

# ***Ion Beam Analysis in Materials Science (Rutherford backscattering and related techniques)***

**Kin Man Yu**

*Electronic Materials Program, Materials Sciences Division,  
Lawrence Berkeley National Laboratory, Berkeley, CA 94720*

(510) 486-6656  
Kmyu@lbl.gov

# Outline



- **Introduction:**
  - **General aspects of ion beam analysis**
  - **Equipment**
- **Rutherford backscattering spectrometry (RBS)**
  - **Introduction-history**
  - **Basic concepts of RBS**
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    - **Scattering cross-section**
    - **Depth scale**
  - **Quantitative thin film analysis**
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- **Non-Rutherford scattering and nuclear reaction analysis**
- **Ion channeling**
  - **Minimum yield and critical angle**
  - **Dechanneling by defects**
  - **Impurity location**

# References



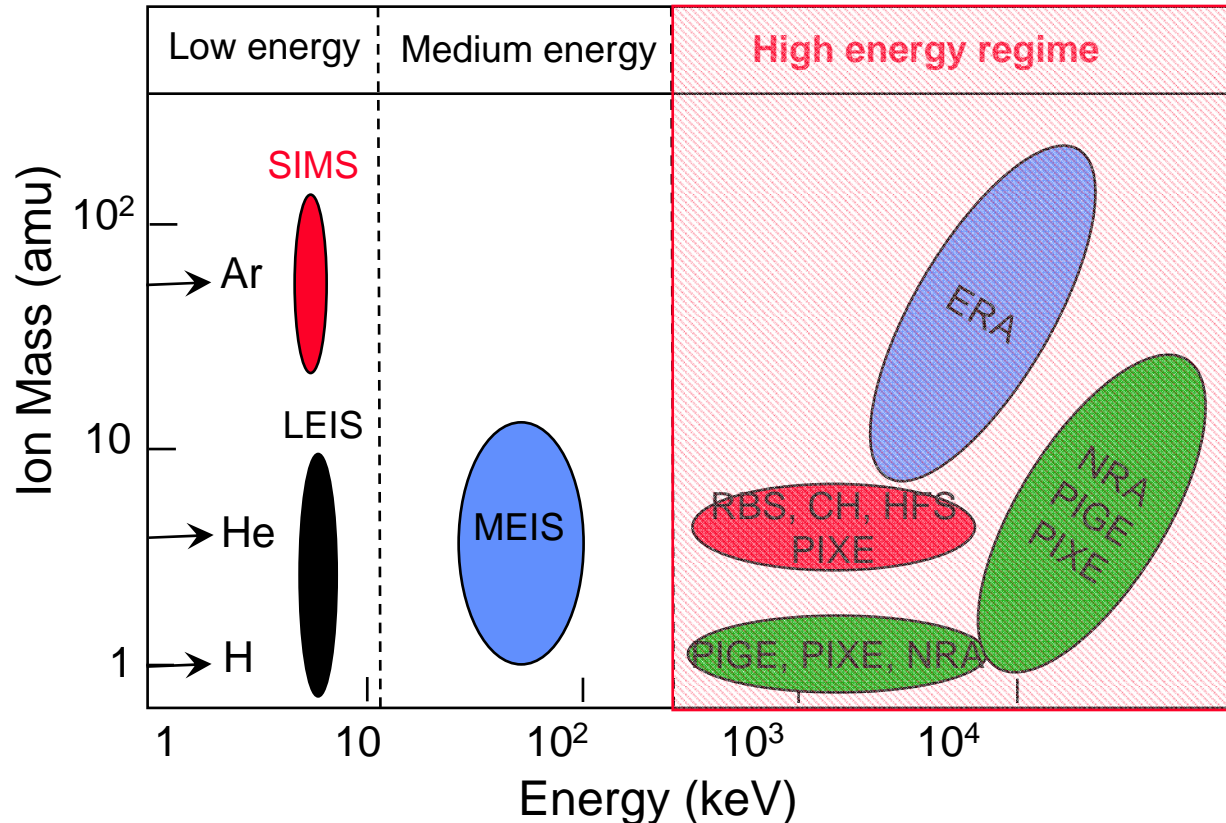
## ▪ Books:

- *Backscattering Spectrometry*, Wei-Kan Chu, James W. Mayer and Marc-A. Nicolet, (Academic Press, New York 1978).
- *Handbook of modern ion beam materials analysis*, edited by J.R. Tesmer and M. Nastasi (MRS, pittsburgh, 1995).
- *Materials Analysis by Ion Channeling*, L. C. Feldman, J. W. Mayer and S. T. Picraux, (Academic Press, New York 1982).
- *Ion Beam Materials Analysis*, edited by J. R. Bird and J. S. Williams, (Academic Press, Australia 1989).
- *Fundamentals of Surface and Thin Film Analysis*, L. C. Feldman and J. W. Mayer (Elsevier Science, New York 1986).
- *Encyclopedia of Materials Characterization*, edited by C. R. Brundle, C. A. Evans, Jr., S. Wilson, (Butterworth-Heinemann, Stoneham, MA 1992).

## ▪ Review Articles:

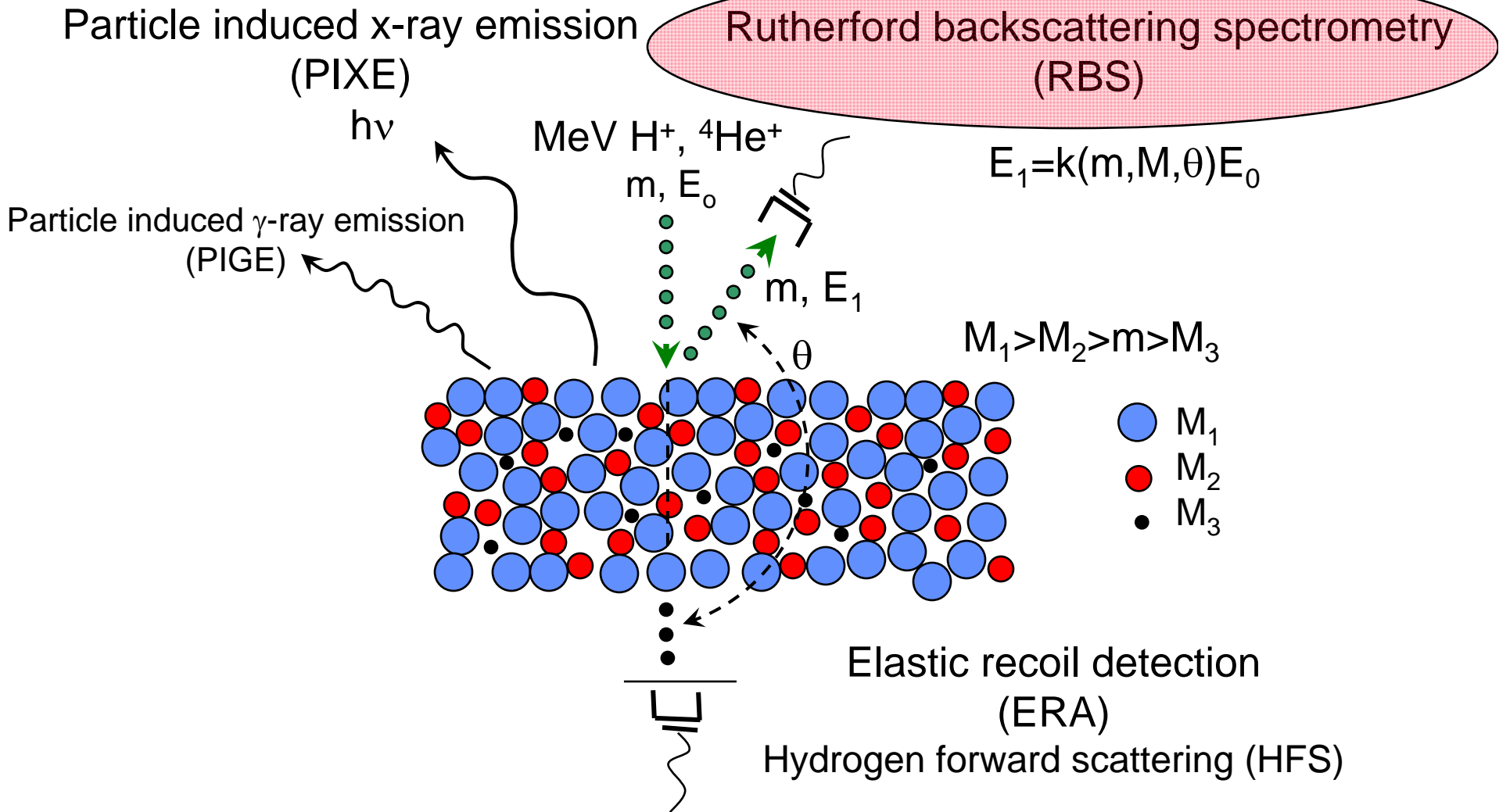
- W-K. Chu, J. W. Mayer, M-A. Nicolet, G. Amsel, T. Buck, and F. H. Eisen, "Principles and applications of ion beam techniques for the analysis of solids and thin films," *Thin Solid Films* **17**, 1 (1973).
  - L. C. Feldman, " MeV ion scattering for surface structure determination," *C. R. C. Crit. Rev. Solid State and Mater. Sci.* **10**, 143 (1981).
  - D. David, "New trends in ion-beam analysis," *Surf. Sci. Rep.* **16**, 333 (1992).
  - W. K. Chu and J. R. Liu, "Rutherford backscattering spectrometry: reminiscences and progresses," *Mater. Chem. Phys.* **46**, 183 (1996).
- <http://www.eaglabs.com/en-US/references/tutorial/rbstheo/cairtheo.html>

# Ion Beam Analysis: General



- |      |                                  |       |                                      |
|------|----------------------------------|-------|--------------------------------------|
| CH   | -Channeling                      | PIGME | -Particle Induced Gamma-ray Emission |
| ERA  | -Elastic Recoil Analysis         | SIMS  | -Secondary Ion Mass Spectrometry     |
| RBS  | -Rutherford Backscattering       | MEIS  | -Medium Energy Ion Scattering        |
| NRA  | -Nuclear Reaction Analysis       | LEIS  | -low energy ion scattering           |
| PIXE | -Particle Induced X-ray Emission | HFS   | -hydrogen forward scattering         |

# MeV Ion Beam Techniques

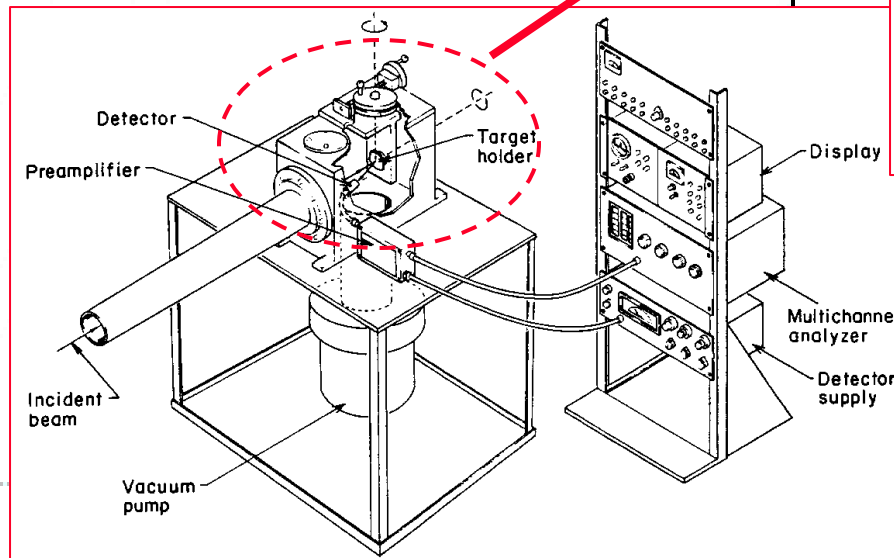
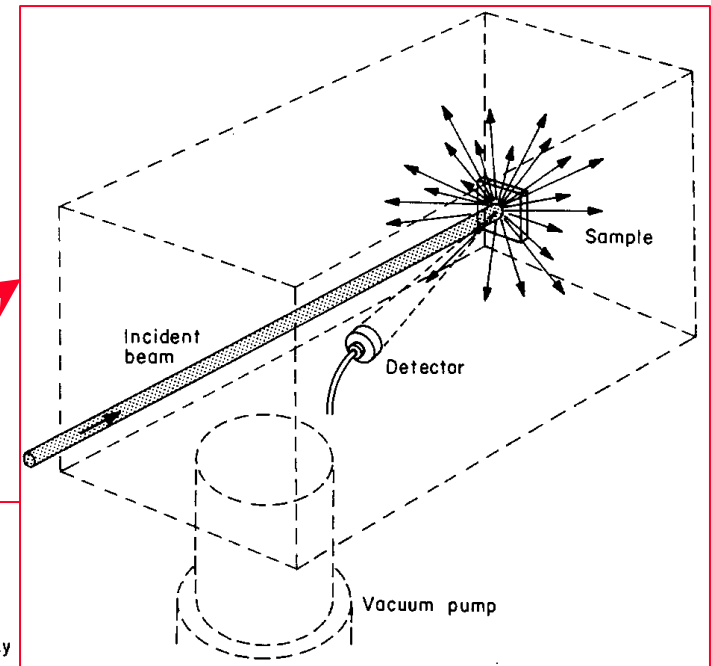
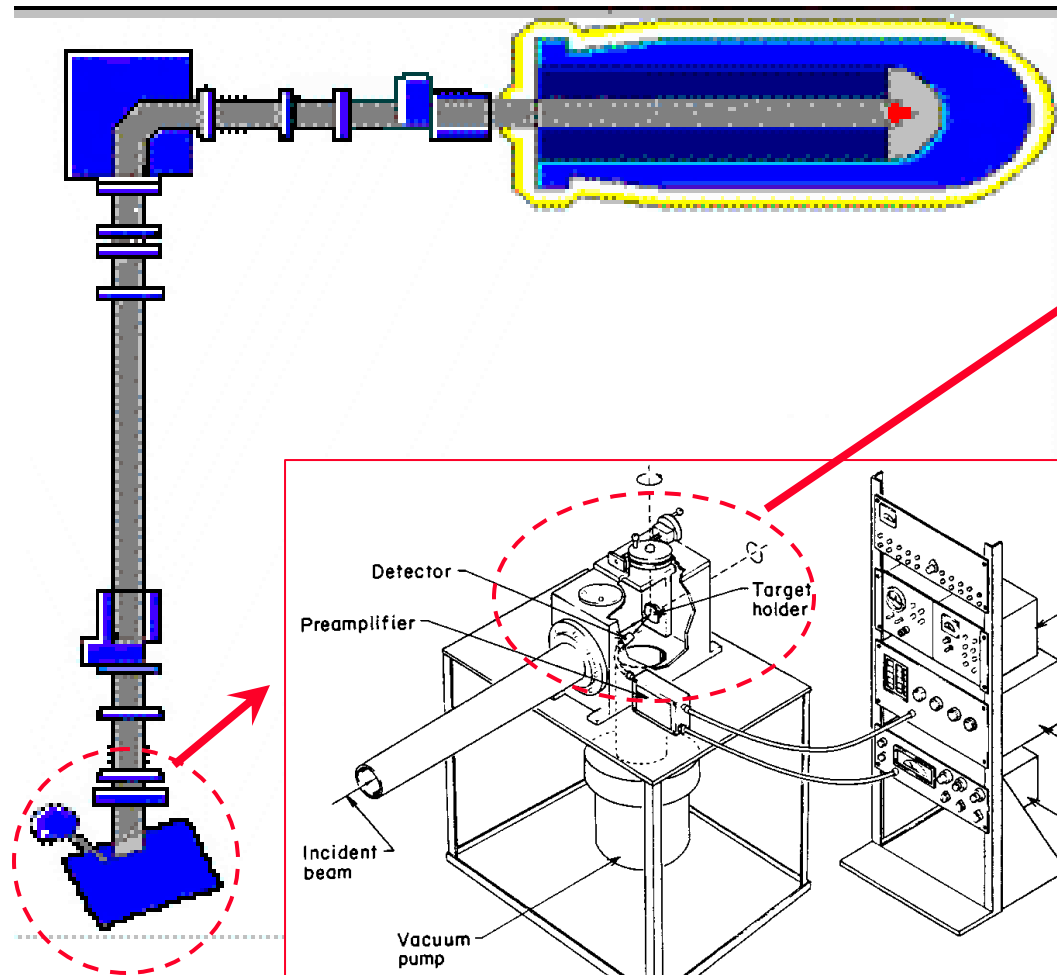


# Ion Beam techniques

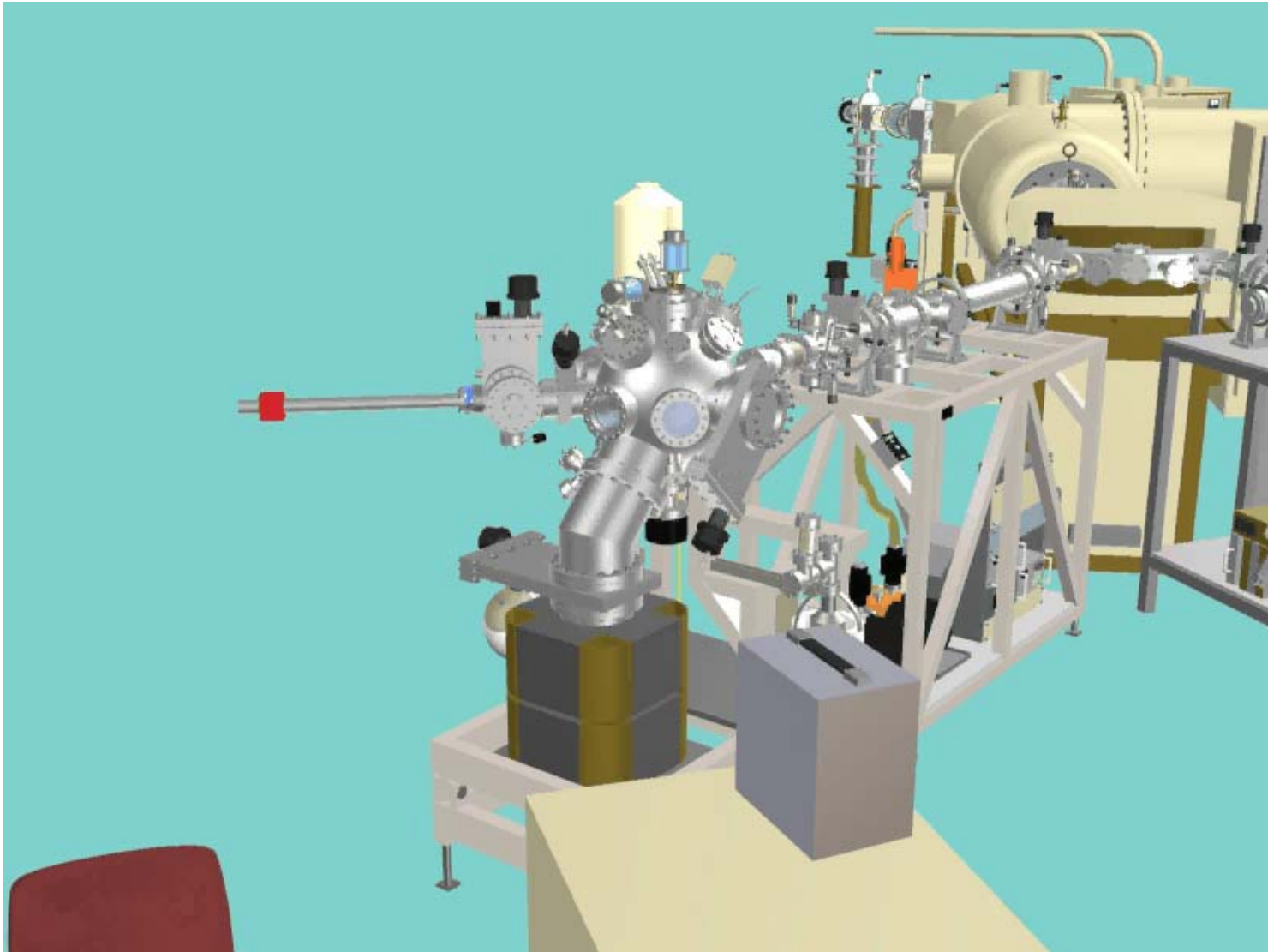


Technique	Typical Applications	Elements detected	Depth probed	Depth resolution	lateral resolution	Detection limit	Quantitative	Depth profiling
RBS	<ul style="list-style-type: none"> <li>•thin film composition and thickness</li> <li>•impurity profiles</li> <li>•thin film interactions and interdiffusions</li> </ul>	B-U	1-2 $\mu\text{m}$	20-200 $\text{\AA}$	0.5-1mm	1-10 at.% $Z < 20$ 0.01-1 at.% 0.01-0.001at.% $Z > 70$	Yes	Yes
PIXE	<ul style="list-style-type: none"> <li>•element identification</li> <li>•impurity analysis</li> </ul>	Al-U	up to 10 $\mu\text{m}$	poor	0.5-1mm	0.001 at.%	Yes	No
HFS	<ul style="list-style-type: none"> <li>•hydrogen or deuterium in thin films</li> </ul>	H, d	1 $\mu\text{m}$	500 $\text{\AA}$	2-3 mm	0.01 at.%	Yes	Yes
non-RBS	<ul style="list-style-type: none"> <li>•Composition of thin oxide, nitride, carbide films</li> </ul>	B, C, N, O, Si	up to 10 $\mu\text{m}$	200 $\text{\AA}$	0.5-1 mm	0.1 at.%	Yes	Yes
NRA	<ul style="list-style-type: none"> <li>•profiling of light elements in heavy matrix</li> </ul>	H, B, C, N, O, F	up to a few $\mu\text{m}$	500-1000 $\text{\AA}$	0.5-1mm	0.001-1 at.%	Yes	Yes
Channeling	<ul style="list-style-type: none"> <li>•crystalline quality of thin films</li> <li>•lattice location of impurity in single crystal</li> <li>•strains in pseudomorphic thin films</li> <li>•implantation damage analysis</li> </ul>	B-U	1-2 $\mu\text{m}$	20-200 $\text{\AA}$	0.5-1mm	0.0001 at.%	Yes	Yes

# Experimental Setup



# An Ideal IBA Laboratory



Arizona State University

LAWRENCE BERKELEY NATIONAL LABORATORY



# A compact RBS System



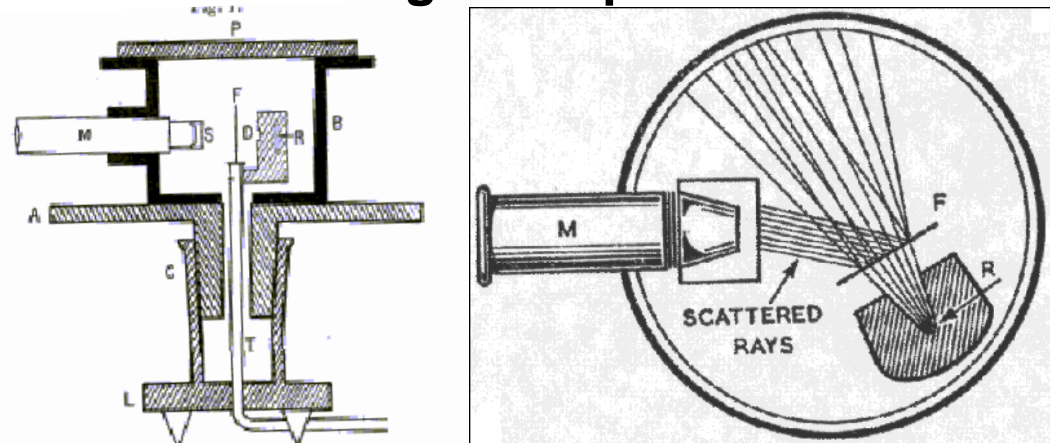
NEC MAS1000

# Rutherford Backscattering Spectrometry (RBS)

# Rutherford Backscattering Spectrometry (RBS)– a brief history



## The original experiment

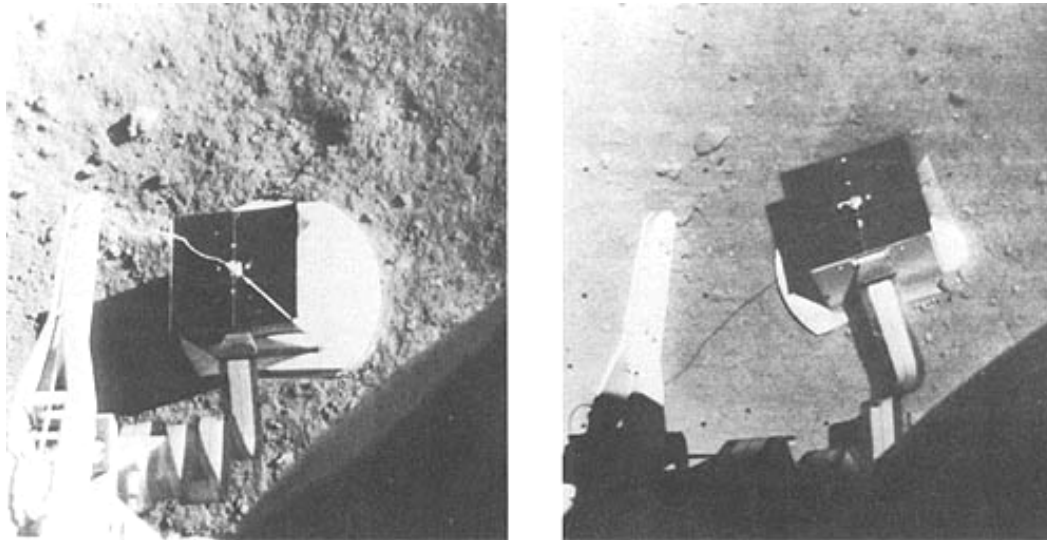


Alpha particles at a thin gold foil experiment by Hans Geiger and Ernest Marsden in 1909.

