Lecture: PHYS-E0525
Microscopy of Nanomaterials

Focused ion beam (FIB) microscopy and applications

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Learning goals

- History and basic working principal of FIB
  Feature, ion source, ion–solid interaction, basic working principal……

- Basic functions with a FIB
  Imaging, milling, deposition and implantation?

- Main applications by a FIB
  Defect analysis, circuit modification, photomask repair and SEM/TEM sample preparation, 3D slicing/EDX mapping
What is FIB?

Focused ion beam (FIB)
✓ Using highly focused ion beams such as Ga⁺ beam to scan and cut a solid material inside a vacuum chamber.
✓ Imaging and micro/nanomachining technique,
History of FIB microscope

- 1975: The first gas field ionization sources (GFIS)-FIB systems based on field emission technology were developed by Levi-Setti and by Orloff and Swanson [1,2].
  Gas ion sources: He, Ne, Ar, N,......

- 1978: The first FIB based on a liquid metal ion source (LMIS) was built by Seliger et al. [3]
  Metal ion sources: Ga, Alloy,

- Late 1980 late:
  Initial purpose of FIB for circuit modification in semiconductor industry.
  First Rossendorf FIB system created in 1987

SEM vs FIB

Single electron beam system

JSM 7500F SEM

@ OtaNan-Nanomicroscopy Center (NMC), Aalto University

Dual beam system

JIB 4700F FIB-SEM

Main difference: additional ion beam column
 Ion sources: LMIS, GIFS (30kV).

- Probe current: 1pA - 90nA (e.g. JIB 4700F)

- Ion beam diameter: ~5 nm to ~0.5 µm.
Gas field ion source (GFIS)

Ionization potential and polarizability of common gases in GFIS vacuum

<table>
<thead>
<tr>
<th>Gas</th>
<th>Ionization potential (eV)</th>
<th>Polarizability ($10^{-24}$ cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>He</td>
<td>24.6</td>
<td>0.20</td>
</tr>
<tr>
<td>Ne</td>
<td>21.6</td>
<td>0.29</td>
</tr>
<tr>
<td>Ar</td>
<td>15.8</td>
<td>1.63</td>
</tr>
<tr>
<td>H$_2$</td>
<td>15.6</td>
<td>0.80</td>
</tr>
<tr>
<td>N$_2$</td>
<td>14.5</td>
<td>1.74</td>
</tr>
<tr>
<td>CO</td>
<td>14.0</td>
<td>1.97</td>
</tr>
<tr>
<td>O$_2$</td>
<td>13.6</td>
<td>1.57</td>
</tr>
<tr>
<td>H$_2$O</td>
<td>12.6</td>
<td>1.43</td>
</tr>
</tbody>
</table>

For He ion GFIS:

✓ Spot size: 0.35 nm for He

✓ Brightness: 5.0E9 A/cm$^2$·Sr

http://www.orsayphysics.com/what-is-fib

Liquid metal ion system (LMIS)

- By applying an electric potential (such as 30kV) between the needle and a downstream metallic extractor.
- A structure known as Taylor cone is formed at the tip of needle.
- Once exceeding a threshold voltage, ion and droplets are extracted from the cone ($E > 1 \times 10^8$ V/cm)
- The extracted ions pass through the hold of extractor.

With Ga ion LMIS:
- Spot size: 3-5 nm
- Brightness: $\sim 3.0 \times 10^6$ A/cm²·Sr
- Probe current 1pA-90nA

Other metal ion sources?
- Au-Si, Au-Ge and Au-Si-Ge
Why Ga ion in LMIS?

- **Low melting point** (29.8°C)
- **Heavy enough** for milling the heavier elements
- **Low volatility** at the melting point (a long source life of about 400 mA-hours/mg)
- **Low vapor pressure**
  --allowing Ga to be used in its pure form instead of in the form of an alloy source.
Why electrostatic lens?

- The ion (positively charged) is much larger and more massive than the electron. $m_i \sim 10^5 m_e$
- Ions travel more slowly
- Larger fields to focus and control ions than electrons:
  Lorentz force: $\vec{F} = q\vec{E} + q\vec{v} \times \vec{B}$
- If magnetic lens, Lorenz force is weaker, so a few km coils will be needed.

