INTRODUCTION to ION BEAM TECHNIQUES

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INTRODUCTION to ION BEAM ANALYSIS (IBA) TECHNIQUES (a.k.a. NUCLEAR ANALYTICAL METHODS)

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• Electrostatic accelerators
• Ion beam analysis with examples
  • RBS
  • ERDA
  • PIXE
• Nuclear microprobe
• Materials modification
ELECTROSTATIC ACCELERATORS
The first ion probe – Rutherford experiment

ERNEST RUTHERFORD
• 1909 – $\alpha$-particle scattering experiment on gold foil
• 1911 – theory of nuclear atom
• had called for "a million volts in a soapbox" to advance nuclear research!
Working in a vacant room at Rutherford's Cavendish Laboratory at Cambridge University, Englishman Cockcroft and Irishman Walton used spare parts to build the world's first nuclear-particle accelerator in 1929.

- high voltage obtained by cascade voltage multiplier
- 1932 the first artificial nuclear reaction $p + ^7\text{Li} \rightarrow ^4\text{He} + ^4\text{He}$
- Nobel prize 1951
ELECTROSTATIC ACCELERATORS

Robert J. Van de Graaff

Princeton University; MIT Boston
- 1929 80 kV
- 1931 7 MV
- after the WWII he founded HVEC – High Voltage Engineering Corporation
ELECTROSTATIC ACCELERATORS

Luis W. Alvares

- WWII – Manhattan project
- Berkeley 1951 – concept of tandem accelerator
- Nobel prize in physics 1968 (bubble chamber)
- Alvarez Hypothesis 1980
Aprox. 20,000 accelerators:

- 90% medicine & industry
  - Medicine
    - Diagnostics (isotope production)
    - Radiation treatment
  - Industry
    - Ion implanters
    - Electron accelerators for radiation processing (e.g. polymer crosslinking, sterilisation...)
- 10% research and education
  - Large scale facilities (e.g. CERN, GSI, etc.)
  - Synchrotron light sources
  - Cyclotrons
  - Electrostatic accelerators (including implanters)
ELECTROSTATIC ACCELERATORS
RBI-AF, Zagreb, Croatia

- 100 keV – 40 MeV
- p, He, Li, C, O, Si, Cl, I, Au....
RBI-AF, Zagreb, Croatia

1.0 MV HVE Tandetron accelerator

6.0 MV EN Tandem Van de Graaff accelerator

PIXE/RBS

In-air PIXE

Dual-beam irradiation

IAEA beam line

TOF ERDA

PIXE crystal spectrometer

Nuclear reactions

Ion microprobe
ION BEAM ANALYSIS

Elastic scattering of incoming ion → Rutherford backscattering spectrometry - RBS

Nuclear reaction → emission of reaction product (particle; gamma ray) → PIGE and NRA techniques

Energy loss of incoming ions → Scanning transmission ion microscopy - STIM

Inner shell ionization → emission of x-ray → PIXE spectroscopy

Elastic scattering → recoil of target nuclei → ERDA depth profiling technique
Non-destructive techniques (most of the time...)

1 BARN (b) = 100 fm²

1 fm = 10⁻¹⁵ m

typical size of the nucleus
Rutherford Backscattering Spectrometry

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RUTHERFORD BACKSCATTERING SPECTROMETRY

For a given scattering angle $\Theta$, known projectile energy $E_{\text{inc}}$ and mass $M_1$ (e.g. 2 MeV $\alpha$), $E_{\text{sc}}$ Can be measured and therefore unknown mass $M_2$ can be determined

$$K = \frac{E_{\text{scattered}}}{E_{\text{incident}}} = \left[\frac{\left(1 - \left(\frac{M_1 \sin \theta}{M_2}\right)^2\right)^{1/2} + \frac{M_1 \cos \theta}{M_2}}{1 + \frac{M_1}{M_2}}\right]^2$$

$E$ Ion energy
$M_1$ Mass of incident ion
$M_2$ Mass of target atom
$\theta$ Scattering angle
RUTHERFORD BACKSCATTERING SPECTROMETRY

cross section

\[ \frac{d\sigma}{d\Omega} = \left( \frac{Z_1 Z_2 e^2}{4E} \right)^2 \cdot \frac{4}{\sin^4 \theta} \cdot \frac{1}{\sqrt{1 - \left( \frac{M_1 \sin \theta}{M_2} \right)^2}} \left( \frac{1}{\sqrt{1 - \left( \frac{M_1 \sin \theta}{M_2} \right)^2}} + \cos \theta \right)^2 \]