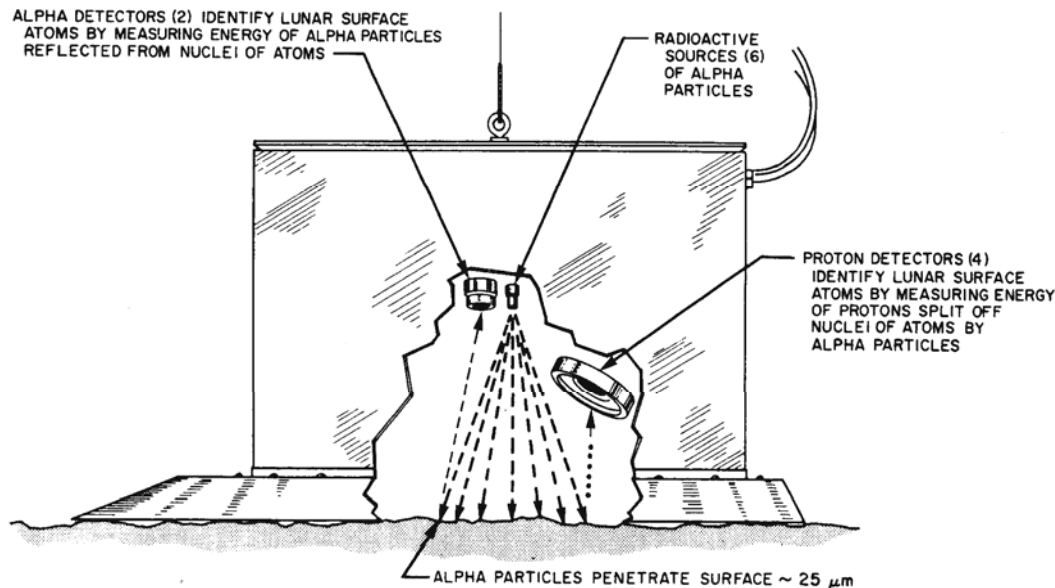
- 1) Almost all of the alpha particles went through the gold foil
- 2) Some of the alpha particles were deflected only slightly, usually  $2^\circ$  or less.
- 3) A very, very few (1 in 8000 for platinum foil) alpha particles were turned through an angle of  $90^\circ$  or more.

"We shall suppose that for distances less than  $10^{-12}$  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



**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**

# Strengths of RBS

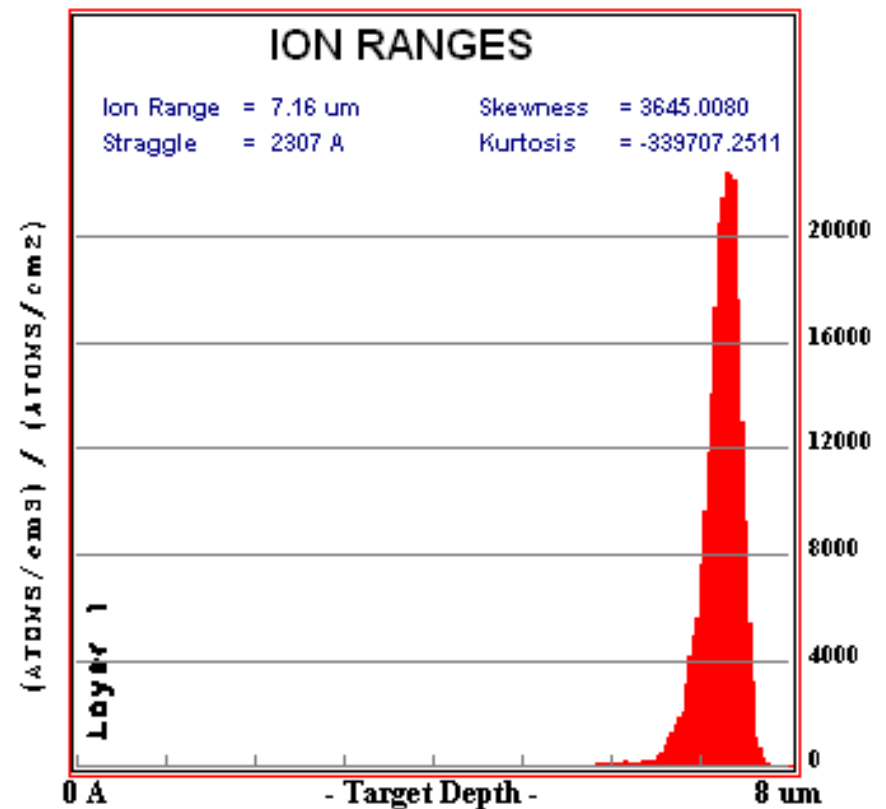
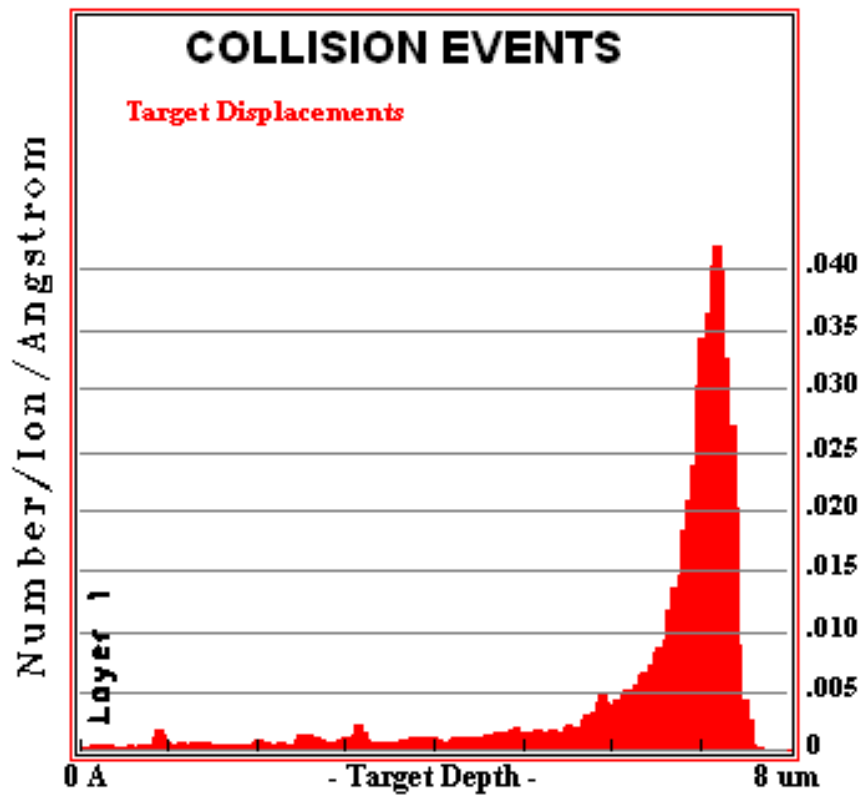


- **Simple in principle**
- **Fast and direct**
- **Quantitative without standard**
- **Depth profiling without chemical or physical sectioning**
- **Non-destructive**
- **Wide range of elemental coverage**
- **No special specimen preparation required**
- **Can be applied to crystalline or amorphous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

# Radiation Damage of RBS



2 MeV  $^4\text{He}^+$  in Si



# Basic concepts of RBS

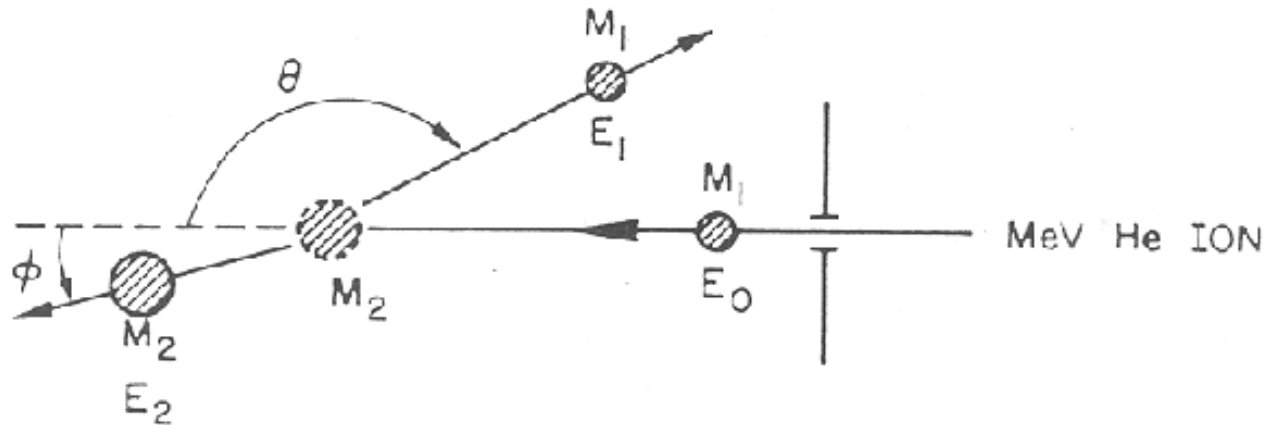


- **Kinematic factor:** elastic energy transfer from a projectile to a target atom can be calculated from collision kinematics
  - mass determination
- **Scattering cross-section:** the probability of the elastic collision between the projectile and target atoms can be calculated
  - quantitative analysis 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:**

$$m_1v = m_1v_1 \cos \theta + m_2v_2 \cos \phi$$

$$m_1v_1 \sin \theta = m_2v_2 \sin \phi$$

$$K_{m_2} = \frac{E_1}{E_0} = \left[ \frac{\sqrt{(m_2^2 - m_1^2 \sin^2 \theta)} + m_1 \cos \theta}{(m_2 + m_1)} \right]^2 = K(\theta, m_2, m_1)$$

# Kinematic Factor



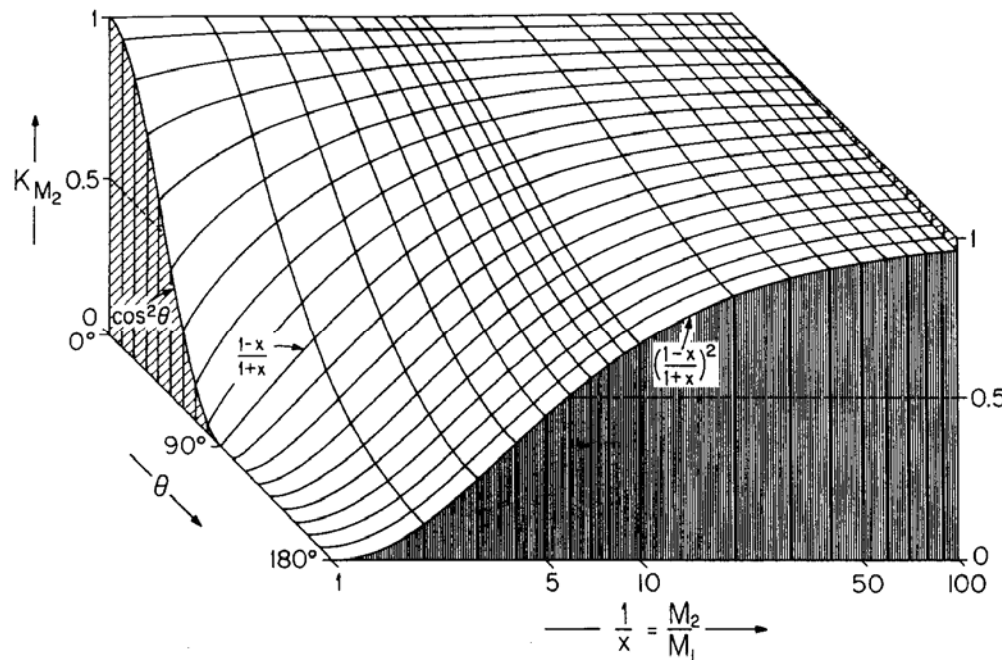
$$K_{m_2} = \frac{E_1}{E_0} = \left[ \frac{\sqrt{(m_2^2 - m_1^2 \sin^2 \theta) + m_1 \cos \theta}}{(m_2 + m_1)} \right]^2$$

$$K_{m_2}(\theta = 180^\circ) = \left[ \frac{(m_2 - m_1)}{(m_2 + m_1)} \right]^2$$

$$K_{m_2}(\theta = 90^\circ) = \frac{(m_2 - m_1)}{(m_2 + m_1)}$$

When  $m_2 \gg m_1$ :

$$K_{m_2}(\theta = 180^\circ) \sim 1$$

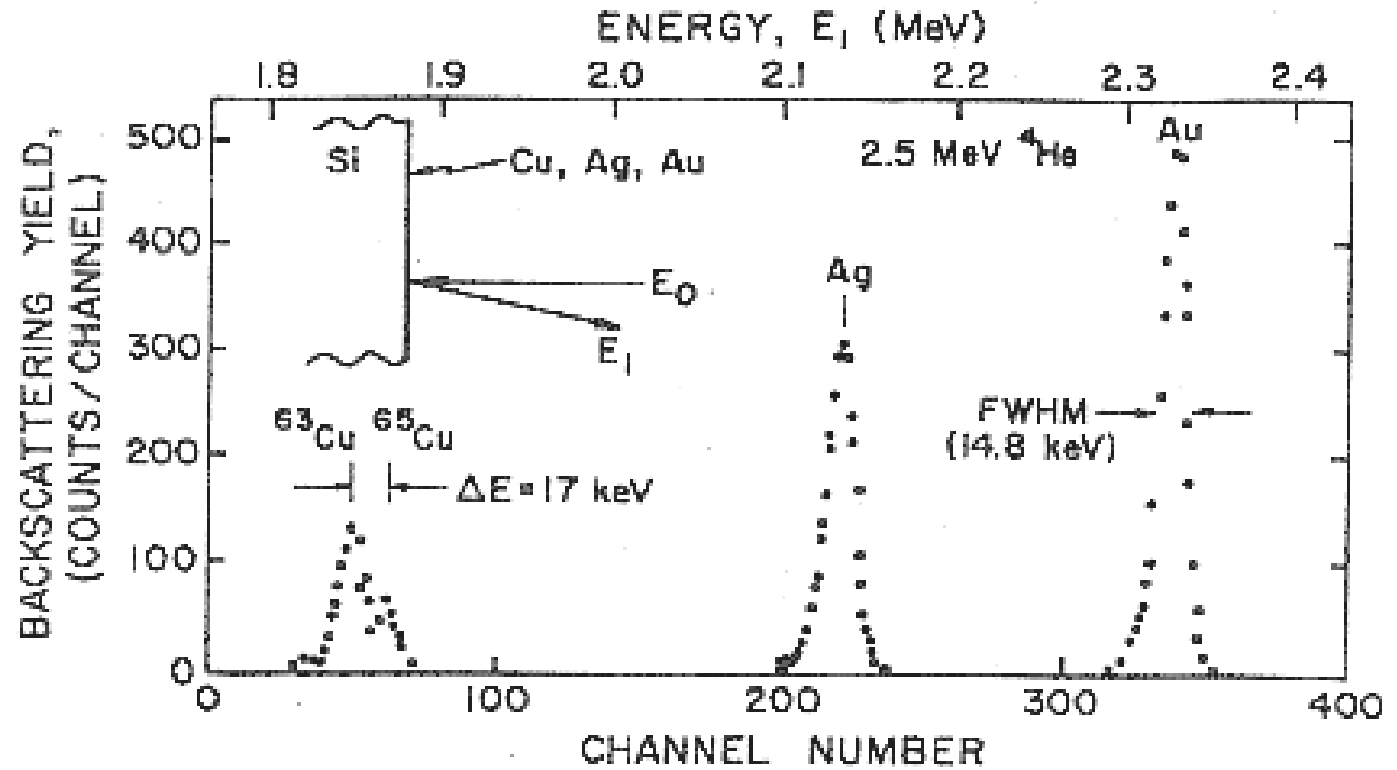


# Element identification



2.5 MeV He ion with  $\theta=170^\circ$

- $K(^{197}\text{Au}) = 0.923$
- $K(^{109}\text{Ag}) = 0.864$
- $K(^{107}\text{Ag}) = 0.862$
- $K(^{65}\text{Cu}) = 0.783$
- $K(^{63}\text{Cu}) = 0.777$

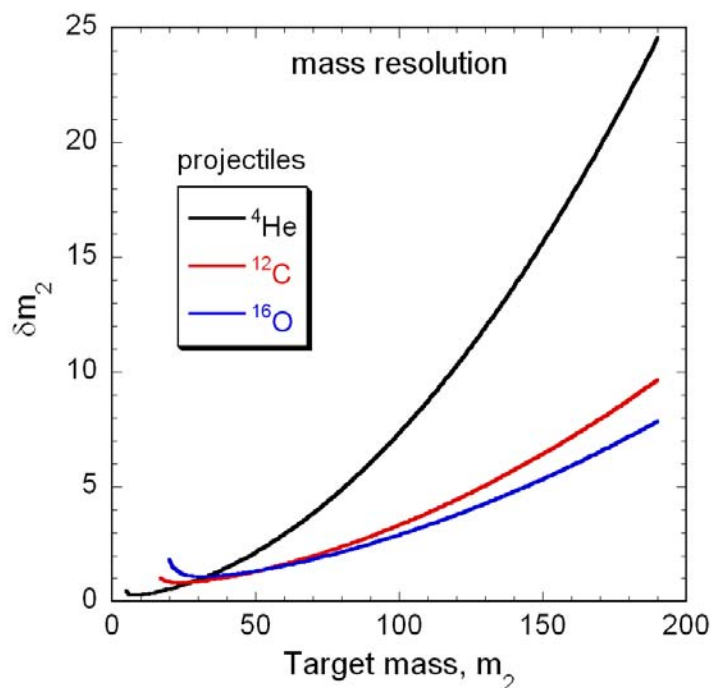


# Mass Resolution, $\delta m_2$



For 180° scattering:  $K_{m_2} = \frac{E_1}{E_0} = \left[ \frac{(m_2 - m_1)}{(m_2 + m_1)} \right]^2$

$$\frac{\delta E_1}{E_0} = \delta \frac{(m_2 - m_1)^2}{(m_1 + m_2)^2} \quad \therefore \delta m_2 = \frac{(m_2 + m_1)^3}{4m_1(m_2 - m_1)} \frac{\delta E_1}{E_0}$$



For a fixed  $\delta E_1/E_0$  ( $\sim 0.01$ ), heavier projectiles result in better mass resolution

