Focused Ion Beam

a) Principles

How does it work..?

Ion source, optics, interaction with the sample

b) Basic Application

Imaging, milling, deposition, typical applications

TEM sample preparation, examples

c) FIB Nanotomography, 3D microscopy
A modern FIB (Focused Ion Beam) system
in a research lab (« lab » systems)

*a complete state of the art (high -performance) SEM equipped with*

a) focused ion column  
b) Gas injector system  
c) micromanipulators

Dual beam ®, crossbeam ®
MSE-704 3D Microscopy and FIB Nanotomography

Ion Beam & Electron Beam

SEM: Imaging and Analysis
- High resolution (1-2nm) SEM
- SE, general imaging, topography contrast
- BSE, chemical (mass density contrast) contrast
- EDX microanalysis (point analysis and element mapping)
- Low voltage SE and BSE imaging (small interaction volume=high resolution), compatible with "non"-conducting and biological specimens

FIB: Nano-machining
- Machining (sputtering)
- chemically assisted deposition and etching (gas injector system)
- Ion beam induced imaging (channeling contrast), SE and SI
- Micromanipulation (multiple micromanipulators) of small objects (<10nm precision)
- Nano-scale "laboratory"

Focused Ion Beam

- Mainly developed in 1970’s and 80’s (Escovitz, Levi-Setti, Orloff, Swanson...)
- Ion column structure similar to that of SEM
- Source: Liquid Metal Ion Source (LMIS). Ex: Ga, Au, Be, Si, Pd, B, P, As, Ni, Sb, alloys ...
- Principle: A strong electromagnetic field causes the emission of positively charged ions

SIM = Scanning Ion Microscope
**Ion Sources**

a) **Gas Field Ion Source**

- **Type of ion source**: Gas field ion source
- **Ion Species**: Ga⁺
- **Virtual source size (nm)**: 50
- **Energy spread, ΔE (eV)**: >4
- **Unnormalized brightness, B (A/cm²sr)**: 3x10⁶
- **Angular brightness (μA/sr)**: 50

b) **Liquid Metal Source**

- **Type of ion source**: Liquid metal
- **Ion Species**: H⁺, H₂⁺, He⁺, Ne⁺...
- **Virtual source size (nm)**: 0.5
- **Energy spread, ΔE (eV)**: ~1
- **Unnormalized brightness, B (A/cm²sr)**: 5x10⁹
- **Angular brightness (μA/sr)**: 35

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**Why use ions instead of electrons?**

- **Electrons**
  - are very small
  - inner shell reactions
  - High penetration depth
  - Low mass -> higher speed for given energy
  - Electrons are negative
  - Magnetic lens (Lorentz force)

- **Ions**
  - Big
  - outer shell reactions (no x-rays)
  - High interaction probability
  - less penetration depth
  - Ions can remain trapped -> doping
  - High mass -> slow speed but high momentum
  - milling !!!
  - Ions are positive
  - Electrostatic lenses
### Comparison of FIB and SEM

<table>
<thead>
<tr>
<th>Particle Type</th>
<th>FIB</th>
<th>SEM</th>
<th>Ratio</th>
</tr>
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<tbody>
<tr>
<td>Particle type</td>
<td>Ga⁺ ion</td>
<td>electron</td>
<td></td>
</tr>
<tr>
<td>Elementary charge</td>
<td>+1</td>
<td>-1</td>
<td></td>
</tr>
<tr>
<td>Particle size</td>
<td>0.2 nm</td>
<td>0.00001 nm</td>
<td>20’000</td>
</tr>
<tr>
<td>Mass</td>
<td>1.2·10⁻²⁵ kg</td>
<td>9.1·10⁻³¹ kg</td>
<td>130’000</td>
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<tr>
<td>Velocity at 30 kV</td>
<td>2.8·10⁵ m/s</td>
<td>1.0·10⁸ m/s</td>
<td>0.0028</td>
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<tr>
<td>Velocity at 2 kV</td>
<td>7.3·10⁴ m/s</td>
<td>2.6·10⁷ m/s</td>
<td>0.0028</td>
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<tr>
<td>Momentum at 30 kV</td>
<td>3.4·10⁻²⁰ kg·m/s</td>
<td>9.1·10⁻₂³ kg·m/s</td>
<td>370</td>
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<tr>
<td>Momentum at 2 kV</td>
<td>8.8·10⁻²¹ kg·m/s</td>
<td>2.4·10⁻²³ kg·m/s</td>
<td>370</td>
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<tr>
<td>Beam Size</td>
<td>nm range</td>
<td>nm range</td>
<td></td>
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<tr>
<td>Energy</td>
<td>up to 30 kV</td>
<td>up to 30 kV</td>
<td></td>
</tr>
<tr>
<td>Current</td>
<td>pA to nA range</td>
<td>pA to uA range</td>
<td></td>
</tr>
<tr>
<td>Penetration depth</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>In polymer at 30 kV</td>
<td>60 nm</td>
<td>12000 nm</td>
<td></td>
</tr>
<tr>
<td>In polymer at 2 kV</td>
<td>12 nm</td>
<td>100 nm</td>
<td></td>
</tr>
<tr>
<td>In iron at 30 kV</td>
<td>20 nm</td>
<td>1800 nm</td>
<td></td>
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<tr>
<td>In iron at 2 kV</td>
<td>4 nm</td>
<td>25 nm</td>
<td></td>
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<tr>
<td>Average electrons signal per 100 particles at 20 kV</td>
<td>secondary electrons</td>
<td>100 - 200</td>
<td>50 - 75</td>
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<tr>
<td></td>
<td>back scattered electron</td>
<td>0</td>
<td>30 - 50</td>
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<tr>
<td></td>
<td>substrate atom</td>
<td>500</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>secondary ion</td>
<td>30</td>
<td>0</td>
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<tr>
<td></td>
<td>x-ray</td>
<td>0</td>
<td>0.7</td>
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</table>

