AP 5301/8301 Instrumental Methods of Analysis and Laboratory

Lecture 11 Ion Beam Analysis

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Lecture 8: outline

- Introduction
 - Ion Beam Analysis: an overview
- Rutherford backscattering spectrometry (RBS)
 - Introduction-history
 - Basic concepts of RBS
 - Kinematic factor (K)
 - Scattering cross-section
 - Depth scale
 - Quantitative thin film analysis
 - RBS data analysis
- Other IBA techniques
 - Hydrogen Forward Scattering
 - Particle induced x-ray emission (PIXE)
- Ion channeling
 - Minimum yield and critical angle
 - Dechanneling by defects
 - o Impurity location





Analytical Techniques



But IBA is typically quantitative and depth sensitive

http://www.eaglabs.com





Ion Beam Analysis: an overview



Ion Beam Analysis



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Why IBA?

- Simple in principle
- Fast and direct
- Quantitative (without standard for RBS)
- Depth profiling without chemical or physical sectioning
- Non-destructive
- Wide range of elemental coverage
- No special specimen preparation required
- Can be applied to crystalline or amorphous materials
- Simultaneous analysis with various ion beam techniques (RBS, PIXE, NRA, channeling, etc.)
- Can obtain a lot of information in one measurement



A typical Ion Beam Analysis Facility

Experimental chamber





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IBA Equipment: accelerator

Van de Graaff (single ended)











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IBA Equipment: tandem pelletron



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Equipment: particle detector





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Rutherford Backscattering Spectrometry (RBS)



- Relatively simple physics
- Requires some experimental skills
- Requires some data simulation to obtain the most out of the spectrum
- The only quantitative and absolute method to determine
 - atomic concentration with no matrix effect
 - composition variation with depth
 - layer thickness
 - damage distribution and impurity lattice location

of multi-elemental, multi-layered films







Rutherford Backscattering Spectrometry (RBS) a brief history



- 1) Almost all of the alpha particles went through the gold foil
- 2) Some of the alpha particles were deflected only slightly, usually 2° or less.
- 3) A very, very few (1 in 8000 for platinum foil) alpha particles were turned through an angle of 90° or more.

"We shall suppose that for distances less that 10⁻¹² cm the central charge and also the charge on the alpha particle may be supposed to be concentrated at a point." (1911)







RBS: The Surveyor V experiment





IDENTIFY LUNAR SURFACE ELERGY OF ALPHA PARTICLES CLEI OF ATOMS CLEI OF ATOMS PARTICLES PROTON DETECTORS (4) IDENTIFY LUNAR SURFACE ATOMS BY MEASURING ENERGY OF PROTONS SPLIT OFF NUCLEI OF ATOMS BY ALPHA PARTICLES UI

ALPHA PARTICLES PENETRATE SURFACE ~ 25 μ m

Surveyor V, first of its spacecraft family to obtain information about the chemical nature of the Moon's surface, landed in Mare Tranquillitatis on September 11, 1967.

"Surveyor V carried an instrument to determine the principal chemical elements of the lunar-surface material," explained ANTHONY TURKEVICH, Enrico Fermi institute and Chemistry Department, University of Chicago. "After landing, upon command from Earth, the instrument was lowered by a nylon cord to the surface of the Moon ..."







General applications of RBS

- Quantitative analysis of thin films
 - thickness, composition, uniformity in depth
 - solid state reactions
 - interdiffusion
- Crystalline perfection of homo- and heteroepitaxial thin films
- Quantitative measurements of impurities in substrates
- Defect distribution in single-crystal samples
- Surface atom relaxation in single crystals
- Lattice location of impurities in single crystals







RBS: basic concepts

 Kinematic factor: elastic energy transfer from a projectile to a target atom can be calculated from collision kinematics

mass determination

 Scattering cross-section: the probability of the elastic collision between the projectile and target atoms can be calculated

> quantitative analysis of atomic composition

- Energy Loss: inelastic energy loss of the projectile ions through the target
 - perception of depth

These allow RBS analysis to give quantitative depth distribution of targets with different masses







Kinematic factor: K



Conservation of energy :

$$\frac{1}{2}m_1v^2 = \frac{1}{2}m_1v_1^2 + \frac{1}{2}m_2v_2^2$$

Conservation of momentum: $m_1 v = m_1 v_1 \cos \theta + m_2 v_2 \cos \phi$ $m_1 v_1 \sin \theta = m_2 v_2 \sin \phi$

$$K_{m_2} = \frac{E_1}{E_o} = \left[\frac{\sqrt{(m_2^2 - m_1^2 \sin^2 \theta)} + m_1 \cos \theta}}{(m_2 + m_1)}\right]^2 = K(\theta, m_2, m_1)$$







Kinematic Factor









Element identification

2.5 MeV He ion with θ =170°





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Mass Resolution, δm_2







Mass Resolution: examples

With system energy resolution $\delta E = 20 \text{keV}$ and $E_0 = 2 \text{MeV}$



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RBS Quantification

Backscattering Yield



- For a given incident number of particles Q, a greater amount of an element present (N_s) should result in a greater number of particles scattered.
- Thus we need to know how often scattering events should be detected (A) at a characteristic energy (E = KE₀) and angle θ, within our detector's window of solid angle Ω.