However,  $\delta E_1$  for heavier projectiles is higher

# Mass Resolution: Examples

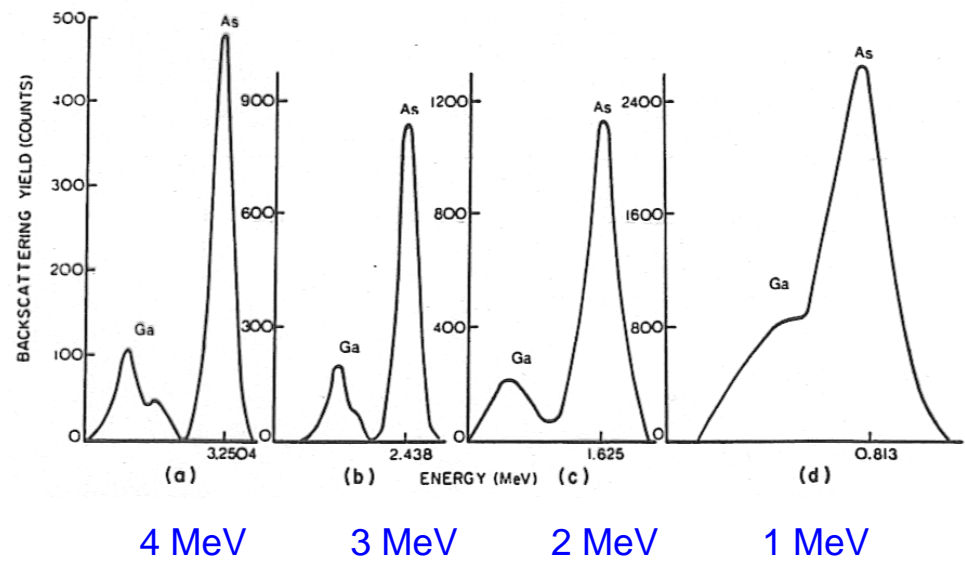
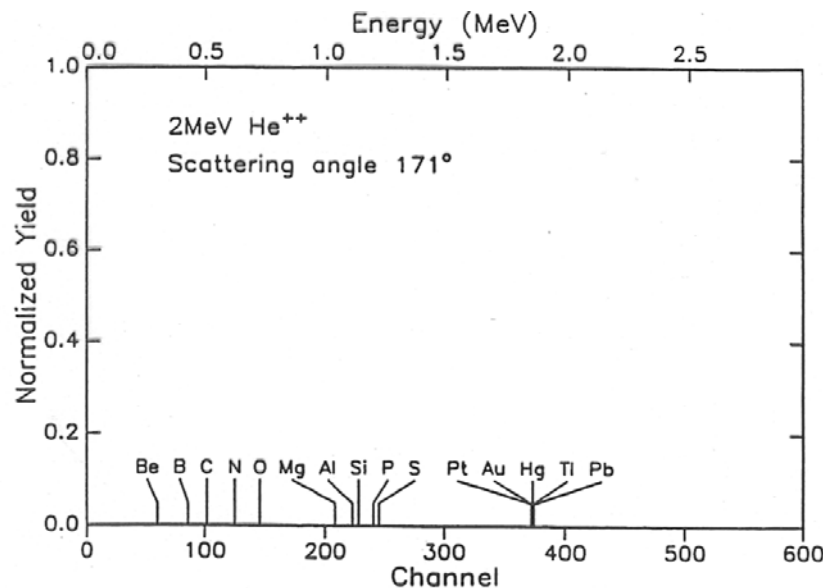


With system energy resolution  $\delta E = 20\text{keV}$  and  $E_0 = 2\text{MeV}$

For  $m_2 = 40$  
$$\delta m_2 = \frac{(40 + 4)^3}{4 \times 4(40 - 4)} \cdot \frac{20}{2000} = 1.48 a.m.u.$$

For  $m_2 = 70$  
$$\delta m_2 = \frac{(70 + 4)^3}{4 \times 4(70 - 4)} \cdot \frac{20}{2000} = 3.84 a.m.u.$$

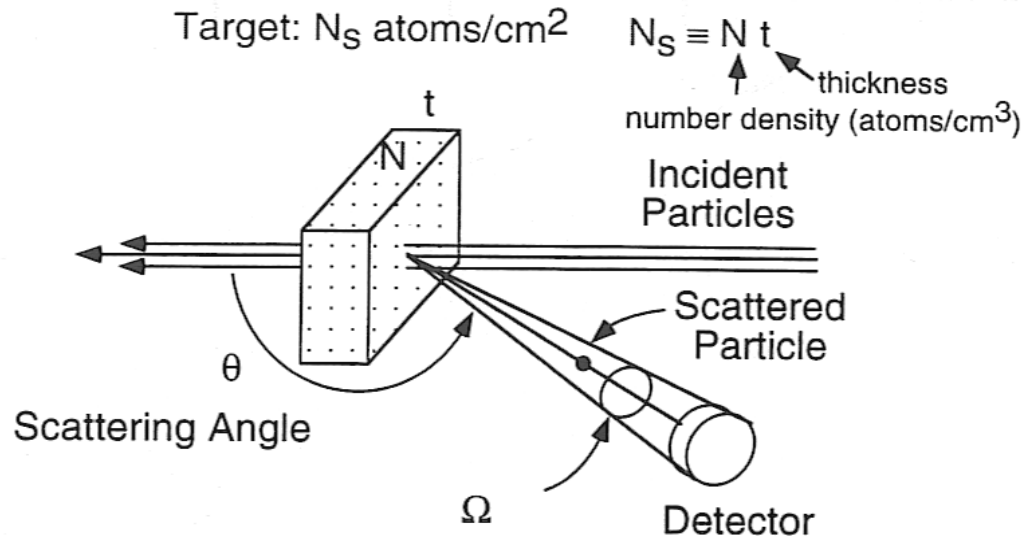
Isotopes of Ga (68.9 and 70.9 a.m.u.) cannot be resolved



# 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 ( $E = KE_0$ ) and angle  $\theta$ , within our detector's window of solid angle  $\Omega$ .

$$A = \sigma \Omega N_s Q$$

# of particles detected

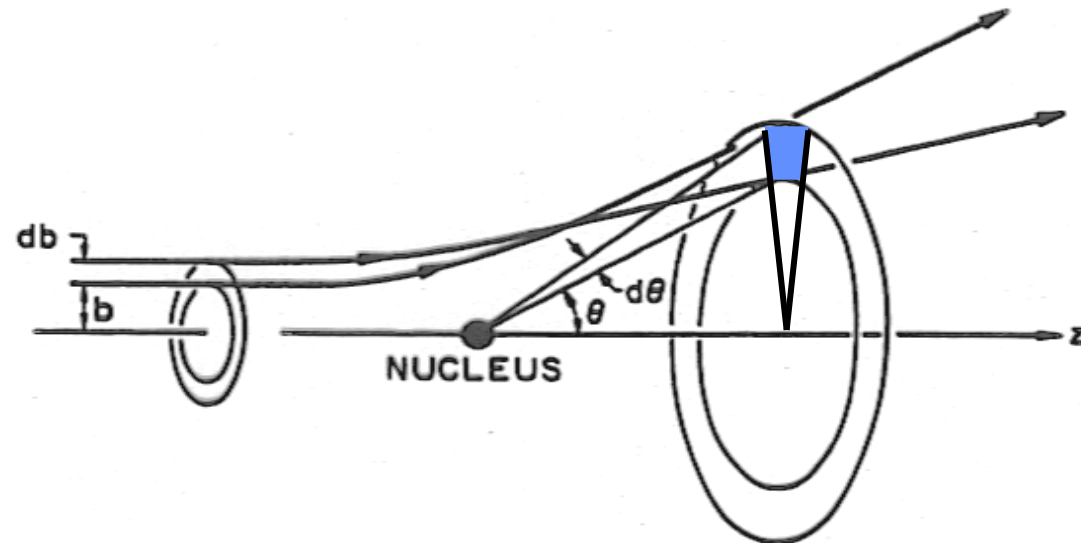
'average cross section'

atoms/cm<sup>2</sup>

detector solid angle

total number of incident particles

# Scattering cross-section



Rutherford cross-section:

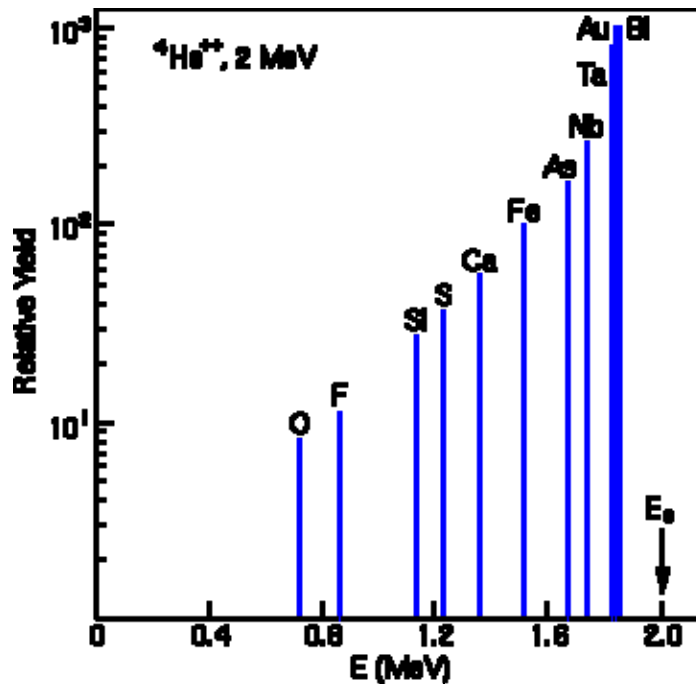
$$\frac{d\sigma}{d\Omega} = \left( \frac{Z_1 Z_2 e^2}{2E_0} \right)^2 \frac{[\cos \theta + (1 - A^2 \sin^2 \theta)^{1/2}]^2}{\sin^4 \left( \frac{\theta}{2} \right) (1 - A^2 \sin^2 \theta)^{1/2}} \quad A = \frac{m_1}{m_2}$$

# Scattering Yield

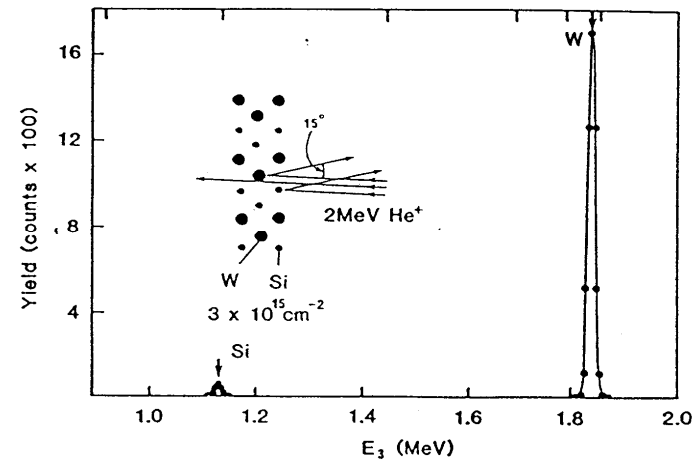


$$Yield, Y \propto \sigma(\theta) = \left( \frac{Z_1 Z_2 e^2}{2E_0} \right)^2 \frac{[\cos \theta + (1 - A^2 \sin^2 \theta)^{1/2}]^2}{\sin^4 \frac{\theta}{2} (1 - A^2 \sin^2 \theta)^{1/2}}$$

$$\propto \left( \frac{Z_1 Z_2}{E_0} \right)^2 \sim 10^{-24} \text{ cm}^2 [\text{barn}]$$



## Example:



Mixed Si and W target analyzed by a 2MeV He ion beam at 165° scattering angle.

$$\sigma(\text{Si}) = 2.5 \times 10^{-25} \text{ cm}^2/\text{str}$$

$$\sigma(\text{W}) = 7.4 \times 10^{-24} \text{ cm}^2/\text{str}$$

Total incident ions  $Q = 1.5 \times 10^{14}$  ions

$\Omega = 1.8 \text{ mstr}$

Area under Si,  $A(\text{Si}) = 200$  counts

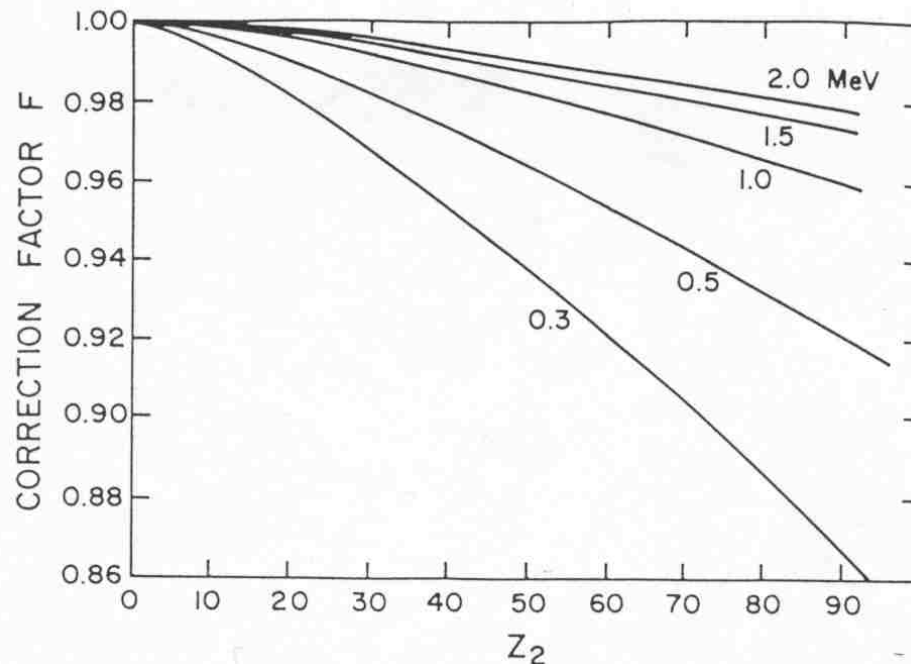
Area under W,  $A(\text{W}) = 6000$  counts

$$(Nt)_{\text{Si}} = 3 \times 10^{15} \text{ atoms/cm}^2$$

$$(Nt)_{\text{W}} = 3 \times 10^{15} \text{ atoms/cm}^2$$



# Deviation from Rutherford scattering



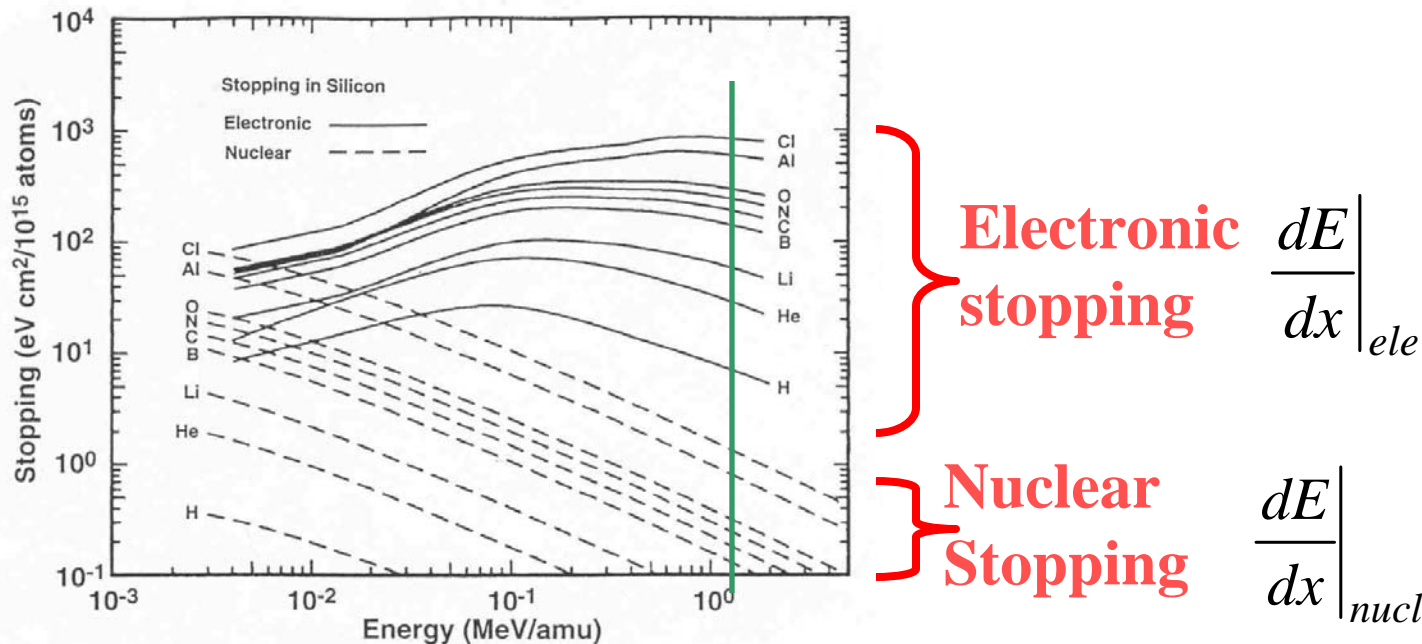
Correction factor F, which describes the deviation from pure Rutherford scattering due to electron screening for He<sup>+</sup> scattering from atoms, Z<sub>2</sub>, at variety of incident kinetic energies.

**At very high energy and very low energy, scattering will deviate from the Rutherford type.**

**At low energy : screening of e<sup>-</sup> must be considered**

**At high energy : nuclear short range force will enhance the cross-section, the so-called “resonance scattering.”**

# Energy Loss



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

- interactions with e<sup>-</sup> by raising them to excited states or even ionizing them.
- Direct ion-nuclei scattering

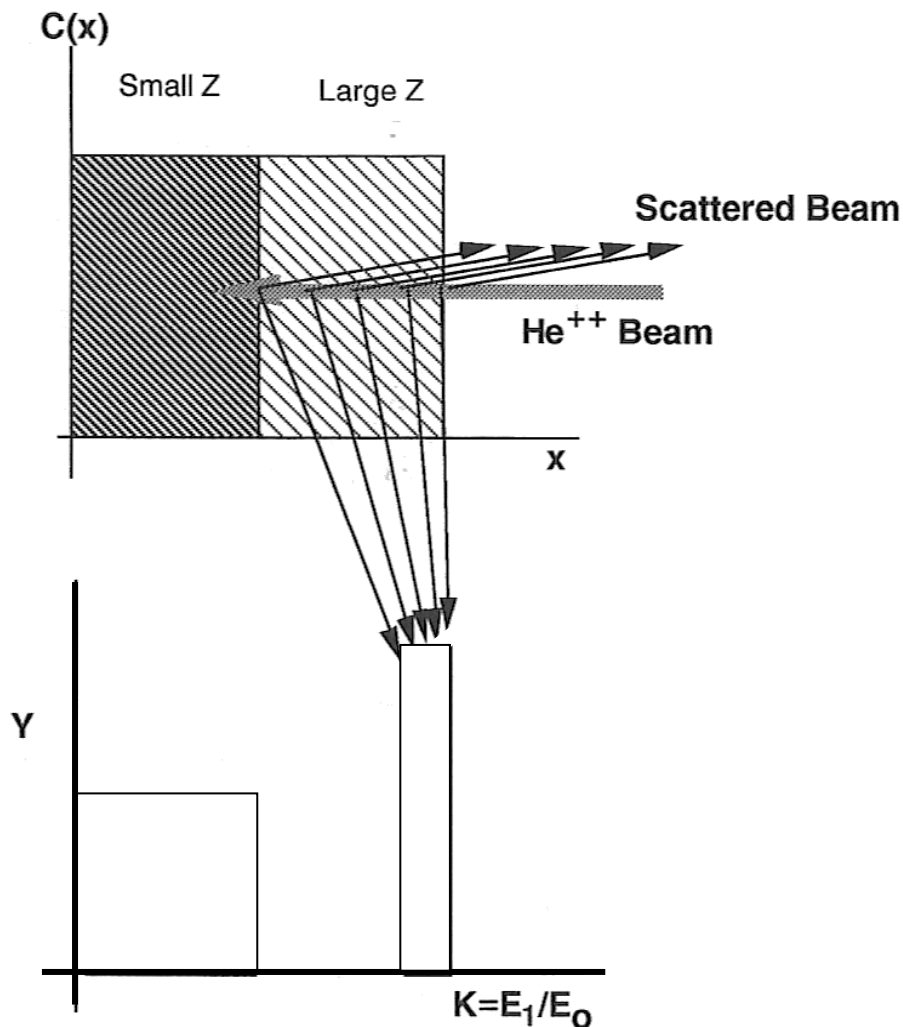
Since the radii of atomic nuclei are so small, interactions with nuclei may be neglected

$$\left. \frac{dE}{dx} \right|_{total} = \left. \frac{dE}{dx} \right|_{ele} + \left. \frac{dE}{dx} \right|_{nucl} \approx \left. \frac{dE}{dx} \right|_{ele}$$

# How to read a typical RBS spectrum

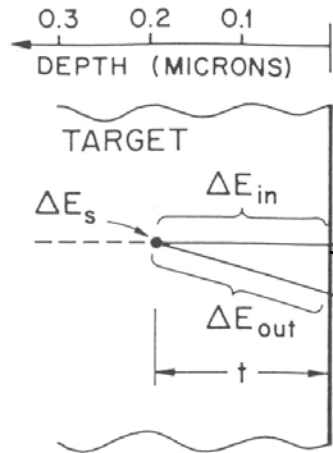


A thin film on a light substrate



- 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 large-angle 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



ENERGY LOSS:

$$\Delta E_{in} \approx \left. \frac{dE}{dx} \right|_{E_0} \cdot t$$

$$E_t = E_0 - \Delta E_{in}$$

$$\Delta E_s = (1-K) E_t$$

$$\Delta E_{out} \approx \left. \frac{dE}{dx} \right|_{E_1} \cdot \frac{t}{\cos \theta}$$

$$E_1 = K(E_0 - \Delta E_{in}) - \Delta E_{out}$$

$$E_1 = K(E_0 - \left. \frac{dE}{dx} \right|_{E_0} \cdot t) - \left( \left. \frac{dE}{dx} \right|_{KE_0} \cdot \frac{t}{\cos \theta} \right)$$

Total energy loss  $\Delta E = KE_0 - E_1$

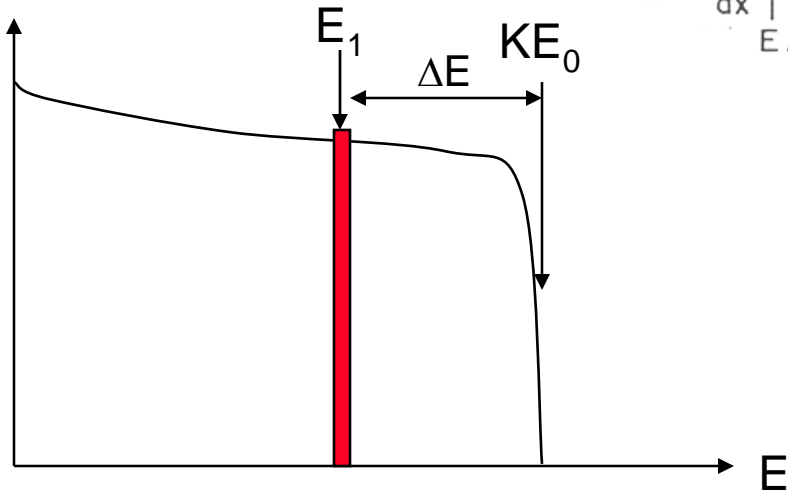
$$\Delta E = \left( \frac{K}{\cos \theta_1} \cdot \left. \frac{dE}{dx} \right|_{E_0} + \frac{1}{\cos \theta_2} \cdot \left. \frac{dE}{dx} \right|_{KE_0} \right) \cdot t = [S_o] \cdot t$$