### Ion Solid interaction

- Secondary electron emission (SE-image)  
  2-3 SE per Ion!

- Surface chemical reactions  
  - deposition  
  - enhanced etching

- Sputtering  
- Damage  
- Implantation
3 basic “operating modes”

- Emission of secondary ions and electrons
  - **FIB imaging** (a) low ion current

- Sputtering of substrate atoms
  - **FIB milling** (b) high ion current

- Chemical interactions (gas assisted)
  - **FIB deposition**
  - Enhanced (preferential) etching (c)

- Other effects:
  - Ion implantation
  - Displacement of atoms in the solid
    - Induced damage
  - Emission of phonons
    - Heating

Monte-Carlo Simulation casino v2.42
http://www.gel.usherbrooke.ca/casino/download2.html

SRIM 2006
http://www.srim.org/
SE image contrast

- e-beam 5kV
- ion-beam 30kV 50pA
- material (sputtering) contrast
- orientational contrast

Channeling contrast

- Secondary Electron Emission Coefficient vs. Angle, (100) Copper 30 keV Ar Ions

G. Carter and J.S. Colligan, Ion Bombardment of Solids, (Elsevier 1968)

- Atom columns align with the ion trajectory = higher penetration
- => less sputtering and less SE electrons
Milling

Milling rate

- Sputter rate for a 10x10x5µm box in Cu
- Typical ion current (high) 10nA
- Sputter Yield for Cu: 0.25µm³/nC

Volume: 500 µm³
Time: Volume/sputter rate = 2000 sec. = 33 min.
Ion-Solid interaction
Sputtering Yield

- Sputtering yield depends on incident angle $\phi$
- Higher probability of collision cascades near the surface at higher $\phi$
- Sputtering yield has maximum for $\phi = 75^\circ$

MSE-704 3D Microscopy and FIB Nanotomography

Milling

FIB milling of steel

X.Xu, et al.
Polishing, at shallow angles

Gas assisted deposition

ion beam (e beam)

adsorbed molecules

substrate
Deposition rate

Nanofabricated structures

Coil 700nm pitch, 80nm line width, diamond-like amorphous carbon, FIB induced CVD

Shinji Matsui, et.al.
(Himeji Institute of Technology, Hyogo, Japan)
b) Basic Applications

- “Industrial” applications (semiconductor industry)
- sectioning for failure analysis
- prototype circuit rewiring
- mask repair
- TEM sample preparation

- Research
  - Micromachining
  - Nanofabricated structures
  - TEM sample preparation
Applications  Chip Modification

Insertion of electrical connection:
1) Removal of isolating layer (milling)
2) Pt deposition (FIB deposition)

FIB-manufactured AFM-tips
Failure analysis

FIB cross-sectioning and SEM imaging

c) TEM preparation in-situ lift-out movie

(downloaded from http://www.feicompany.com/)
Site specific TEM lamella extraction

5nm Si Nano-wire
M. Pavius, V. Pott, CMI
M. Cantoni, CIME

TEM sample “grid”, diameter 3mm

Si-Nanowire
TEM, HRTEM

poly-silicon

SiO$_2$ amorph

5nm
TEM lamellae by FIB

Focused Ion Beam adds a new dimension to TEM specimen preparation

- Large (10x5um) flat areas with uniform thickness (50-80 nm)
- Preparation of heterogeneous samples with “difficult” material combinations becomes possible
- Precise selection of the lamella position possible (devices)

Take care of artifacts !!!
3D Microscopy

FIB Nanotomography
Problem of serial sectioning:
3D-reconstruction of disordered microstructures

2D Volume fraction

?? Nr of particles ??
?? Shape ??

