The Focused Ion Beam – Scanning Electron Microscope
A tool for sample preparation, two and three dimensional imaging

Bowen, Jacob R.

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The Focused Ion Beam – Scanning Electron Microscope: A tool for sample preparation, two and three dimensional imaging

Jacob R. Bowen
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• 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
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
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

10 nA image

Image width ~100 µm
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
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 $\rightarrow$ effects gas pressure
• Choice of probe current $\rightarrow$ 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
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 $\rightarrow$ 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
In-situ manipulation
Image intensity changes on manipulator contact

When lamella is cut free its intensity changes
Touch down and lamella attachment
Final thinning to electron transparency
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

SE2

In-lens
<table>
<thead>
<tr>
<th>Location for TEM observation – 5 kV</th>
</tr>
</thead>
</table>

![Image of TEM observation](attachment:TEM_image.jpg)
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

30 kV ions

5 kV ions

DTU Energy Conversion, Technical University of Denmark
Extraction of polymer solar cell sample for synchrotron X-ray experiment

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.
Focused ion beam tomography
Now inaccessible 3D electrode parameters are available
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
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
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
Ni, YSZ and TPB phases for 1400 & 1500°C 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 – PLD epitaxial barrier layer example

Knibbe et al., J. Am. Ceramic Society. 93 p2877 (2010)
Effect of probe current on EBSD patterns

Average band contrast

- STN99 (30kV milling)
- STN99 (5kV polish)
- STN99 (mechanical Polish)
- STN94 (30kV milling)
- STN94 (mechanical Polish)
- coated STN94 (30kV milling)
- coated 8YSZ (30kV milling)

Effect of ion beam damage on EBSD patterns

(a) 30 kV
(b) 5kV
(c) Mechanical polish (different location)

30kV milling + 5kV mechanical polish

30kV
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

(a) Grain ID

(b) Phases

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

FIB-SEM

Electrons
- Normal SEM
  - Cause electron emission
  - Secondary electron imaging
- Secondary electron imaging
- Cause ion emission
  - Secondary ion emission
- Structure
- Remove contamination
- Create new surfaces

Ions
- Damage sample
- Write patterns
  - Lithography
- Cross-sections
  - Circuit edits
- Top-down milling
  - 3D Microscopy
- Create structures
  - TEM Sample prep
- Devices / circuit edits
- Thin sections
  - Lithography
- Serial Sections
-Join materials
  - TEM sample prep
- No X-ray production
- Deposition
  - Write patterns
  - Create structures
  - Join materials

Cause molecule breakdown
Thank you for your attention