$[S_o]$  is the effective stopping power

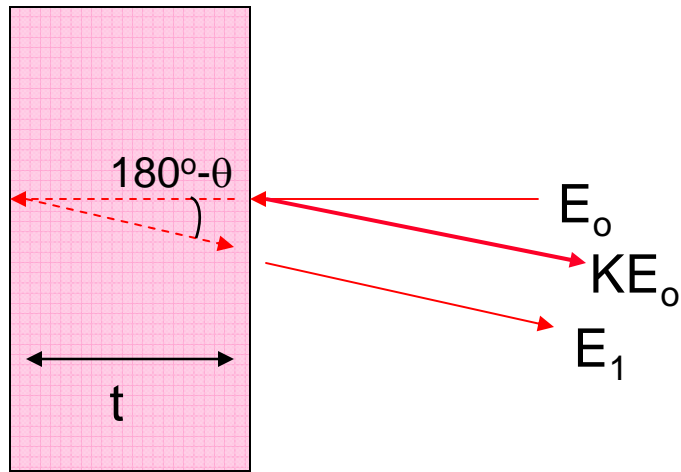
## Stopping cross-section

$$\varepsilon = \frac{1}{N} \frac{dE}{dx} : \frac{\text{eV cm}^3}{\text{cm atom}} = \frac{\text{eV cm}^2}{\text{atom}}$$

$$\Delta E = \left( \frac{K}{\cos \theta_1} \cdot N \varepsilon_{in} + \frac{1}{\cos \theta_2} \cdot N \varepsilon_{out} \right) \cdot t = N [\varepsilon_o] \cdot t$$



# Depth scale: thin film

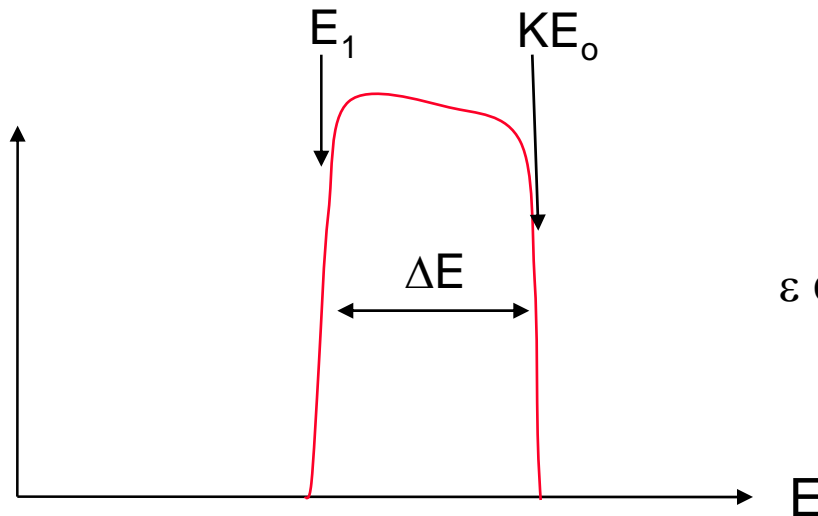


Thickness of film:

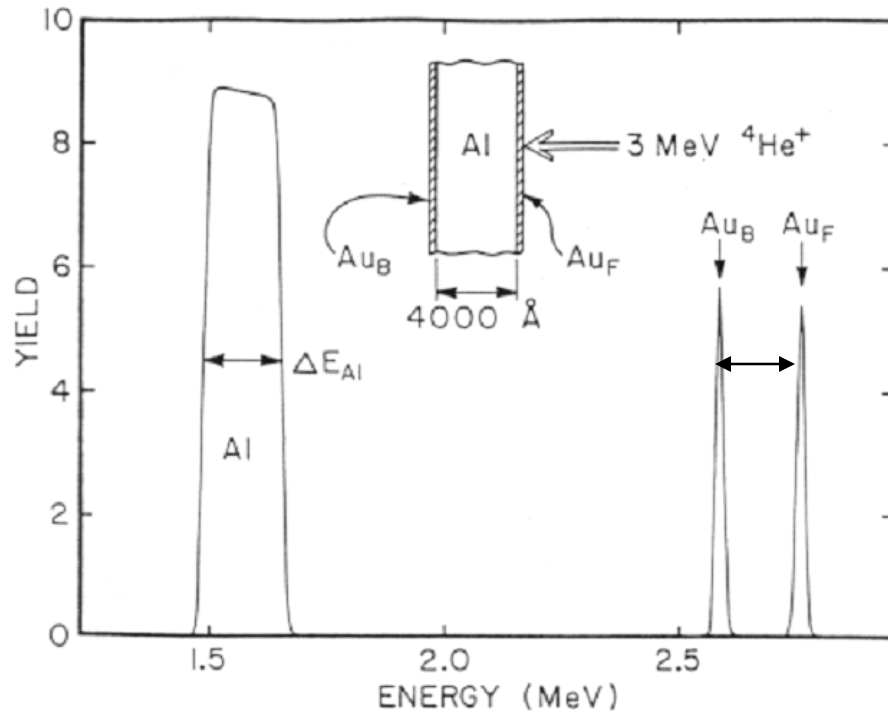
$$\Delta E = \left( K \frac{dE}{dx} \Big|_{E_0} + \frac{dE}{dx} \Big|_{KE_0} \right) \cdot t = [S_o] \cdot t$$

$$t = \frac{\Delta E}{[S_o]} = \frac{\Delta E}{N[\epsilon_o]}$$

$\epsilon$  obtained from TRIM code or empirical energy loss data fitting



# Example: layer thickness



$$\Delta E_{Al} = 165 \text{ keV}$$

$$\Delta E_{Au} = 175 \text{ keV}$$

$$K_{Au} = 0.9225$$

$$K_{Al} = 0.5525$$

## consider the Au markers

$$\begin{aligned} \Delta E_{Au} &= E_{AuF} - E_{AuB} \\ &= [K_{Au} dE/dx |_{E_0 + 1 / (\cos 10^\circ)} \cdot dE/dx |_{EAuB}] \cdot t \end{aligned}$$

$$\begin{aligned} dE/dx |_{3\text{MeV}} &= N_{Al} \epsilon_{Al} |_{3\text{MeV}} \\ &= 6.02 \times 10^{22} \cdot 36.56 \times 10^{-15} \\ &= 2.2 \times 10^9 \text{ eVcm}^{-1} \end{aligned}$$

$$\begin{aligned} dE/dx |_{EAuB} &= N_{Al} \epsilon_{Al} |_{2.57\text{MeV}} \\ &= 6.02 \times 10^{22} \times 39.34 \times 10^{-15} \\ &= 2.37 \times 10^9 \text{ eVcm}^{-1} \end{aligned}$$

$$t = 3945 \text{ \AA}$$

## consider the Al signals

$$\Delta E_{Al} = [K_{Al} dE/dx |_{E_0 + 1 / (\cos 10^\circ)} \cdot dE/dx |_{KE_0}] \cdot t$$

$$t = 3937 \text{ \AA}$$

# Energy Loss: Bragg's rule



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

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

## Example:

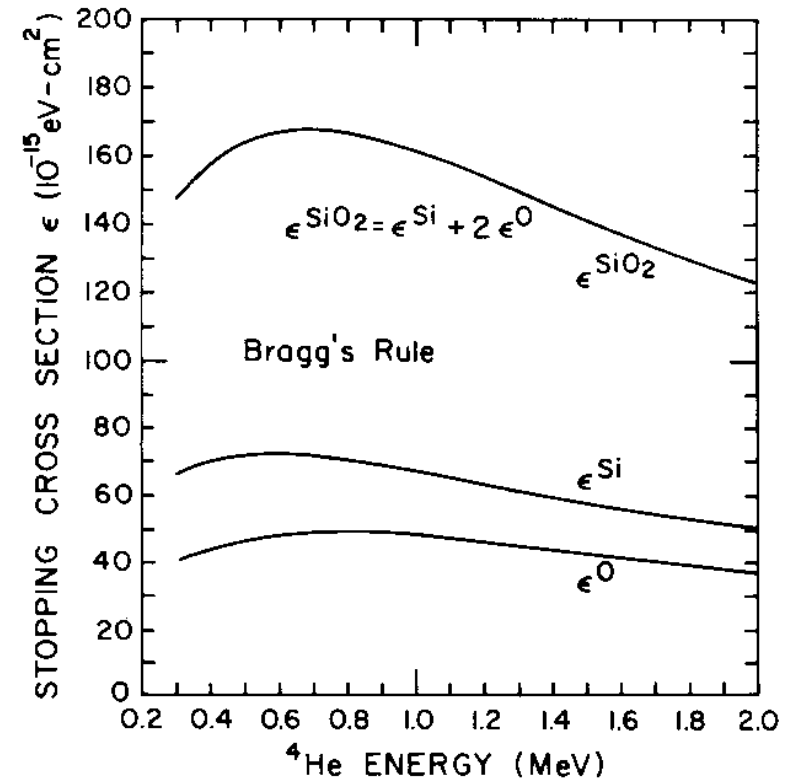
the stopping cross-section  $\epsilon^{Al_2O_3}$  of  $Al_2O_3$ .

Given:  $\epsilon^{Al} = 44 \times 10^{-15} \text{eVcm}^2$

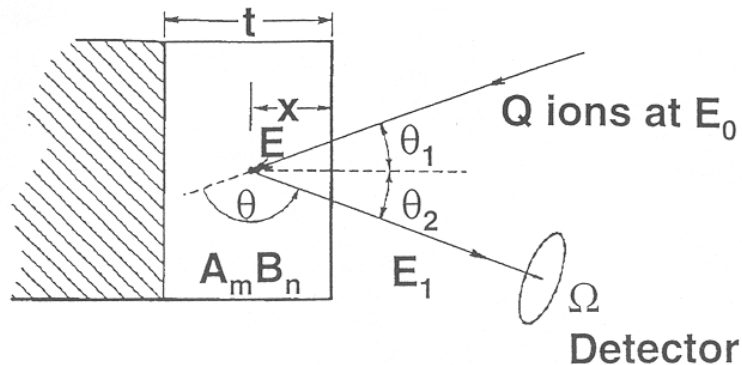
$$\epsilon^O = 35 \times 10^{-15} \text{eVcm}^2$$

$$\begin{aligned} \epsilon^{Al_2O_3} &= 2/5 \times \epsilon^{Al} + 3/5 \times \epsilon^O \\ &= (2/5 \times 44 + 3/5 \times 35) \times 10^{-15} \\ &= 38.6 \times 10^{-15} \text{eV-cm}^2/\text{atom} \end{aligned}$$

$$\begin{aligned} dE/dx(Al_2O_3) &= N \epsilon^{Al_2O_3} = (1.15 \times 10^{22})(38.6 \times 10^{-15}) \text{eV/cm} \\ &= 44.4 \text{eV/\AA} \end{aligned}$$



# Quantitative analysis: composition and thickness

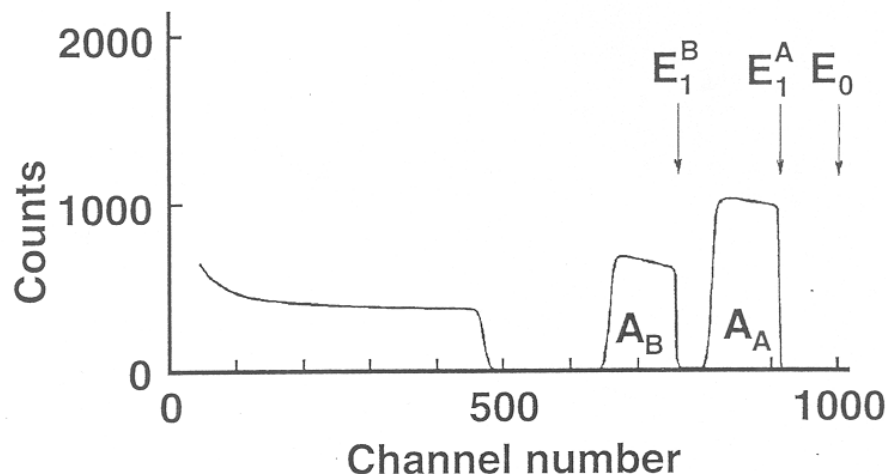


$$A_A = \sigma_A \cdot \Omega \cdot Q \cdot (Nt)_A$$

$$A_B = \sigma_B \cdot \Omega \cdot Q \cdot (Nt)_B$$

$$\frac{A_A}{A_B} = \left( \frac{\sigma_A}{\sigma_B} \right) \cdot \left( \frac{(Nt)_A}{(Nt)_B} \right) = \left( \frac{Z_A}{Z_B} \right)^2 \cdot \frac{m}{n}$$

$$\frac{m}{n} = \left( \frac{A_A}{A_B} \right) \cdot \left( \frac{Z_B}{Z_A} \right)^2$$

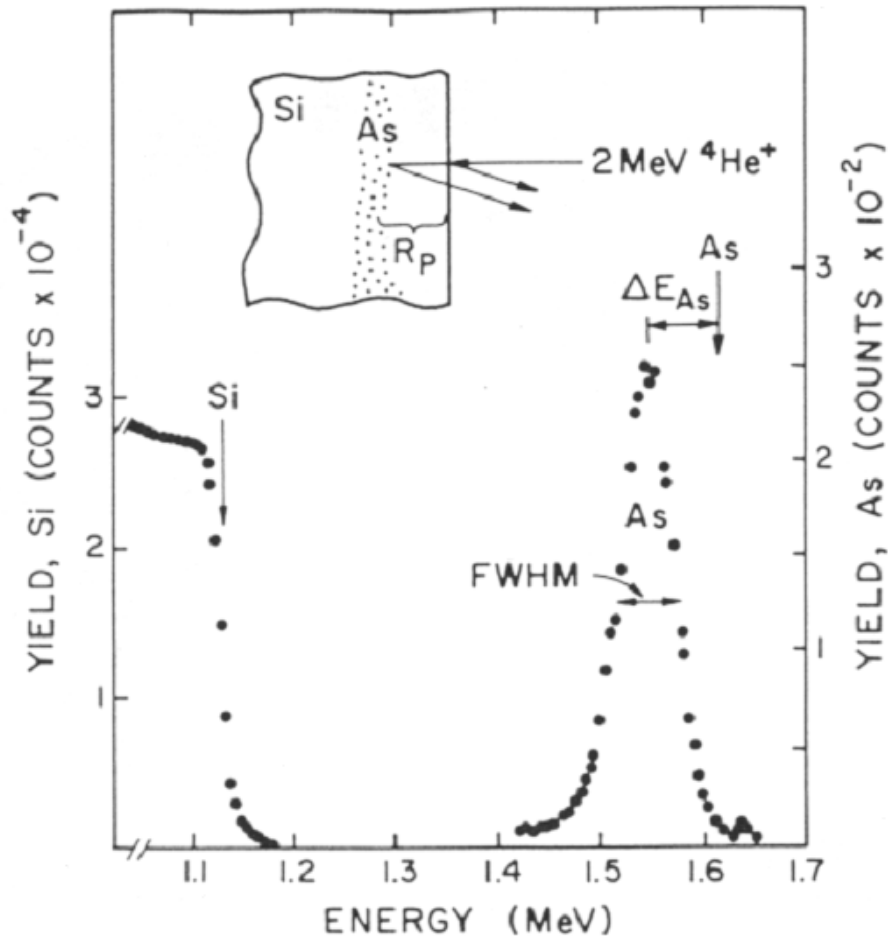


$$t = \frac{(\Delta E)_A}{[S_o]_A^{A_m B_n}} = \frac{(\Delta E)_B}{[S_o]_B^{A_m B_n}}$$

$$t = \frac{(\Delta E)_A}{N[\epsilon_o]_A^{A_m B_n}} = \frac{(\Delta E)_B}{N[\epsilon_o]_B^{A_m B_n}}$$



# Example: As implanted Si



$$K_{As} = 0.809; K_{Si} = 0.566$$

$$[\epsilon_o]_{Si}^{Si} = 92.6 \times 10^{-15} \text{ eV} - \text{cm}^2 / \text{atom}$$

$$[\epsilon_o]_{As}^{Si} = 95.3 \times 10^{-15} \text{ eV} - \text{cm}^2 / \text{atom}$$

$$\Delta E_{As}^{Si} = 68 \text{ keV}$$

$$(FWHM)_{As} = 60 \text{ keV}$$

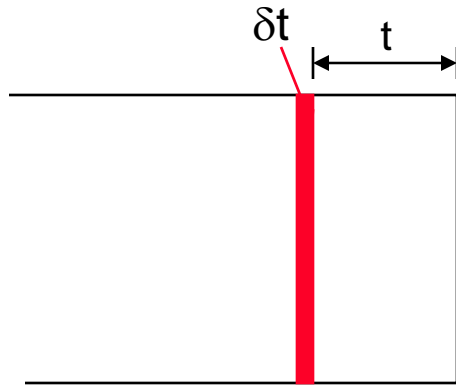
$$R_p = \frac{\Delta E_{As}^{Si}}{N[\epsilon_o]_{As}^{Si}} = 1420 \text{ \AA}$$

$$\Delta R_p = \frac{(FWHM)_{As}}{2.355 \cdot N[\epsilon_o]_{As}^{Si}} = 540 \text{ \AA}$$

Total As dose:

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

# Quantitative Analysis



When  $Y(t)$  corresponds to the yield for one energy division in the RBS spectrum:

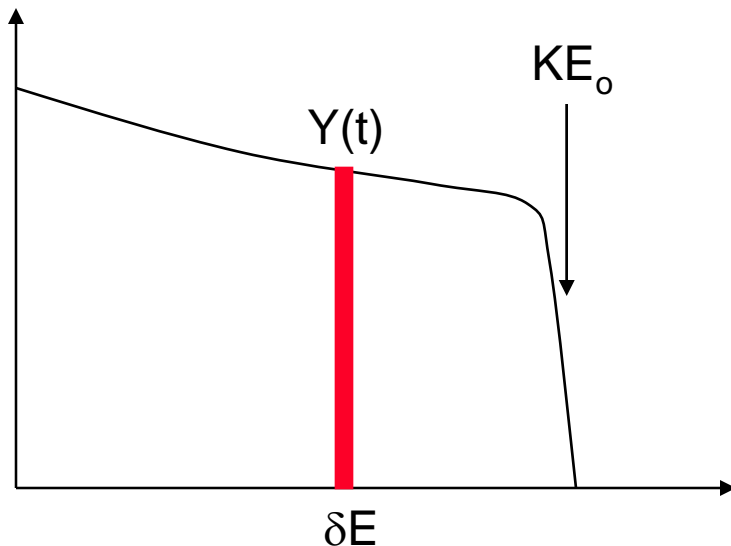
$$Y(t) = H(E)$$

$$H(E) = \sigma \cdot \Omega \cdot Q \cdot N \cdot \delta t$$

$$= \sigma \cdot \Omega \cdot Q \cdot N \cdot \delta E / [S_o]$$

$$= \sigma \cdot \Omega \cdot Q \cdot N \cdot \delta E / N[\epsilon_o]$$

where  $\delta E$  is the energy/channel in the RBS spectrum



Consider the As implanted Si example:

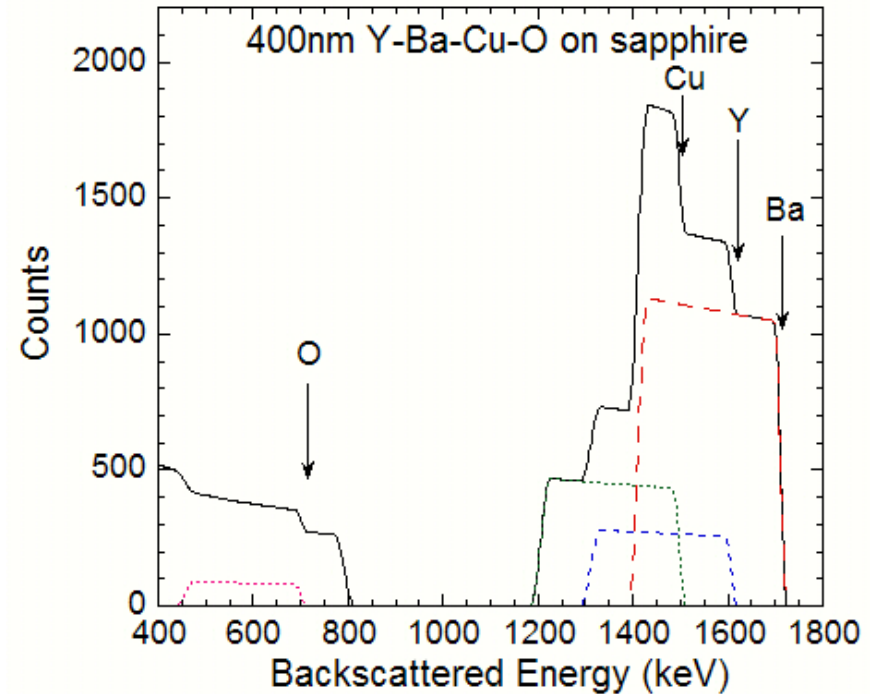
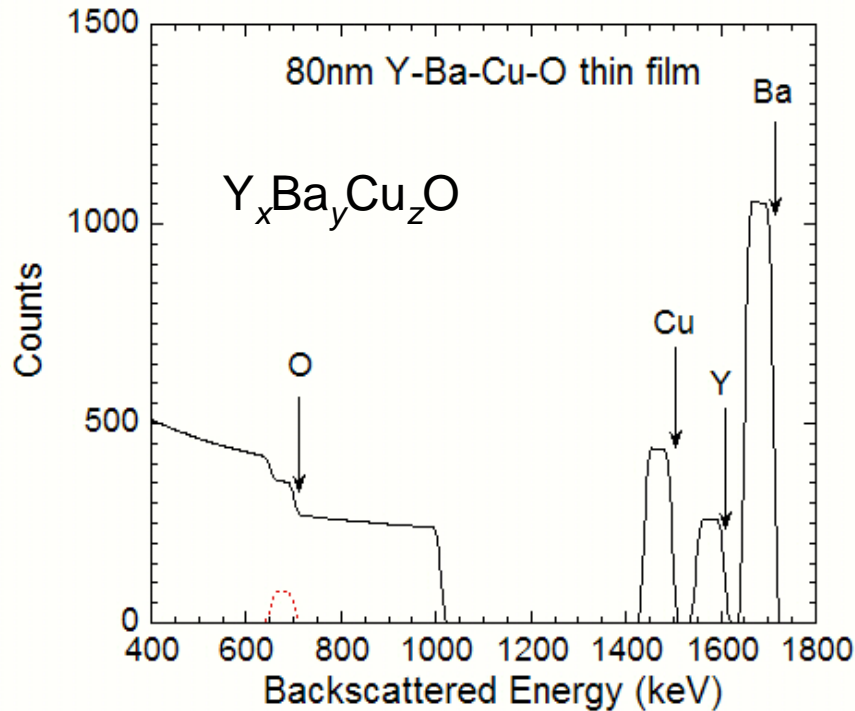
$$A_{As} = \sigma_{As} \cdot \Omega \cdot Q \cdot (Nt)_{As}$$

$$H_{Si} = \sigma_{Si} \cdot \Omega \cdot Q \cdot N \frac{\delta E}{N[\epsilon_o]_{Si}^{Si}}$$

$$\frac{As_{As}}{H_{Si}} = \left( \frac{Z_{As}}{Z_{Si}} \right)^2 \cdot \frac{(Nt)_{As}}{\delta E / [\epsilon_o]_{Si}^{Si}}$$