Voxel, Resolution, Pixel size

From: J.C.Russ, 1998

<table>
<thead>
<tr>
<th>Smeallest element</th>
<th>Mag</th>
<th>Ideal z-spacing</th>
<th>Beam current</th>
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<td>nm</td>
<td>kX</td>
<td>nm</td>
<td>pA</td>
</tr>
<tr>
<td>1000</td>
<td>5</td>
<td>100</td>
<td>500</td>
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<td>50</td>
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<tr>
<td>100</td>
<td>50</td>
<td>10</td>
<td>50</td>
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<table>
<thead>
<tr>
<th>Smallest element</th>
<th>Mag</th>
<th>X</th>
<th>Y</th>
<th>Pixel size</th>
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<tbody>
<tr>
<td>nm</td>
<td>kX</td>
<td>pm</td>
<td>pm</td>
<td></td>
</tr>
<tr>
<td>1000</td>
<td>5</td>
<td>60.0</td>
<td>65.73</td>
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<td>500</td>
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<td>300</td>
<td>15</td>
<td>20.0</td>
<td>21.91</td>
<td>19.5</td>
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<tr>
<td>200</td>
<td>30</td>
<td>10.0</td>
<td>10.95</td>
<td>9.8</td>
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<tr>
<td>100</td>
<td>50</td>
<td>6.0</td>
<td>5.18</td>
<td>5.9</td>
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</table>
3D slicing of multifilament Nb$_3$Sn superconductor

Preparing for slicing

Automated milling and imaging of 170 slices (10h)
align and crop

3D volume rendering, reconstruction:
Orthogonal slices

ImageJ
Image Processing and Analysis in Java
The choice of the right detector

SE detector (TLD)

BSE detector (TLD)

Ion beam imaging (SE)

Ion beam for slicing and imaging requires stage movement...!

Nb3Sn multifilament Superconductors

Materials & grain contrast

Nb3Sn superconductor multifilament cable:
14’000 Nb3Sn filaments (diameter ~5um) in bronze matrix
1.8kV EsB detector
Chemistry and orientation

**Chemistry and orientation**

**Nb$_3$Sn multifilament Superconductors**

*Materials & grain contrast*

2048x1536x1700 (10x10x10nm voxel)

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**What is the spatial resolution in BSE imaging...?**

**Scatter range in Nb$_3$Sn:**

Monte-Carlo Simulation of electron trajectories

*backscattered electrons*
Interaction volume at 1.5kV, BSE escape depth

Monte-Carlo Simulation of electron trajectories in Cu and C backscattered electrons

Orientation contrast
identification of grain texture

Tribology: wear trace on steel
Tribo-corrosion
J. Perret, S. Mischler
IMX-LMCH
Grain orientation contrast of small grains
(grain size < 100nm)

2048x1536x1200 volume: 20x15x12um
10x10x10nm voxel
growth of ZnO films, photovoltaics

10x10x10nm voxel size, 2048x1536x2200 pixel/slices

C. Balif, S. Nicolay, D. Alexander
Pb-free solder: “one detector is not enough”

M. Maleki,
EPFL-LMAF
10x10x10nm voxel size, 2048x1536x2000 pixel/slices
2 images (3Mb) / slice ...... 12Gb data

Phase 1
Dark in EsB image
White in SE-InLens

Phase 2
White in SE-InLens - Dark in EsB image

10x10x10nm voxel size, 2048x1536x2000 pixel/slices
2 images (3Mb) / slice ...... 12Gb data
Quantitative microstructure analysis

→ Algorithms
→ object recognition
→ stereological correction of boundary truncation
→ extraction of statistical data (particle shape and size distribution)

Particle recognition: Edge detection in 3D, Watershed for separation

Voxel: 75nm
Cube:
40*
20*
15 μm

Size, 3D-shape, geometrical relationships between particles
Quantitative microstructure analysis → Algorithms

Münch and Holzer 2006
FIB-nt of particulate systems – part II:
Object recognition and effect of boundary truncation

FIB-NT compared with other 3D-techniques

New possibilities in 3D-microscopy:
Combination with quantitative analytical SEM techniques:
EDX, EBSD