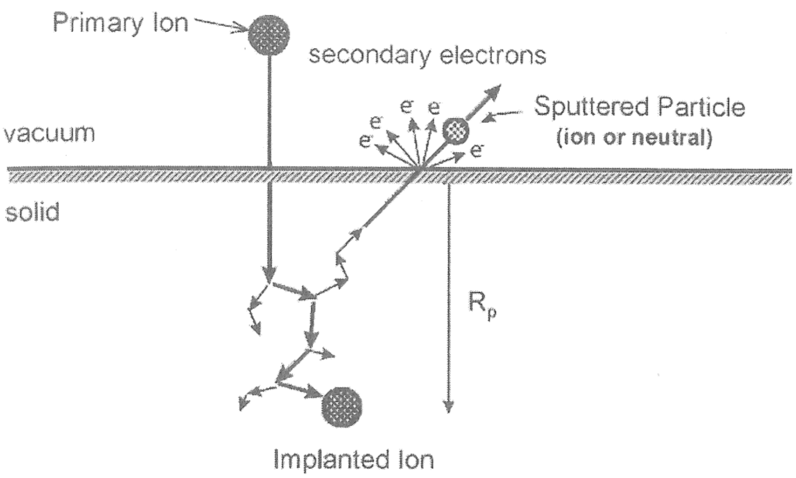
Ion-solid interactions

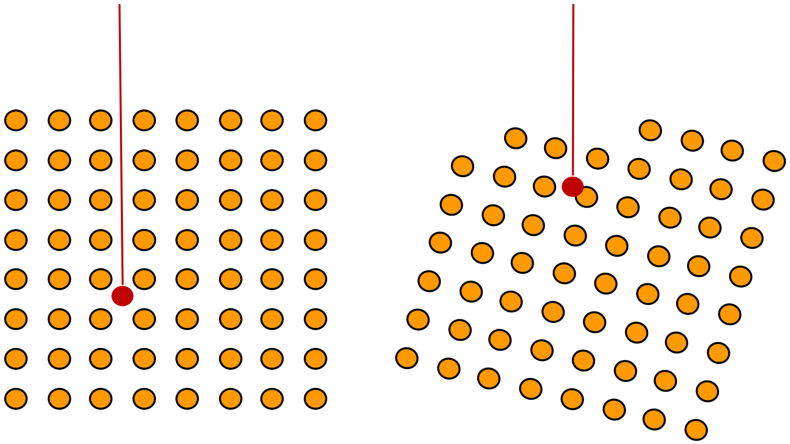


Schematic of the collision cascade



Ion Solid Interactions





Number of SE2/PI > 2

For deeply penetrating ions SE1 >>> SE2

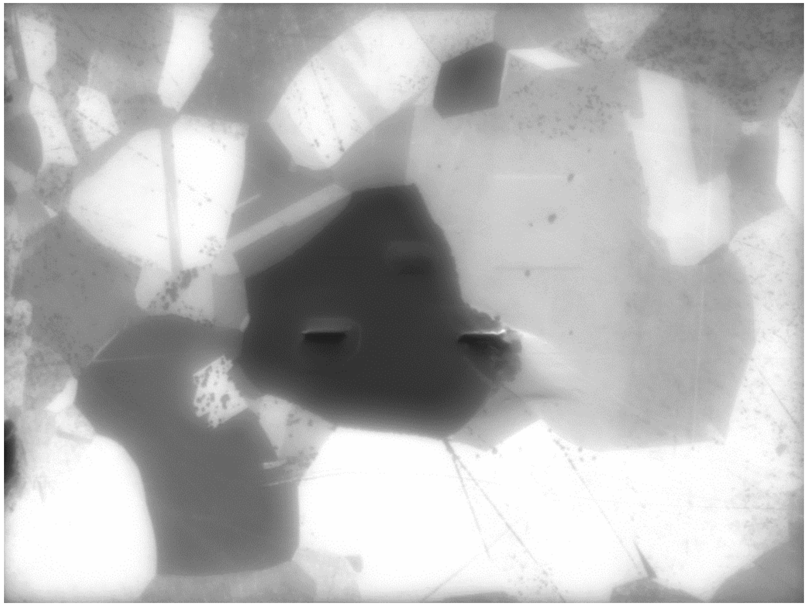
See PDF from David Joy on He ion microscopy

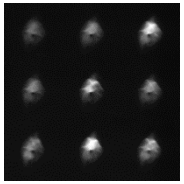
Channeling Contrast & Milling Rate

50 pA image

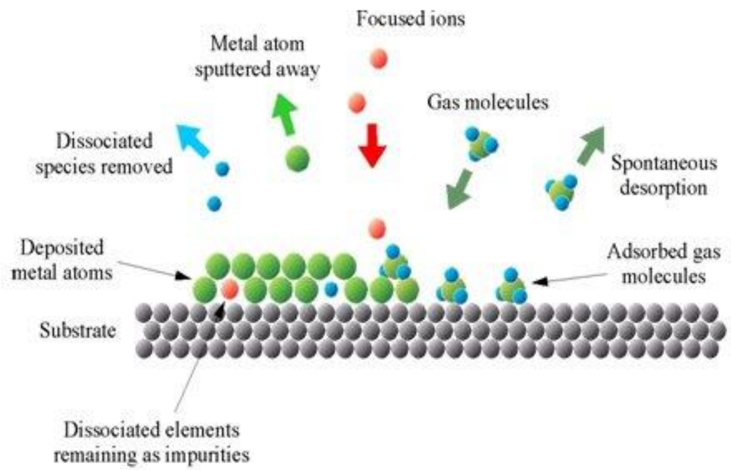
Image width ~100 µm

10 nA image

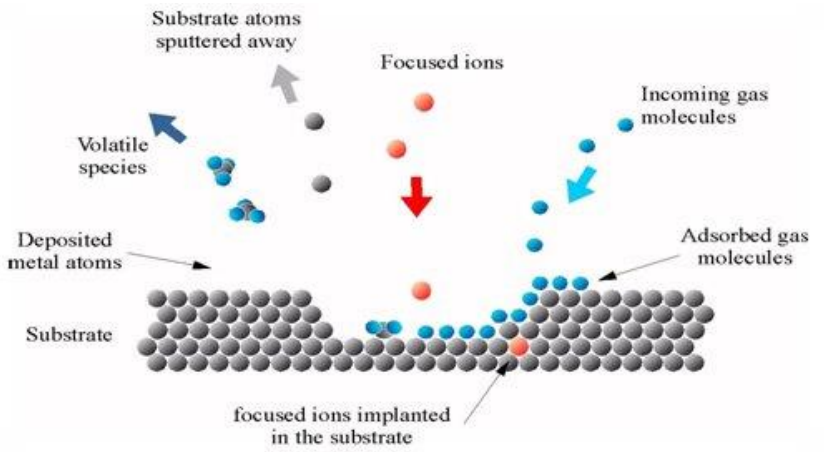




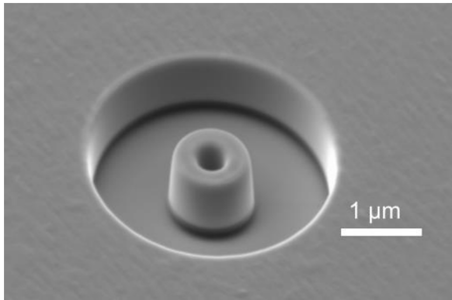
Deposition & gas assisted etching



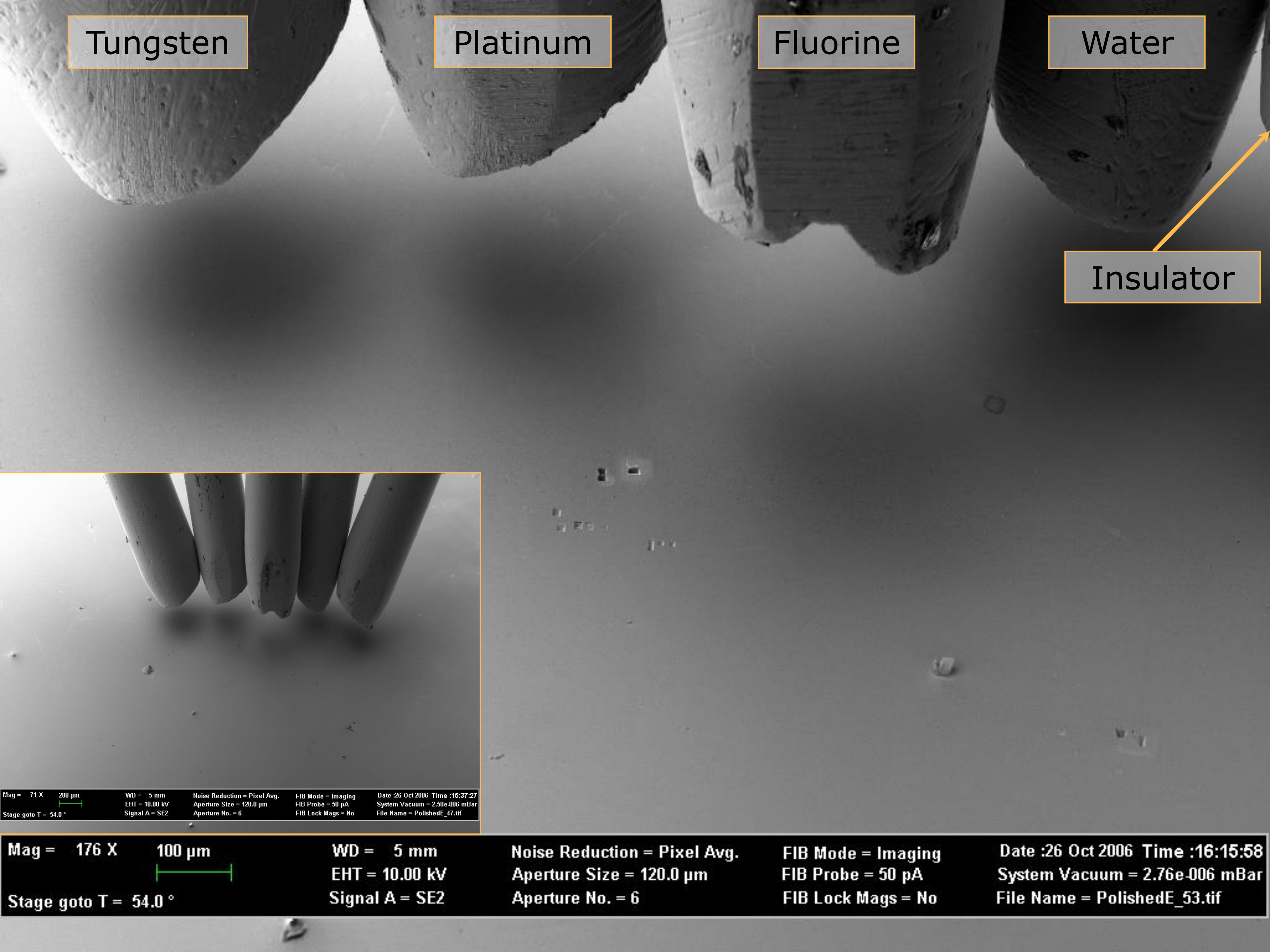
1. Adsorption of the precursor molecules on the substrate
2. Ion beam induced dissociation of the gas molecules
3. Deposition of the material atoms and removal of the organic ligands



1. Adsorption of the gas molecules on the substrate
2. Interaction of the gas molecules with the substrate
   Formation of volatile and non volatile species
3. Evaporation of volatile species and sputtering of non volatile species



Deposition and patterns


Tungsten
Platinum
Fluorine
Water
Insulator
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200 µm
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WD = 5 mm
EHT = 10.00 kV
Signal A = SE2
Noise Reduction = Pixel Avg.
Aperture Size = 120.0 µm
Aperture No. = 6
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FIB Probe = 50 pA
FIB Lock Mags = No
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System Vacuum = 2.50e-006 mBar
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100 µm
Stage goto T = 54.0 °
WD = 5 mm
EHT = 10.00 kV
Signal A = SE2
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FIB Lock Mags = No
Date :26 Oct 2006 Time :16:15:58
System Vacuum = 2.76e-006 mBar
File Name = PolishedE_53.tif

Uses of deposition pre-cursors

- Pt & W
  - Surface protection layers
  - Conductive connections & integrated circuit edits (mostly Pt)
  - Microwelding (mostly Pt)
  - W precursor gas can give FIB column contamination issues
  - Amorphous and contain carbon
- Insulator (SiO2)
  - Insulating sections of integrated circuit edits (mostly Pt)
  - Surface protection layers
- Carbon
  - Surface protection layers
  - Microwelds
  - Difficult to control system vacuum
- Water & Flouring
  - Reactive etching for polymers & Si

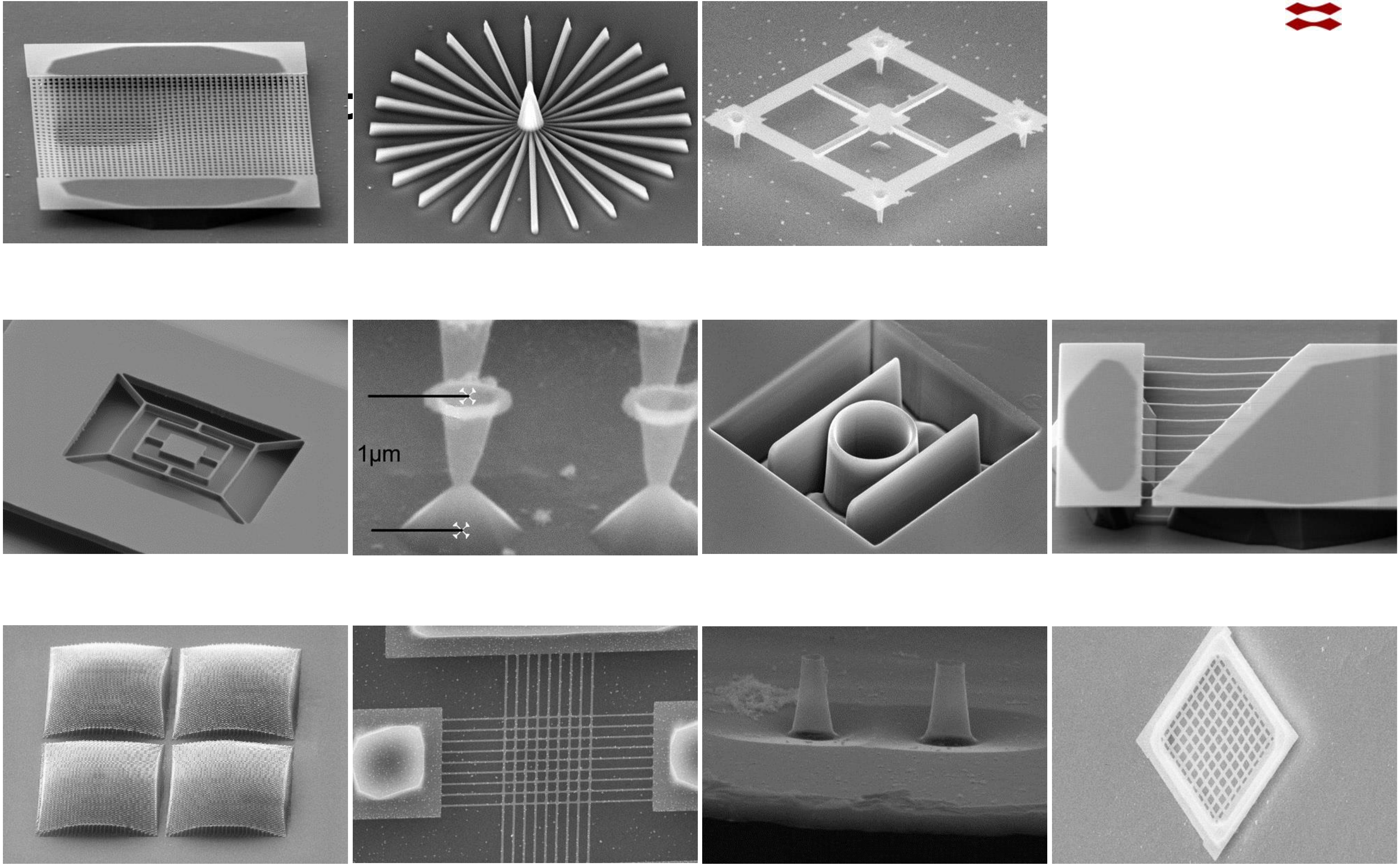
Take minute to discuss with your neighbour

- What parameters control deposition rate and quality of deposition?

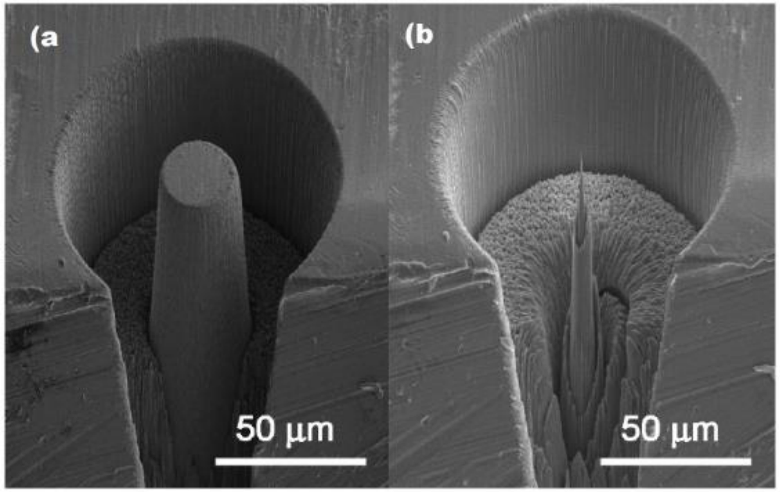
- Heating and cooling of pre-cursor reservoir → effects gas pressure
- Choice of probe current → milling versus deposition
- Area of milling job
- Angle of sample to injection needle
- Beam scan frequencies in X & Y

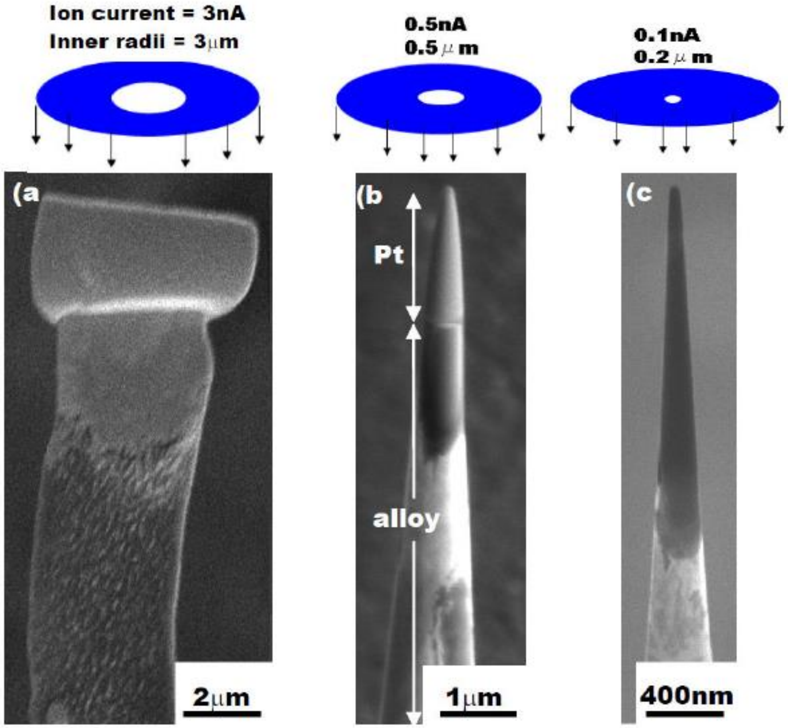
Deposition

- Fine balance between deposition & milling
- W & Pt deposit amorphously
- SiO2 deposits are crystalline
- Deposits contain significant quantities of C & Ga
- Some times necessary to use e-beam deposition to protect small surface features