Scattering cross-section



If particles are coming in with impact parameters between b and b +db, they will be scattered through angles between θ and θ +d θ . For central forces, we have circular symmetry.

$$2\pi bdb = -\sigma(\theta) \cdot 2\pi \sin\theta d\theta$$

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Rutherford cross-section:

$$\frac{d\sigma}{d\Omega} = \left(\frac{Z_1 Z_2 e^2}{2E_o}\right)^2 \frac{\left[\cos\theta + (1 - A^2 \sin^2\theta)^{1/2}\right]^2}{\sin^4\left(\frac{\theta}{2}\right)(1 - A^2 \sin^2\theta)^{1/2}} \sim \left(\frac{Z_1 Z_2}{E_o}\right)^2 \qquad A = \frac{m_1}{m_2}$$





Scattering Yield

Yield,
$$Y \propto \sigma(\theta) = \left(\frac{Z_1 Z_2 e^2}{2E_0}\right)^2 \frac{\left[\cos\theta + (1 - A^2 \sin^2\theta)^{1/2}\right]^2}{\sin^4 \frac{\theta}{2} (1 - A^2 \sin^2 \theta)^{1/2}}$$

 $\propto (\frac{Z_1 Z_1}{E_0})^2 \sim 10^{-24} \, cm^2 [barn]$









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Energy Loss



When an He or H ion moves through matter, it loses energy through

- interactions with e⁻ by raising them to excited states or even ionizing them.
- Direct ion-nuclei scattering

Since the radii of atomic nuclei are so small, interactions with nuclei may be neglected

$$\frac{dE}{dx}\Big|_{total} = \frac{dE}{dx}\Big|_{ele} + \frac{dE}{dx}\Big|_{nucl} \approx \frac{dE}{dx}\Big|_{ele}$$





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How to read a typical RBS spectrum

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A thin film on a light substrate



- Most projectile ions experience electronic stopping that results in a gradual reduction of the particle's kinetic energy (dE/dx).
- At the same time a small fraction of projectile ions come close enough to the nucleus for largeangle scattering (KE).
- A detected backscattered particle has lost some energy during initial penetration, then lost a large fraction of its remaining energy during the large-angle scattering event, then lost more energy in leaving the solid.



Depth Scale





Depth scale: thin film



Thickness of film:

$$\Delta E = \left(K\frac{dE}{dx}\Big|_{E_0} + \frac{\frac{dE}{dx}\Big|_{KE_0}}{\cos\theta}\right) \bullet t = [S_o] \bullet t$$

$$t = \frac{\Delta E}{\left[S_o\right]} = \frac{\Delta E}{N[\varepsilon_o]}$$

Ε

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 ϵ obtained from TRIM code or empirical energy loss data fitting





Example: layer thickness



consider the Au markers $\Delta E_{Au} = E_{AuF} - E_{AuB}$ $= [K_{Au} dE/dx |_{Eo} + 1 / (cos10^{\circ}) \cdot dE/dx |_{EAuB}] \cdot t$

 $dE/dx |_{3MeV} = N_{AI} \varepsilon_{AI} |_{3MeV}$ = 6.02x10²² • 36.56x10⁻¹⁵ = 2.2x10⁹eVcm⁻¹

 $dE/dx |_{EAuB} = N_{AI} \varepsilon_{AI} |_{2.57MeV}$ = 6.02x10²² x 39.34x10⁻¹⁵ = 2.37x10⁹ eVcm⁻¹ t = 3945Å

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consider the AI signals $\Delta E_{AI} = [K_{AI} dE/dx|_{E0} + 1 / (cos10^{\circ}) \cdot dE/dx|_{KE0}] \cdot t$ t = 3937Å



Energy Loss: Bragg's rule

For a target $A_m B_n$, the stopping crosssection is the sum of those of the constituent elements weighted by the abundance of the elements.