**Independent of Q and  $\Omega$**

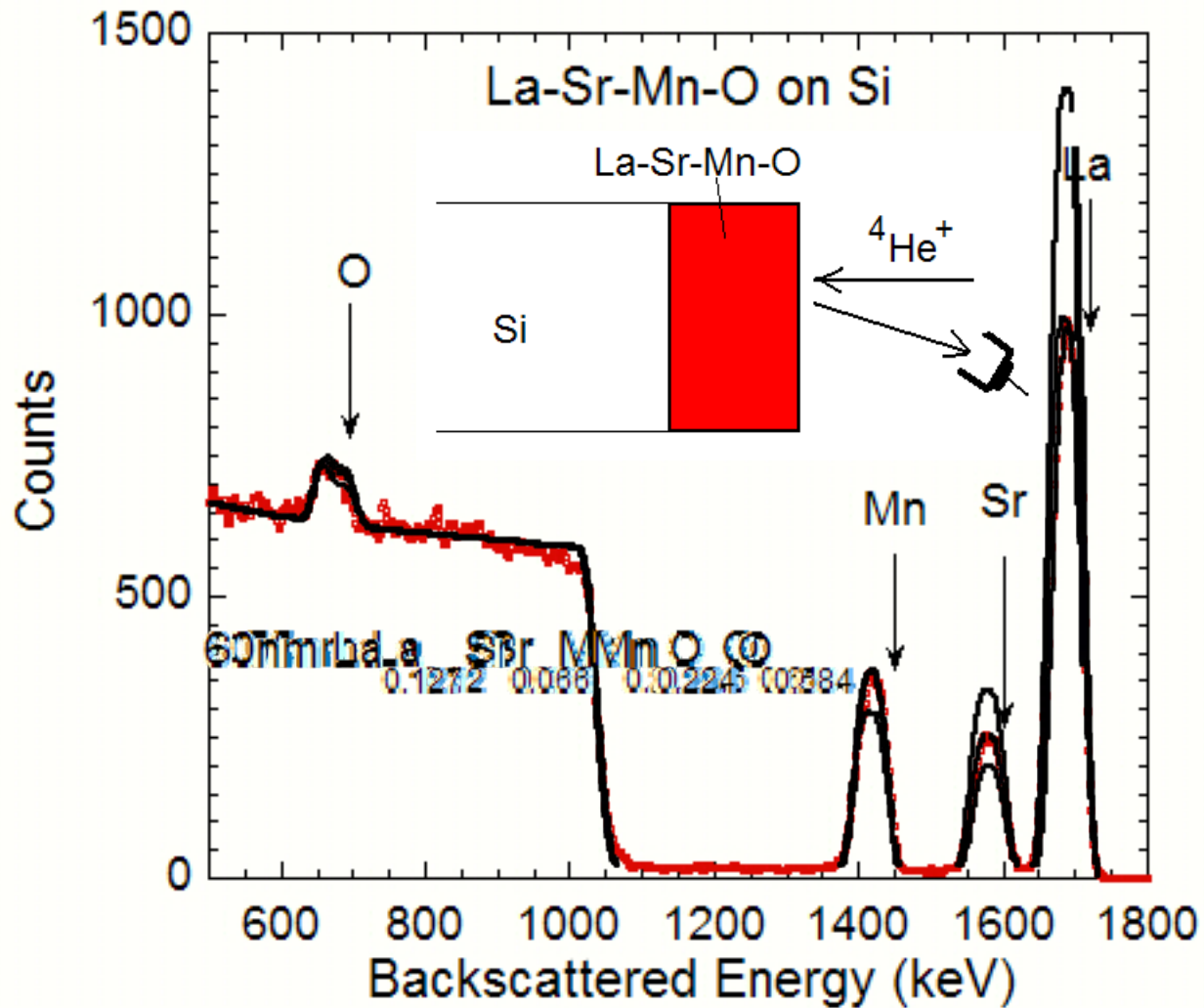
# Thin film analysis



$$\frac{y}{x} = \frac{A_Y}{A_{Ba}} \cdot \left(\frac{Z_{Ba}}{Z_Y}\right)^2$$

$$\begin{aligned} \frac{y}{x} &= \frac{H_Y}{H_{Ba}} \cdot \left(\frac{Z_{Ba}}{Z_Y}\right)^2 \cdot \frac{[\epsilon_o]_Y^{YBCO}}{[\epsilon_o]_{Ba}^{YBCO}} \\ &\approx \frac{H_Y}{H_{Ba}} \cdot \left(\frac{Z_{Ba}}{Z_Y}\right)^2 \end{aligned}$$

# RBS simulation



# Example: silicide formation

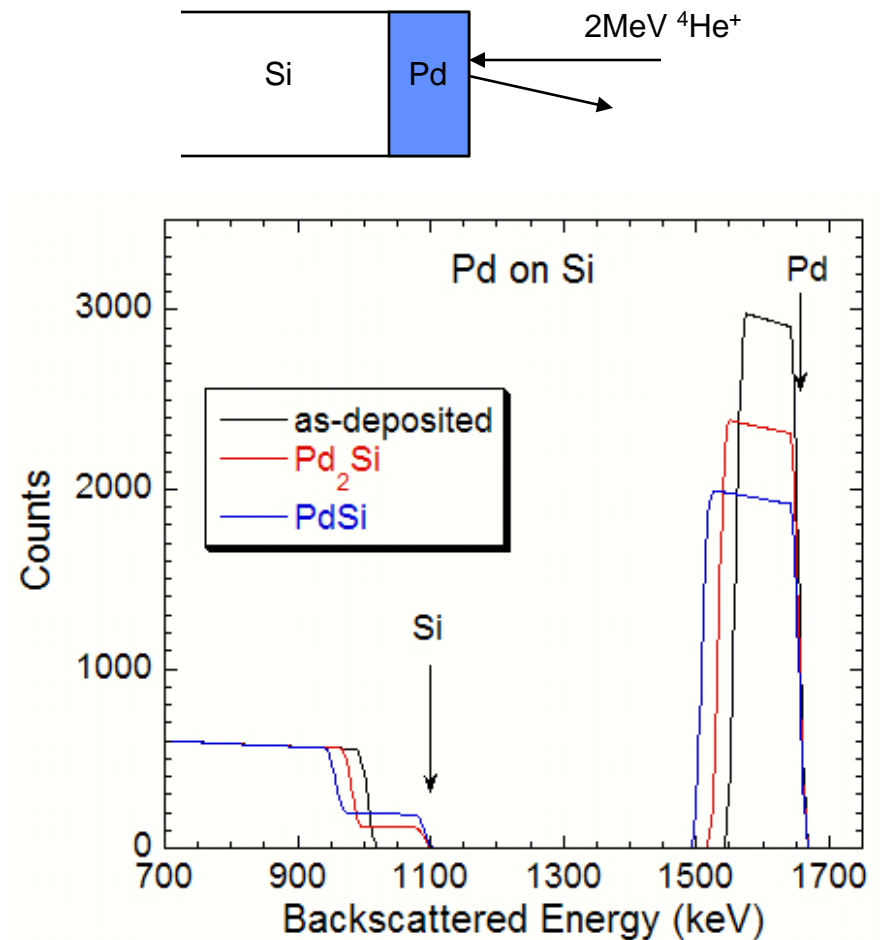
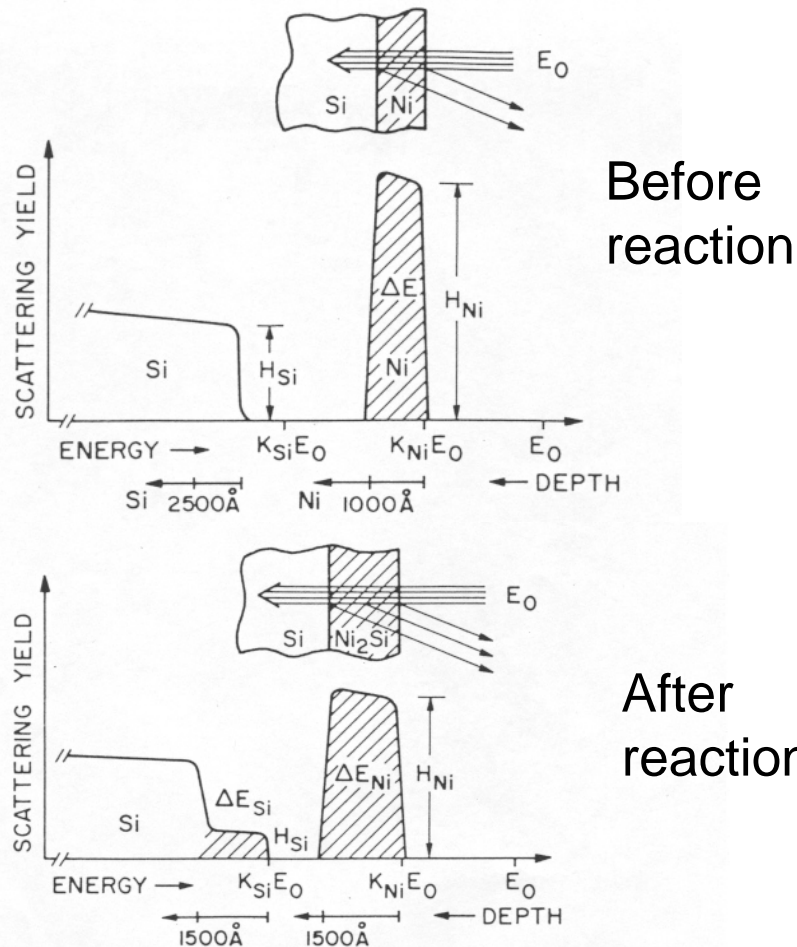
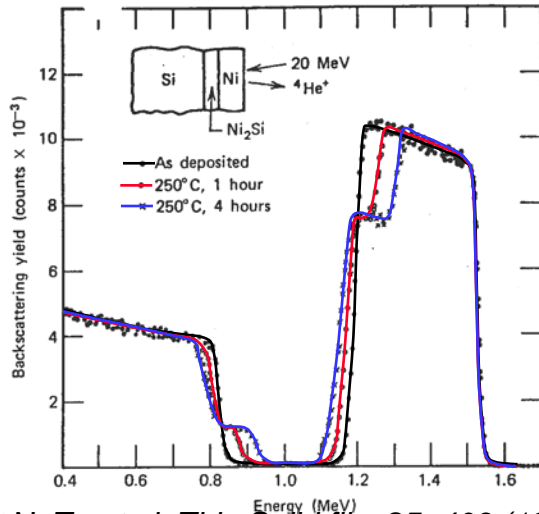
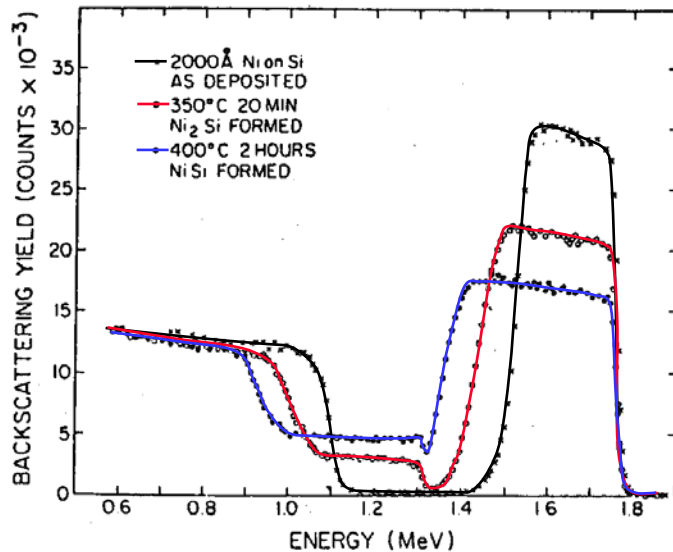


Figure 3.10 Schematic backscattering spectra for MeV  $^4\text{He}$  ions incident on 1000 Å Ni film on Si (top) and after reaction to form  $\text{Ni}_2\text{Si}$  (bottom). Depth scales are indicated below the energy axes.

# RBS application: silicide formation

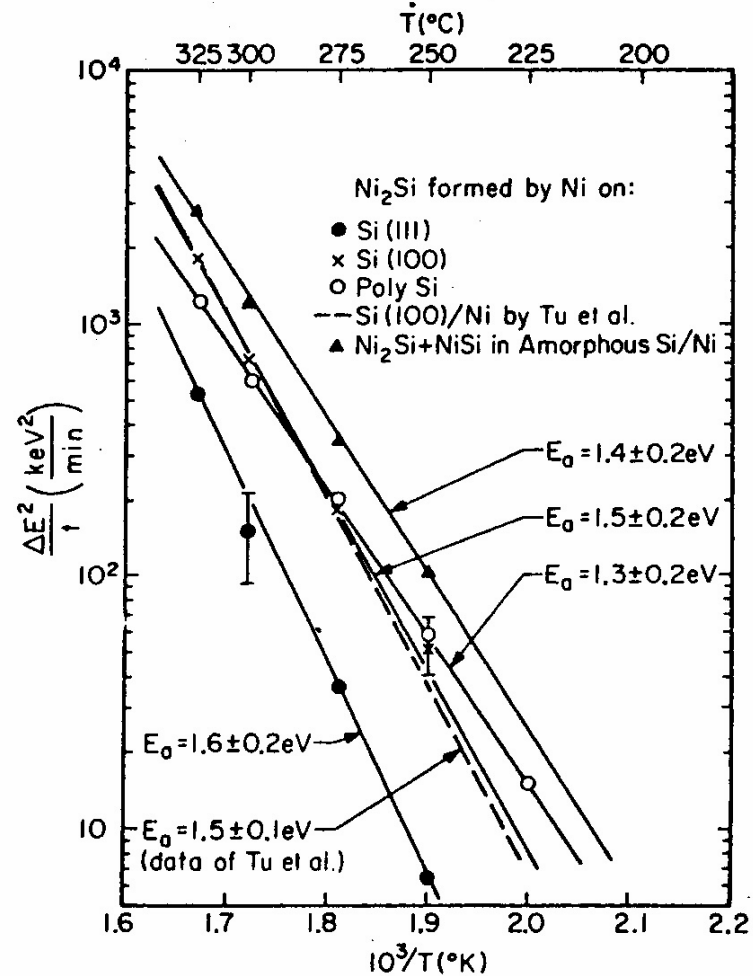


K. N. Tu et al. *Thin Solid film* **25**, 403 (1975).



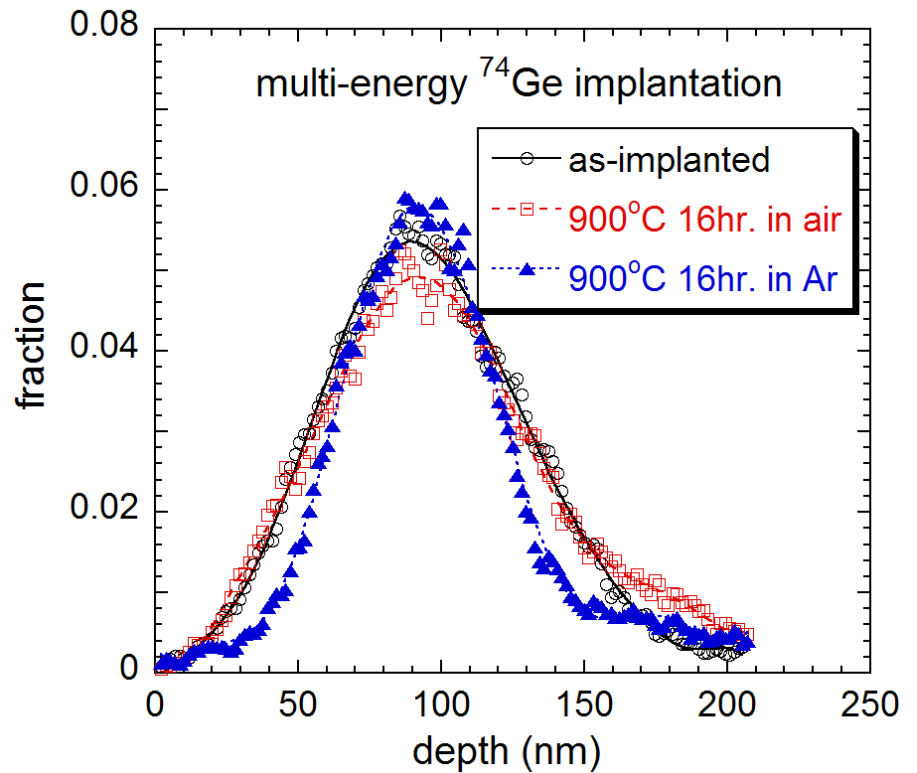
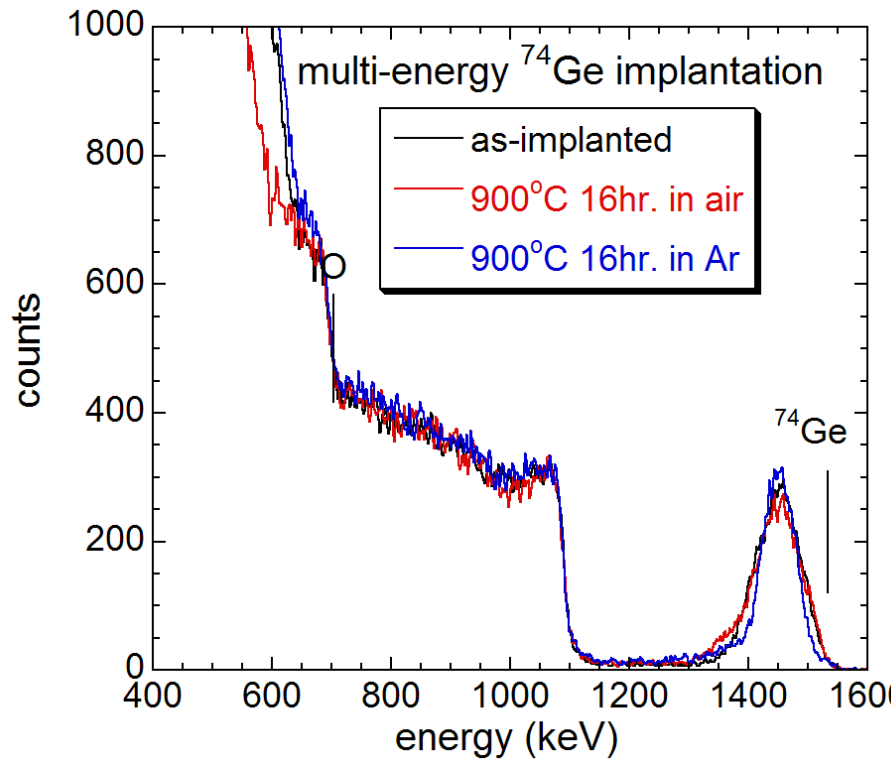
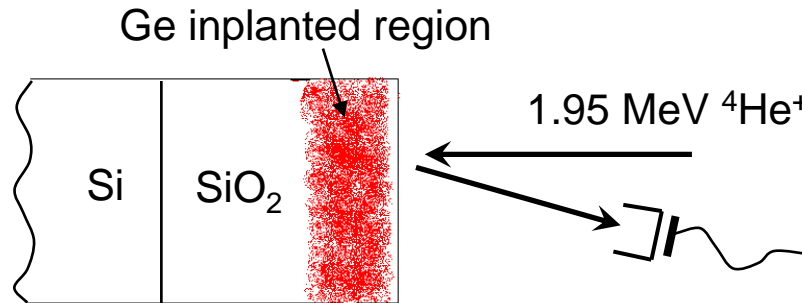
K. N. Tu et al. *Japn. J. Appl. Phys. Suppl.* **2 Pt 1** 669 (1974).

