Part II: Application of Ion Beams

- Sputtering (depth profiling)
- Rutherford backscattering spectrometry (RBS)
- Elastic recoil detection (ERD)
- Secondary Ion Mass Spectrometry (SIMS)
- How to fabricate via ion implantation

Sputter Yield and Ion Mixing

- **Sputtering** is the removal of surface atoms due to energetic ion bombardment
- The sputtering is described by a "Sputtering Yield", $Y$, which is defined as the mean number of sputtered target atoms per incident ion

$$Y(E, \theta, m) = \frac{\text{atoms removed}}{\text{incident ion}}$$

$10^{-5} \leq Y \leq 10^3$
Sputtering yields vs energy, angle and ion mass

- Ne, 20keV
- Ar, 40°
- Sputtered Si atoms/ion
- Angle [degrees]
- Incident energy [keV]

Sputtering yield measurements

- Helium
- Neon
- Argon
- Krypton
- Xenon
- 20keV, 40°
Preferential sputtering in depth profiling

For a target composing of 20Å SiO₂/ 50Å Si/5Å Ta/ 10 µm C and using Ar @ 10keV, 45°

<table>
<thead>
<tr>
<th>Element</th>
<th>10000 Ar⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.73</td>
</tr>
<tr>
<td>O</td>
<td>2.90</td>
</tr>
<tr>
<td>Ta</td>
<td>0.0005</td>
</tr>
<tr>
<td>C</td>
<td>0.0032</td>
</tr>
</tbody>
</table>

Distribution of elements after sputtering

• Significant ion mixing is observed due to sputtering processes
Summary: factors affecting depth profiling

- **Sample characteristics:**
  - Depth information (IMFP)
  - Original surface roughness
  - Alloys, compounds, second phases (preferential sputtering and induced roughness)

- **Ion bombardment-induced effects:**
  - Atomic mixing
  - Sputtering-induced roughness
  - Preferential sputtering and decomposition of compounds
  - Enhanced diffusion and segregation

Rutherford Backscattering Spectroscopy

- **Typical parameters:** 500keV-4MeV H⁺, He⁺
- **Ion range:** 1.5-14 μm
- **Ion beam is close to surface normal**
- **Small fraction of incident ions will scatter back**

- **Introduction**
- **History**
- **Scattering geometry and kinematics**
- **Rutherford cross section and limitations (non-Rutherford)**
- **RBS spectra from thin and thick films**
- **Stopping power and energy loss - aside**
- **Detector resolution**
- **Energy straggling**
- **Non-Rutherford cross sections**
RBS- Rutherford Backscattering Spectrometry

- Widely used for near-surface layer analysis of solids
- Elemental composition and depth profiling of individual elements
- Quantitative without reference sample (unlike SIMS, XPS with depth profiling)
- Non-destructive (unlike SIMS, XPS with depth profiling)
- Analyzed depth: ~ 2\(\mu\)m for He ions
- Very sensitive for heavy elements: ~ ppm
- Less sensitive for light elements \(\Rightarrow\) ERD
- Available at UWO!

History

Sir Ernest Rutherford (1871 - 1937)
- 1911: Rutherford’s scattering experiments: \(^4\)He on Au
  - Atomic nucleus, nature of the atom

RBS as materials analysis method

“Nuclear scattering and nuclear reactions induced by high energy protons and deuterons have been applied to the analysis of solid surfaces. The theory of the scattering method, and determination of O, Al, Si, S, Ca, Fe, Cu, Ag, Ba, and Pb by scattering method are described. C, N, O, F, and Na were also determined by nuclear reactions other than scattering. The methods are applicable to the detection of all elements to a depth of several \(\mu\)m, with sensitivities in the range of \(10^{-8}\) to \(10^{-6}\) g/cm\(^2\).”
RBS: Scattering geometry and kinematics

\[ E_I = k E_o \]

\[ k = \frac{E_I}{E_o} = \left[ \frac{(M_2^2 - M_1^2 \sin^2 \theta)^{1/2} + M_1 \cos \theta}{M_2 + M_1} \right]^2 \]

\[ \alpha: \text{incident angle} \]
\[ \beta: \text{exit angle} \]
\[ \theta: \text{scattering angle} \]