 $\varepsilon^{AmBn} = m \varepsilon^{A} + n \varepsilon^{B}$

$\begin{array}{l} \mbox{Example:} \\ \mbox{the stopping cross-section ϵ^{Al2O3} of Al_2O_3.} \\ \mbox{Given:} $\epsilon^{Al} = 44 \times 10^{-15} eVcm^2$ \\ $\epsilon^{O} = 35 \times 10^{-15} eVcm^2$ \\ $\epsilon^{Al2O3} = 2/5 \times \epsilon^{Al} + 3/5 \times \epsilon^{O}$ \\ $= (2/5 \times 44 + 3/5 \times 35) \times 10^{-15}$ \\ $= 38.6 \times 10^{-15} eV-cm^2/atom$ \\ \mbox{dE/dx}(Al_2O_3) = N \epsilon^{Al2O3} = (1.15 \times 10^{22})(38.6 \times 10^{-15}) eV/cm$ \\ $= 44.4 eV/Å$ \end{array}$









Quantitative analysis: composition and thickness



$$A_{A} = \sigma_{A} \bullet \Omega \bullet \Omega \bullet (Nt)_{A}$$

$$A_{B} = \sigma_{B} \bullet \Omega \bullet \Omega \bullet (Nt)_{B}$$

$$\frac{A_{A}}{A_{B}} = \left(\frac{\sigma_{A}}{\sigma_{B}}\right) \bullet \left(\frac{(Nt)_{A}}{(Nt)_{B}}\right) = \left(\frac{Z_{A}}{Z_{B}}\right)^{2} \bullet \frac{m}{n}$$

$$\frac{m}{n} = \left(\frac{A_{A}}{A_{B}}\right) \bullet \left(\frac{Z_{B}}{Z_{A}}\right)^{2}$$

$$t = \frac{(\Delta E)_{A}}{[S_{o}]_{A}^{A_{m}B_{n}}} = \frac{(\Delta E)_{B}}{[S_{o}]_{B}^{A_{m}B_{n}}}$$

$$t = \frac{(\Delta E)_{A}}{N[\varepsilon_{o}]_{A}^{A_{m}B_{n}}} = \frac{(\Delta E)_{B}}{N[\varepsilon_{o}]_{B}^{A_{m}B_{n}}}$$





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Example: As implanted Si



K_{As}=0.809; K_{Si}=0.566 $[\varepsilon_o]_{Si}^{Si} = 92.6 \times 10^{-15} eV - cm^2 / atom$ $[\varepsilon_{o}]_{As}^{Si} = 95.3 \times 10^{-15} eV - cm^{2} / atom$ $\Delta E_{As}^{Si} = 68 keV$ $(FWHM)_{As} = 60 keV$ $R_p = \frac{\Delta E_{As}^{Si}}{N[\varepsilon_o]_{As}^{Si}} = 1420\text{\AA}$ $\Delta R_p = \frac{(FWHM)_{As}}{2.355 \bullet N[\varepsilon_o]_{As}^{Si}} = 540\text{\AA}$ Total As dose:

$$(Nt)_{As} = \frac{A_{As}}{(\sigma_{As} \bullet \Omega \bullet Q)}$$





RBS Application: impurity profile



Thin film analysis









Example: silicide formation (1970-80s)

Before reaction

After reaction



Wei-Kan Chu, James W. Mayer and Marc-A. Nicolet, <u>Backscattering Spectrometry</u>, (Academic Press, New York 1978).





RBS application: silicide formation





J. O. Olowolafe et al. Thin Solid film 38, 143 (1976).





IBA Data Analysis

- Starts with a simple guess sample structure (film thickness and composition)
- Simulates spectrum and directly compare to experimental data
- Iterates simulated structure to fit experimental data (either automatically or manually)
- An ideal software:
 - able to handle different data files
 - have large data base for various ion-solid interaction: resonant scattering, nuclear reaction, etc.
 - User friendly interface
 - Fast simulation
 - Can simulate sample non-uniformity (roughness, compositional gradient, etc.)
 - Able to correct for electronic errors (pile up, dead time, charge up, etc)



Thin Film Analysis: single layer



Thin film analysis: multilayers











Impurity profile



Strengths of RBS

- Simple in principle
- Fast and direct
- Quantitative without standard
- Depth profiling without chemical or physical sectioning
- Non-destructive
- Wide range of elemental coverage
- No special specimen preparation required
- Can be applied to crystalline or amorphous materials
- Simultaneous analysis with various ion beam techniques (PIXE, PIGE, channeling, etc.)





