Ion Beam Induced Excitation Methods
A. Particle Induced X-Ray Emission (PIXE)
B. Rutherford Backscattering Spectroscopy (RBS)
C. Secondary Ion Mass Spectrometry (SIMS)
D. Ion Induced Secondary Emission Microscopy
E. Helium Ion Microscopy
Secondary Ion Microscopy in Biology

Figure 2

MIMS images of *Teredinibacter turnerae*, a nitrogen-fixing bacterium inhabiting a shipworm gill. The $^{12}\text{C}^{15}\text{N}/^{12}\text{C}^{14}\text{N}$ image (left) allows the uptake of $^{15}\text{N}$ by these organisms to be quantified. On the right, the image of $^{12}\text{C}^{15}\text{N}^+$ ions shows that sensitivity is sufficient to image the flagellum of this bacterium (arrowed) even though the flagellum diameter is estimated from the ion signal to be only about 10 nm. From [1].

Time of flight SIMS

Figure 1. Schematic diagram of the Time-of-Flight secondary ion microscope.

Figure 5. Secondary ion images of Al\(^+\) (a) and \(^{28}\)Si\(^+\) (b) taken from a 250 \(\mu\)m diameter surface region of an Al–Si integrated circuit. The transmission reduction due to lateral energy filtering was approximately 30% for elemental secondary ion species.

Commercial SIMS Instrument/ CAMECA
Ion Microprobe, CAMECA NanoSIMS 50L

The Microanalysis Center for Geochemistry and Cosmochemistry at Caltech took the delivery of a CAMECA NanoSIMS 50L in last December and finished the installment and on-site tests in this past April. Now the instrument is fully operational and running well. The CAMECA NanoSIMS 50L is a new ion microprobe, developed for trace element and isotopic analysis of ultra-fine features. Among the unique new features offered by the NanoSIMS 50L are: 1) The ability to extend the SIMS analysis to extremely small areas or volumes (∼35 nm size in cesium, ∼150 nm in oxygen) while maintaining extremely high sensitivity at high mass resolution (HMR). This derives from the revolutionary coaxial optical design of the ion gun and secondary ion extraction, and from a new design of the magnetic sector mass analyzer. 2) The capability of simultaneously measuring up to 7 masses (ions), ensuring more efficient and precise isotopic ratios from the same small volume, or better ion image superimposition in imaging mode. Along with the 7 electron multiplier (EM) detectors, 4 Faraday cups (FC) are also installed on the Caltech NanoSIMS 50L, enabling to achieve the precision and external reproducibility of isotope ratio measurements down to the low sub-permil level.
Segregation and diffusion of elements in YAG (Yttrium Aluminium Garnet)

The NanoSIMS is used here to contribute to the understanding of the segregation and diffusion of elements in polycrystalline materials.

In the first example, the strategy is to make use of $^{18}$O stable isotope tracers in order to image and quantify the incorporation of oxygen. A sintered transparent YAG (yttrium aluminum garnet) is oxidized in a $^{18}$O$_2$ atmosphere at 1400°C. The $^{18}$O oxygen atoms diffuse inside the ceramics through the grain boundaries as evidenced from the two complementary images of $^{16}$O (base element of the oxide) and $^{18}$O (incorporated during the oxidation sequence).

Dr. Hajime Haneda, NIMS, Tsukuba, Japan
Extracellular Proteins Limit the Dispersal of Biogenic Nanoparticles

John W. Moreau,†,⊥, Peter K. Weber, Michael C. Martin, Benjamin Gilbert, Ian D. Hutcheon, and Jillian F. Banfield
ION INTERACTIONS

Three main components

1. "nuclear" energy loss, dominates at low energies when \( E < A \text{ keV} \)

2. electronic energy loss
   Interactions with atomic electrons
   (like "inelastic" electron scattering)

3. charge exchange
   \( \leq 10\% \) of total

\[
\frac{\mathrm{d}E}{\mathrm{d}x}
\]

\[
A(\text{keV}) \quad E(\text{keV})
\]
He Ion Beam Microscope
(combine SEM with RBS)

- The scanned beam is 35 keV $\text{He}^+$ ions rather than electrons
- The scanned He ions are launched from a single metal atom on the ion source needle
- The helium ion source brightness of $3.6 \times 10^9 \text{ A/cm}^2\text{str}$ and a 1:1 column demagnification provide for:
  - 0.3 nm resolution at a 8 mm working distance
  - A 2.5 $\mu$m depth of focus
- Two detectors collect $\text{He}^+$ induced secondary electrons (SE mode) or Rutherford Backscattered Ions (RBI mode) to form two very different images simultaneously
- Ion induced SE’s have an energy mean of ~ 2eV with ~1nm mean free path in conductors,
  - hence SE mode images are extremely surface sensitive
- Electron flood interface, while HIM imaging, neutralizes any positive surface charge, hence:
  - $\text{Samples need not be coated and}$
  - Dielectrics can be imaged at the highest beam energy and resolution
Field Ion Microscopy

Fig. 1 (left). Schematic drawing of a field ion microscope. Fig. 2 (center). A polarized helium atom is attracted to the metal tip, is slowed down in a number of hops, and is ionized in the ionization zone (0.2 Å thick) over a protruding atom. The helium ion is accelerated toward the screen (see Fig. 1). Fig. 3 (right). (A) The potential funnel of an atom in a strong electric field; (B) the potential of an electron near the metal surface. Field ionization of the gas atom occurs when the electron from the ground state tunnels along the dotted line into the metal to the left.