1µm

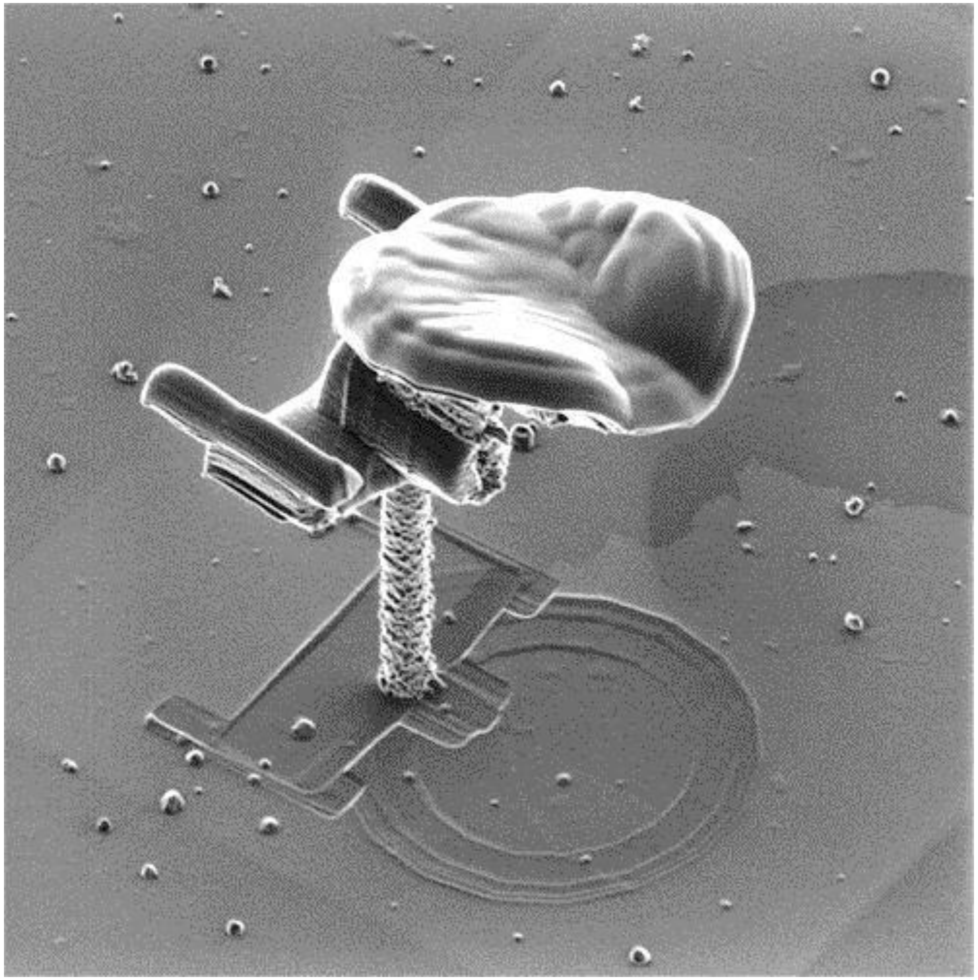
Annular milling & Atom probe tip manufacture



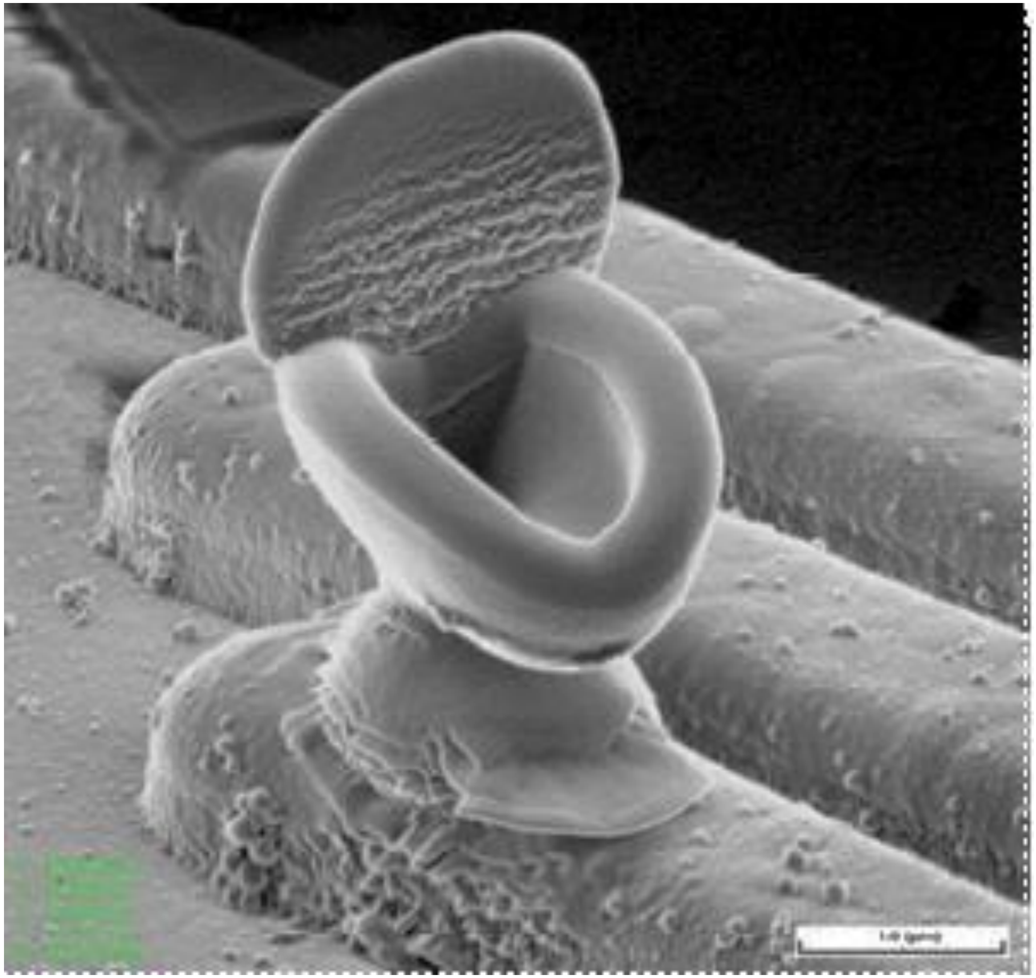


Re-deposition effects on shape

Not so useful structure = nano art?



Another structure?



Take a minute to discuss with your neighbour

- What is the most desirable shape of an ion beam?

  - Small diameter (full-width half-maximum)
  - Top hat intensity profile compared to bell curve intensity profile
  - Small tails
  - Circularly symmetric

Probes and alignment

Probe currents

- Low currents for imaging
  - Typically 50 pA but can use 10 pA to 500 pA
    - Low current → high noise
- Low currents for writing / lithography
  - 2 pA upwards depending on the scale of structure
- Low currents for final polishing to TEM electron transparrency
  - 20-50pA depending what kind of TEM is needed
- Intermediate currents for serial sectioning
  - 200 pA to 2 nA depending on required volume and resolution
- High currents for removing material
  - >10 nA for pre-trenching
  - > 500 pA for fine milling

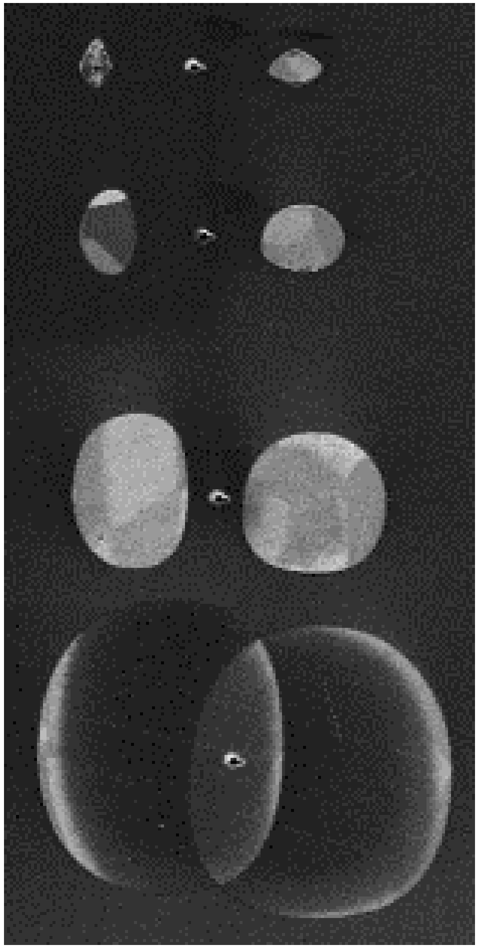
Probe currents

- Depending on microscope each probe current needs to be aligned
  - Condenser voltage
  - Aperture size
  - Specimen current
  - Focus
  - Stigmatism
  - Relative beam shift

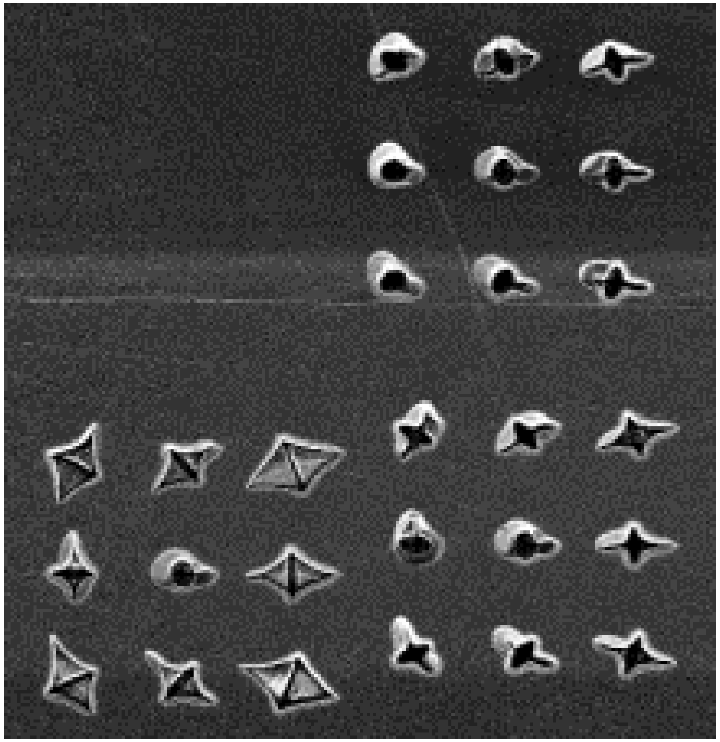
FIB beam alignment

Focus adjustment

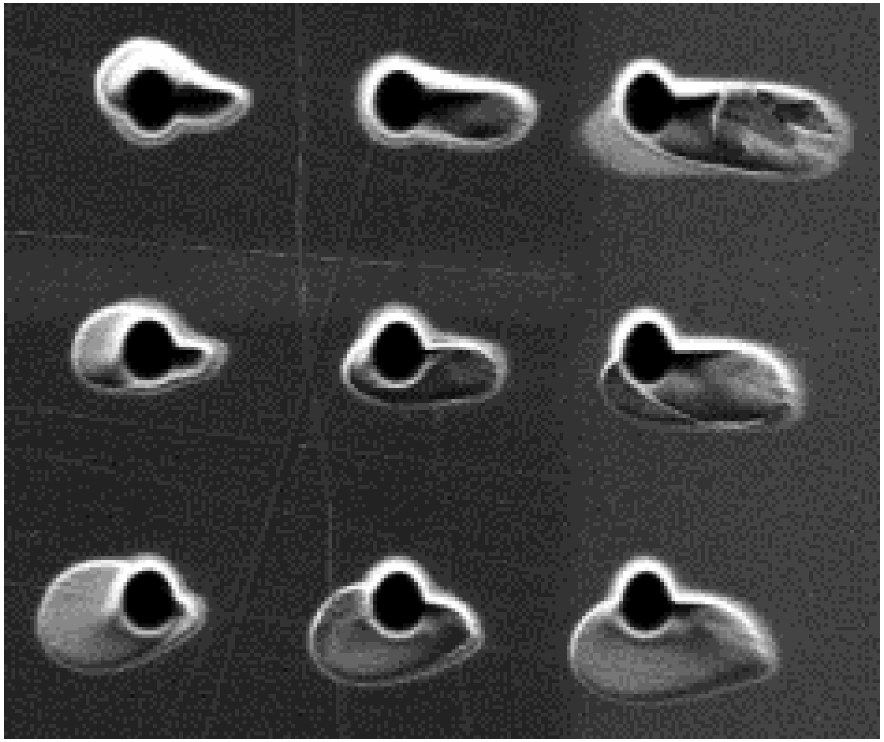
Spot spacing = 20 µm (10 nA probe, 50 pA images)



FIB Beam alignment



Stigmation adjustment

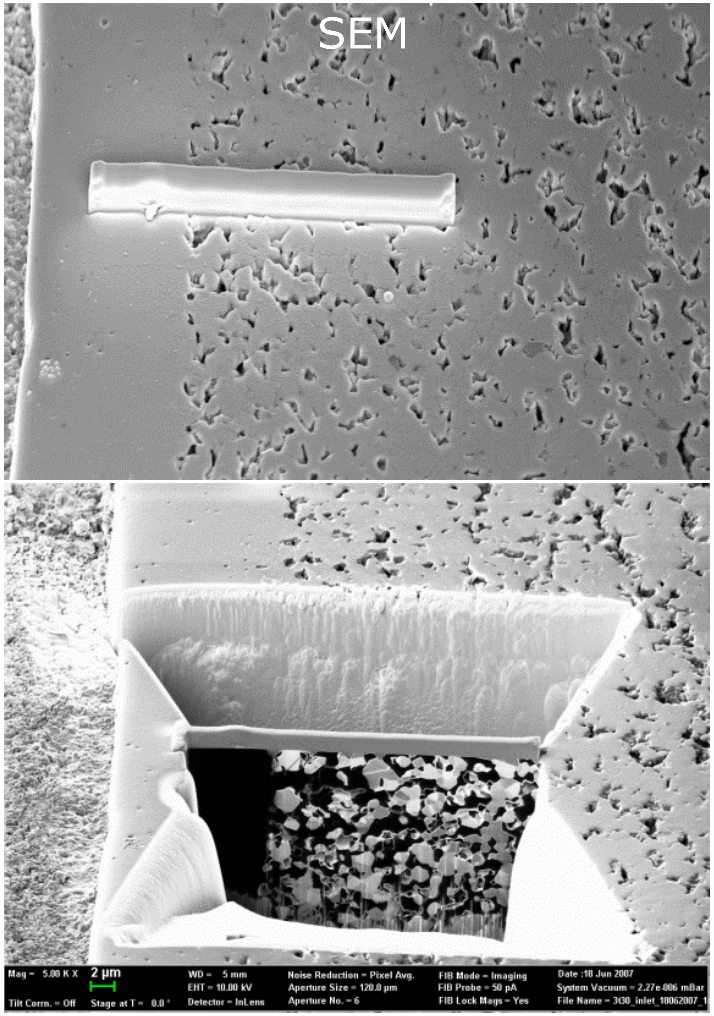


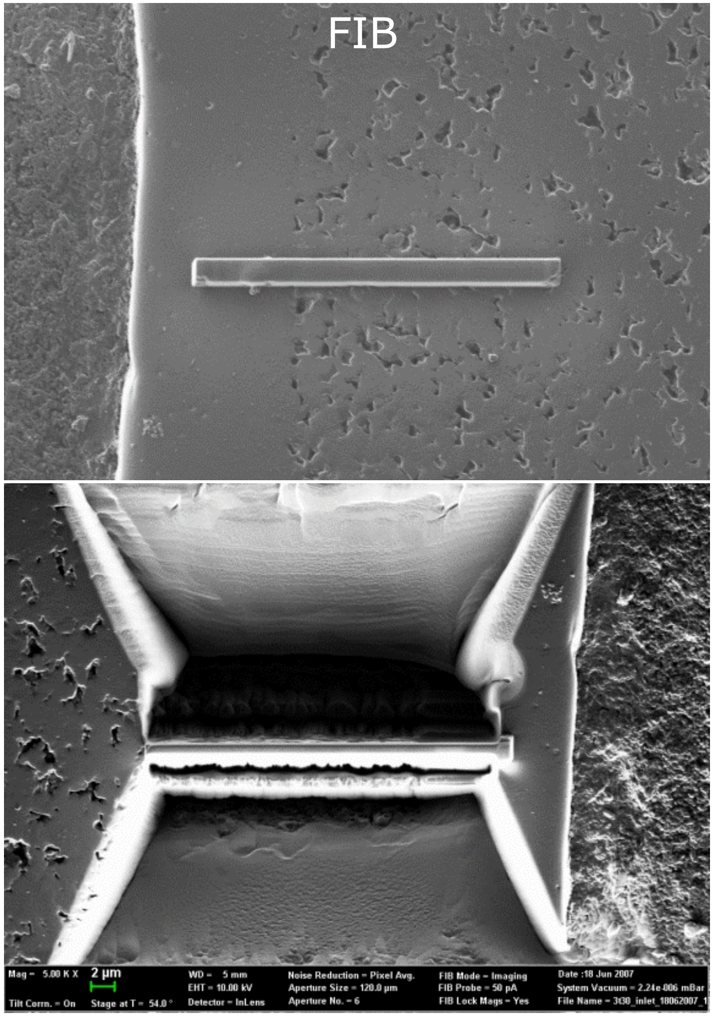
Aperture alignment

Spot spacing = 10 µm (10 nA probe, 50 pA images)

TEM lamella preparation

Cross-sectioning & TEM lamella lift-out





Automation of milling jobs

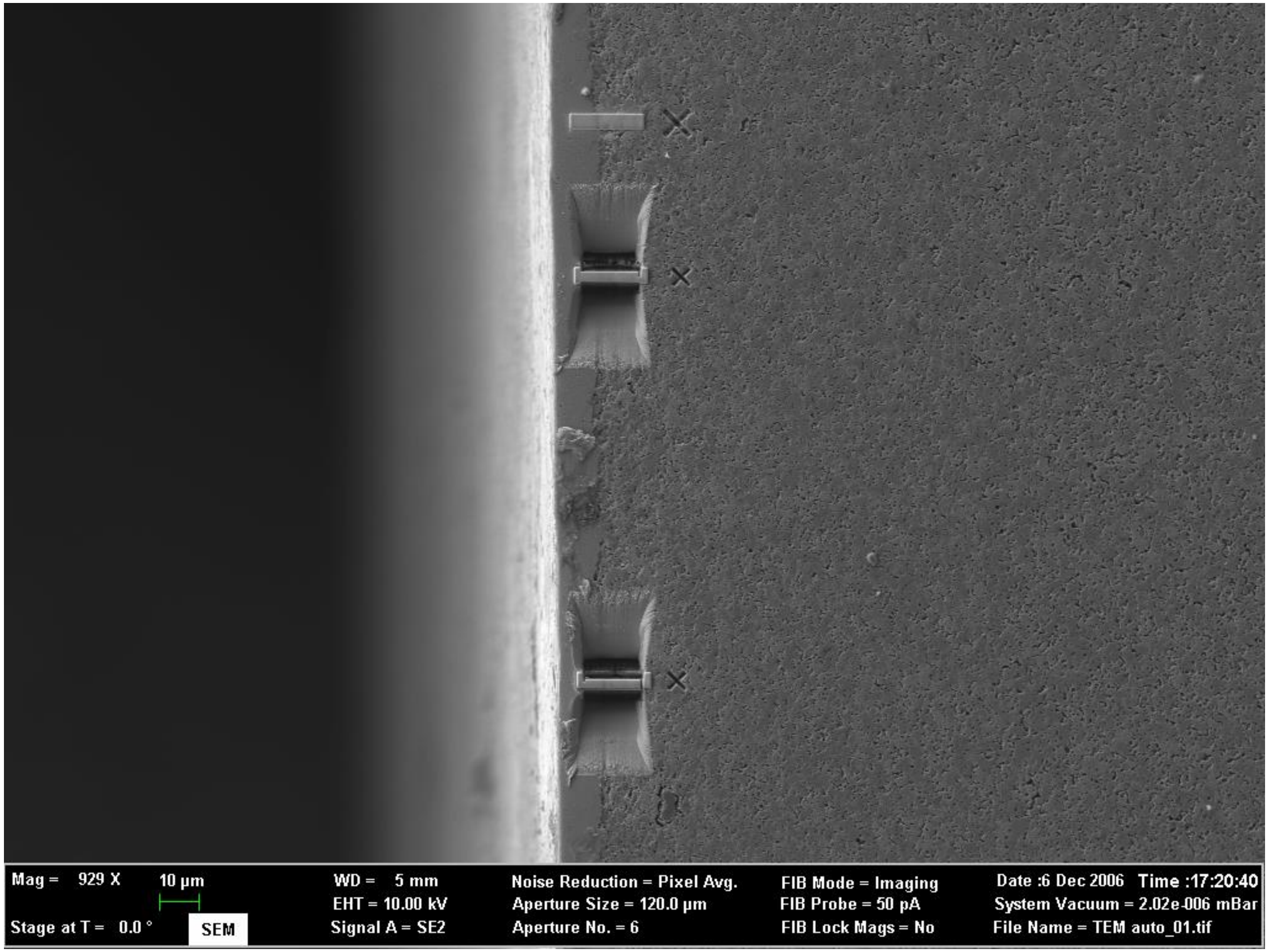
- Define a milling object
  - Probe current, X, Y & Z dimensions, material
- Add to job list
- Add successive jobs to list
- Optionally set up drift correction