- $Z_1$: Atomic number of incident ion
- $Z_2$: Atomic number of target atom
- $E$: Energy of incident ion
- $M_1$: Mass of incident ion
- $M_2$: Mass of target atom
- $\theta$: Angle of incidence

Graph showing relative yield for different elements at 2 MeV.
Proton beam (2 MeV)
Detector positioned at $\Theta=165^0$,
Sample: thin TiO$_2$ film on Si substrate
TaSi layers of 590 and 230 nm deposited on Si substrate as seen by 2 MeV alpha RBS.
Sample:
thin film a-Si solar cell
(amorphous silicon)

5.1 MeV Li$^{2+}$ beam
$\Theta=170^\circ$
In situ RBS:
Ion beam: 2 MeV Li$^7$
Sample: AlCuFe thin film
Observation of layer intermixing
Effect of high temperature deposition on CoSi2 phase formation
- Identification of phase transition from CoSi to CoSi$_2$
ERDA - ELASTIC RECOIL DETECTION ANALYSIS

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ERDA - ELASTIC RECOIL DETECTION ANALYSIS

Geometry: transmission (problems due to energy straggling) or reflection (small sampling depth)

Experimental setup:

Stopping foil – by selection of appropriate thickness, system is optimized for one particular element (e.g. Hydrogen using He ion beam)

$\Delta E$, $E$ detector: - scattered and recoiled particles are discriminated by different $dE/dx$! (energy straggling ?)

TOF, $E$ detector:
- scattered and recoiled particles are discriminated by measurement of time of flight (with minimal straggling) – best depth resolution

+ Magnetic spectrometer (expensive)
TOF - ERDA

Acc. grid

DLC
ion

Mirror grid

MCP

$\Delta t \sim 200$ ps
Heavy ion beam – e.g. 20 MeV Iodine ions
- sensitivity $10^{15}$ /cm$^2$
- 5 nm depth resolution, up to 500 nm probe depth
- all elements are resolved

Sample:
20 nm multilayers TiN/AlN
Corrosion of ancient glass found at the fort Sokol (close to Dubrovnik airport)
PARTICLE INDUCED X-RAY EMISSION SPECTROSCOPY

Elastic scattering of incoming ion → Rutherford backscattering spectrometry - RBS

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Elastic scattering → recoil of target nuclei → ERDA depth profiling technique
Simple quantification for thin targets:
\[ Y_i = \frac{Q}{e} C_i \Omega \varepsilon \sigma_i \]
- \( Q/e \) – fluence
- \( \Omega \varepsilon \) – detector solid angle and efficiency
- \( \sigma_i \) – production cross section
- \( \sigma_i = \sigma_{ii} \omega \), where \( \sigma_{ii} \) is ionization cross section and \( \omega \) fluorescence yield
For thick targets, quantification is becoming more complicated!!

\[ Y_i = \frac{Q}{e} \int_{0}^{d} c(x) \sigma_i(E(x)) e^{-\mu x / \sin \phi} \, dx \]

Yield depends on composition due to ion stopping & x-ray absorption:
a) Iterative procedure, or
b) Matrix composition from other techniques (RBS)!!
PIXE ANALYSIS

air pollution monitoring

Nucleopore Track-Etched Membrane

Figure 2. Representative PIXE spectra of the fine airborne particulate matter collected in the sampling site.
### PIXE Analysis

#### Air Pollution Monitoring

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PIXE ANALYSIS
air pollution monitoring
In air PIXE ANALYSIS

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<th>ion/energy</th>
<th>range in air (mm)</th>
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<tr>
<td>p, 2 MeV</td>
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<td>p, 3 MeV</td>
<td><strong>140.52</strong></td>
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<tr>
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</tr>
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<td>5.21</td>
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<td>$^{28}$Si, 6 MeV</td>
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Analysis of helmet

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<th>P</th>
<th>S</th>
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</tbody>
</table>
Simple external beam setup:
- Robust Al foil exit window
- No additional vacuum pump required
- Classical Si(Li) detector
- Computer controlled XYZ table

X-ray detector

ION BEAM
(1 mm diameter)
Analysis of technology used to make Roman silver plate (found recently in town Vinkovci, Croatia)

Proton beam collimated to $\phi < 1$ mm; Scanned area 3 x 3 cm
Why we need microbeams?