Helium Ion Source
(111) W TIP (atomic view)
Field Ion Micrograph of the Unshaped Emitter
**Probe Size:**
\[ d_p = \sqrt{M \cdot d_g^2 + d_i^2 + d_s^2 + d_d^2} \]

**Demagnified source:**
\[ d_{so} = M \cdot d_g \]

**Spherical aberration:**
\[ d_s = 0.5 C_s \alpha_i^3 \]

**Chromatic aberration:**
\[ d_c = C_c \frac{\Delta U}{U} \alpha_i \]

**Diffraction Error:**
\[ d_d = 0.6 \frac{\lambda}{\alpha_i} \]

---

**Superposition of the aberration discs**
Probe Size: \[ d_p = \sqrt{(M \cdot d_g)^2 + d_s^2 + d_c^2 + d_d^2} \]

Demagnified source: \[ d_{50} = M \cdot d_g \]

Spherical aberration: \[ d_s = 0.5 C_s \alpha_i^3 \]

Chromatic aberration: \[ d_c = C_c \frac{\Delta U}{U} \alpha_i \]

Diffraction Error: \[ d_d = 0.6 \frac{\lambda}{\alpha_i} \]

Superposition of the aberration discs
Schematics of a field ion microscope
- FIM tip created via chemical etching

- ALIS tip formed with additional reshaping

- 3 atom shelf called the “trimer” created through field evaporation

- Single atom selected to form the final probe

- Results in a sub-angstrom virtual source with high brightness \((4 \times 10^9 \text{ A}/(\text{cm}^2 \text{ sr}))\) and low energy spread \((<1\text{ eV})\)
SE1 are produced directly by the primary beam
SE2 are produced by the backscattered beam as it exits sample
<table>
<thead>
<tr>
<th>Secondary Electrons</th>
<th>Backscattered Ions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Surface Information</strong>&lt;br&gt;Yield varies with material (range ~2-8)&lt;br&gt;SE image provides both topographic information and material contrast&lt;br&gt;Typical Energy ~5 eV&lt;br&gt;SE Yield Ranges from 3 to 8&lt;br&gt;Escape Depth ~ nm</td>
<td><strong>Material Information</strong>&lt;br&gt;Scatter yield varies as $Z^2$ of target&lt;br&gt;Scatter energy varies with mass of target (similar to RBS)&lt;br&gt;Typical Energy ~ several keV&lt;br&gt;Typical Yield ~ 0.1% to 3%&lt;br&gt;Minimal Topographic Information&lt;br&gt;Immune to surface charging</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Transmitted Ions</th>
<th>Other Contrast Mechanisms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Provides material information.&lt;br&gt;Scatter cross section varies with $Z^2$ of the target&lt;br&gt;Provide crystallographic information</td>
<td><strong>Channeling Contrast</strong>&lt;br&gt;<strong>Voltage Contrast</strong>&lt;br&gt;Photon Imaging “Ion luminescence”</td>
</tr>
</tbody>
</table>
sample courtesy of Al Lysse, Carl Zeiss SMT Inc., US
Eutectic Alloy of Pb, Sn, In, and Bi

Depth of Field inversely proportional to Half Angle ($\alpha_i$) of incident beam
- contacted nanowire
- device bonded to its chip carrier
- WD = 9.5 mm!

- best imaging WD @ 6-8 mm
- advantage for imaging samples with big topography if ROI is not highest (e.g. curved samples)
Note how the RBI image contains very little edge information

- Carbon
- Nickel
- Copper
- Gold
SE image                                              He Ion Backscattered image

From P. Gnauck, Zeiss
- Transmission Ion imaging performed with a "Conversion Style" detector

- Transmission image of EUV mask clearly shows the absorber stack, the cap layer, and the multi-layers

- An improved detector in development which will allow both bright and dark field imaging
Channeling Contrast

Contrast differences due to grain orientation
Backscattered ions have high energy and are impervious to surface charging.

Charge control in Secondary electron mode using an electron flood gun.
Asbestos Fiber on Holey Carbon Film

Self supporting

On carbon support
- Virtually no X-ray production $\rightarrow$ use backscattered helium for materials characterization

- Cross section for scattering (RBI Yield, Imaging)

$$\sigma \propto \frac{Z^2}{E_0^2} \frac{1}{\sin^4(\theta)}$$

- Backscatter Energy for spectroscopy

$$E_B = E_0 \left( \frac{\sqrt{M_2^2 - M_1^2 \sin^2 \theta + M_1 \cos \theta}}{M_1 + M_2} \right)^2$$
Figure 1: Helium backscatter spectra from a set of ZrO$_x$ films on silicon. The legend indicates the number of ALD cycles used to grow the various films. Samples provided courtesy of Dr. Steffen Teichert, Qimonda (Dresden, Germany).
Correlation between low voltage RBS and MeV RBS