- Account for re-deposition of material when designing jobs

- TEM liftout process is can be almost fully automated

- Jobs can be batched for e.g. production of multiple TEM samples
  - In semiconductor industry TEM sample site is registered with CAD diagrams of integrated circuits

Auto lamella example



Mag = 929 X
10 µm
Stage at T = 0.0 °
SEM
WD = 5 mm
EHT = 10.00 kV
Signal A = SE2
Noise Reduction = Pixel Avg.
Aperture Size = 120.0 µm
Aperture No. = 6
FIB Mode = Imaging
FIB Probe = 50 pA
FIB Lock Mags = No
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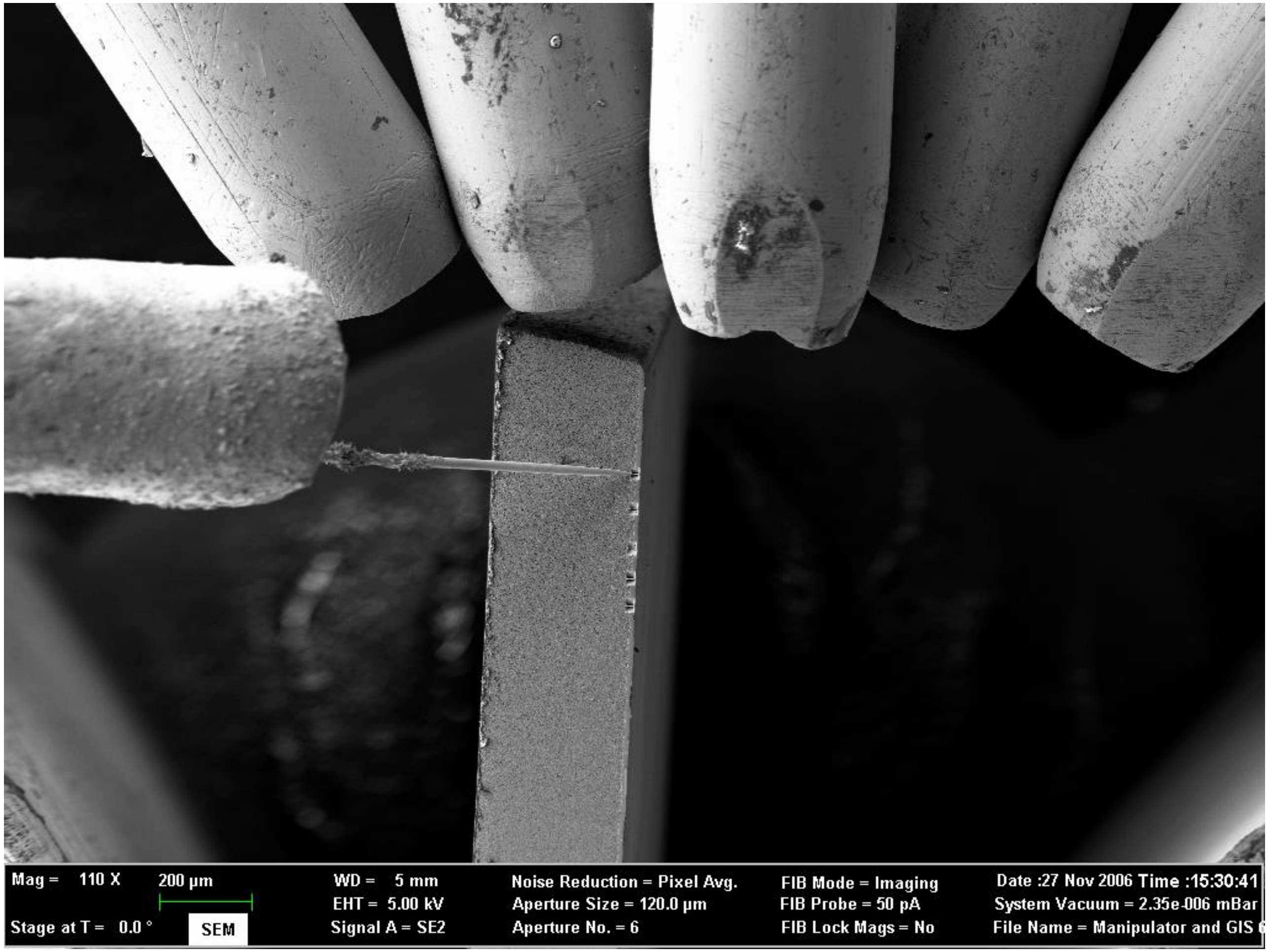
System Vacuum = 2.02e-006 mBar
File Name = TEM auto_01.tif

In-situ manupulation

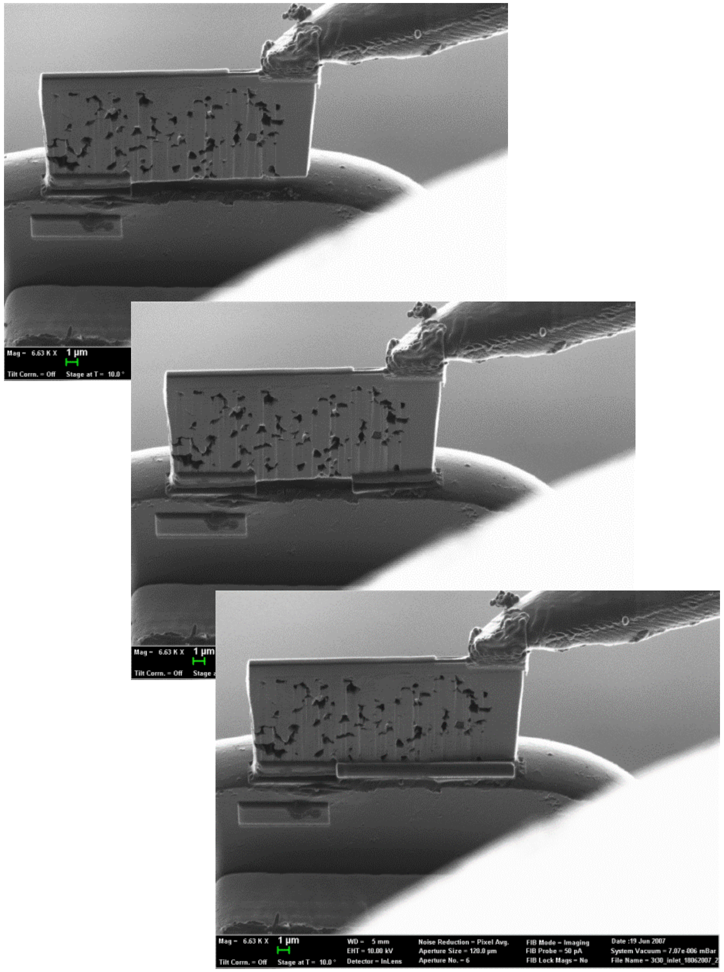


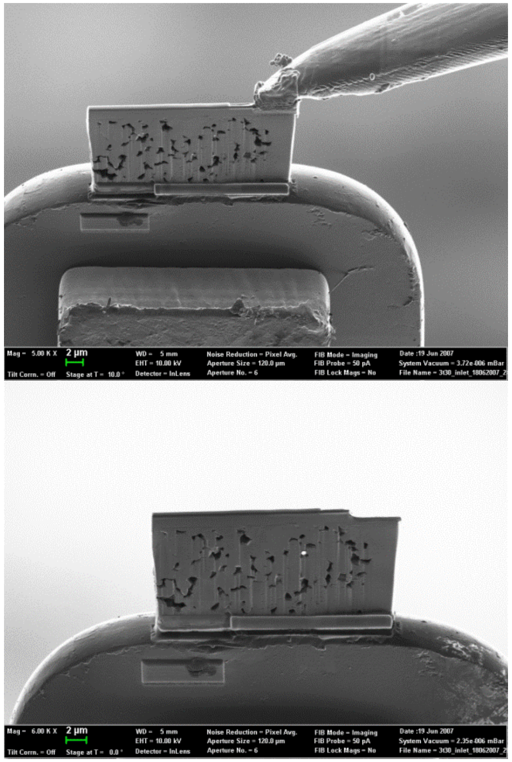
Lift-out

Image intensity changes on manipulator contact

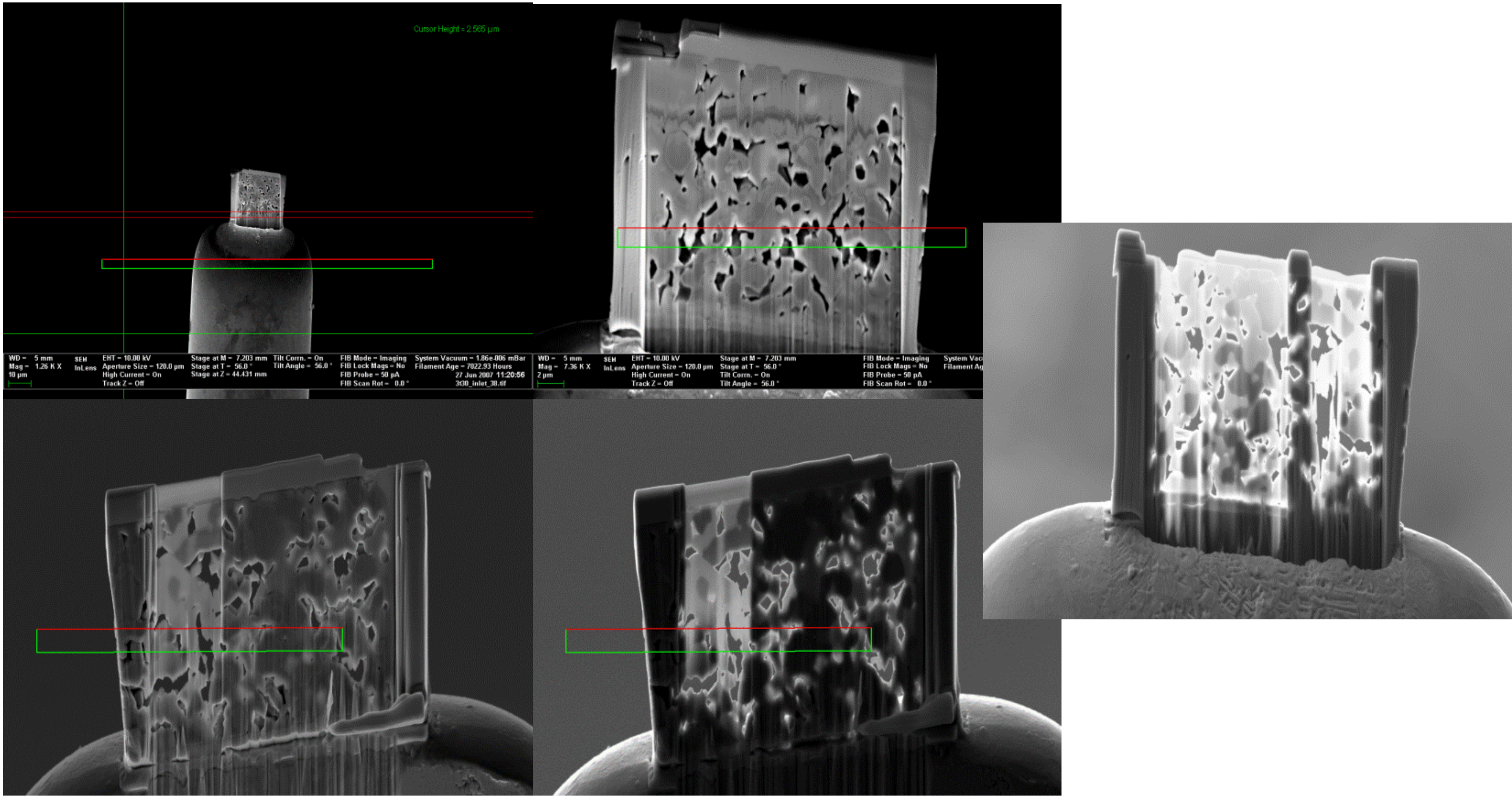
When lamella is cut free its intensity changes

Touch down and lamella attachment



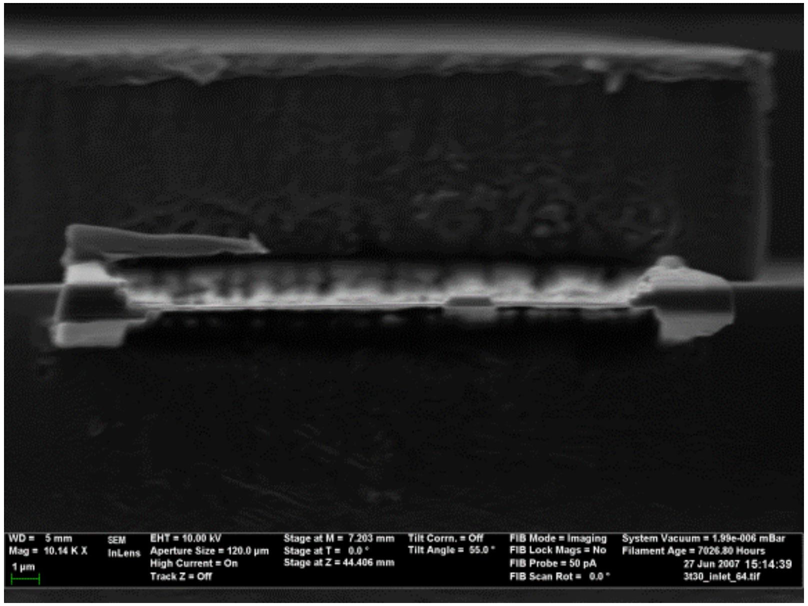


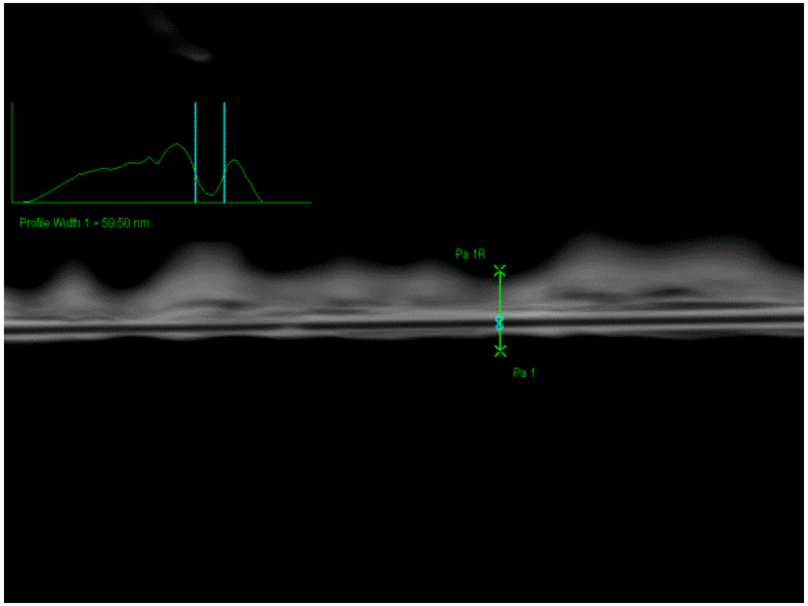
Final thinning to electron transparrency