- Analysis of microscopic samples!
- Imaging of elemental composition!
Available configurations at RBI:
Doublet (Dx = 11  Dy = 67)
Triplet (Dx = 30  Dy = 102)
Quintuplet (Dx=90  Dy=110)
NUCLEAR MICROPROBE

Load lock

Beam in

XYZ translator

Sample holder
Analysis of single airparticulates for identification of sources of pollution: Na, Cl – sea salt
Seawater pollution influence on sea-urchin (microbeam PIXE imaging)
PIXE and STIM maps of a skin section treated with ZnO nanoparticles
Z. Sziksai et al., NIM B 269 (2011) 2278
Painting by Hans Georg Geiger from the St. Mihael Ch., Gracani
The **light red** layer exhibits high Hg and S concentrations (HgS – cinnabar), while the **dark red** layer beneath shows presence of Pb, Al, Ca, but without Hg (either *minium*, or *carmine*).

2D element distribution of the pigment cross section sample taken from the red area of the painting.
**OTHER IBA TECHNIQUES**

- Elastic scattering of incoming ion → Rutherford backscattering spectrometry - RBS
- Inner shell ionization → emission of x-ray → PIXE spectroscopy
- Nuclear reaction → emission of reaction product (particle; gamma ray) → PIGE and NRA techniques
- Energy loss of incoming ions → Scanning transmission ion microscopy - STIM
- Elastic scattering → recoil of target nuclei → ERDA depth profiling technique
OTHER IBA TECHNIQUES

- RBS in channeling (RBS/c)
- MeV-SIMS
- Secondary electrons SE imaging
- Ion beam induced charge (IBIC)
- Ionoluminescence (IL)
- P-p & C-C scattering
- High resolution HR-PIXE
- Elastic scattering of incoming ion → Rutherford backscattering spectrometry - RBS
- Nuclear reaction → emission of reaction product (particle; gamma ray) → PIGE and NRA techniques
- Energy loss of incoming ions → Scanning transmission ion microscopy - STIM
- Inner shell ionization → emission of x-ray → PIXE spectroscopy
- Elastic scattering → recoil of target nuclei → ERDA depth profiling technique
STIM & MeV-SIMS

Ion beam: 9 MeV O$^{4+}$, image size: 85x85 µm$^2$ (≈300 nm/pixel)

DUAL BEAM FOR in situ RBS/C ANALYSIS

5 MeV Si $\rightarrow$ SiO$_2$ quartz
RBS/c: 1 MeV protons

M. Karlušić et al., unpublished
MATERIALS MODIFICATION USING ION BEAMS

**Ion implantation:**
- a) Injection of foreign atoms
- b) Displacement of atoms

**Single ion tracks:**
Fast and heavy ions (~MeV/amu) create latent tracks of damage used as a template in nanostructuring

**Irradiation with protons:**
Produce homogeneous radiation damage that can be used for lithography, defect engineering, etc..
ION ENERGY LOSS

Energy loss of Xe ion in silicon (SRIM)

\[-\frac{dE}{dx} = \frac{4\pi n z^2}{m_e v^2} \cdot \left(\frac{e^2}{4\pi \varepsilon_0}\right)^2 \cdot \left[\ln \left(\frac{2m_e v^2}{I}\right)\right]\]

Bethe – Bloch formula
APPLICATIONS
RADIATION DAMAGE IN CAF$_2$

- Multitechnique approach to analyse nanoscale radiation damage!

TEM, RBS/c, AFM & ERDA

RIPPLES BY GRAZING INCIDENCE SHI


AFM & GISAXS

23 MeV I
15 MeV Si
6 MeV Si
3 MeV O
ION BEAM ASSISTED ORDERING OF QDs

In situ ToF-ERDA ANALYSIS OF ION TRACKS

AFM & ERDA


PERFORATING GRAPHENE

AFM & Raman

O. Ochedowski et al., Nanotechnology (2015)

GANIL
84 MeV Ta

RBI
23 MeV I

15 MeV Si
CONDUCTIVE CHANNELS IN DIAMOND

Implantation with three-dimensional masking

After thermal annealing (900 °C) damaged regions are becoming conductive
Problem: How to make reliable connection?

Si pin diode