Optimized mass resolution for:

\[ 160^\circ < \theta < 170^\circ \]

Scattering kinematics: example 1

2MeV \(^4\text{He}^+\), \(\theta=165^\circ\)
Backscattered from
C, O, Fe, Mo, Au
3\times10^{16} \text{ atoms/cm}^2 \text{ each}\n
on Si substrate
Key features of RBS

Ability to quantify depth profile of buried species with a precision of ~ 3%

Qualitative information: **kinematic factor, \( k \)**

\[
k = \frac{E_i}{E_o} = \left[ \frac{M_2^2 - M_1^2 \sin^2 \theta)^{1/2} + M_1 \cos \theta}{M_2 + M_1} \right]^2
\]

Quantitative: **scattering cross section, \( \sigma \)**

\[
\frac{d\sigma}{d\Omega} = \sigma(\theta) = \left( \frac{Z_1 Z_2 e^2}{4E \sin^2 \left( \frac{\theta}{2} \right)} \right)^2
\]

Scattering kinematics: example 2

⇒ Decreased mass resolution for heavier elements
Rutherford Cross Section

- Neglecting shielding by electron clouds
- Distance of closest approach large enough that nuclear force is negligible

\[ \frac{d\sigma}{d\Omega} = \sigma(\theta) = \left( \frac{Z_1 Z_2 e^2}{4E \sin^2 \left( \frac{\theta}{2} \right)} \right)^2 \]

Scattering kinematics: example 1

2MeV 4He⁺, \( \theta = 165° \)
Backscattered from C, O, Fe, Mo, Au
3x10^{16} atoms/cm² each on Si substrate

- Increased sensitivity for heavier elements
- Good for heavier elements on lighter substrates
- Bad for light elements on heavier substrates
Shielded Rutherford Cross Sections

Shielding by electron clouds gets important at
• low energies
• low scattering angles
• high Z

Shielding is taken into account by a shielding factor $F(E, \theta)$

$$\sigma(E, \theta) = F(E, \theta)\sigma_R(E, \theta)$$

$F(E, \theta)$ is obtained by solving the scattering equations for a shielded interatomic potential:

$$V(r) = \frac{Z_1 Z_2 e^2}{r} \varphi\left(\frac{r}{a}\right)$$

$\varphi$: Screening function

Use Thomas-Fermi or Lenz-Jenssen screening function

$a$: Screening radius

$$a = 0.885 a_0 \left(\frac{Z_1^{2/3} + Z_2^{2/3}}{2}\right)^{1/2}$$

$a_0$: Bohr radius

RBS spectra from thin and thick films

The integrated peak count $A_i$ for each element on the surface can be calculated using this equation:

$$A_i = (N t)_i \times Q \times \Omega \times \frac{\sigma(E, \theta)}{\cos \theta}$$

where
$(N t)_i$: areal density, atoms per unit area;
$Q$: ion beam fluency;
$\Omega$: solid angle of the detector;
$\sigma(E, \theta)/\cos \theta$: cross section of an element
Ion dose (fluence), solid angle, cross section

- Ion dose (fluence), the number of incident particles (collected charge)
  - measured by Faraday cup
  - \( Q = I \times t \)

- Solid angle, in steradians, sr
  - stays constant for a particular detector/detector slit
  - need to be verified by the calibration standard measurements

- Cross section (or differential cross section), in cm\(^2\)/sr of the element
  - well known (tabulated) in Rutherford cross section regime