TEM sample prep video

Thickness measurement





At this stage things can go horribly wrong

→ Advice: stop milling and give to TEM operator before it is too late

The sample can always be put back in the FIB for further thinning

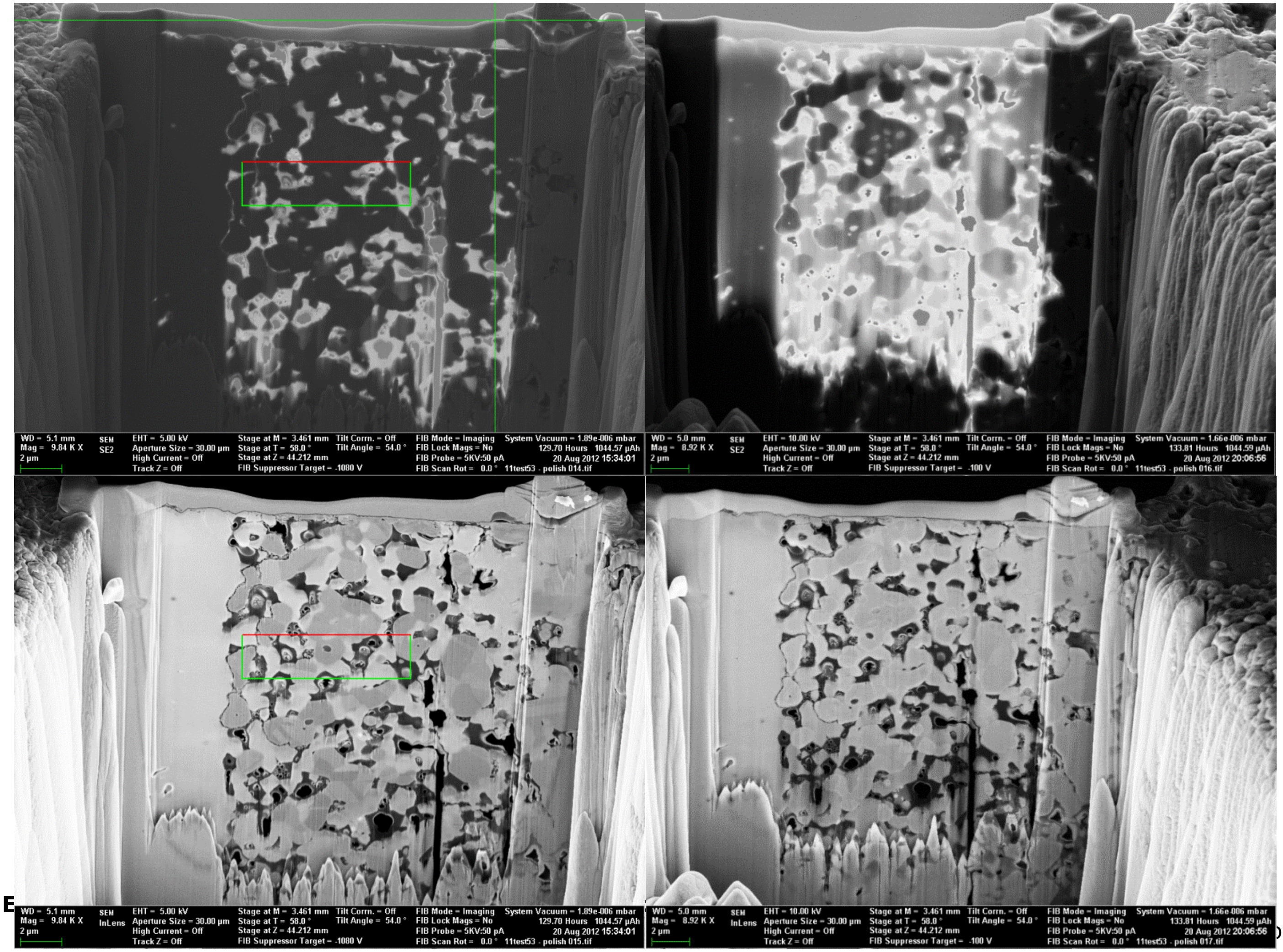
Thickness measurement

5 keV

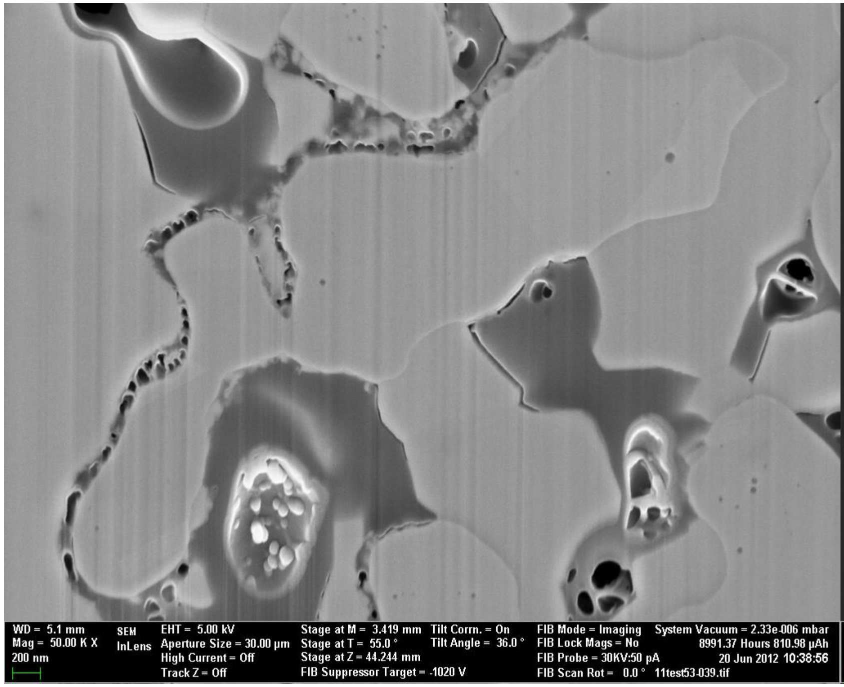
10 keV

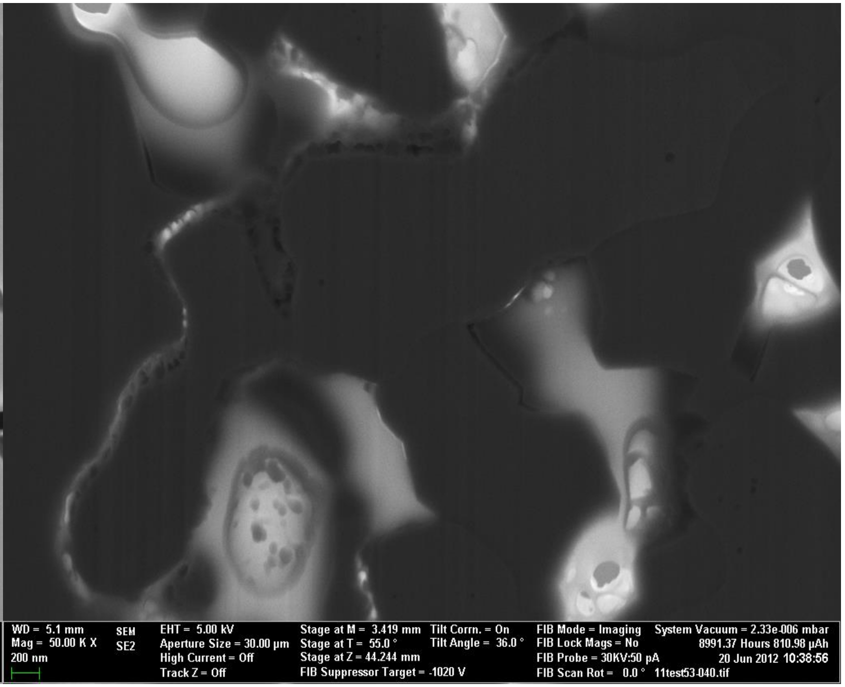
SE2

In-lens



Location for TEM observation – 5 kV





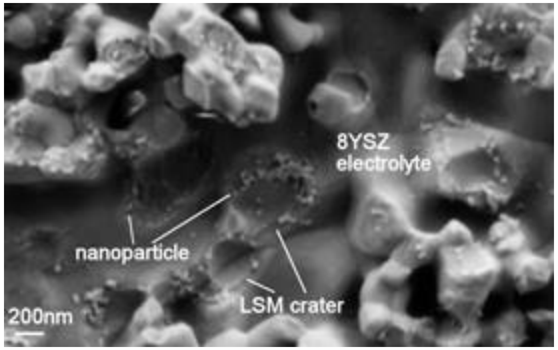
Take a minute to discuss with your neighbour

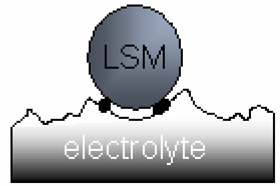
- What is the minimum obtainable TEM lamella thickness?

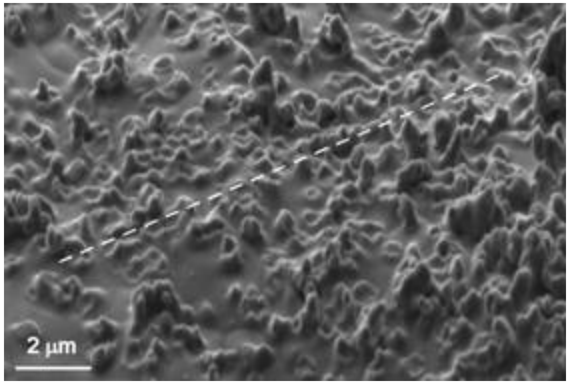
  - At least three times the thickness of the damage layer
  - E.g. Silicon damage layer is about 20 nm with 30 keV Ga ions

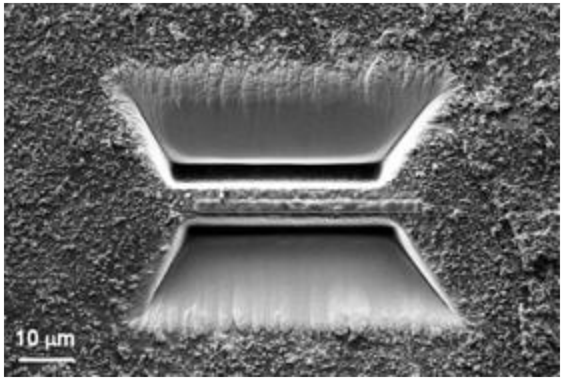
Some examples of investigations of FIB prepared samples

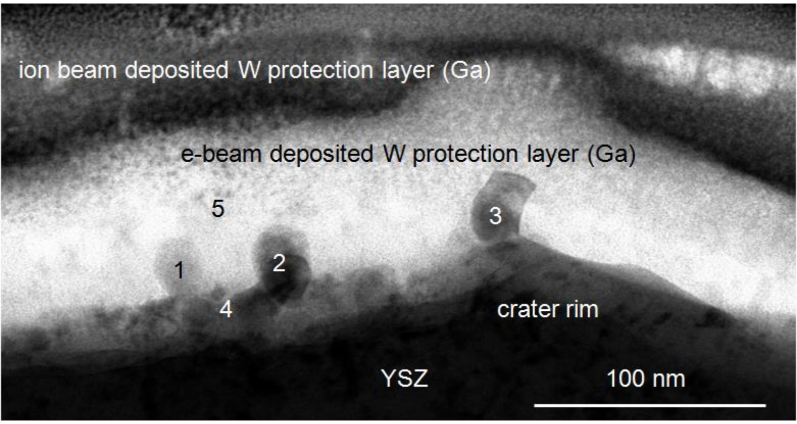
Site specific TEM prep: SOFC cathode impurity nano particle

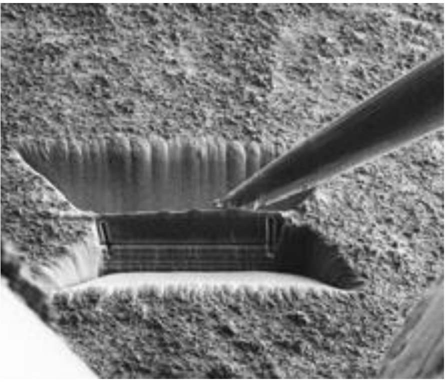


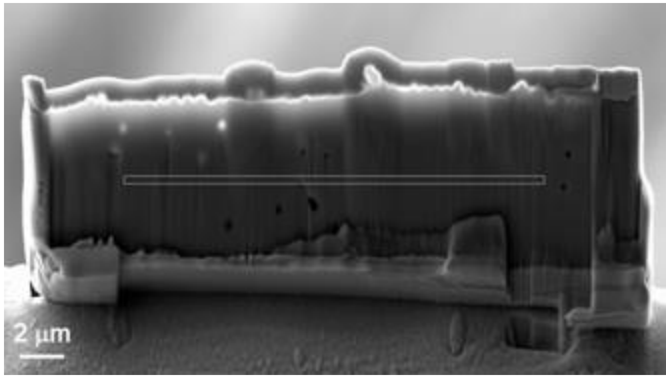




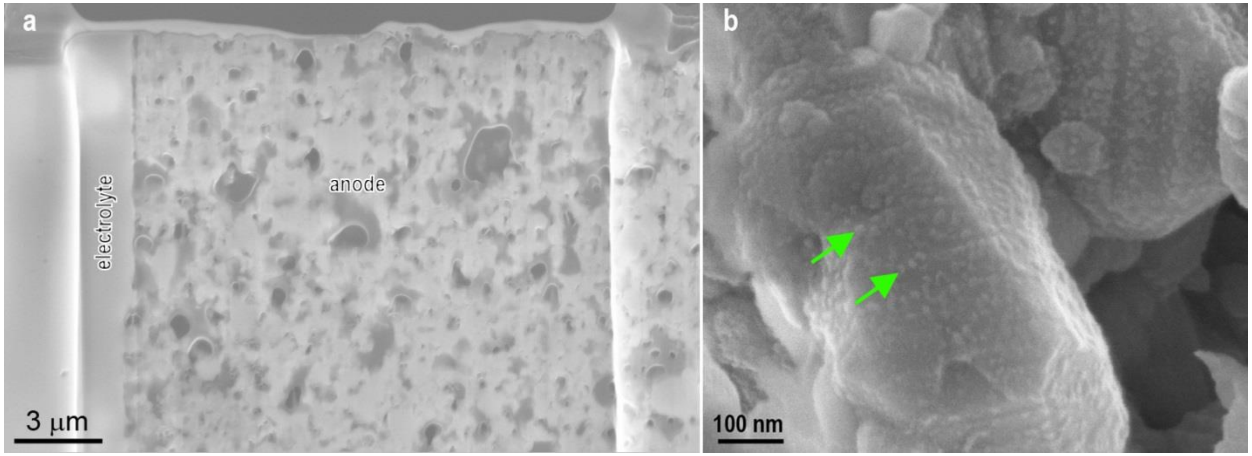


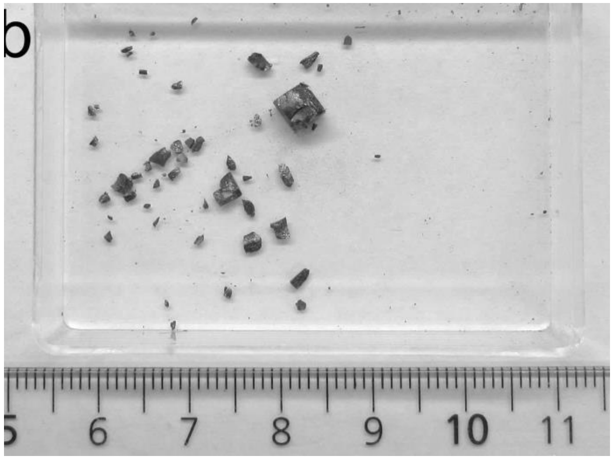


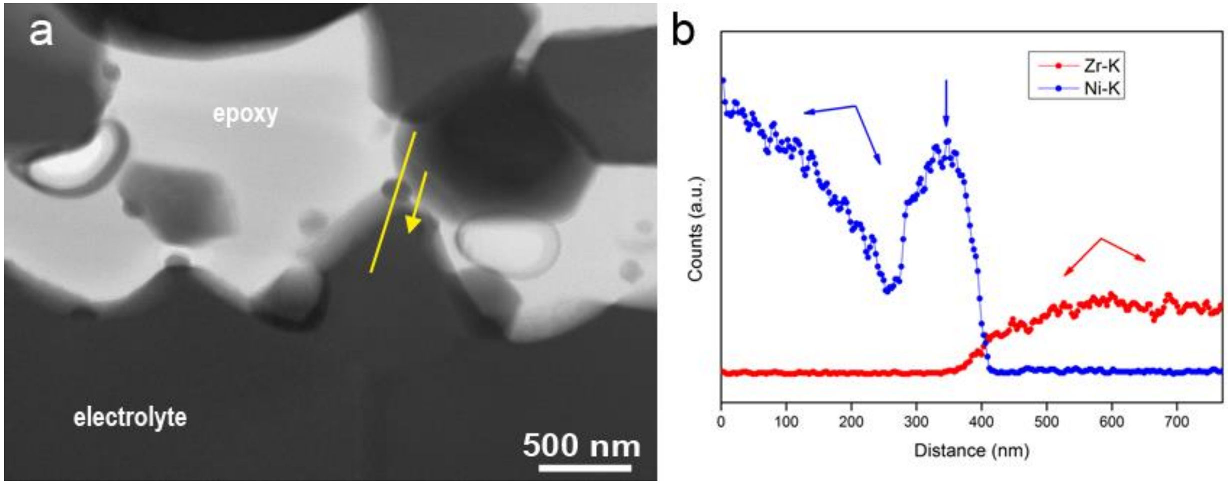


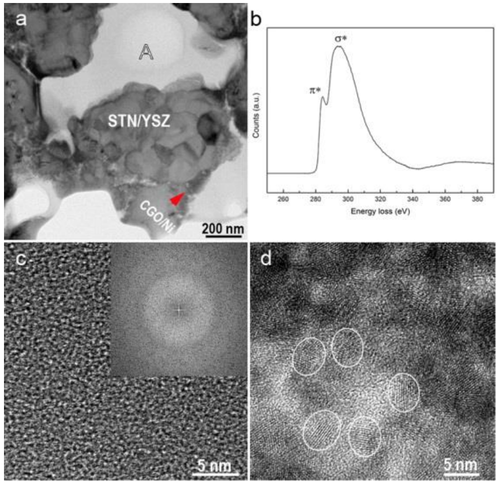


Protecting nano-particles on porous substrates using epoxy impregnation



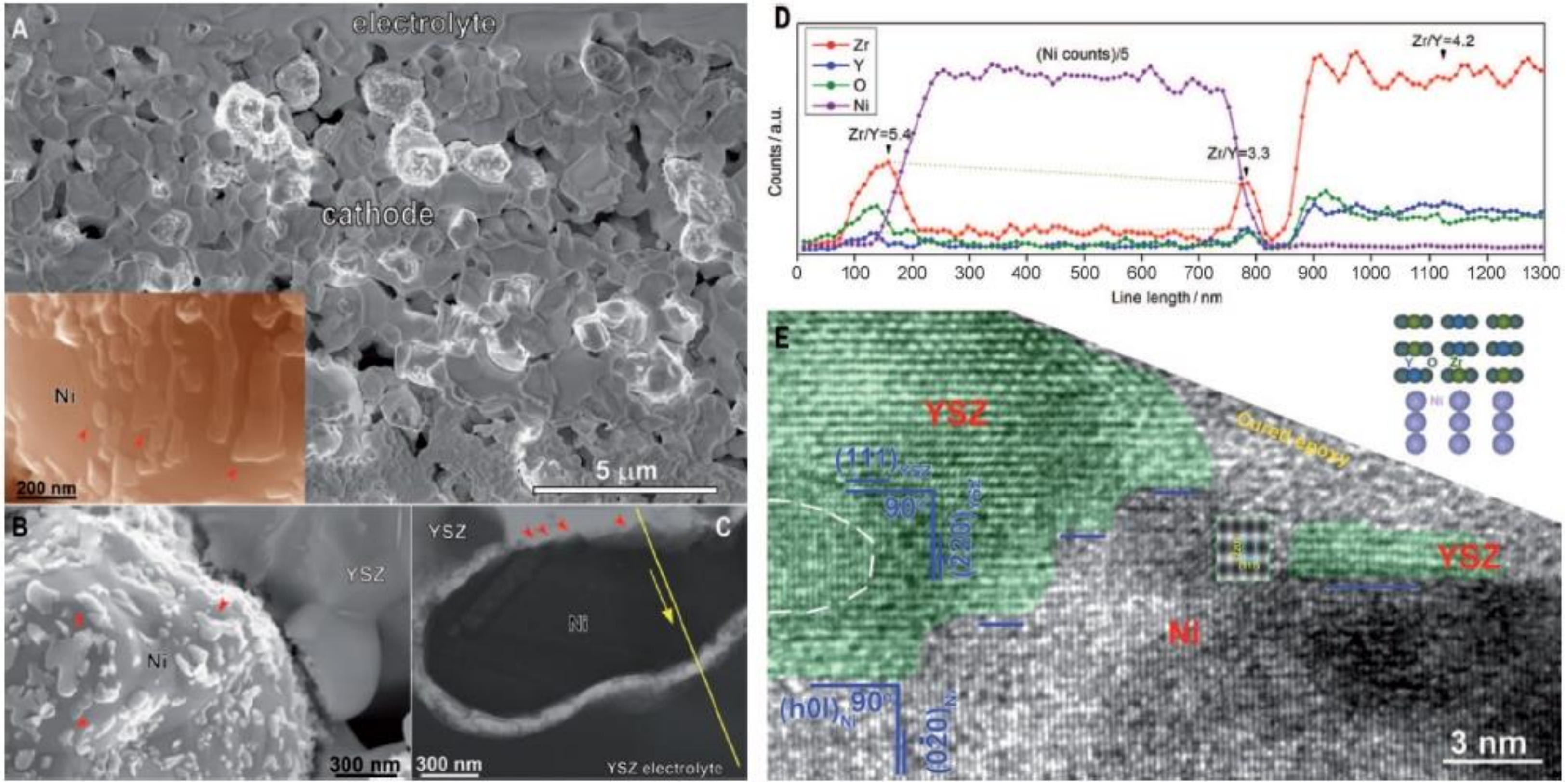






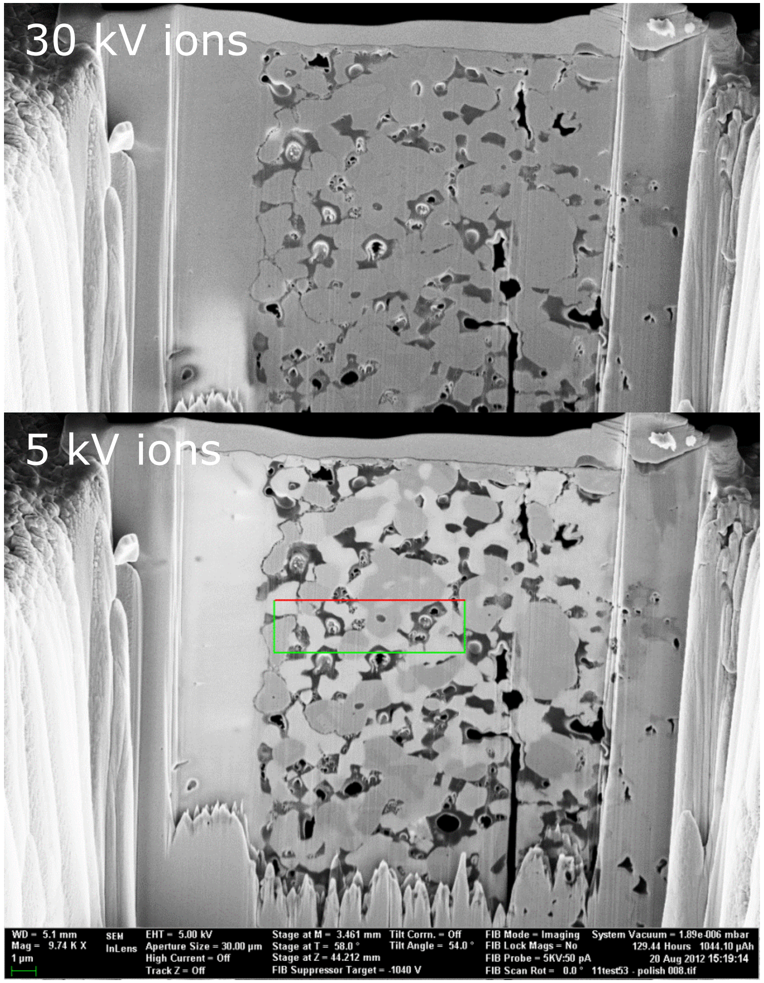
Zhang et al. 2013 http://dx.doi.org/10.1017/S1431927613000019