Aberrations in ion and electron optics depend on the same factors.
Electron-solid interaction

- Secondary electrons (SE),
- Backscattered electrons (BSE)
- Cathodoluminescence
- Auger electrons
- Characteristic x-ray
- Interaction volume: $\mu$m

Ion-solid interaction

**Nuclear processes**
- Sputtered particles (ions or neutrals)
- Backscattered ions

**Electronic processes:**
- Ion induced secondary electron (iSE),
- X-rays or Auger electrons (low yield)
- Interaction volume: **tens of nm**

Collision cascade (Ga ions)

30 keV Ga\(^+\) ion

Sputtered particles

Emitted e\(^-\)

Solid Collision cascade model

TRIM or SRIM (transport, or stopping range ions in matter)—Monte Carlo simulation

- Projected range \(R_p\) : 10-100 nm
- Lateral range \(R_l\) : 5-50 nm

C. A. Volkert et al., MRS bulletin 32, 389, 2007
Basic functions in a FIB

- FIB imaging
  - iSEs

- FIB sputtering (milling)
  - Primary ion

- FIB assisted deposition
  - Primary ion

- Ion implantation
  - Primary ion
ISE imaging in a FIB–SEM

Contrast mechanism of iSE imaging
- Ion “channels” parallel to crystal planes, fewer electrons are emitted.
- Heavier samples typically result in more ISEs (and SEs).
- Surface topography can lead to increases in the number of ISEs (and SEs).
- Offering complementary information about a sample surface.

Mainly detecting iSEs for imaging in a FIB
A few 1-10 iSEs (10eV) / Ga ion (5-30kV)

Drawbacks by iSE imaging
- Surface damage and ion implantation
  - Channeling effect in iSE imaging is obvious!
  - Imaging resolution: ~5 nm

SE and iSE images from a FIB-cut brass
C.A.Volkert et al., MRS bulletin 32, 389, 2007

Crystal orientation (100)  Crystal orientation (110)  Heavier atoms (110)  Surface geometry (110)
FIB milling

JIB-4700F

FIB-SEM (Dual-FIB)

Ion Milling ⇒ Observation by SEM image

To optimize the following parameters for efficient milling:

1. Ion beam parameters (ion energy, probe current, and beam diameter),

2. Processing parameters (dwell time, beam overlap, ion dose, scanning mode)

3. Target materials (mass, density, and crystallographic orientation)
Energy dependence of sputtering yield of Au and Si target substrates by three types of ions at normal incidence.

- Sputtering yield “saturates” at ~100keV.
- Higher energy leads to significant implantation.

**Ar: Z=18**
**Ga: Z=31**
**As: Z=33**
At higher probe current (i.e. larger spot size), higher sputtering yield but lower resolution, vice versa.
Focus and astigmatism

Much necessary with **good focus** and **low astigmatism** before milling!

- With astigmatism and out-of-focus ion beam, each spot may become elliptical and elongated. Thus, the distorted beam finally causes the unwanted milling.

![Image](image_url)
Effect of incidence angle:

✓ Maximum sputtering yields (Sputtered atoms per incoming ion) at angles in the range of 75° to 80°.

✓ FIB milling is usually done at normal incidence for vertical trench profile.

✓ No longer ‘normal’ once the milling starts-inclined incidence on tampered sidewall.
Effects of channeling on the FIB milling of a Cu 10°/100 twist bicrystal at (a) 0° tilt and (b) 10° tilt.

**Channeling effect**

- **Ion channeling effects:**
  - Reducing sputtering yield,
  - Low processing efficiency,
  - Groove-like morphology of the surface
  - High surface roughness,

- **Relevant factors:**
  - Angle of incidence of the ion beam
  - Characteristics of the ion
  - Orientation of the target.

C. A. Volkert et al., MRS bulletin 32, 389, 2007
Pixel size vs. beam size

For continuous non-wavy milling, $p_s/d_f$ should be less than 0.638

Ion flux distribution along a scan line with $p_s/\sigma = 3.0$ (top), 1.5 (bottom), $d_f = 2.35\sigma$.

“Recent developments in micromilling using focused ion beam technology”, Tseng, 2004
Merits with spiral scan:

✓ The unwanted beam exposure or etching are much smaller in the case of the spiral scan.

✓ Shape produced by the spiral scan is much more symmetric.