## Reaction kinetics:



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

# RBS Application: impurity profile



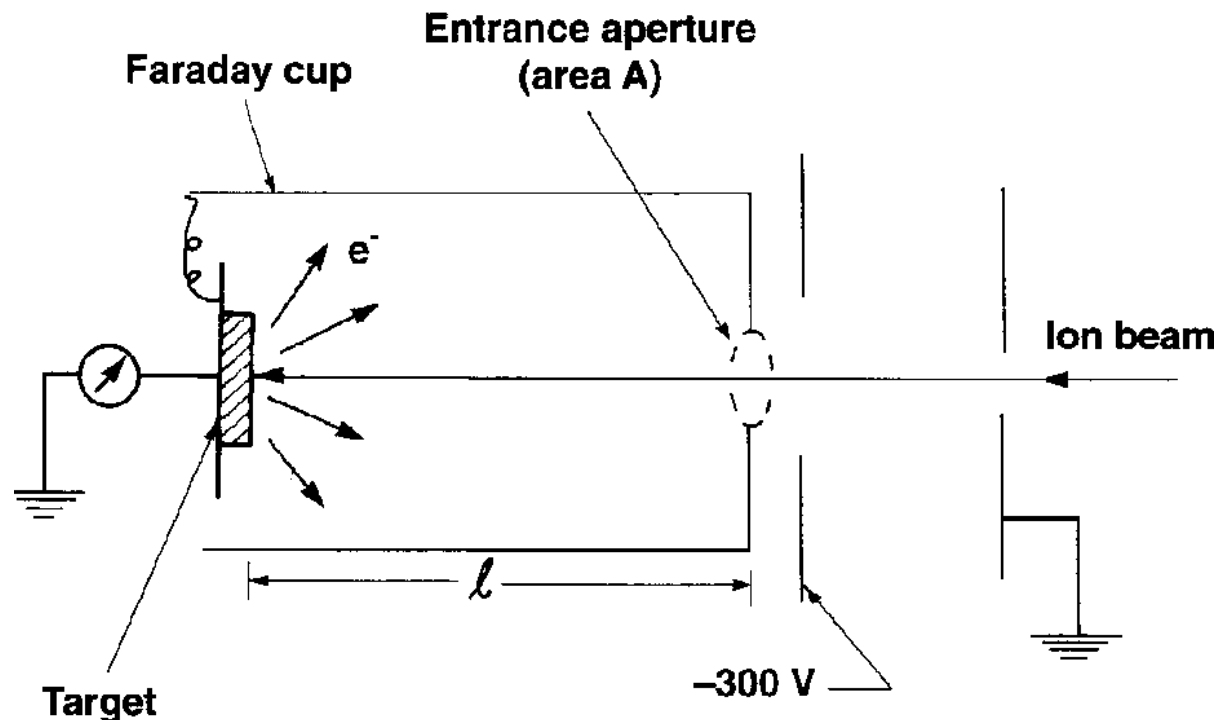
I. Sharp et al., LBNL (2004)

# Pitfalls in IBA



- **Charge Integration**

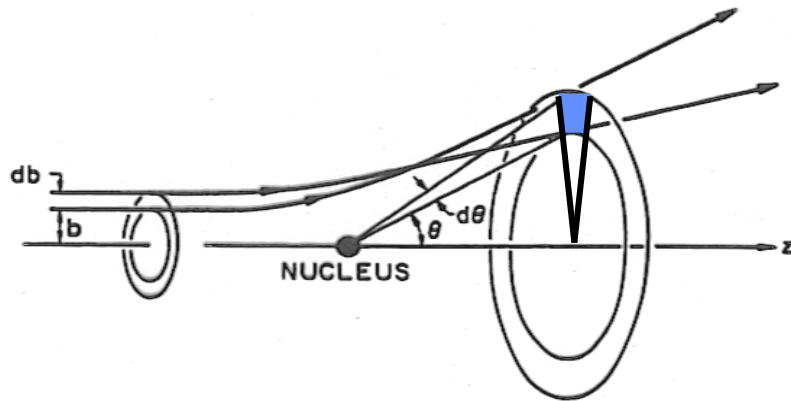
- Accurate charge integration is important for absolute quantitative measurements
  - Good faraday cup design





# Pitfalls in IBA (cont.)

- Deviation from Rutherford cross-section:



$b = \text{distance of closest approach} = Z_1 Z_2 e^2 / E$

$r_n = \text{nuclear radius} = 1.4 Z_2^{1/3} \times 10^{-5}$

$r_K = \text{atomic K-shell radius} = 0.5 / Z_2$

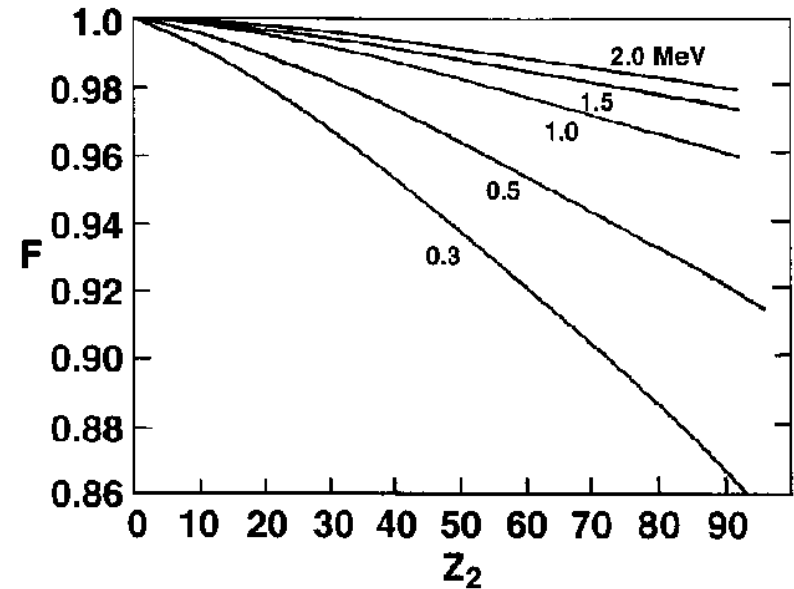


FIG. 12.5. Screening-correction factor,  $F$ , to the Rutherford cross section for  $^4\text{He}$  backscattering as a function of  $Z_2$  and  $E$ . (T. Ermer *et al.* 1979)

In order to minimize electron screening and maintain a point charge approximation:

$$0.5r_K > b > 3r_n$$

**For typical RBS: Rutherford cross section is valid (~4%)**

# Pitfalls in IBA (cont.)



## ■ Insulating samples: charging effect

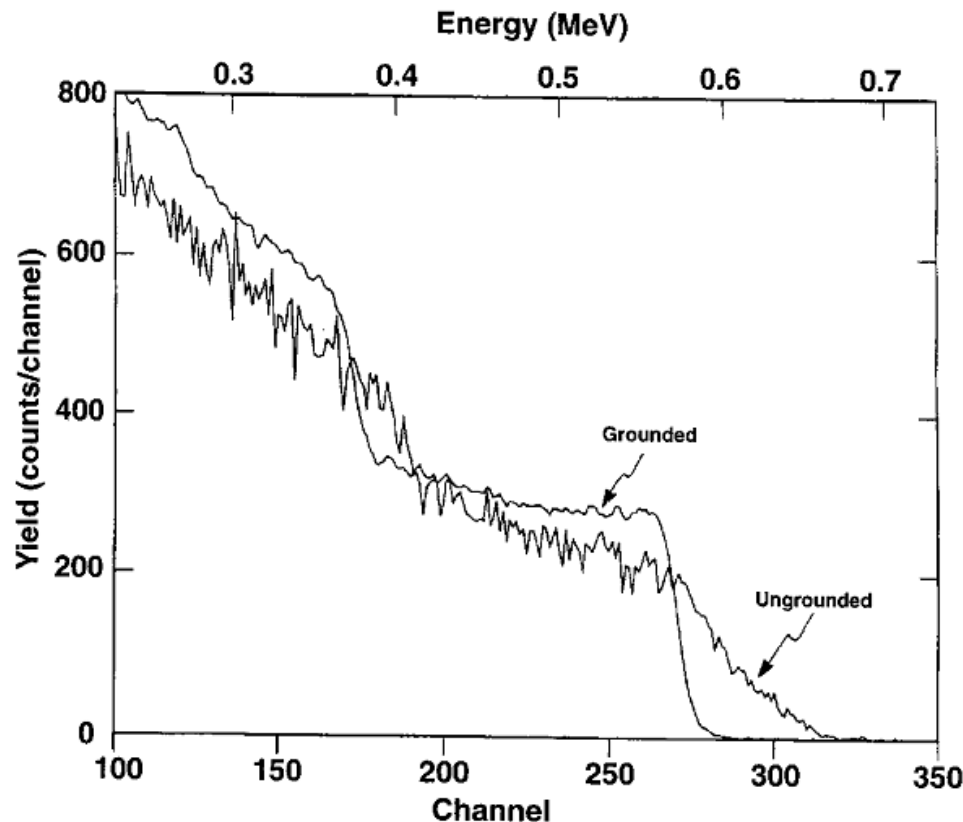


FIG. 12.8. Surface charging effect. Comparison of RBS spectra from a quartz target using 1 MeV  $^4\text{He}$ : a) ungrounded; b) grounded via a thin conductive surface layer of graphite by rubbing a pencil lightly across the surface (Almeida and Macauley-Newcombe, 1991).

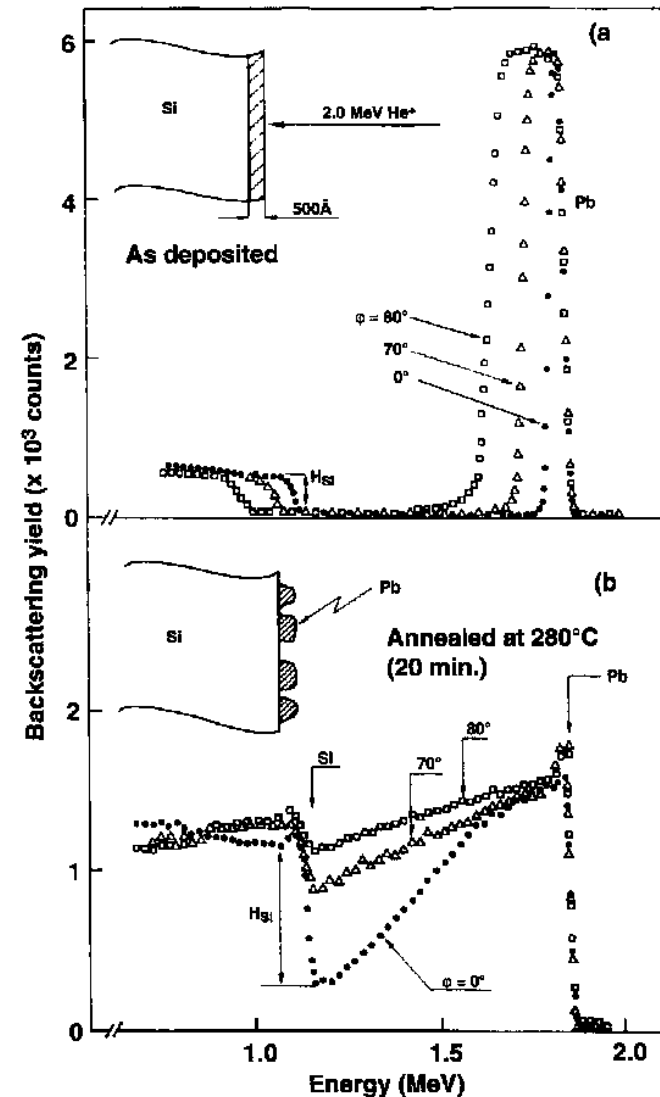
### Severely distort the RBS spectrum

- 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

# Pitfalls in IBA (cont.)



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



Campisano et al., 1978

# Weaknesses of RBS

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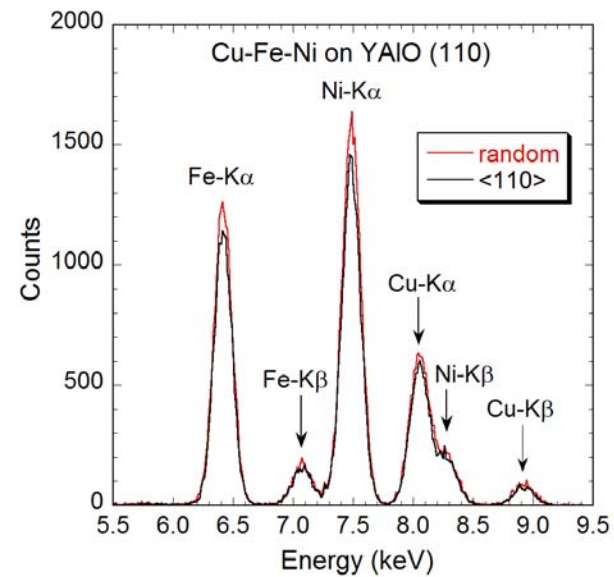
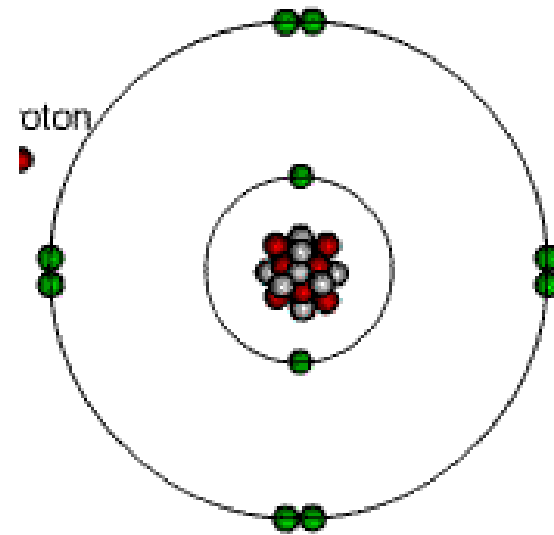
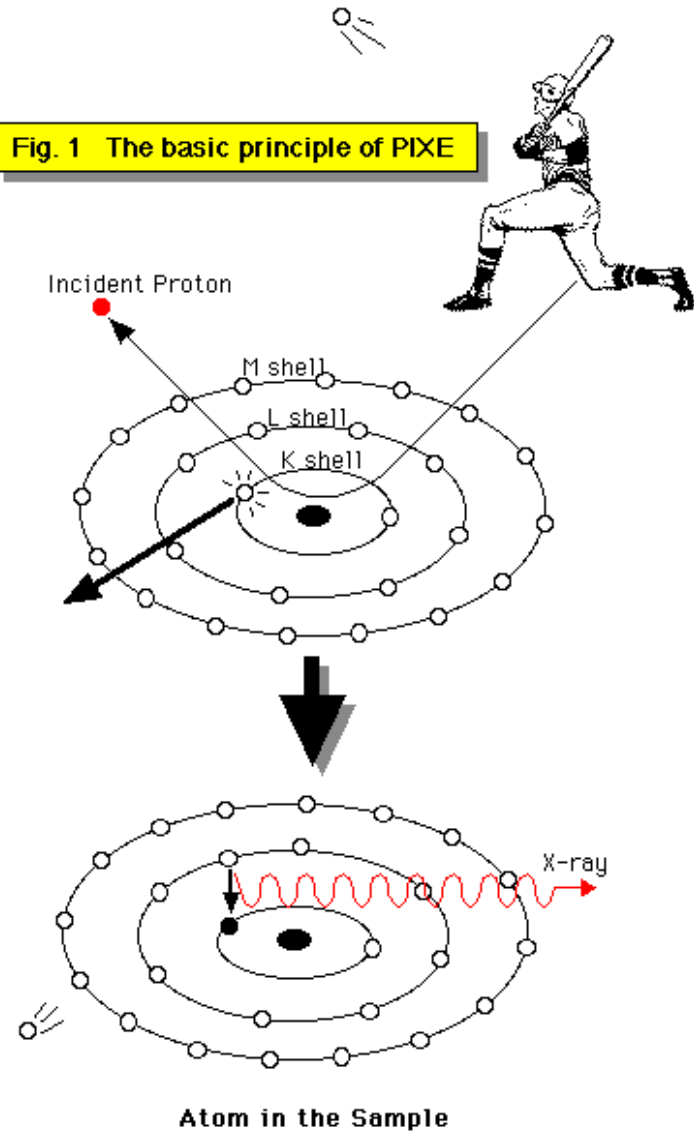
- **Poor lateral resolution (~1mm)**
- **Moderate depth resolution (~100Å)**
- **No microstructural information**
- **No phase identification**
- **Poor mass resolution for target mass heavier than 70amu**
- **Detection of light impurities in a heavy matrix difficult (e.g. C, O, B in Si)**

# Particle Induced X-ray Emission (PIXE)

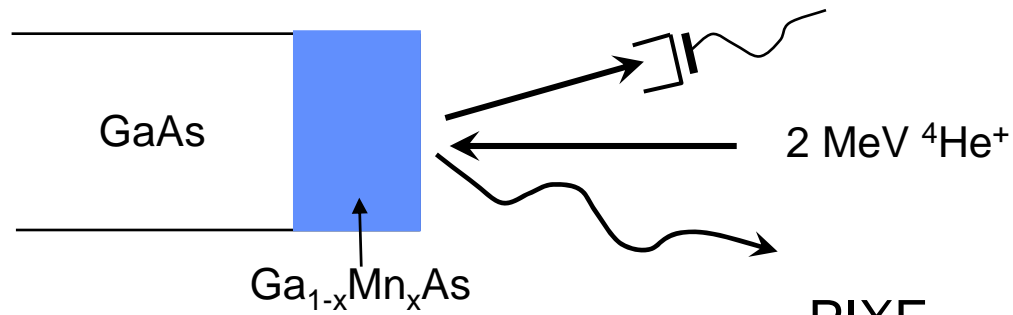
# Particle Induced X-ray Emission (PIXE)



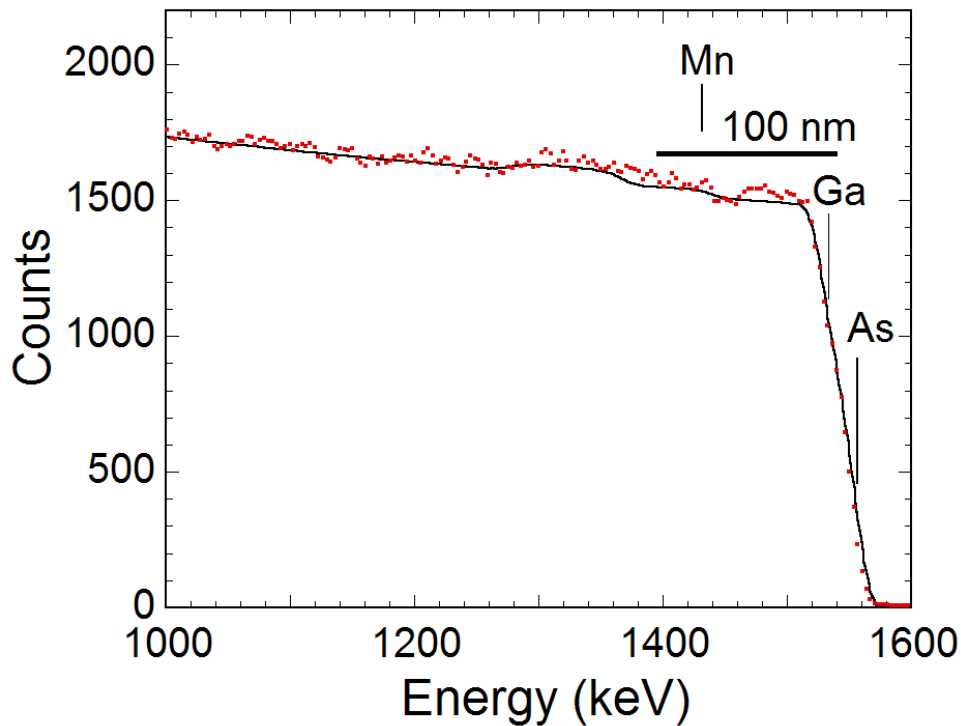
Fig. 1 The basic principle of PIXE



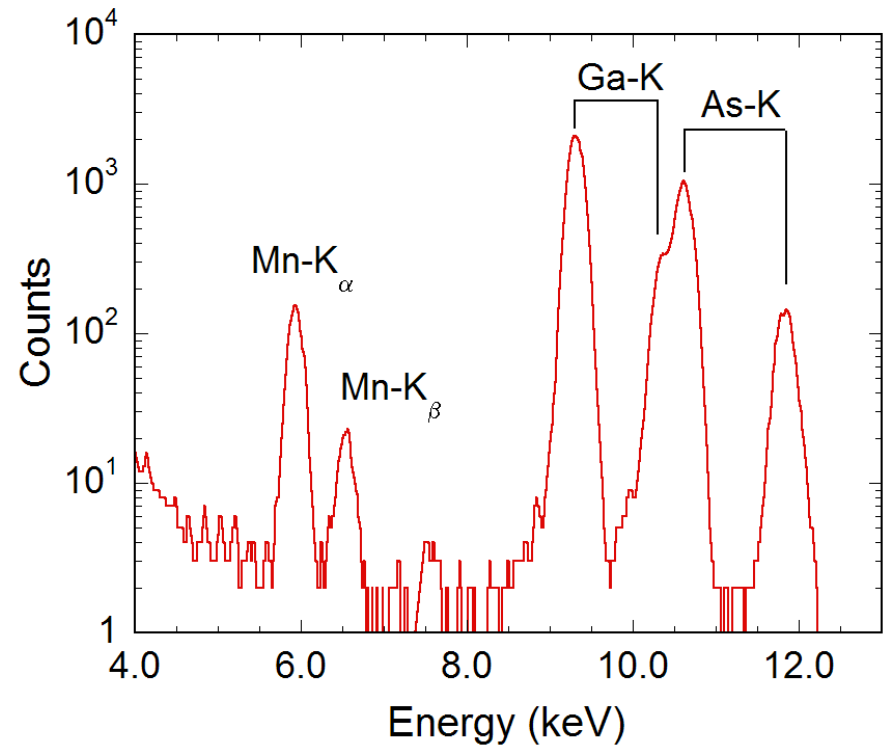
# PIXE: Light impurity in heavy matrix



RBS



PIXE



*K. M. Yu et al. 2002.*

# PIXE Application: Geology, Art, Archeology, Biology



## External Beam

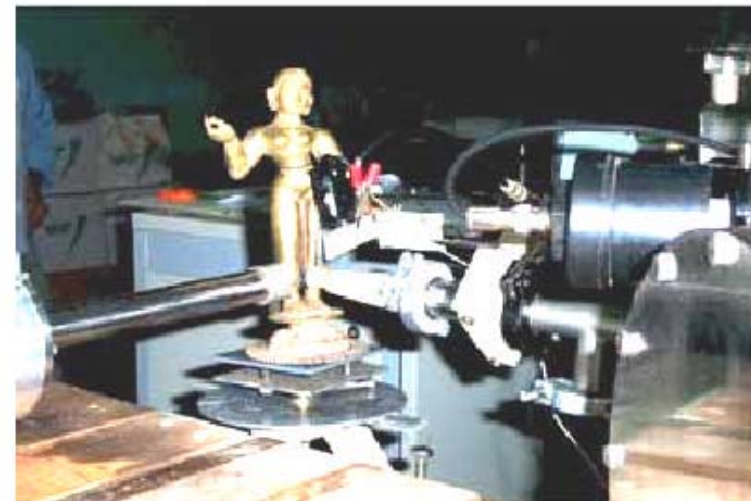
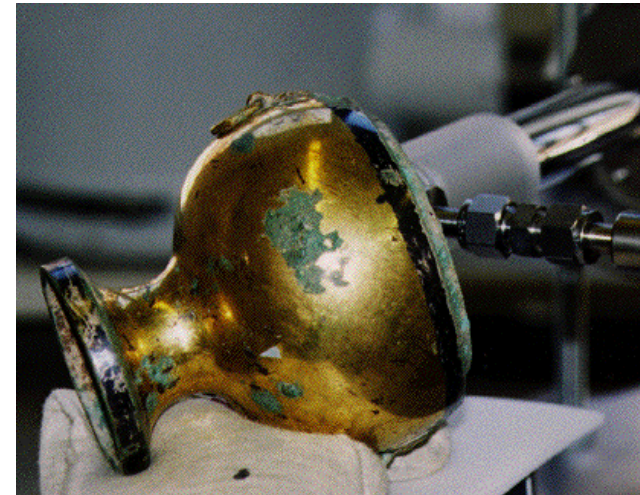
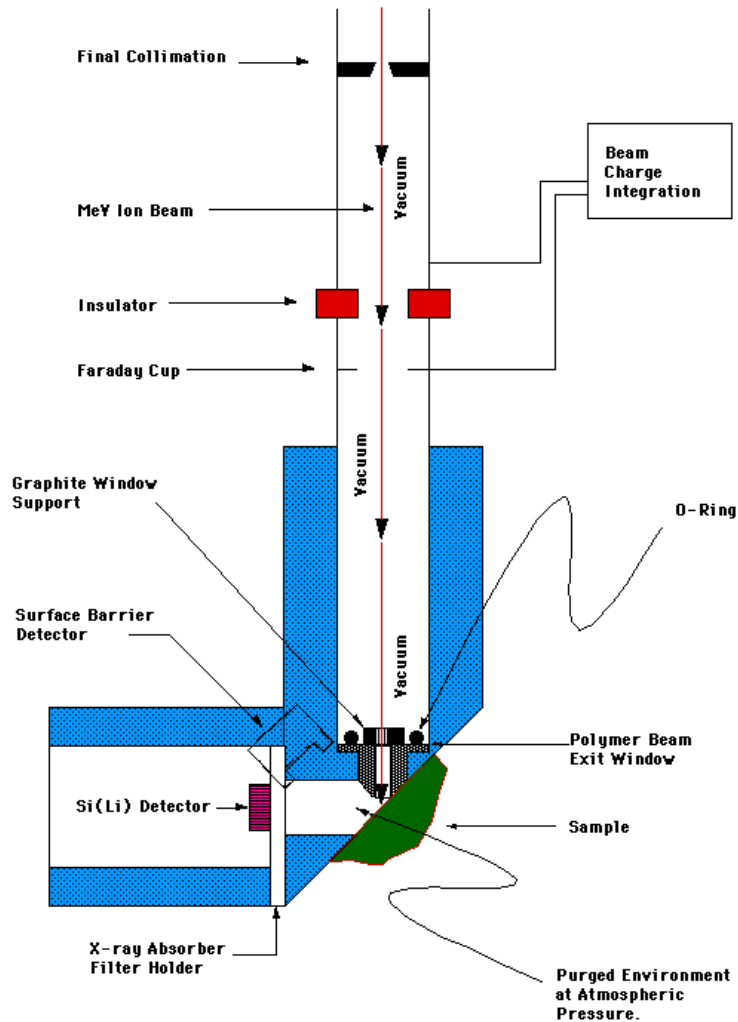


Figure 1. External PIXE set-up at IOP.