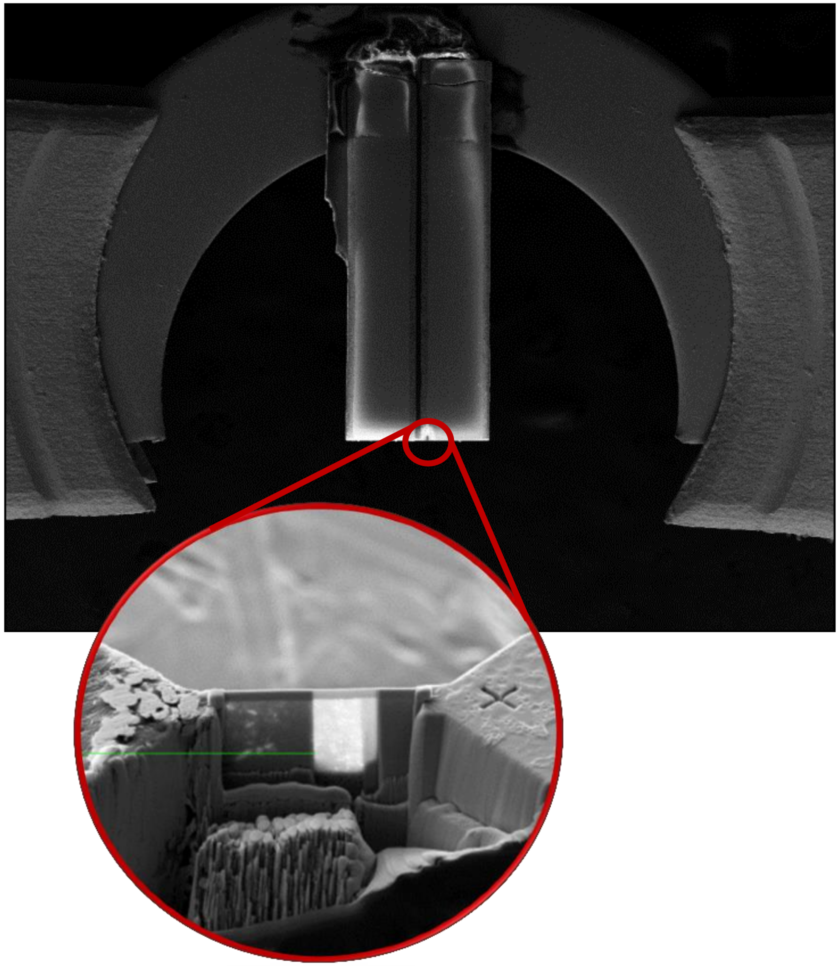
Epitaxy of Zirconia nano-particles



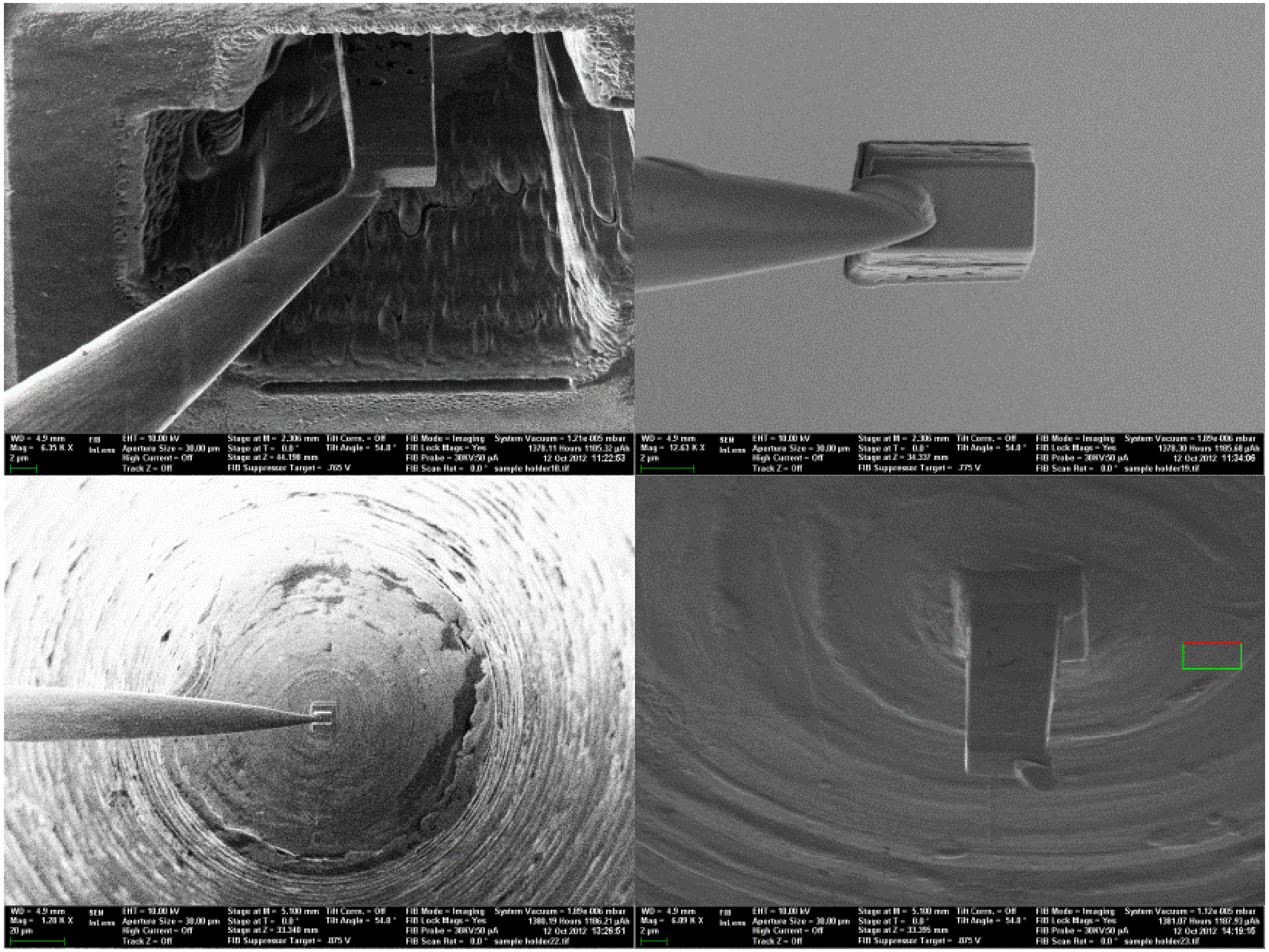
Zhang et al. 2014 http://dx.doi.org/10.1002/celc.201300045

An example of ion beam damage





Extraction of polymer solar cell sample for synchrotron X-ray experiment



DTU Energy Conversion, Technical University of Denmark

Courtesy: K. Thydén

Serial sectioning & 3D microscopy

Name some parameters available from 3D structures

- Phase fraction
- Particle size & distribution
- Particle number density
- Connectivity / percolation
- Tortuosity
- Particle shape / pathway local shape e.g. constrictions
- Surface area (total and phase/interface specific)
- Surface curvature & roughness
- Length of linear feature and linear density (e.g. TPB)
- Location of specific particles e.g. clustering
- …

2006: First 3D reconstruction of SOFC electrode published

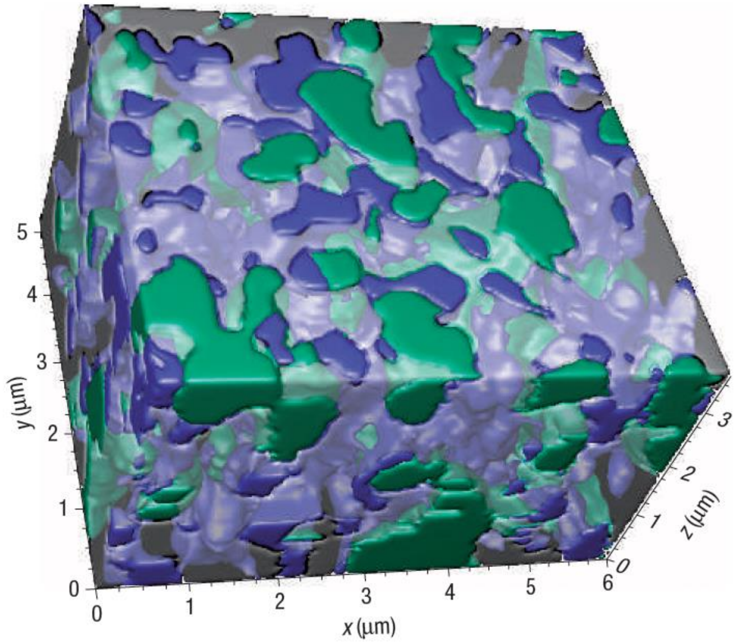


Figure 2 **3D anode reconstruction.** A view of the 3D reconstruction showing the Ni (green), YSZ (translucent/grey), and pore (blue) phases.

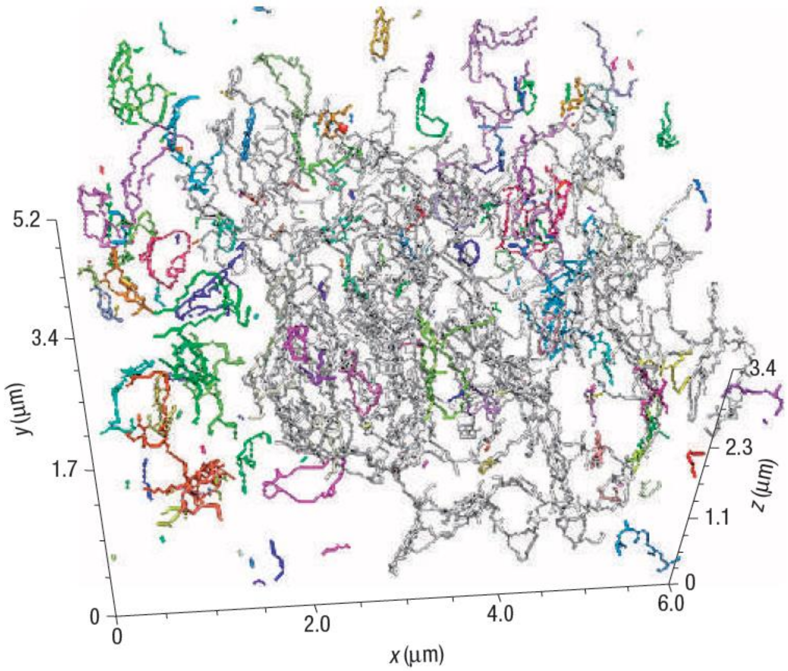
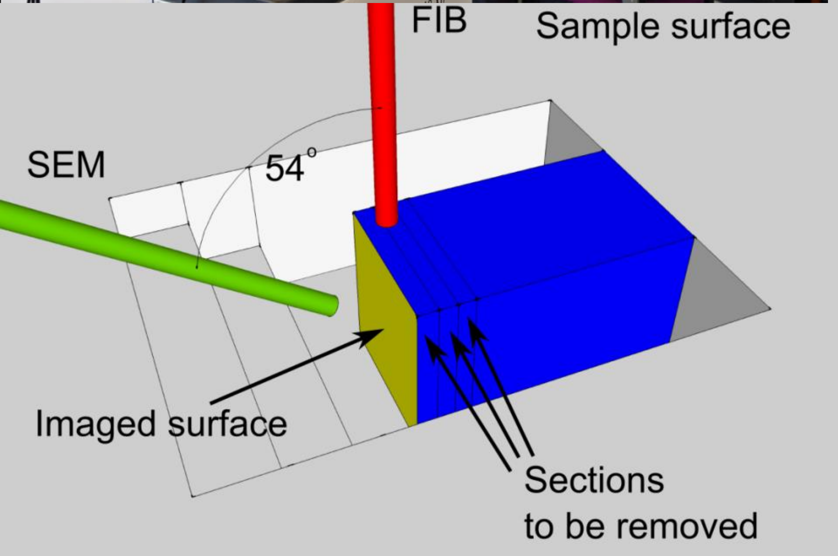
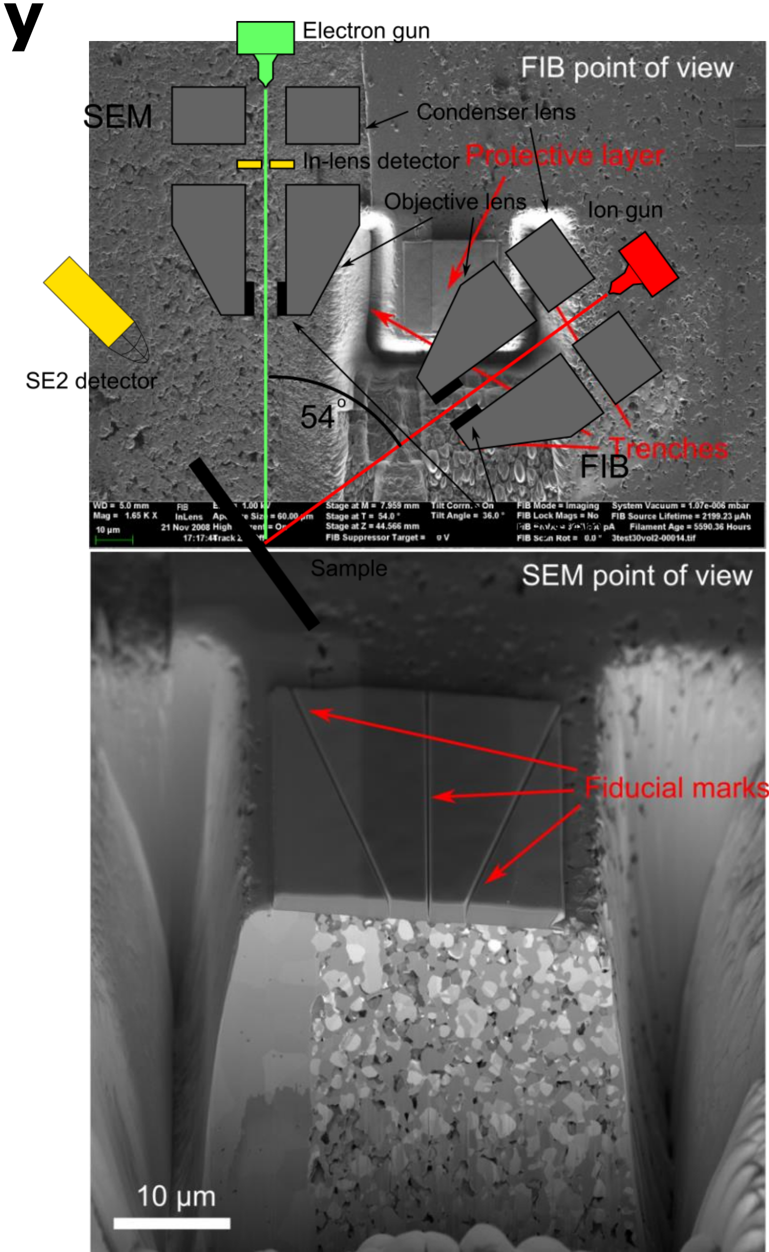


Figure 3 **3D map of the three-phase boundaries in the anode.** Each colour represents a set of contiguous TPBs. The majority of the TPB length (63%) is connected (coloured white/grey). The remaining length consists of shorter, disconnected TPB segments (having colours other than white/grey). A fraction of these intersect the sample boundaries, and hence may be connected to larger segments existing outside the sample volume. However, a substantial fraction (19%) of the TPBs contact neither the highly interconnected white/grey TPBs nor the sample boundaries, that is, they are actual short segments.