RBS Spectrum of a thick film

- Target is divided into thin sublayers ("slabs")
- Calculate backscattering from front and back side of each sublayer taking energy loss into account
- For each isotope of each element in sublayer
RBS spectrum: bad choice of the substrate

Very thin layer of Al, Fe, Ag and Au (mixed)

RBS Spectrum: Stoichiometry

2MeV $^4$He$^+$, backscattered from ceramic films on Si substrate

Ba$_{0.05}$Cd$_2$KSi$_2$NaO$_4$/Si
Ion channeling and blocking

Si (diamond structure)

- Si(111) (011)
- Si(111) – side view

Use crystal structure of the substrate

- Substrate can be aligned to a major crystallographic direction to minimize background signal in some cases
**Silicon detector resolution**

**Principle of operation:**
- Creation of electron-hole pairs by charged particles
- Separation of electron-hole pairs by high voltage $V$

$\Rightarrow$ Number of electron-hole pairs $\propto$ Particle energy

$\Rightarrow$ Charge pulse $\propto$ Particle energy

**Limited energy resolution ($\sim 1\text{keV}/100\text{Å}$) due to:**
- Statistical fluctuations in energy transfer to electrons and phonons
- Statistical fluctuations in annihilation of electron-hole pairs

**Additional energy broadening due to:**
- Preamplifier noise
- Other electronic noise

**Energy Straggling**

Slowing down of ions in matter is accompanied by a spread of beam energy

$\Rightarrow$ energy straggling

*Electronic or nuclear energy loss straggling* due to statistical fluctuations in the transfer of energy to electrons or nuclear energy loss

Energy after penetrating a layer $\Delta x$: $<E> = E_0 - s \Delta x$

$<E>$ - mean energy; $s$ - stopping power

$\Rightarrow$ only applicable for mean energy of many particles
Non-Rutherford cross sections

Typical problem for light elements: overlap with thick layers of heavier elements
⇒ High cross sections wishful!!!
Secondary Ion Mass Spectrometry (SIMS)

Secondary Ion Mass Spectrometry (SIMS) is the mass spectrometry of ionized particles emitted from a solid surface when it is bombarded by primary energetic particles.

**Primary particles:** ions or neutrals

**Emitted particles:** electrons, neutral atoms or molecules, atomic or molecular ions

**SIMS:** based on m/e ratio of ejected particle under ion bombordment

The emission of particles from the surface is also known as sputtering (depends on the energy of the impinging ions, collision angles, the electronic properties of the ion and the solid, and the chemical environment of the solid).

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Dynamic SIMS and static SIMS

**Dynamic SIMS** and **static SIMS** are two main operation modes of SIMS.

**Dynamic SIMS:**
- a high incident ion current density (1 A/cm²)
- it gives elemental composition vs depth
- used extensively in semiconductor industry

**Static SIMS:**
- a low incident ion beam density (1 nA/cm²)
- the ions do not disturb the composition and structure of the surface
- possible to get composition of the first few monolayers
Formation of molecular ions

- During sputtering, molecular ions, even not present in solid can be observed
- Two models exist to explain formation of molecular ions:
  - atomic combination model (individually sputtered atoms recombine to form molecular ion if they possess sufficient kinetic energy to participate in chemical reaction)
  - direct emission model (characteristic cracking patterns)
**SIMS of aluminium**

- maximum for Al$^+$
- next abundant species are Al$^{2+}$ and Al$^{3+}$

**Sputtering Yield**

- **Sputtering Yield**, $Y$
  - the energy of the incident ion; the mass of the incident particles; material properties (ionization potential (IP) and electron affinity (EA))
  
- Both **positive** and **negative** ions can be formed and used
  - Several models predict exponential dependence of the secondary ion yield on IP or EA
- Secondary ion yield strongly depend on the chemical environment (**matrix effect**)
  
  Si$^+$ (in SiO$_2$) = 2500 Si$^+$ (in Si)
Positive Secondary Ion Enhancement: $O^{2+}$ beams