HARVARD PIXE SYSTEM

LAWRENCE BERKELEY NATIONAL LABORATORY

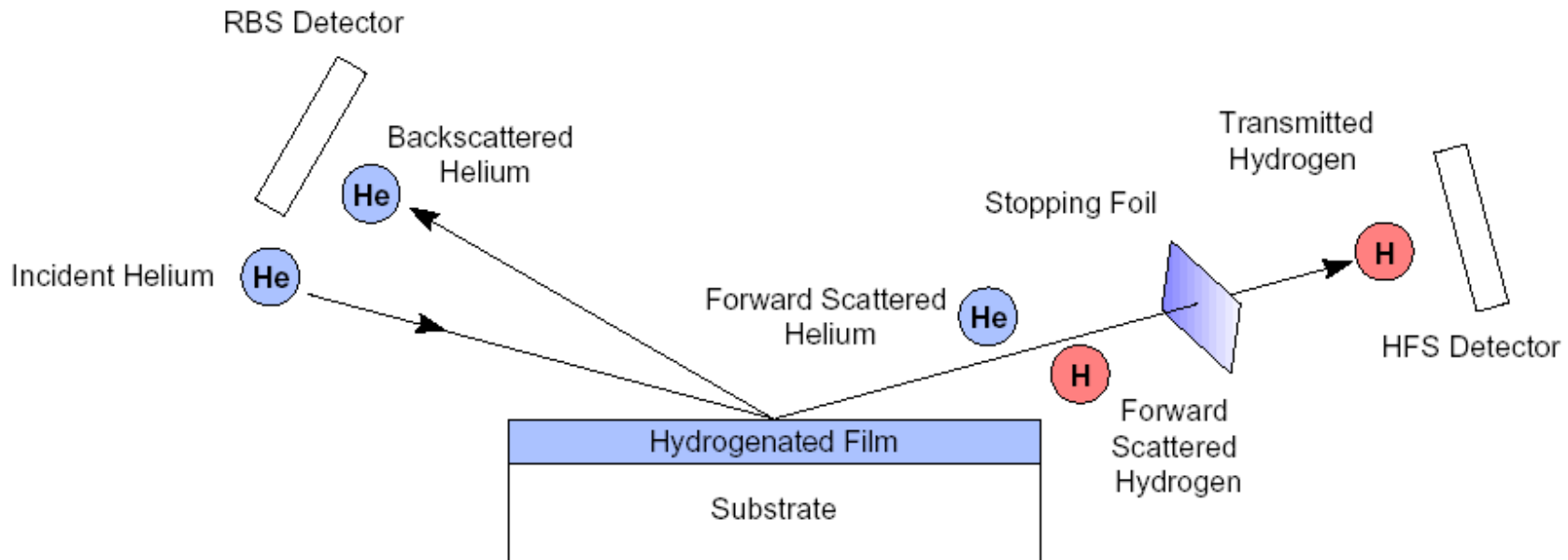


# Hydrogen Forward Scattering (HFS) (Elastic Recoil Detection Analysis ERDA)

# Hydrogen Forward Scattering (HFS)



Generally known as Elastic Recoil Detection (ERD)



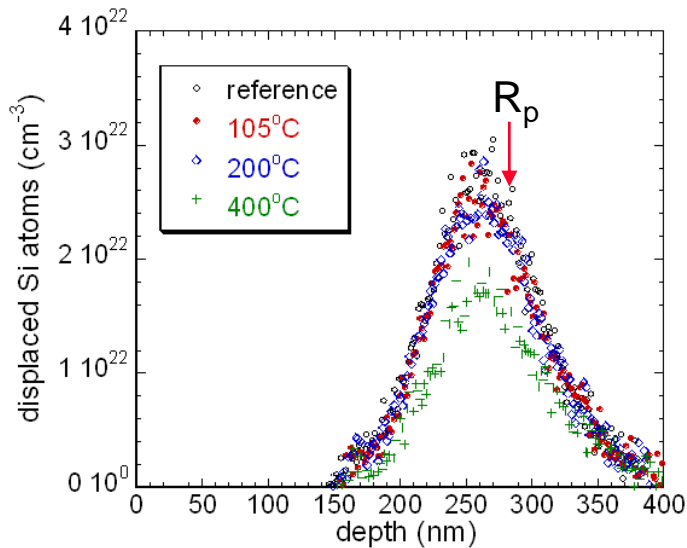
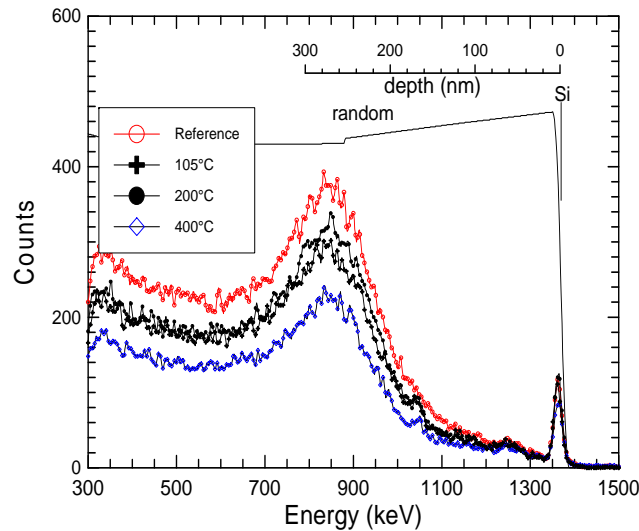
Charles Evans and Assoc., RBS APPLICATION SERIES NO. 3

- Quantitative hydrogen and deuterium profiling
- Good sensitivity ( $\sim 0.01\text{at\%}$  of H)
- Can be performed simultaneously with RBS and PIXE
- Profiling with any light element in solid (using heavy ion beam, ERD)

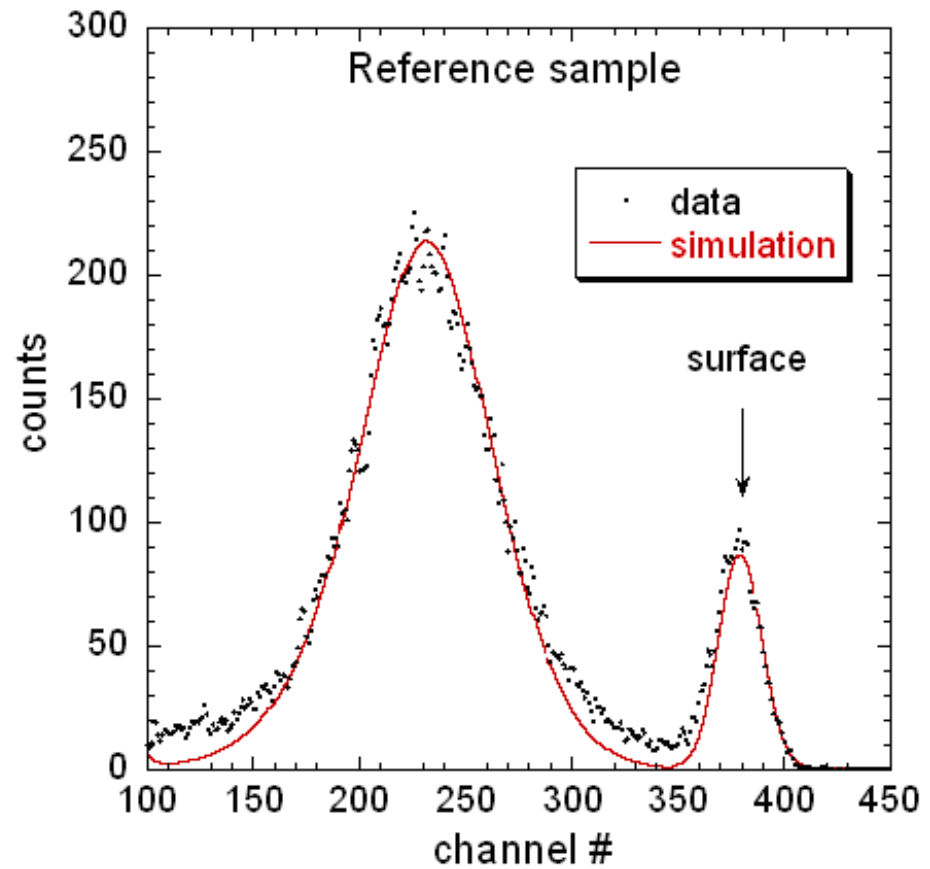
# HFS: H implanted Si



## Channeling-RBS



## HFS

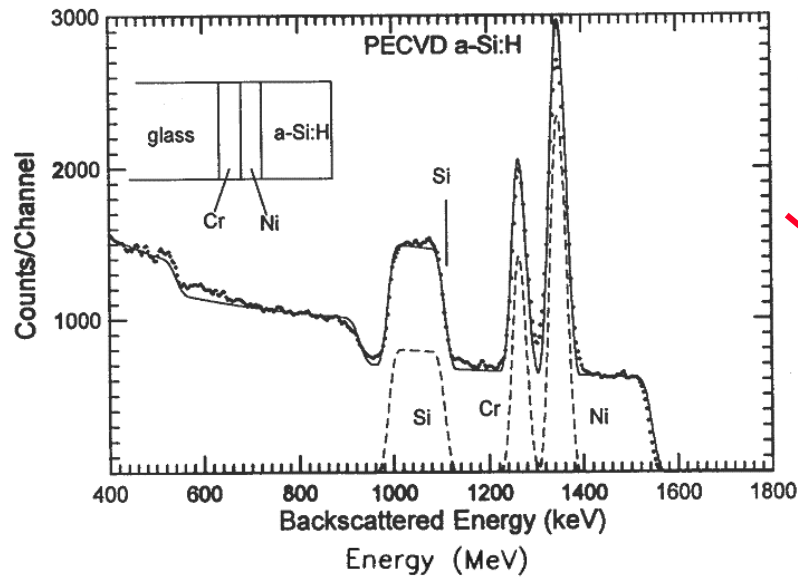


$[Nt]_H = 2.1 \times 10^{17} / \text{cm}^2$   
 $R_p = 280 \text{ nm}$   
 $\Delta R = 140 \text{ nm}$

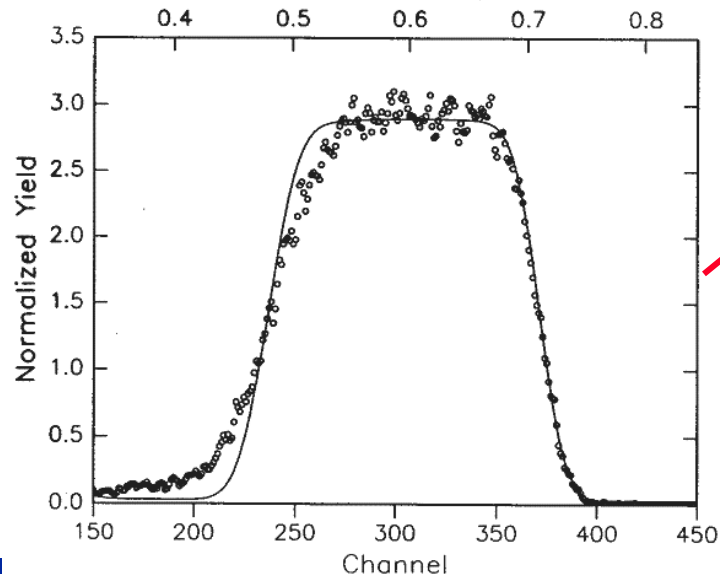
# HFS: a-Si:H film



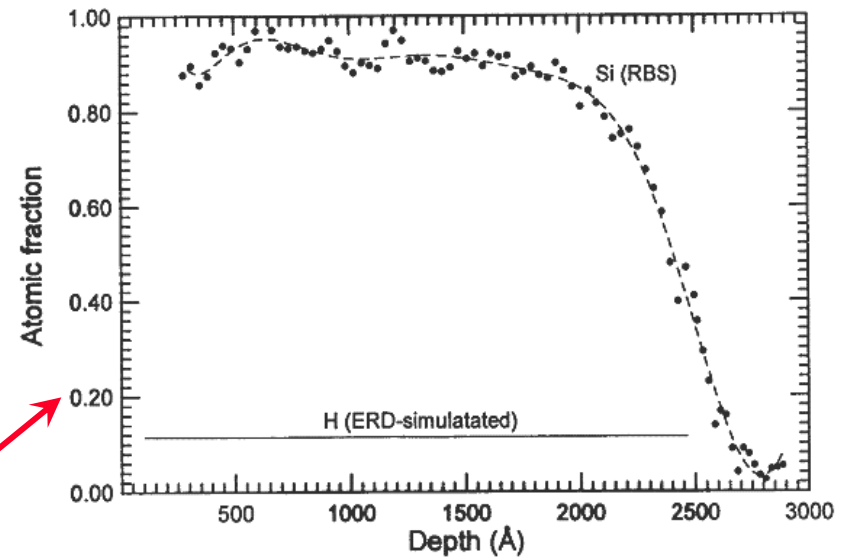
RBS



HFS



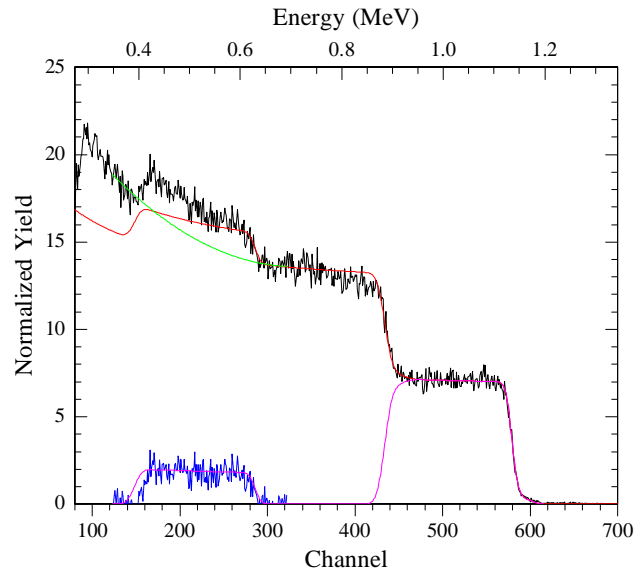
atomic profiles



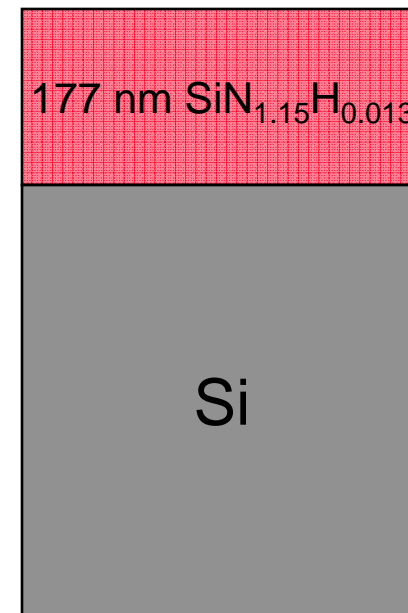
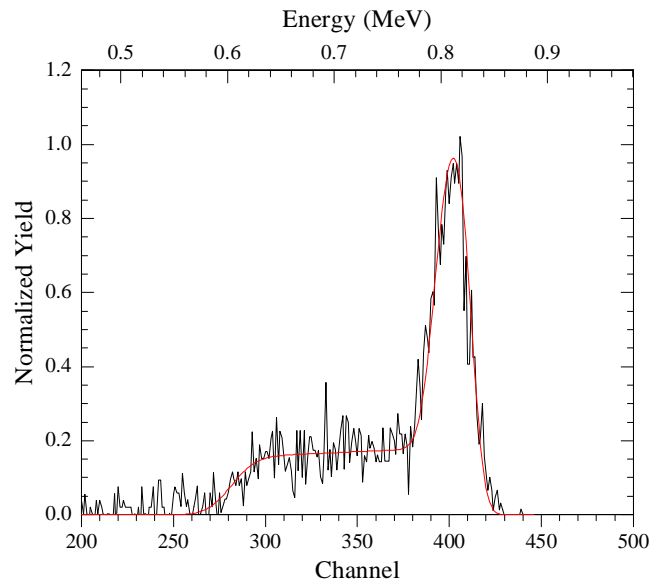
# HFS: a-SiN:H film



RBS



HFS



Courtesy: F. Hellman group, 2008



# Non-Rutherford Scattering and Nuclear Reaction Analysis

# Non-Rutherford Scattering



Non-Rutherford elastic scattering cross sections appear when the ion energy is so high that the ion starts penetrate the Coulomb barrier of the target atom. When the ion penetrates the Coulomb barrier of the target atom, the scattering is from the target atom's nuclear potential and the effect of the nuclear forces for the scattering then become significant.

- For MeV ion beams, this phenomenon can be observed in low  $Z$  projectile/target system where the Coulomb barrier is small.
- The cross-section for such nuclear resonance scattering can be many times greater than the Rutherford values.
- Such non-Rutherford scattering has been used to some extent for the detection of light elements (C, N, O, Si) in heavy matrixes

# Non-Rutherford Cross-Sections



## Summary of Literature on Proton Elastic Scattering Cross Sections Relevant to Low-MeV Proton Backscattering Analysis

Target nucleus	Energy range	Scattering angle	Ref.	Cross section, remarks
$^2\text{H}$	2.0—2.8	165 c	51	$\sigma/\sigma_R \approx 130\text{—}260$ ; data at intervals of 100 keV
$^4\text{He}$	1.7—3.0	168 c	51	$\sigma/\sigma_R \approx 100\text{—}300$ ; data at intervals of 250 keV
$^6\text{Li}$	1.2—3.1	164 l	52	$\sigma/\sigma_R \approx 1.2$ and 15 at 1.2 and 3.0 MeV; a broad peak in the excitation curve (width $\Gamma = 500$ keV, $\sigma/\sigma_R \approx 30$ ) at $E_p = 1.8$ MeV
$^7\text{Li}$	0.5—1.4	160 c	53	$\sigma/\sigma_R \approx 4\text{—}8$ and 45—70 in the smooth regions of the excitation curve at energies $E_p = 1.2\text{—}1.8$ and 2.4—3.0 MeV; broad peak ( $\Gamma \approx 200$ keV $\sigma/\sigma_R \approx 40$ ) at 2.05 MeV; good agreement between data
	0.9—3.7	164 l	51	
	1.3—3.0	167 l	54	
$^9\text{Be}$	0.2—1.7	161 c	55	$\sigma/\sigma_R \approx 1\text{—}2$ and 10—11 in the smooth regions below 0.9 and 1.4—2.0 MeV, respectively; the data of Ref. 55 are significantly lower than those of Ref. 56; a broad peak ( $\Gamma \approx 300$ keV $\sigma/\sigma_R \approx 40^{55}$ or $\sigma/\sigma_R \approx 30^{57}$ ; for the peak, see also Ref. 57
	1.6—3.0	146 c	55	
	0.8—2.6	160 c	56	
$^{10}\text{B}$	1.0—3.0	156 c	58	$\sigma/\sigma_R$ increases smoothly from 2—7 for energies 1.0—2.0 MeV; a broad peak ( $\Gamma \approx 200$ keV $\sigma/\sigma_R \approx 12$ ) at 2.2 MeV; smooth increase of $\sigma/\sigma_R$ from 6 (at 2.4 MeV) to 13 (at 3.0 MeV)