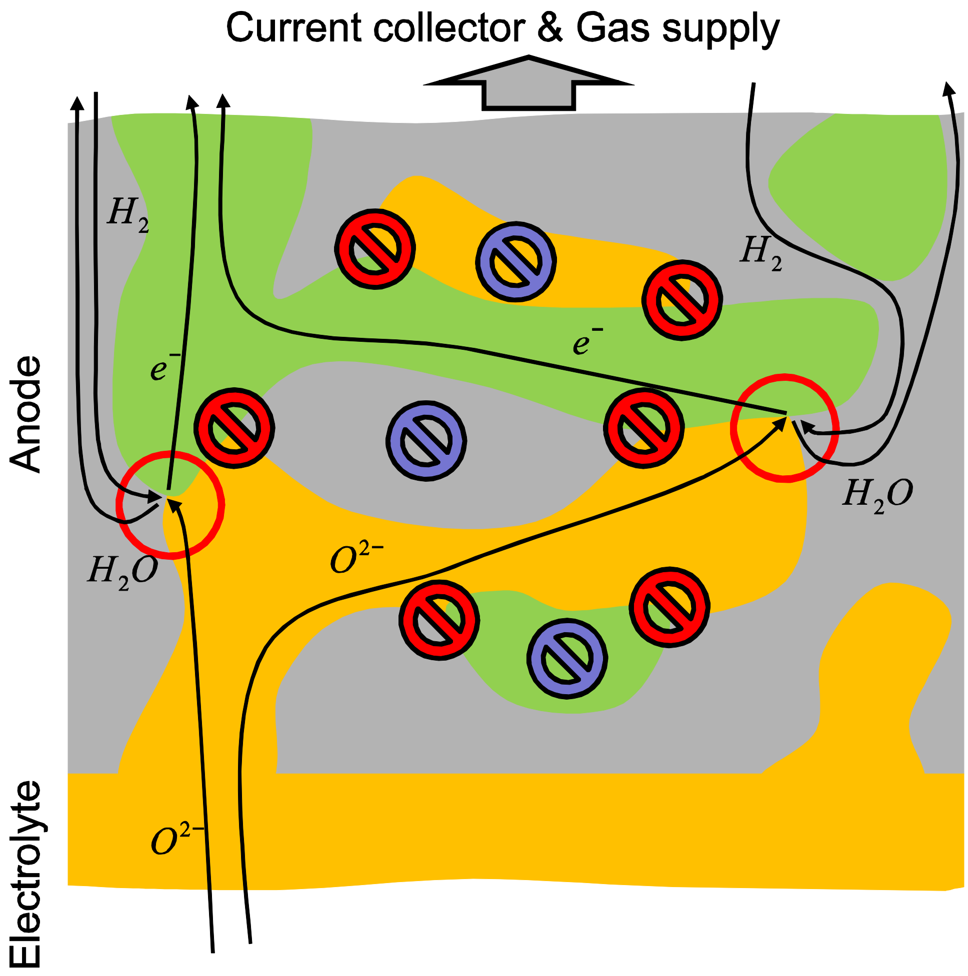
Wilson et al. 2012 http://dx.doi.org/10.1038/nmat1668

Focused ion beam tomography





Now inaccessible 3D electrode parameters are available



○ Active triple phase boundary

🟩 Electron conducting phase

⬜ Gas phase

🟨 Ion conducting phase

⊘ Non-percolating triple phase boundary

⊘ Non-percolating phase structure

FIB serial sectioning factors

- SEM drift → Image alignment
- Curtain effects & top surface roughness
- FIB drift → milling artefacts
- Z dimension calibration (slice interval)
- Z resolution (given by electron interaction volume)
- Image intensity correction
- Maintaining SEM focus & stigmatism
- Charging effects & SEM image distortion
- Tilt correction
- Intensity saturation for both ROI & fiducial marks
- Volume & resolution versus time
- Choice of FIB beam probe current
- Y dimension artefacts with milling depth
- Re-deposition on trench side walls
- SEM image acquisition mode (frame averaging, FIB on/off)

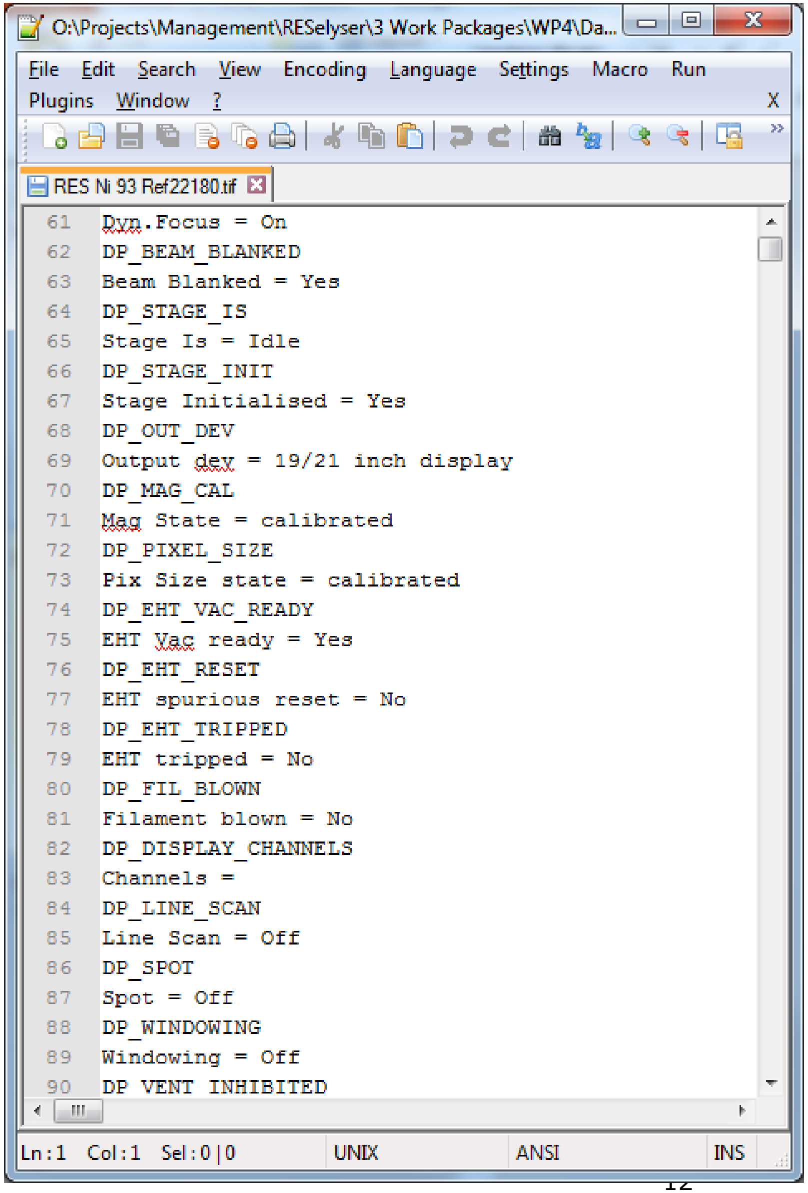
Image acquisition

- Synchronous imaging
  - Mill slice → stop FIB → take SEM image
  - Usually a built-in function in control system
- Asynchronous imaging (partial slice contained in images)
  - Image whilst milling (usually requires rapid frame rate and frame averaging to reduce noise)
    - Acquire a video
      - Choose frame rate
      - Choose video resolution (limited)
      - Choose compression
      - No tiff header
    - Acquire single frames (requires a macro)
  - Pause milling (can use single slow scans no FIB interference)
    - Acquire single frames at specified time intervals (requires macro)

- Choose type according to sample charging and sample stability etc.

Microscope parameters

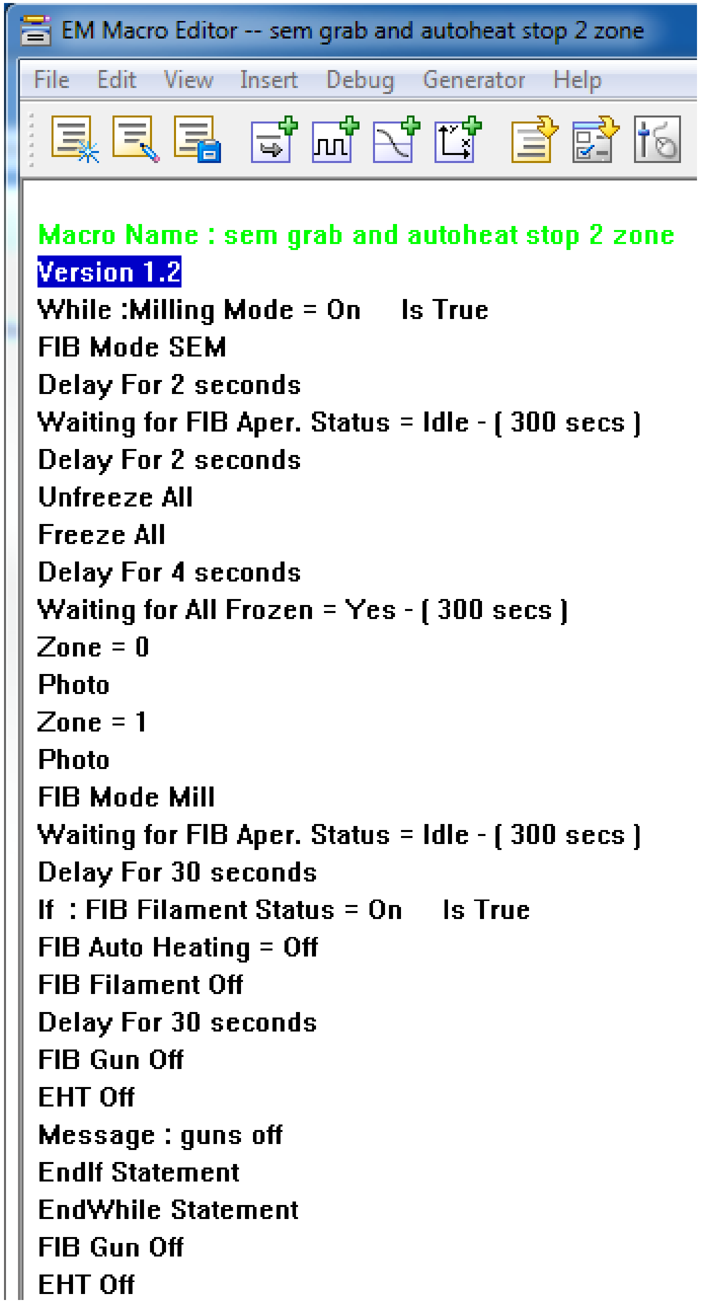
- On new Zeiss and most modern machines all microscope parameters are encoded in images
- Very useful for tracking microscope conditions over time
- The more metadata the better
- Can be incorporated into image processing programs



```
Dyn.Focus = On
DP_BEAM_BLANKED
Beam Blanked = Yes
DP_STAGE_IS
Stage Is = Idle
DP_STAGE_INIT
Stage Initialised = Yes
DP_OUT_DEV
Output dev = 19/21 inch display
DP_MAG_CAL
Mag State = calibrated
DP_PIXEL_SIZE
Pix Size state = calibrated
DP_EHT_VAC_READY
EHT Vac ready = Yes
DP_EHT_RESET
EHT spurious reset = No
DP_EHT_TRIPPED
EHT tripped = No
DP_FIL_BLOWN
Filament blown = No
DP_DISPLAY_CHANNELS
Channels =
DP_LINE_SCAN
Line Scan = Off
DP_SPOT
Spot = Off
DP_WINDOWING
Windowing = Off
DP_VENT_INHIBITED
```

Macro control

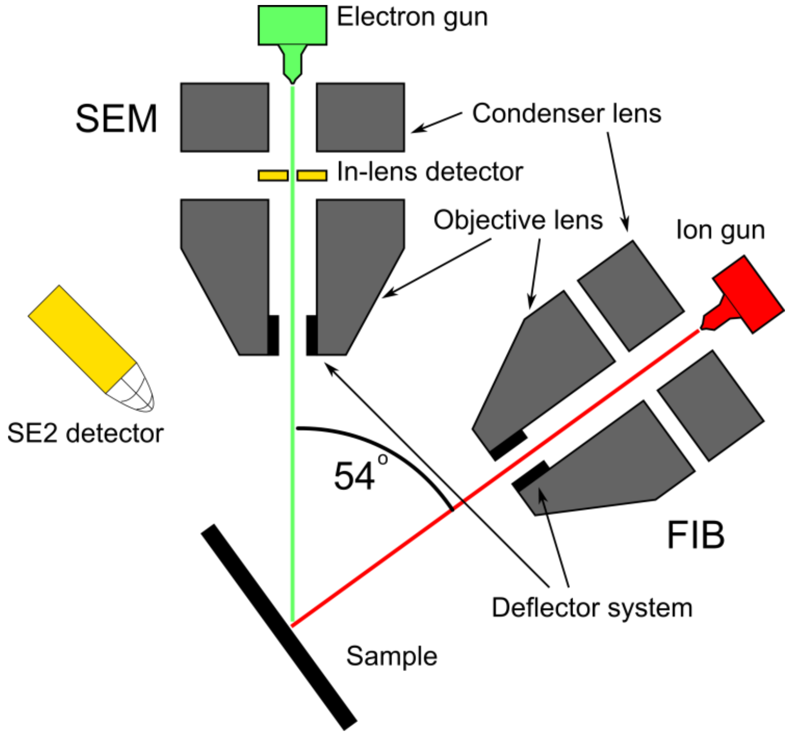
- Useful to customise image acquisition
- Very useful for microscope safety in unattended operation



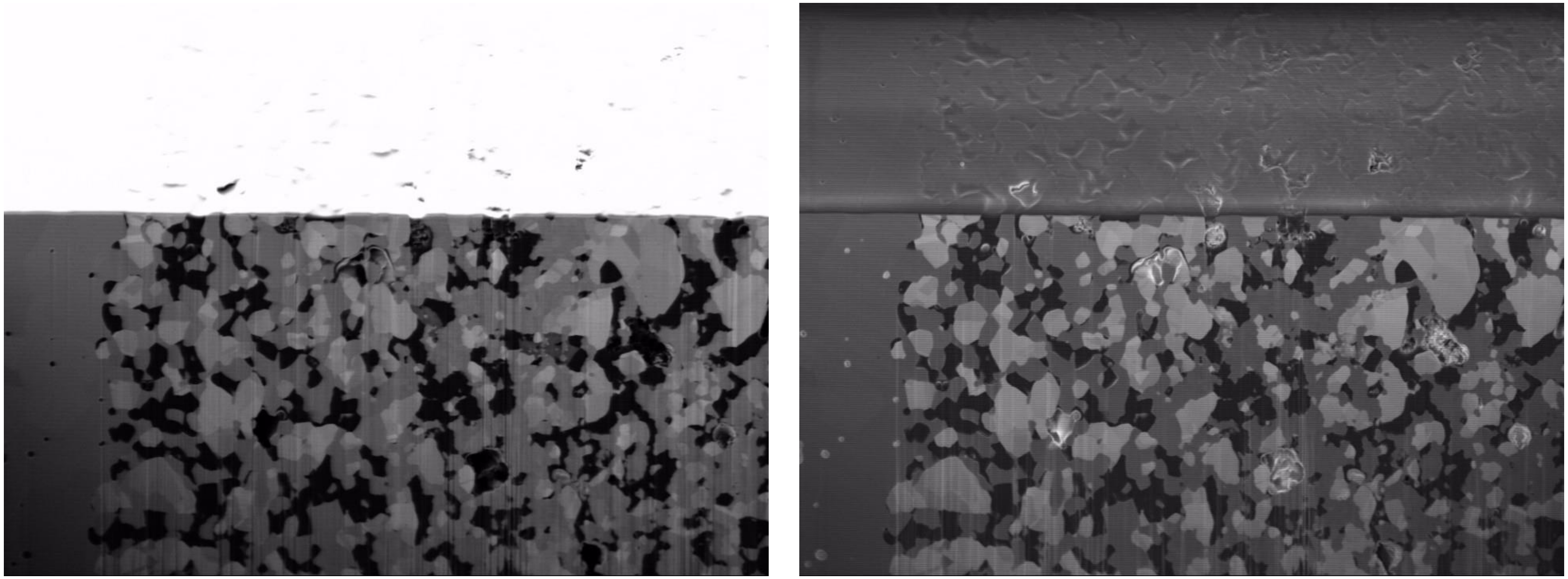
```
Macro Name : sem grab and autoheat stop 2 zone
Version 1.2
While :Milling Mode = On     Is True
FIB Mode SEM
Delay For 2 seconds
Waiting for FIB Aper. Status = Idle - ( 300 secs )
Delay For 2 seconds
Unfreeze All
Freeze All
Delay For 4 seconds
Waiting for All Frozen = Yes - ( 300 secs )
Zone = 0
Photo
Zone = 1
Photo
FIB Mode Mill
Waiting for FIB Aper. Status = Idle - ( 300 secs )
Delay For 30 seconds
If  : FIB Filament Status = On     Is True
FIB Auto Heating = Off
FIB Filament Off
Delay For 30 seconds
FIB Gun Off
EHT Off
Message : guns off
EndIf Statement
EndWhile Statement
FIB Gun Off
EHT Off
```

Maintaining image sharpness

- Accelerating voltage & aperture
  - Controls depth of field
- Dynamic focus
  - Controls depth of focus in sectioning plane
- Tracking working distance
  - Controls slice focus as function of slice number



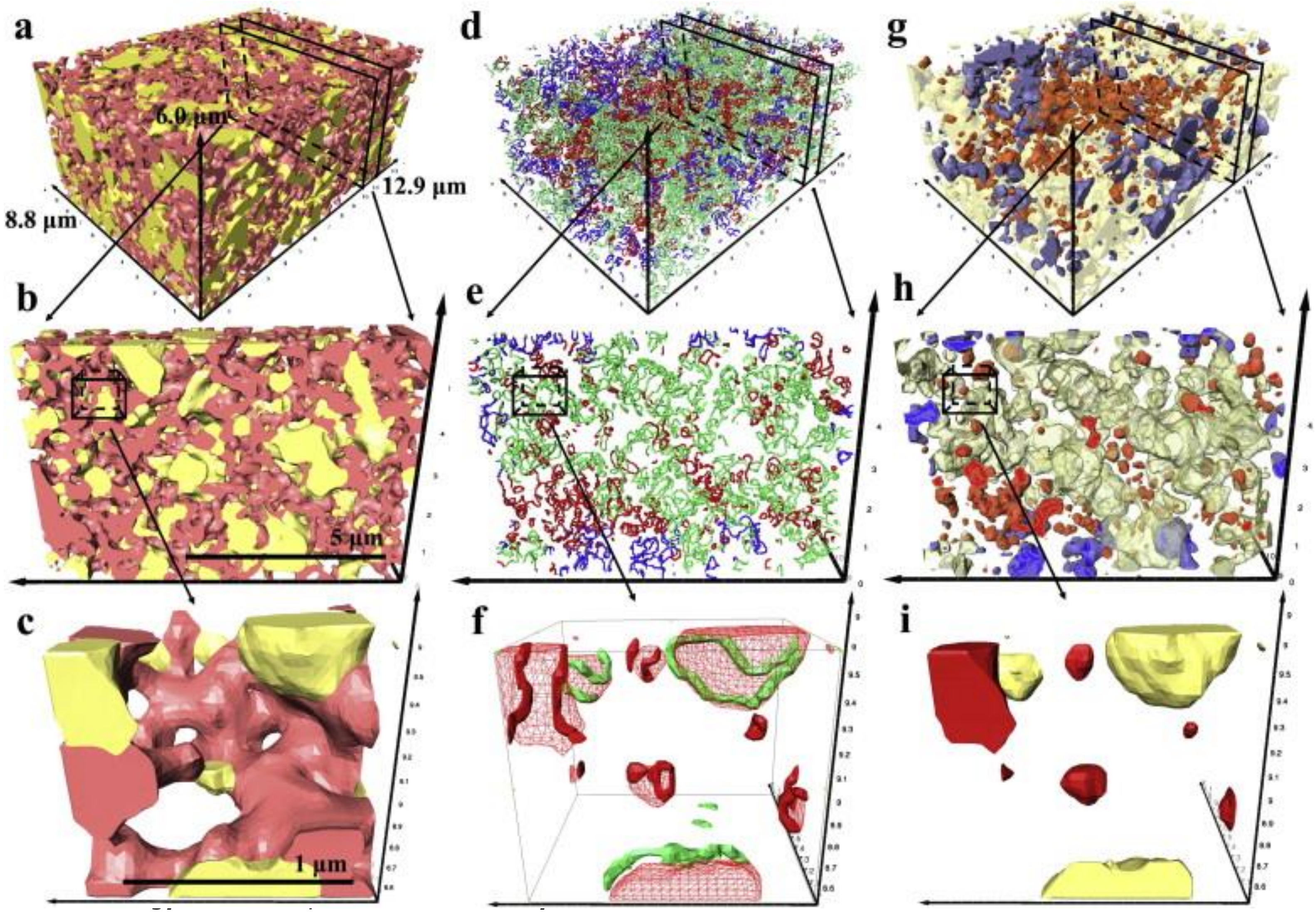
Obtaining phase contrast



Supporting porous structures

- Epoxy impregnation under vacuum
- Supports thin protrusions into pores
- Generates contrast between closed and open porosity