**Figure 5.3.** The variation of positive ion yield as a function of atomic number for 1 nA 13.5 keV $O$-bombardment: $\circ$ from elements; $\square$ from compounds. Reproduced with permission from H.A. Storms, K.F. Brown, and J.D. Stein, *Anal. Chem.*, 49, 203 (1977). (Copyright (1977) American Chemical Society)

Negative Secondary Ion Yield: $Cs^+$ bombardment

**Figure 6.30.** The ratio of negative ion yield ($M^-$) under $Cs^+$ bombardment to positive ion yield ($M^+$) under $O^-$ bombardment as function of atomic number. Reproduced from data in H.A. Storms, K.F. Brown, and J.D. Stein, *Anal. Chem.*, 49, 203 (1977). Copyright (1977) American Chemical Society
Secondary Ion Yields (Primary Beam Effects)

Other factors affecting the secondary ionization efficiencies in SIMS:
- **Oxygen bombardment** increases the yield of positive ions
- **Cesium bombardment** increases the yield of negative ions.

The increases can range up to four orders of magnitude.

### Quantitative analysis of SIMS

- The intensity of secondary ions can be expressed by the following
  \[
  I_{A^+}^T = j_p \times A \times Y_{A^+}^T \times f \times C_{A^+}^T
  \]

  - \( I \) – measured ion current of \( A^+ \) in the matrix \( T \)
  - \( j_p \) – primary ion current density
  - \( A \) - area of analysis
  - \( Y \) – secondary ion yield in the matrix \( T \)
  - \( f \) - instrumental transmission factor for \( A \)
  - \( C \) – atomic concentration of \( A \) in the matrix \( T \)

- can simplify if use standards
  \[
  I_{A^+}^T = S_{A^+}^T \times C_{A^+}^T
  \]

  - \( S \) – sensitivity factor for \( A^+ \) in the matrix \( T \)
Mass Analysers

- Quadrupole
- Magnetic sector
- Time-of-Flight

<table>
<thead>
<tr>
<th>Type</th>
<th>Resolution</th>
<th>Mass range</th>
<th>Transmission</th>
<th>Mass detection</th>
<th>Relative sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quadrupole</td>
<td>$10^2 - 10^3$</td>
<td>$&lt;10^3$</td>
<td>0.01–0.1</td>
<td>Sequential</td>
<td>1</td>
</tr>
<tr>
<td>Magnetic sector</td>
<td>$10^4$</td>
<td>$&gt;10^4$</td>
<td>0.1–0.5</td>
<td>Sequential</td>
<td>10</td>
</tr>
<tr>
<td>Time-of-flight</td>
<td>$&gt;10^3$</td>
<td>$10^3 - 10^4$</td>
<td>0.5–1.0</td>
<td>Parallel</td>
<td>$10^4$</td>
</tr>
</tbody>
</table>

How to fabricate via ion implantation

- Semiconductor doping
- SOI technology
- Wafer bonding methods
- Formation of nanocrystals
Semiconductor-on-insulator technology

FIGURE 1 A CROSS-SECTION OF THE TOSHIBA SOI POWER IC STRUCTURE

Wafer bonding, “smart cut” process

1. H$_2$/He$_2$-Implantation
2. Direct Waferbonding
3. Splitting by annealing
4. Layer on 'Host'-Substrate; Polishing/Etching (with A precision)
Tandetron Accelerator Laboratory (WSC G49)

1.7 MV Tandetron Accelerator

- ERD Chamber
- Rutherford Backscattering System (RBS)
- Sputter Source (for He)
- Sputter Source (for all others)
- PIXE system
- Striker gas
- Quadrupoles
- Injector Magnet
- High Energy Magnet
- Group III, V Molecular Beam Epitaxy System
- Group IV Molecular Beam Epitaxy System
- Raster Scanner
- Implant Stage
- Medium Energy (MEIS) Ion Scattering System