✓ Redeposited material can be better removed from the sidewalls as the beam progresses from the center of a hole outward,

❖ Spiral scan is better for milling holes or complexly patterning!
❖ Serpentine scan is suitable for milling a feature with sharp angles (like square pattern).
Sputtering yield of different materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Sputter rate [μm^3/nC]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.27</td>
</tr>
<tr>
<td>Thermal Oxide</td>
<td>0.24</td>
</tr>
<tr>
<td>TEOS</td>
<td>0.24</td>
</tr>
<tr>
<td>Al</td>
<td>0.3</td>
</tr>
<tr>
<td>Al2O3</td>
<td>0.08</td>
</tr>
<tr>
<td>GaAs</td>
<td>0.61</td>
</tr>
<tr>
<td>InP</td>
<td>1.2</td>
</tr>
<tr>
<td>Au</td>
<td>1.5</td>
</tr>
<tr>
<td>TiN</td>
<td>0.15</td>
</tr>
<tr>
<td>Si3N4</td>
<td>0.2</td>
</tr>
<tr>
<td>C</td>
<td>0.18</td>
</tr>
<tr>
<td>Ti</td>
<td>0.37</td>
</tr>
<tr>
<td>Cr</td>
<td>0.1</td>
</tr>
<tr>
<td>Fe</td>
<td>0.29</td>
</tr>
<tr>
<td>Ni</td>
<td>0.14</td>
</tr>
<tr>
<td>Cu</td>
<td>0.25</td>
</tr>
<tr>
<td>Mo</td>
<td>0.12</td>
</tr>
<tr>
<td>Ta</td>
<td>0.32</td>
</tr>
<tr>
<td>W</td>
<td>0.12</td>
</tr>
<tr>
<td>MgO</td>
<td>0.15</td>
</tr>
<tr>
<td>TiO</td>
<td>0.15</td>
</tr>
<tr>
<td>Fe2O3</td>
<td>0.25</td>
</tr>
<tr>
<td>Pt</td>
<td>0.23</td>
</tr>
<tr>
<td>PMMA</td>
<td>0.4</td>
</tr>
</tbody>
</table>

- Sputtering yield varies with material, orders of magnitude difference across periodic table.
- Actual rate much lower due to re-deposition of sputtered material.
Re-deposition:
During ion milling, a portion of the ejected atoms bump back into the already sputtered surface and redeposit onto it.

Re-deposition depends on:
• Kinetic energy of atoms leaving surface
• Sticking coefficient of target
• Sputtering yield of target
• Geometry of feature being milled

Factors that increase sputtering rate tend to increase re-deposition:
i) FIB milling is performed in a confined trench.
ii) FIB milling is performed in a high-aspect-ratio trench.
iii) Higher ion beam currents are used.

Re-deposition can be greatly reduced by broadening trench width, decreasing probe current and multiple passes scanning!
Curtaining effect (by non-planar milling of the surface)
Due to competition between smoothing by surface diffusion or viscous flow and roughening because of surface curvature-dependent sputter yields.

i) Rough surface

ii) A surface with uneven chemical composition.

iii) Composites of hard and soft materials.

iv) Height steps (e.g. patterned structures in semiconductors)

v) A porous structure.

vi) Curtaining effect increases with lower acceleration voltages which is used for high quality samples.
FIB milling

Ion Beam artifacts—curtaining effect

To eliminate the curtaining artifacts:

i) Making sure that the ion beam meets only homogeneous material:
   i.a) Remove all material with different sputtering behavior before starting the FIB steps.
   i.b) Change the geometry in a way that the ion beam comes from a homogeneous direction.

ii) Use thick, uniform and dense protection cap.

iii) Rocking the sample during FIB milling process.

iv) Infiltrate the samples, which have porous structures, with low viscosity resin.

For example:
✓ Stream of a gaseous organometallic platinum or polymeric carbon compound

✓ Ga⁺ beam (mild current avoiding a high rate of sputtering) causes the cleavage of the platinum or carbon from the volatile components of the precursor compounds.
FIB–Deposition

FIB deposition compared to CVD and PVD

✓ Locally

✓ Site specially

✓ No purity (organic residues)

✓ Just a few precursor gases are available for the deposition of Pt, W, SiO2, and C.

Circuit modification

Welding for lift-out process
FIB/SEM–Deposition

Applications of FEBID


Mask repair
Liang, T et al., (Vol. 6283, p. 62830K). International Society for Optics and Photonics.

Soldering carbon nanotubes onto electrodes, Madsen, D. N., Nano Letters, 3(1), 47-49.