LSM YSZ SOFC cathode



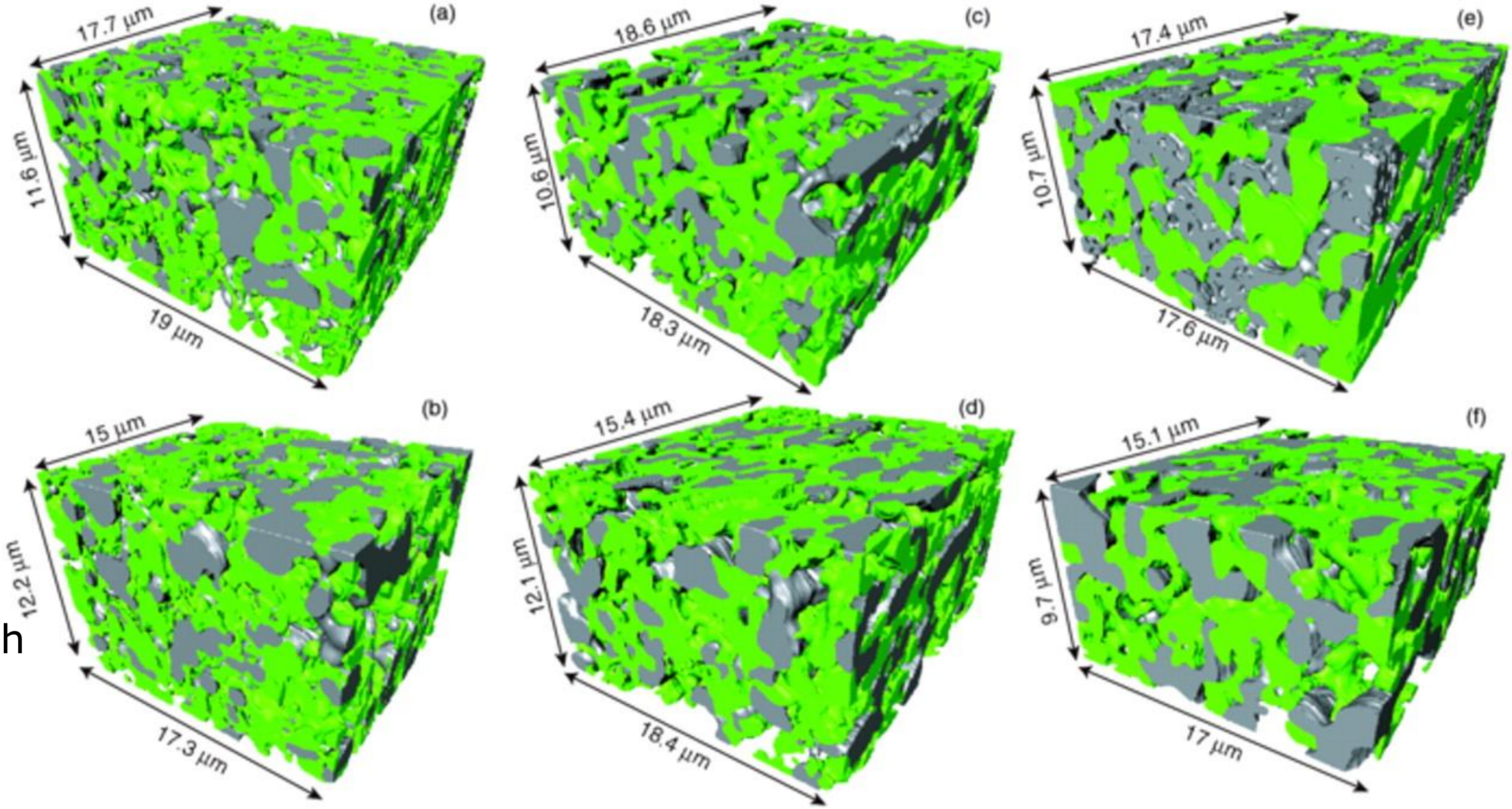
Wilson et al. 2009 http://dx.doi.org/10.1016/j.elecom.2009.03.010

3D reconstructions of SOFC anodes as a function of sintering temperature

1400 C 1450 C 1500 C

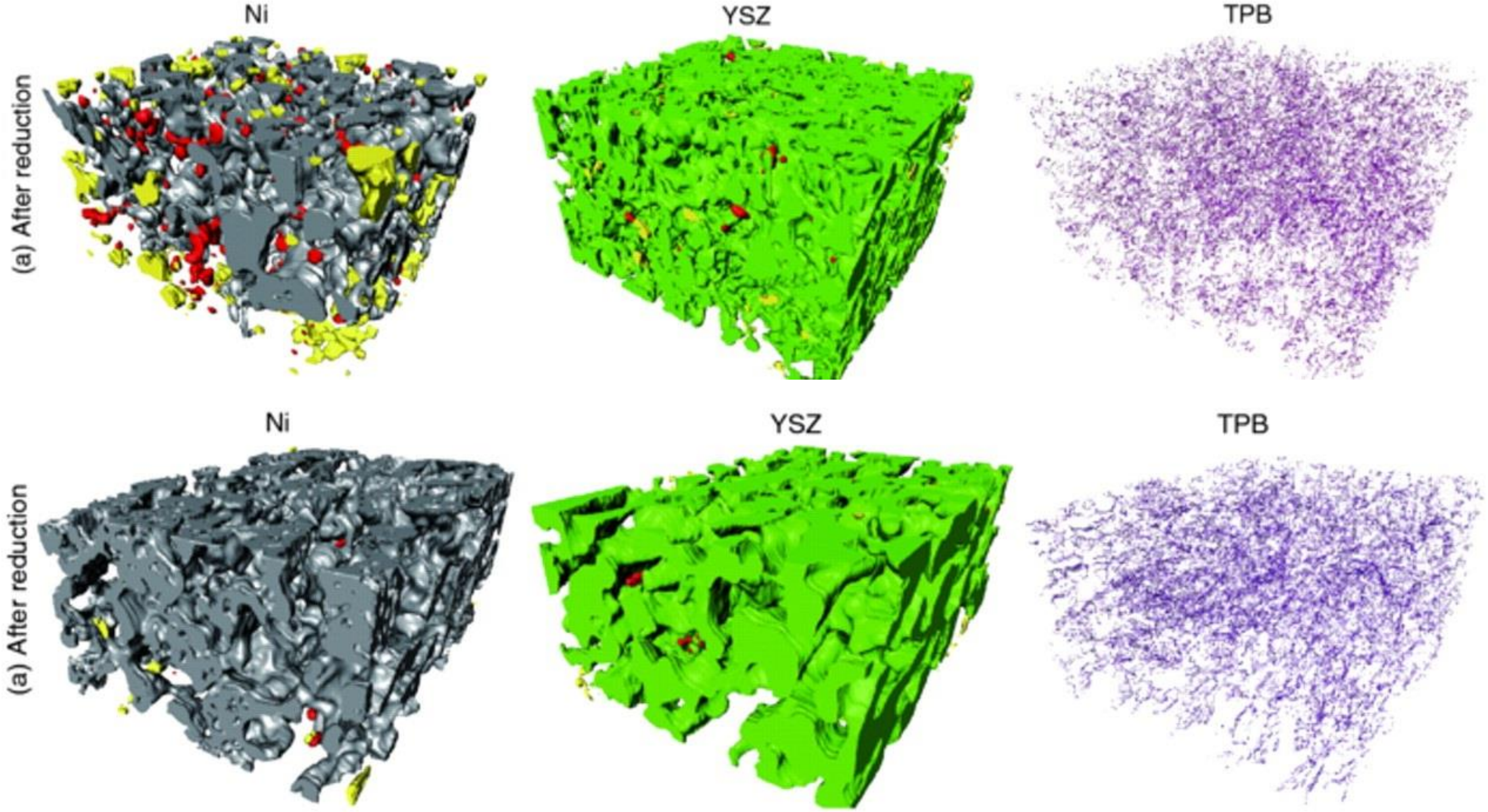
Before operation

After 100h operation

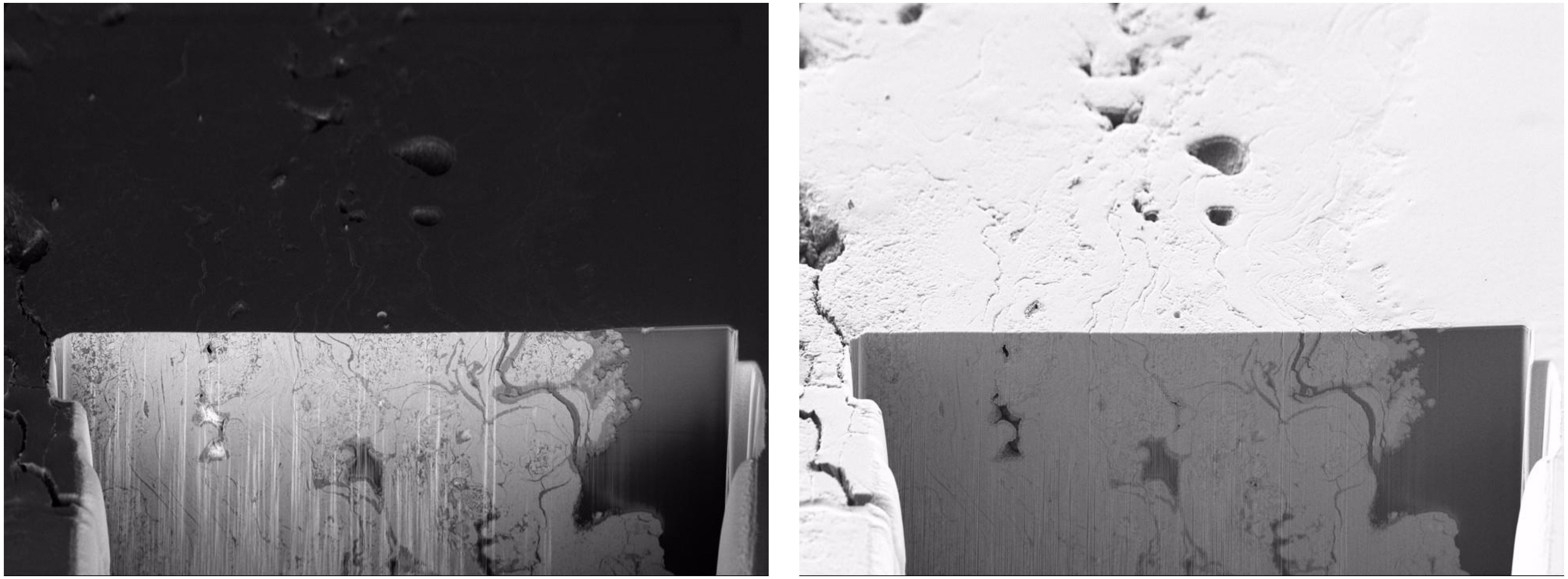


Jiao et al. 2012 http://dx.doi.org/10.1149/2.056207jes

Ni, YSZ and TPB phases for 1400 & 1500C anodes



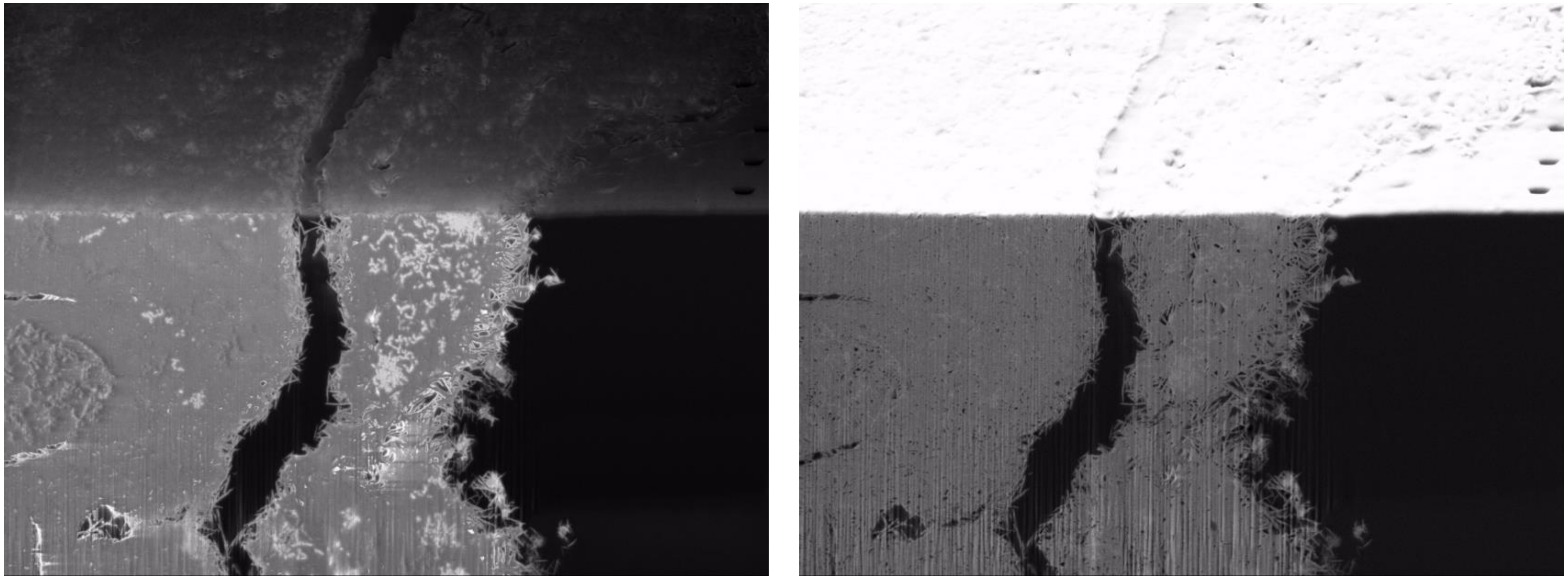
Large milling jobs



Vacuum plasma sprayed Raney Ni H_2 alkaline water electrolysis electrode

Image width = 100 µm, pixel size ~50 nm

Highly heterogeneous structures

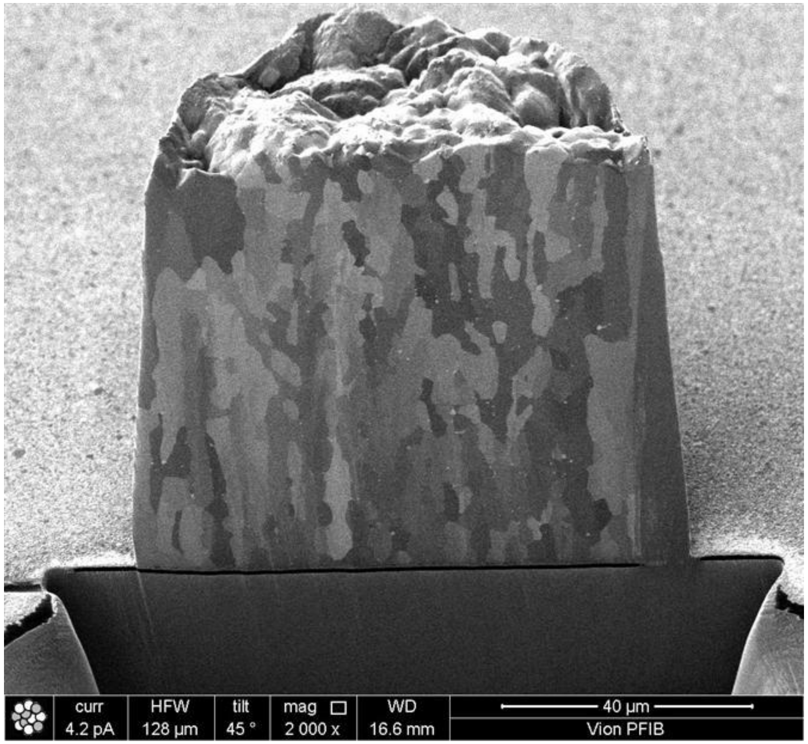


Vacuum plasma sprayed Raney Ni H_2 alkaline water electrolysis electrode

Image width = 20.48 µm, pixel size = 10 nm

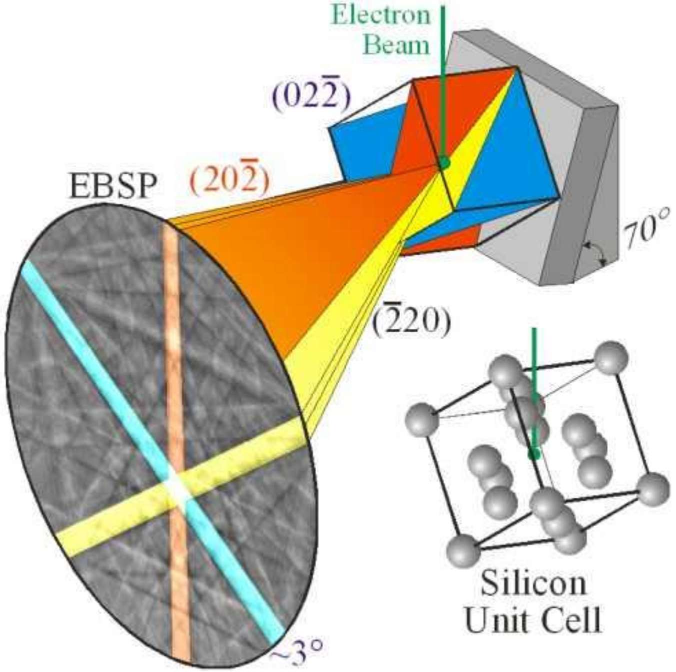
Rapid large volume milling

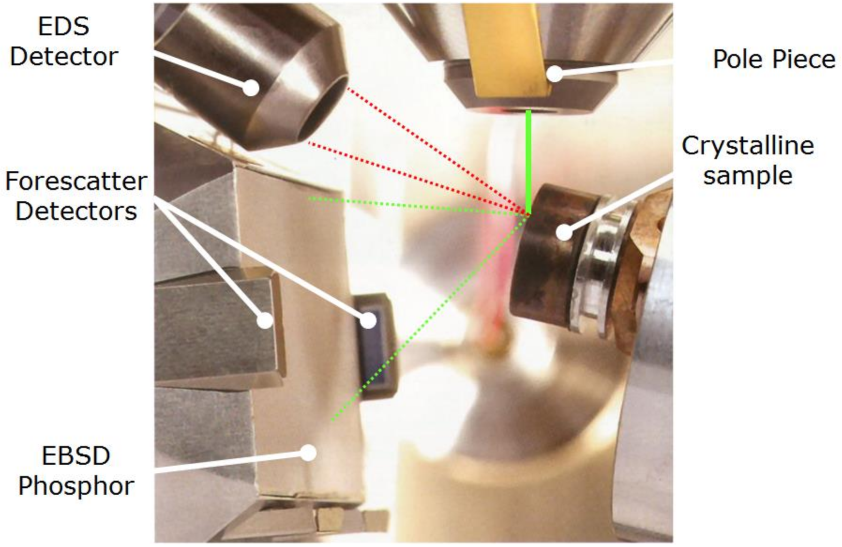
- 80 um wide and 100 um tall bump cross-sectioned with Vion in 20 minutes.



3D-EBSD

Electron backscatter diffraction





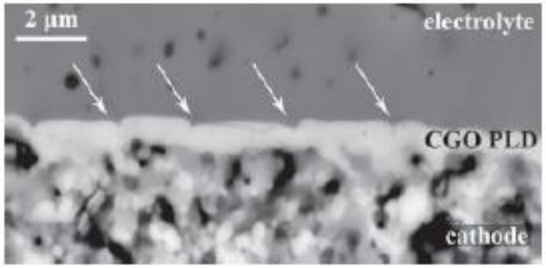
Pixel size resolution limit typically ~25 nm

New transmission method claims 1-2 nm!