Weaknesses of RBS

- Poor lateral resolution (~0.5-1mm)
- Moderate depth resolution (>50Å)
- No microstructural information
- No phase identification
- Poor mass resolution for target mass heavier than 70amu (PIXE)
- Detection of light impurities more difficult (e.g. Li, B, C, O, etc) (non-Rutherford scattering, Nuclear Reaction Analysis)
- Data may not be obvious: require knowledge of the technique (simulation software)





Hydrogen Forward Scattering (HFS) (Elastic Recoil Detection Analsyis ERDA)







Hydrogen Forward Scattering (HFS)

Generally known as elastic recoil detection analysis (ERDA)







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Hydrogen Forward Scattering (HFS)

Generally known as Elastic Recoil Detection (ERD)



Charles Evans and Assoc., RBS APPLICATION SERIES NO. 3

- Quantitative hydrogen and deuterium profiling
- Good sensitivity (~0.01at% of H)

long Kong

- Can be perform simultaneously with RBS and PIXE
- Profiling with any light element in solid (using heavy ion beam, ERD)







HFS: a-SiN:H film







Energy (MeV)



Courtesy: F. Hellman group, 2008





Particle Induced X-ray Emission (PIXE)



Atom in the Sample



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PIXE: Light impurity in heavy matrix



PIXE Application: Geology, Art, Archeology, Biology







Figure 1. External PIXE set-up at IOP.



HARVARD PIXE SYSTEM

城市大學

City University

of Hong Kong



Ion channeling









Ion Channeling



Kobelco Steel Group





Ion Channeling







Ion Channeling: minimum yield and critical angle



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Two important parameters to characterize channeling results:

1. Minimum yield:

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$$\chi_{\min} = \frac{Y_{channeled}}{Y_{random}}$$

~0.02-0.06

2. Critical half-angle, $\psi_{1/2}$ indicates presence of defects responsible for beam dechanneling



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Ion Channeling: minimum yield and critical angle

Minimum Yield, χ_{min}



$$\chi_{\min} = \frac{Y_{channeled}}{Y_{random}}$$
$$\chi_{\min} \approx \frac{\pi r_{\min}^2}{\pi r_o^2} \sim 0.02 - 0.05$$

Critical half-angle, $\psi_{1/2}$



$$\Psi c = \frac{1}{\sqrt{2}} \left(\frac{2Z_1 Z_2 e^2}{Ed}\right)^{\frac{1}{2}} \left\{ \ln\left[\left(\frac{Ca}{\rho}\right)^2 + 1\right] \right\}^{\frac{1}{2}}$$

where d is the distance of atoms in a row, a is the Thomas-Fermi screeing distance, r is rms thermal vibration

$$\Psi_{1/2} \sim \Psi_c \sim (\frac{2Z_1Z_2}{E})^2 \sim 0.5 - 1^o$$





Dechanneling by defects







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Channeling: Impurity Lattice Location



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Experimental techniques : combined channeling RBS/PIXE



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Channeling: Ga_{1-x-y}Be_yMn_xAs



Homo- and Heteroepitaxy

港城市大學

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Channeling: Heteroepitaxy





Z. Liliental-Weber

550mm min on 210 mm Ga

K. M. Yu et al., LBNL 2004.





Amorphous layer analysis







Strengths of Ion Beam Analysis Techniques

- Simple in principle
- Fast and direct
- Quantitative (without standard for RBS)
- Depth profiling without chemical or physical sectioning
- Non-destructive
- Wide range of elemental coverage
- No special specimen preparation required
- Can be applied to crystalline or amorphous materials
- Simultaneous analysis with various ion beam techniques (RBS, PIXE, channeling, etc.)







Pitfalls in IBA

Yield:
$$Y_i = N_i (\frac{d\sigma(E,...)}{d\Omega}) * Q\Omega$$

- Q-total charge
 - accurate charge integration
- Ω-solid angle
 - $d\sigma/d\Omega$ detection angle, double/plural scattering
- Other issues:
 - Radiation damage
 - Sputtering
 - Detector response
 - Surface roughness
 - Non-uniformity
 - Charging on insulators
 - Count rate effects





Charge integration

- Charge Integration
 - Accurate charge integration is important for absolute quantitative measurements
 - Good faraday cup design







Deviation from Rutherford scattering



- At very high energy and very low energy, scattering will deviate from the Rutherford type.
- At low energy : screening of e⁻ must be considered
- At high energy : nuclear short range force will enhance the cross-section, the so-called "resonance scattering."



Charging effect for insulating samples



FIG. 12.8. Surface charging effect. Comparison of RBS spectra from a quartz target using 1 MeV ⁴He: a) ungrounded; b) grounded via a thin conductive surface layer of graphite by rubbing a pencil lightly across the surface (Almeida and Macauley-Newcombe, 1991).

Severely distort the RBS spectrum

- Provide a supply of low-E electrons from a small, hot filament located nearby
- Coating the surface with a very thin layer of conducting material





Target non-uniformity

- Surface roughness and interface roughness cannot be distinguished
- Target non-uniformity will resemble diffusion









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