Hall magnetic sensor
Gallium implantation

✓ Alternation of the specimen’s local composition within the interaction volume.

✓ Leading to structural changes, as well as alteration in, e.g., thermal, electrical, optical, and mechanical properties.

Annealing effect of Ga-implanted ZnO$_2$ nanorods

FIB patterning vs EBL patterning

✓ FIB Milling allows for creating cross sections or developing structures with desired geometries to control not only the lateral position but also local depth.
✓ It does not require the use of masks.
Applications

FIB bitmap vector patterning

Maskless lithography and nanostructuring
Applications

Cross Sectional SEM sample

Before milling  Deposition  Rough milling  Fine milling

Standard spec. (60nA)  Option (90nA)

Deposition  Rough milling  Fine milling

Processing time

80 min.  35 min.

Milled cross section (SIM image)

From JEOL
How to prepare a successful TEM lamella?

- Mechanical polishing
- Ion polishing by FIB

JEOL 2200FS TEM with double correctors

Probe-based

Chip-based

HE150 electrical probing holder (by Nanofactory AB)

DENSsolutions

Challenging work!
Applications

FIB-cut TEM lamella (in-situ lift-out technique)

- TEM lamella can be prepared site-specifically with a spatial accuracy as fine as 30 nm.

- Compared to other techniques (microtomy, low-energy ion milling, dimpling, etc.), it costs a short time (a few hours).

- Applicable for broad material systems including hard, soft, life materials with cryo-stage.
TEM specimen preparation (overview)

(a) Formation of protection layer
(b) Rough milling
(c) Fixation of thin section to TEM grid
(d) Fine milling (Thinning)
(e) TEM observation

https://www.jeol.co.jp
Prior to FIB, a ~30 nm Pt layer was pre-deposited on the top by sputtering machine.

**Pre-cutting**
- Pattern size: 10 µm L x 2 µm H,
- Depo (Pt): Beam 10 (30pA), 1 µm thick
- Grooving: Beam 4 (10 nA), Depth 6 µm

**Undercutting**
- Side cutting
  - Pre-thinning
  - Under cutting
- Beam 5 (3 nA), Depth 6 µm
FIB-cut Steps

Lifting-out

T: 0°

Deposit

T: 0°

SEM

FIB

For welding:
Carbon deposition,
Beam 10 (30 pA),
2-3 minutes

FIB-cut Steps

Fine milling

Fine milling:
30 kV, Beam 7 (500 pA), until 750 nm
30 kV, Beam 8 (300 pA), until 300 nm
+/- 2°

Fine polishing:
5 kV, Beam 7 (50 pA) or Beam 8 (30 pA)
< 100 nm
+/- 1.5°

Cleaning:
3 kV, Beam 7, < 2 mins
+/- 5°

Note: in order to have a robust in-situ TEM sample, it would be better to prepare a wedging shape lamella.
Surface cleaning inside TEM
STEM imaging after cleaning

10 nm

2 nm
3D observation and analysis

Technique features of FIB 3D observation and analysis
✓ Simultaneously cross-sectioning (by ion beam) and monitoring and/or SE or BSE imaging (by electron beam)
✓ Two modes
  i) Dynamic mode: SEM imaging in real time during milling process.
  ii) Static mode: SEM imaging, EDX mapping/FIB imaging after each milling. High resolution imaging.
✓ Resolution: lateral ~1nm, z-resolution 10-100nm
✓ FIB tomography fills in the gap between TEM tomography and X-ray tomography.
Applications

Procedure of FIB–Tomography (video)

**FIB tomography**

**Advantage:**
- Compared to serial TEM or serial tomography, the main advantage is the size of the volume that can be acquired.
- With a close to one thousand-fold increase in favor to the FIB-SEM.
3D EDX mappings

Spatial distribution of chemical elements in dentine

Materials 2018, 11(9), 1493
Key points to remember

✓ FIB technologies have been widely used in micro/nano manufacturing, with unique advantages of high fabrication resolution, high flexibility, maskless processing, and rapid prototyping.

✓ FIB technologies have a significant impact in various areas, such as semiconductor industry, micro-/nano-optics, surface engineering, biotechnology, and nanotechnology.

![Diagram with various imaging techniques and applications related to FIB technologies.](image-url)
Key references

- *Focused ion beam systems—basics and applications*, edited by Nan Yao, Cambridge University, New Jersey, online 2010.

Thanks for your attention!