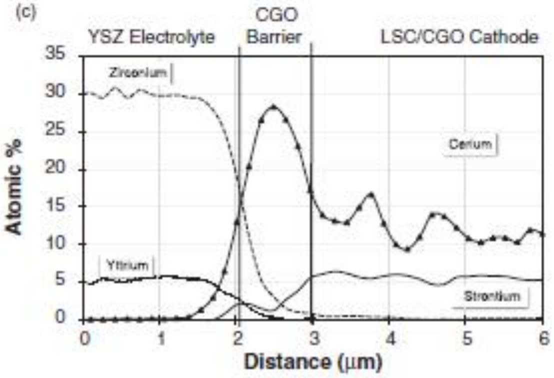
EBSD introduction @EBSD.com

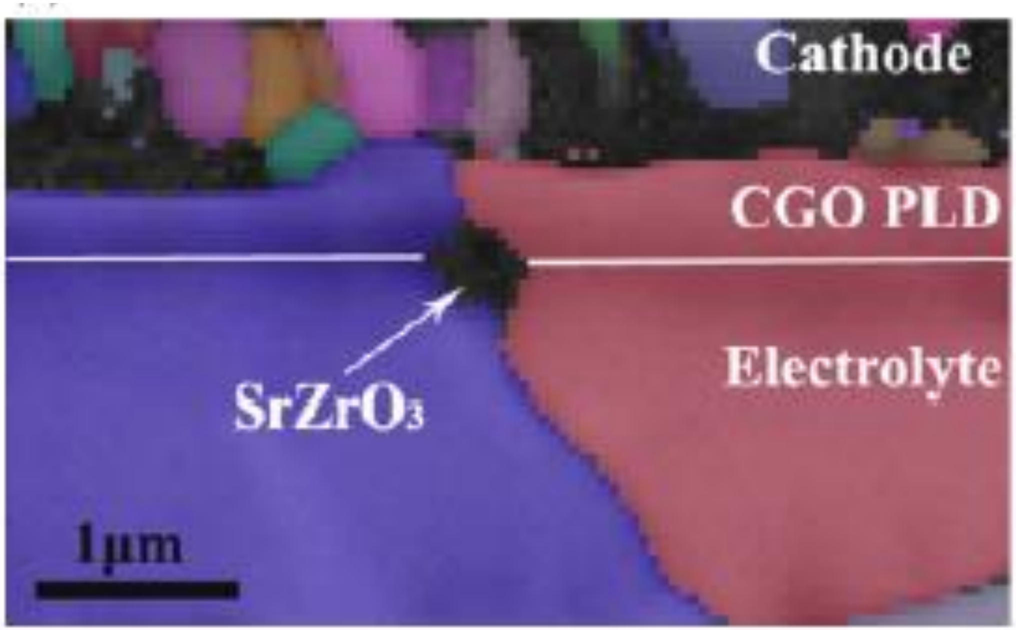
EBSD – PLD epitaxial barrier layer example

(a)



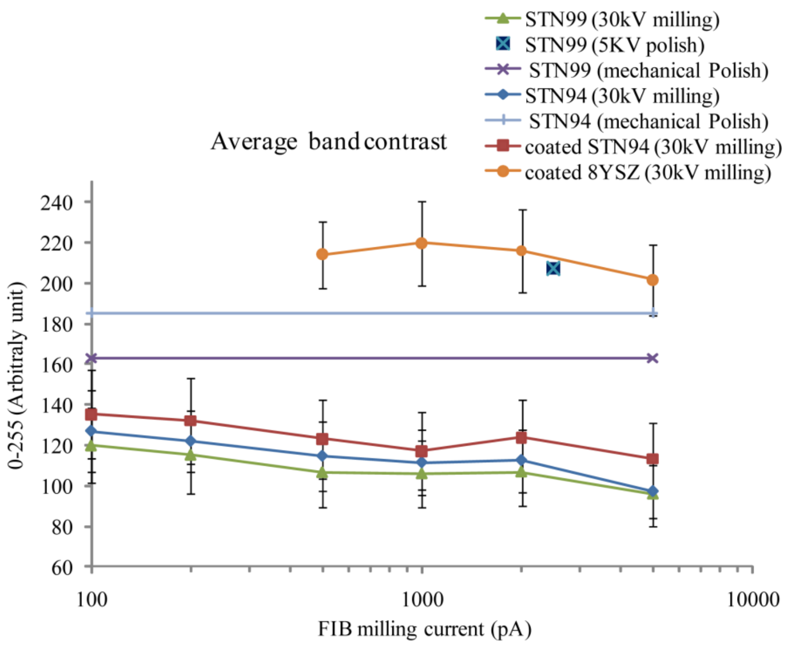
(c)

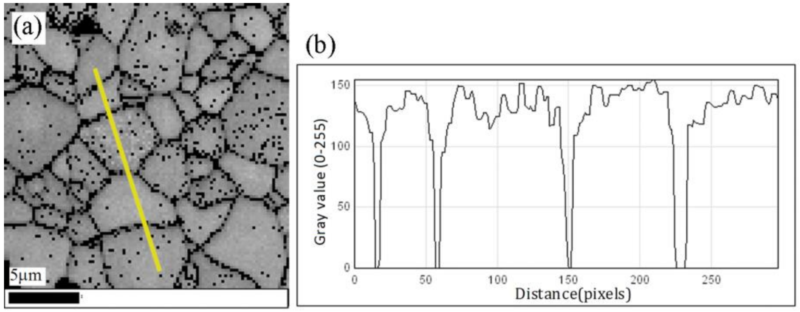


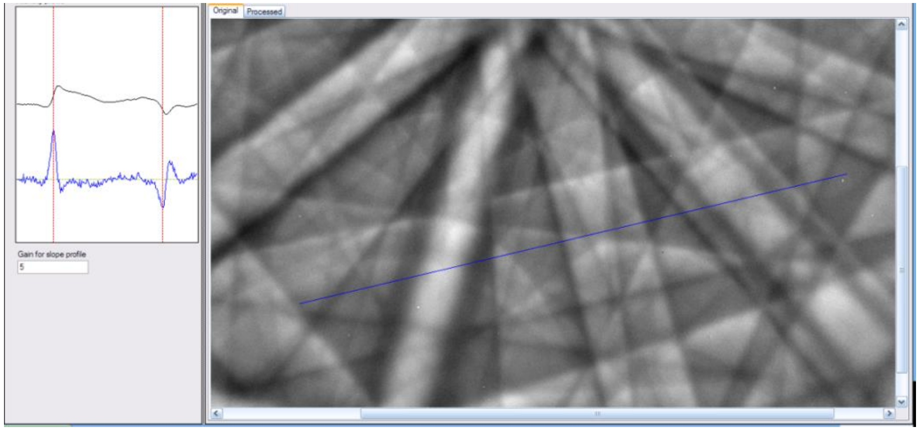


Knibbe et al., J. Am. Ceramic Society. 93 p2877 (2010)

Effect of probe current on EBSD patterns

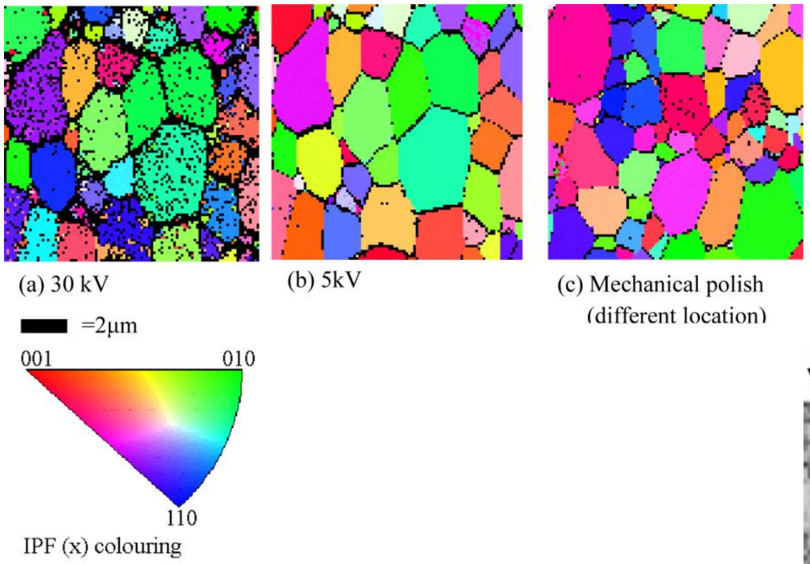






Saowadee et al. 2012 http://dx.doi.org/10.1111/j.1365-2818.2012.03616.x

Effect of ion beam damage on EBSD patterns

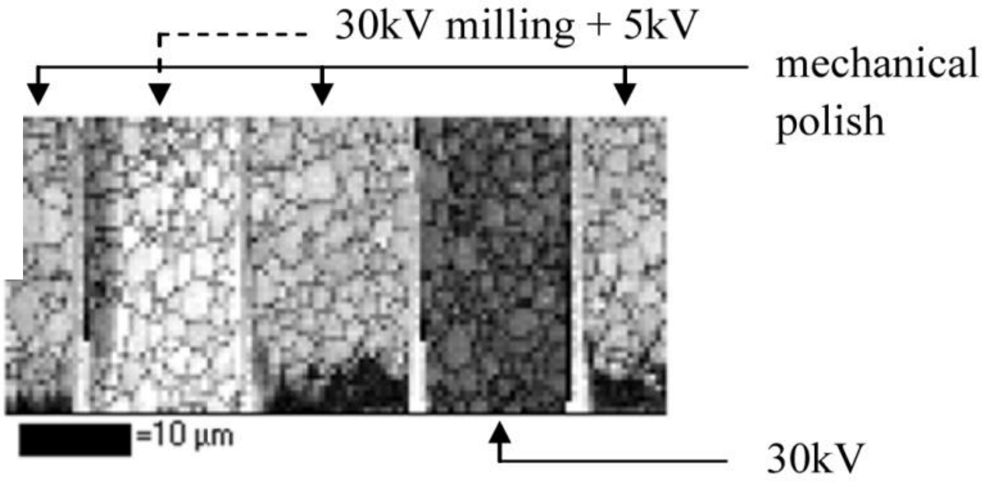


(a) 30 kV

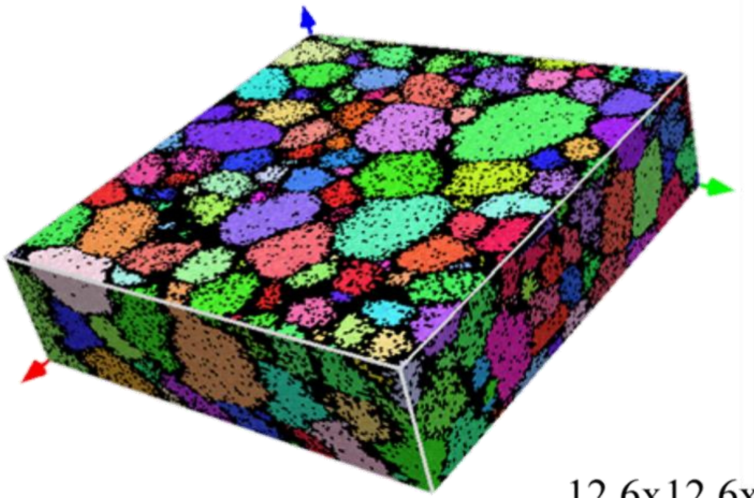
(b) 5kV

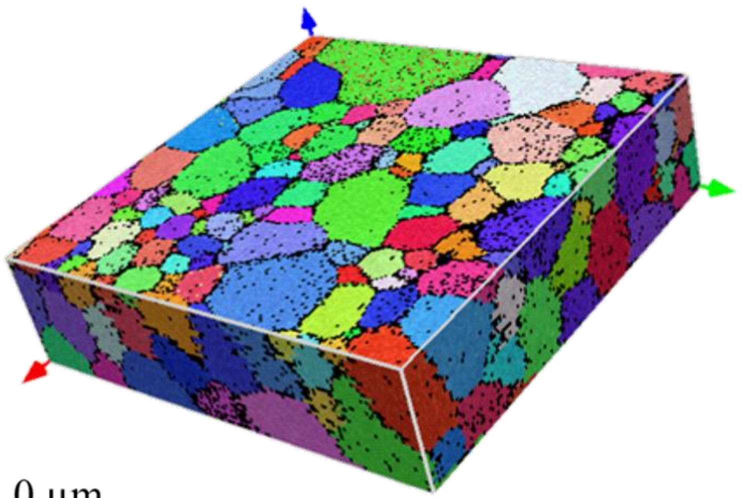
(c) Mechanical polish (different location)

IPF (x) colouring



3D-EBSD – La doped STN

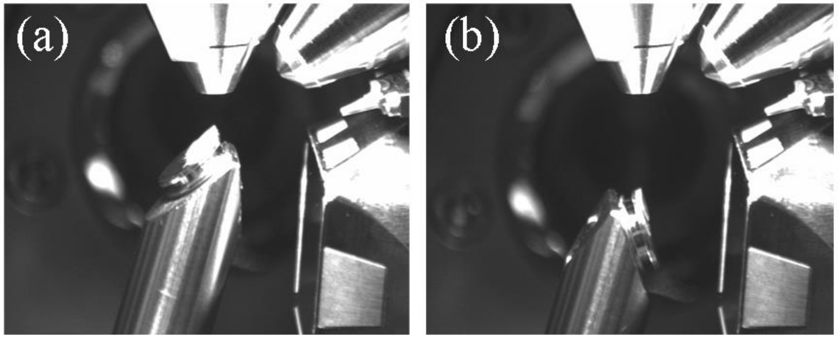


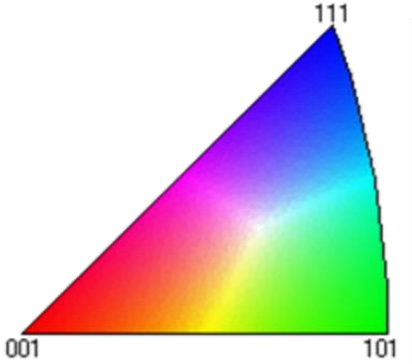


12.6x12.6x3.0 µm

(a)

(b)

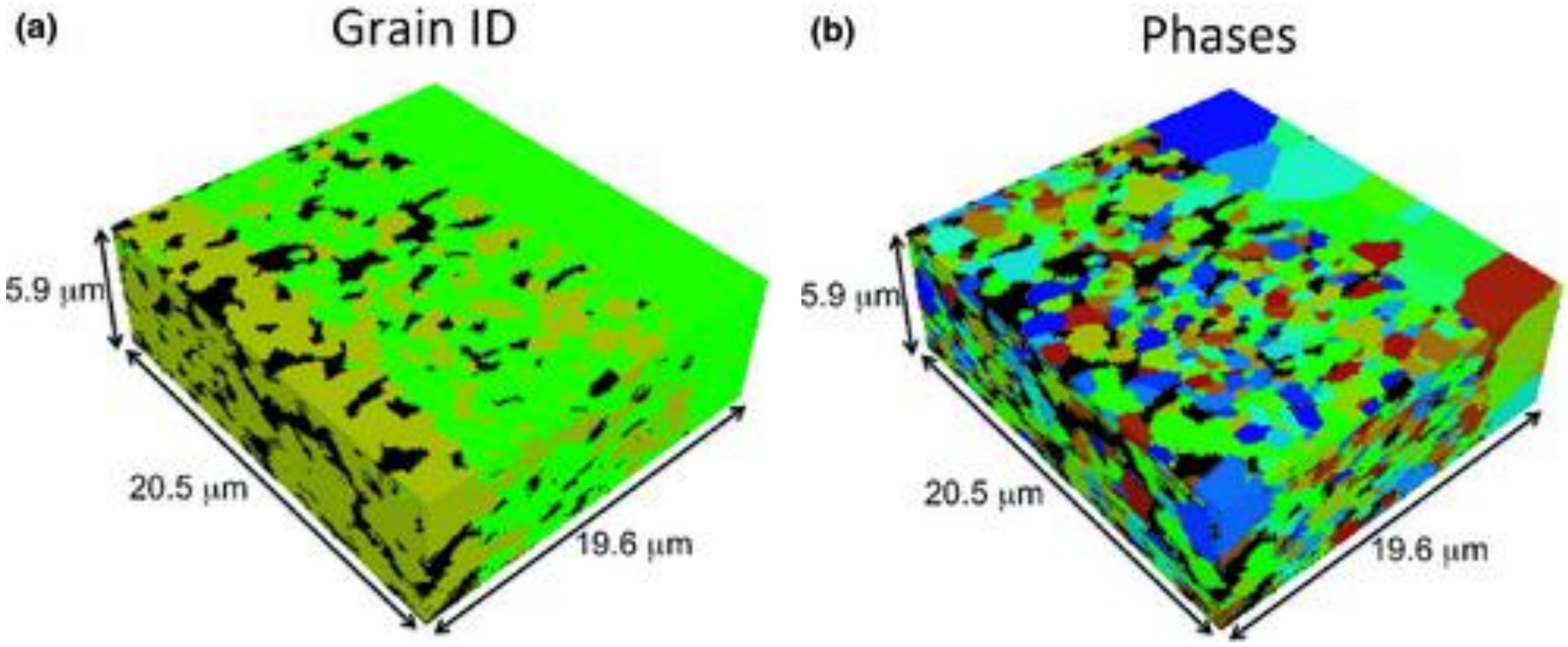




IPF - x

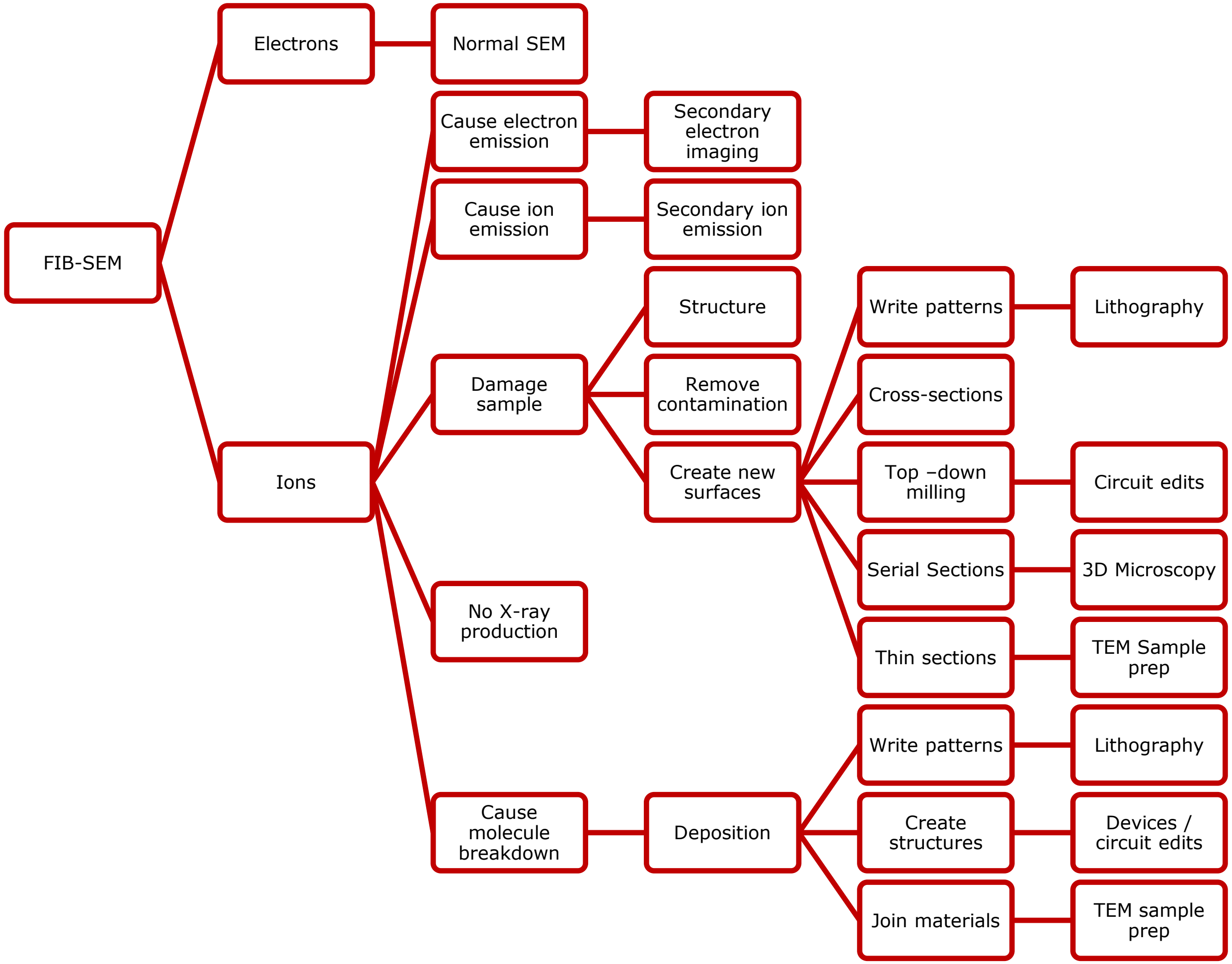
Courtesy: N. Saowadee

Two phase 3D EBSD mapping of LSM-YSZ SOFC cathode



Dillon et al. 2011 http://dx.doi.org/10.1111/j.1551-2916.2011.04673.x

Summary



FIB-SEM

Electrons

Normal SEM

Ions

Cause electron emission

Secondary electron imaging

Cause ion emission

Secondary ion emission

Damage sample

Structure

Remove contamination

Create new surfaces

Write patterns

Lithography

Cross-sections

Top –down milling

Circuit edits

Serial Sections

3D Microscopy

Thin sections

TEM Sample prep

No X-ray production

Cause molecule breakdown

Deposition

Write patterns

Lithography

Create structures

Devices / circuit edits

Join materials

TEM sample prep


Thank you
for your
attention