# Non-Rutherford Cross-Sections (cont.)



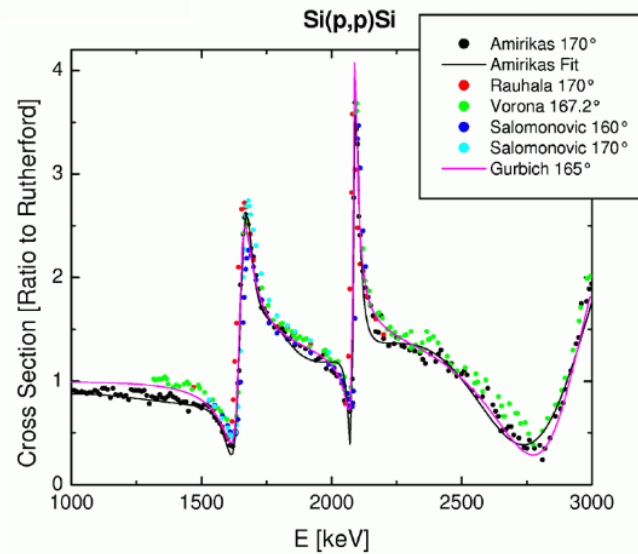
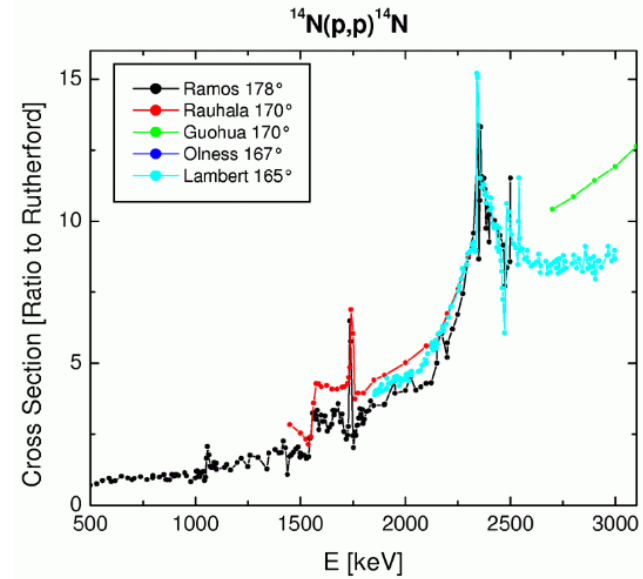
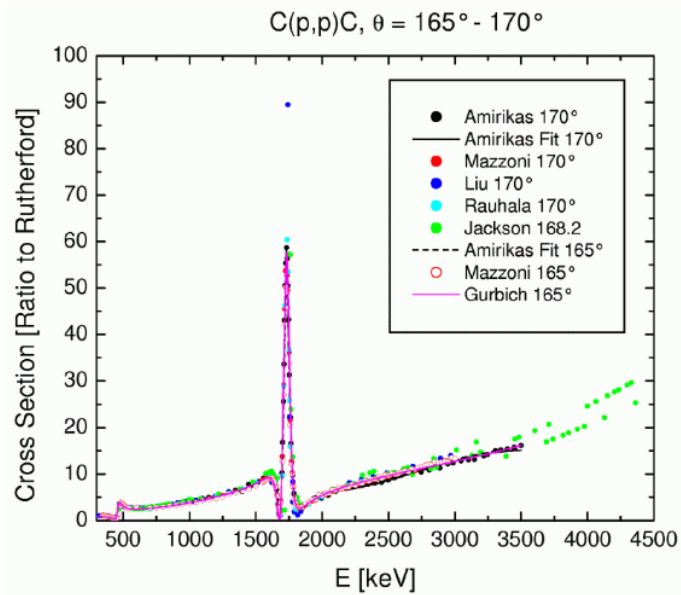
Target nucleus	Energy range	Scattering angle	Ref.	Cross section, remarks	
$^{11}\text{B}$	0.6—2.0	153 c	59	$\sigma/\sigma_R$ decreases from $\approx 2$ to 6 in the smooth region above 0.8 MeV	
$^{12}\text{C}$	0.7—2.5	170 l	46	$\sigma$ <b>non-Rutherford</b> at least above 0.3 MeV; $\sigma/\sigma_R \approx 2.4$ —10 and 2.4—15 in the smooth regions at 0.7—1.6 and 1.8—4.3 MeV, respectively; $\sigma/\sigma_R \approx 10$ at 2.5 MeV; the strong resonance peak ( $\Gamma \approx 40$ keV, $\sigma/\sigma_R \approx 60$ ) at 1.74 MeV has been used for carbon detection; a low $\sigma/\sigma_R \approx 2$ minimum at 1.69 MeV	
	0.3—4.0	164 l	60		
	0.4—4.3	169 c	61		
$^{14}\text{N}$	1.4—2.3	170 l	46	$\sigma$ <b>non-Rutherford</b> at least above 0.6 MeV; $\sigma/\sigma_R \approx 4$ —4.5 and 4—9 in the smooth regions at 1.58—1.73 and 1.85—2.3 MeV; strong narrow isolated resonances at 1.74 MeV ( $\Gamma \approx 5$ keV, $\sigma/\sigma_R \approx 10$ ) and at 3.2 MeV ( $\Gamma \approx 10$ keV, $\sigma/\sigma_R \approx 50$ )	
	0.9—4.0	161 c	62		
	1.0—4.1	168 c	63		
	0.6—1.8	160 c	64		
	1.9—3.0	166 c	65		
$^{16}\text{O}$	0.8—2.5	170 l	45	$\sigma$ <b>non-Rutherford</b> above 0.7 MeV, $\sigma/\sigma_R$ increases smoothly from 1.0 at 0.7 MeV to 5.7 at 2.5 MeV; another smooth region where $\sigma/\sigma_R \approx 6.6$ —10 between 2.7 and 3.4 MeV; resonances at 2.66 and 3.48 MeV; for angular distributions and the 2.66 MeV resonance, see Ref. 69	
	0.8—2.0	172 c	66		
	0.6—4.5	169 l	67		
	0.6—2.0	160 l	68		
$^{19}\text{F}$	1.4—3.8	168 c	77	Another smooth region where $\sigma/\sigma_R$ decreases from $\approx 1.8$ to 0.5 at 2.15—2.8 MeV; natural Si target in Ref. 46	
	1.0—2.0	150 l	78		
	0.8—1.9	165,153 l	42,70		$\sigma/\sigma_R \approx 1.4$ and 2—1.3 in the smooth regions at 1.0—1.3 and 1.5—1.65 MeV; a resonance at 1.45 MeV
	0.5—1.8	160 c	71		
	0.5—2.1	160 c	72		

# Non-Rutherford Cross-Sections (cont.)



Target nucleus	Energy range	Scattering angle	Ref.	Cross section, remarks
<sup>23</sup> Na	0.6—1.5	158 c	73	$\sigma/\sigma_R \approx 0.9$ —1.1 in the smooth region at 1.0—1.4 MeV; <sup>73</sup> the data from Ref. 56 are significantly lower than those of Ref. 73
	0.5—1.0	160 l	56	
<sup>24</sup> Mg	0.7—2.5	170 l	47	$\sigma$ non-Rutherford above 0.8 MeV; $\sigma/\sigma_R \approx 1.05$ —1.1 and 1.3—1.5 in the smooth regions at 0.85—1.4 and 1.66—1.85 MeV; resonances at 0.83, 1.48, 1.62, and above 1.9 MeV; natural Mg used as target in Ref. 47
	0.4—3.9	164 l	74	
<sup>27</sup> Al	1.0—2.4	170 l	48	$\sigma$ non-Rutherford at least above 1.0 MeV; many sharp overlapping resonances, no smooth regions wider than 50 keV above 1.2 MeV; $\sigma/\sigma_R < 3$ up to 2.4 MeV; yield curve without absolute cross section scale in Ref. 75
	1.4—2.3	164 l	75	
<sup>28</sup> Si	1.5—2.2	170 l	46	$\sigma$ non-Rutherford at least above 1.5 MeV; $\sigma/\sigma_R$ decreases smoothly from $\approx 2.2$ to 1 for energies from 1.7 to 2.05 MeV ( $\Gamma \approx 15$ keV), isolated resonance at $E_{He} = 4.79$ MeV; see also Refs. 118 to 120
	2.0—5.0	165 c	76	
<sup>31</sup> P	1.0—2.0	165 l	79	$\sigma$ non-Rutherford at least above 1.0 MeV; $\sigma/\sigma_R \approx 1.05$ and 1.2 in the smooth regions at 1.0—1.2 and 1.3—1.45 MeV. Resonances at 1.25, 1.52, 1.59, 1.72—1.74, and 1.90 MeV

# Non-Rutherford cross sections



# Non-Rutherford scattering: Example 1: SiO<sub>2</sub>

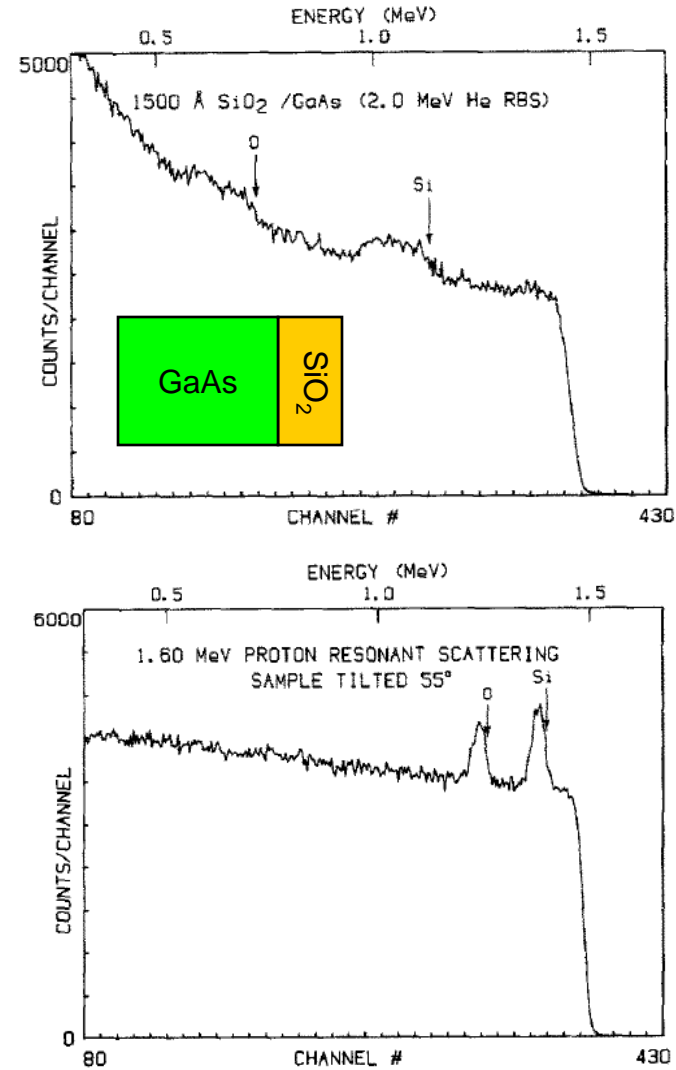
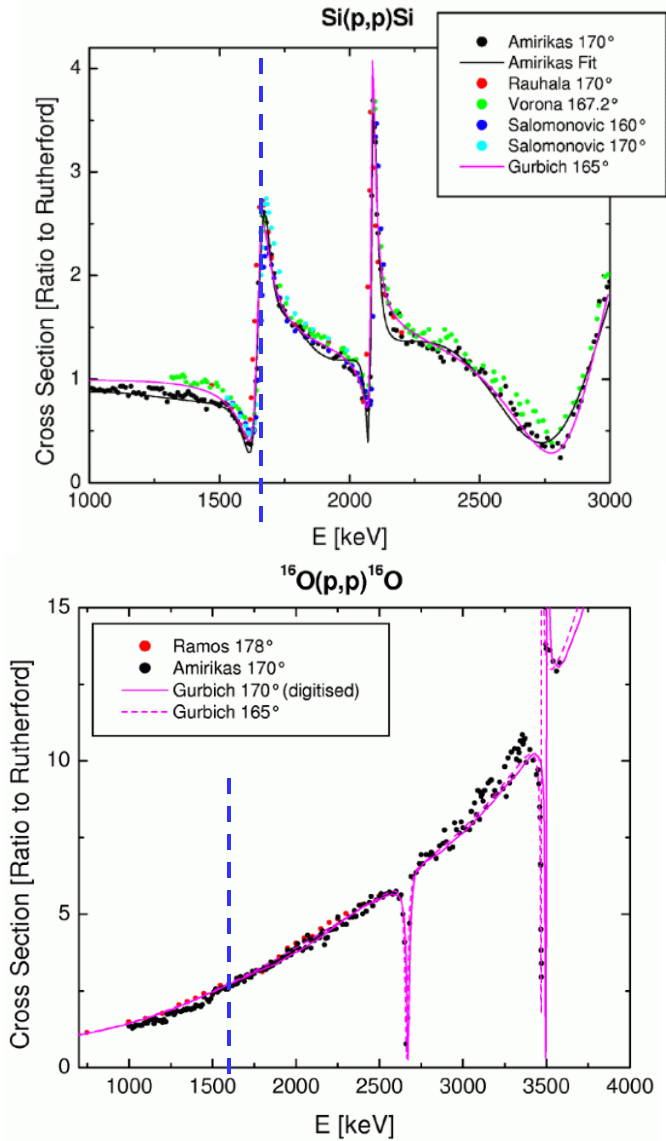


Fig. 4. Backscattering spectra of 2.0 MeV He-RBS (a) and 1.60 MeV PRS (b) of a 1500 Å SiO<sub>2</sub> film on GaAs.

K. M. Yu et al., Nucl. Instrum. Meth. B30 (1988) 551-556

# Non-Rutherford scattering: Example 1: SiC

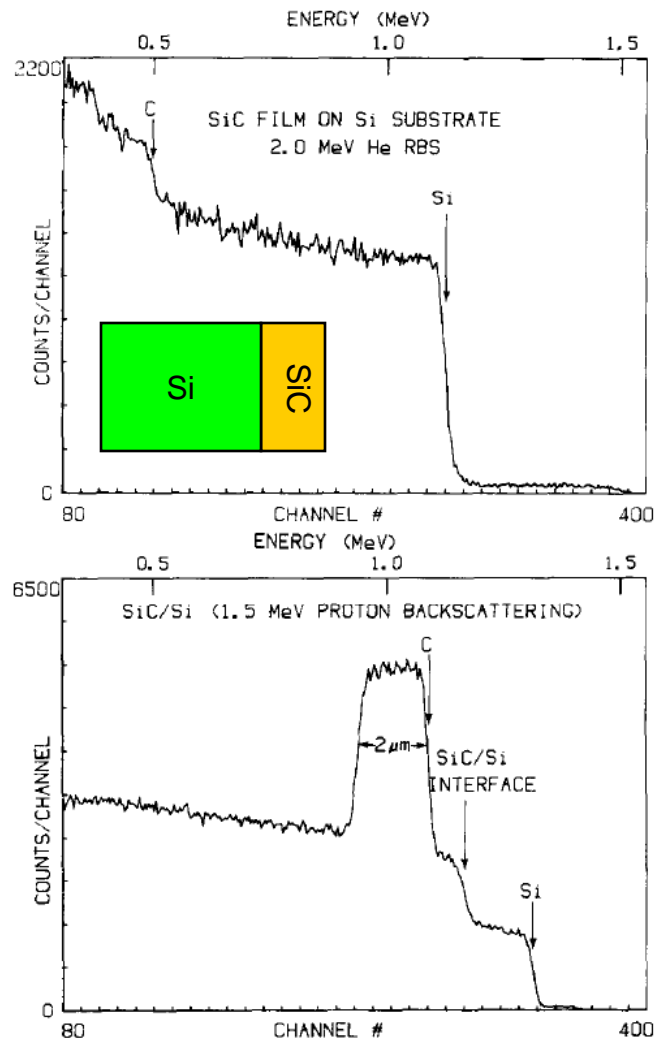
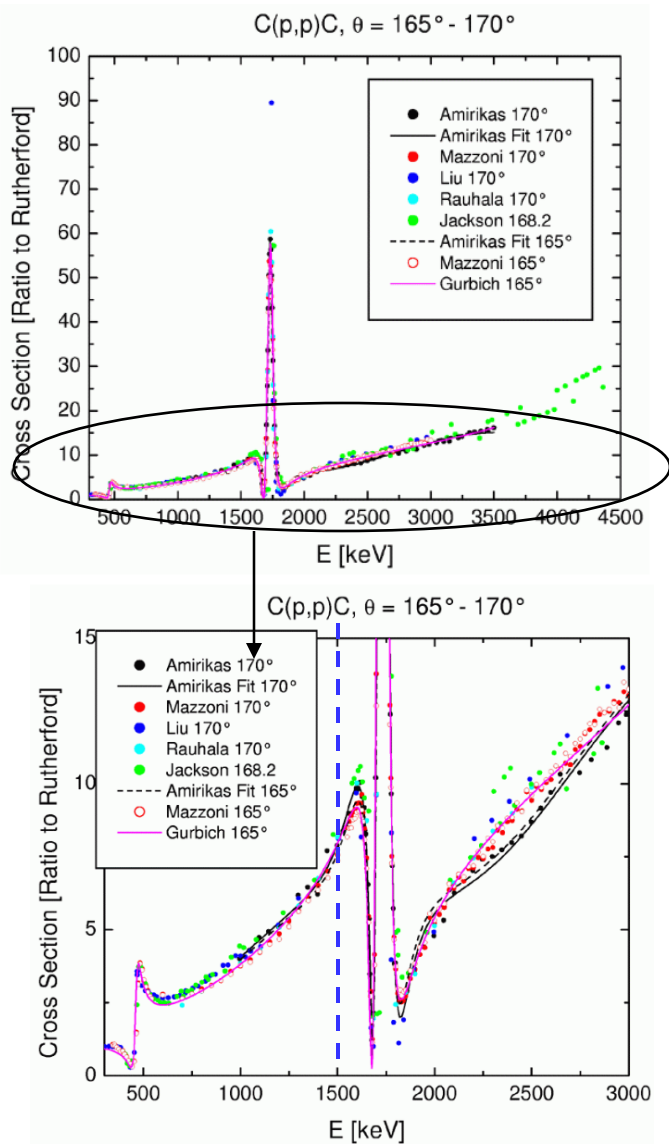
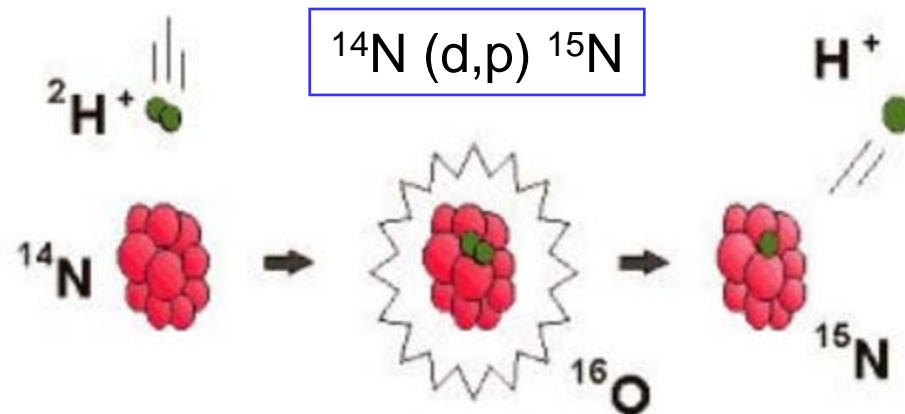


Fig. 5. Backscattering spectra with 2.0 MeV He particles (a) and 1.50 MeV protons (b) from a sample of  $2\mu\text{m}$  SiC film on Si substrate.

K. M. Yu et al., Nucl. Instrum. Meth. B30 (1988) 551-556

# Nuclear Reaction Analysis (NRA)



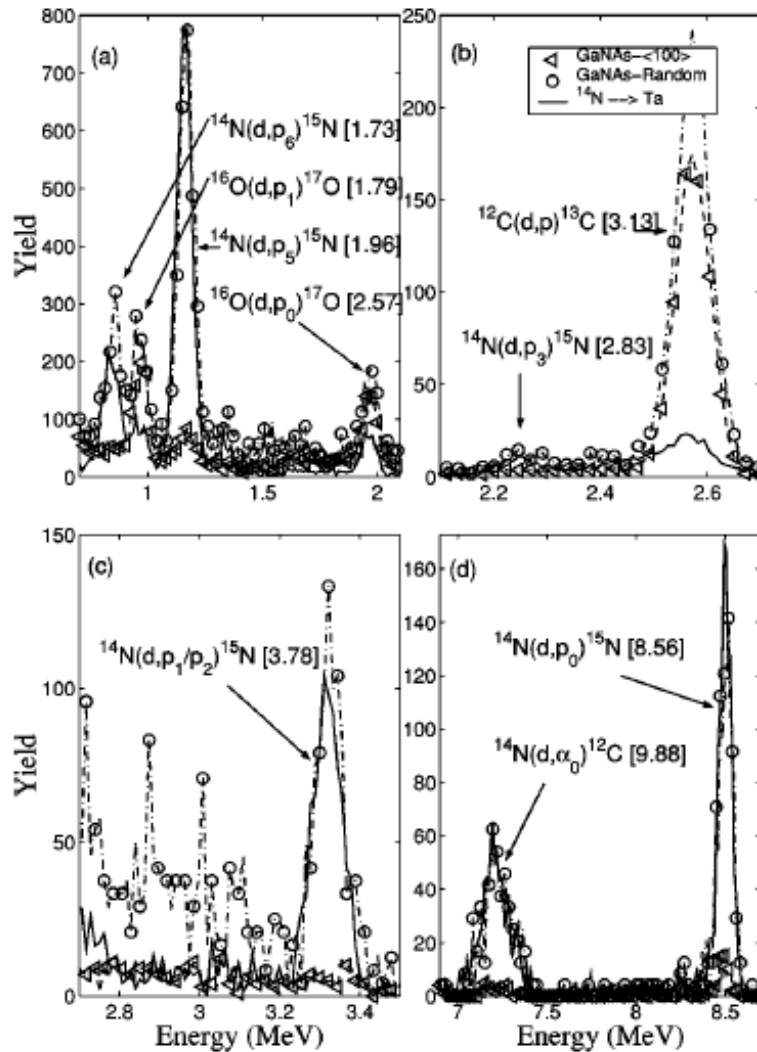
- When the incident beam energy exceeds a certain threshold value, other energetic particles appear in the spectrum.
- The detection of these particles usually provide information which is not obtainable from RBS.
- The NRA technique is very useful as a tool for the detection and profiling of light elements in heavy matrix.
- In many cases such particle-particle NRA can be carried out in a RBS setup with only minor modifications.

# Some useful particle-particle reactions



Nucleus	Reaction	Incident Energy (MeV)	Emitted Energy (MeV)	Approx. cross section (mb/sr)
$^2\text{H}$	$^2\text{H} (d,p) ^3\text{H}$	1.0	2.3	5.2
$^2\text{H}$	$^2\text{H} (3\text{He},p) ^4\text{He}$	0.7	13.0	61
$^6\text{Li}$	$^6\text{Li} (d,\alpha) ^4\text{He}$	0.7	9.7	35
$^7\text{Li}$	$^7\text{Li} (p,\alpha) ^4\text{He}$	1.5	7.7	9
$^{11}\text{B}$	$^{11}\text{B} (p,\alpha) ^8\text{Be}$	0.65	5.57 ( $\alpha_0$ )	0.7
		0.65	3.70 ( $\alpha_1$ )	550
$^{12}\text{C}$	$^{12}\text{C} (d,p) ^{13}\text{C}$	1.2	3.1	35
$^{15}\text{N}$	$^{15}\text{N} (p,\alpha) ^{12}\text{C}$	0.8	3.9	15
$^{18}\text{O}$	$^{18}\text{O} (p,\alpha) ^{15}\text{N}$	0.73	3.4	15
$^{19}\text{F}$	$^{19}\text{F} (p,\alpha) ^{16}\text{O}$	1.25	6.9	0.5
$^{23}\text{Na}$	$^{23}\text{Na} (p,\alpha) ^{20}\text{Ne}$	0.592	2.238	4
$^{31}\text{P}$	$^{31}\text{P} (p,\alpha) ^{28}\text{Si}$	1.514	2.734	16

# NRA Example: GaNAs dilute nitride



1.3 MeV deuterium ions  
 $^{14}\text{N}(d,p)^{15}\text{N}$  and  $^{14}\text{N}(d,\alpha)^{12}\text{C}$  reactions

GaNAs film with ~4% N

FIG. 1. Experimental NRA yields from GaNAs ( $\langle 100 \rangle$  and random direction) and  $^{14}\text{N}$  implanted Ta. Triangles and rings denote  $\langle 100 \rangle$  and random yields, respectively. The values in the square brackets are the initial particle energies in mega-electron-volts before the mylar foil in front of the detector. Figures (a), (b), (c), and (d) corresponds to the reaction yield in different regions of particle energy.

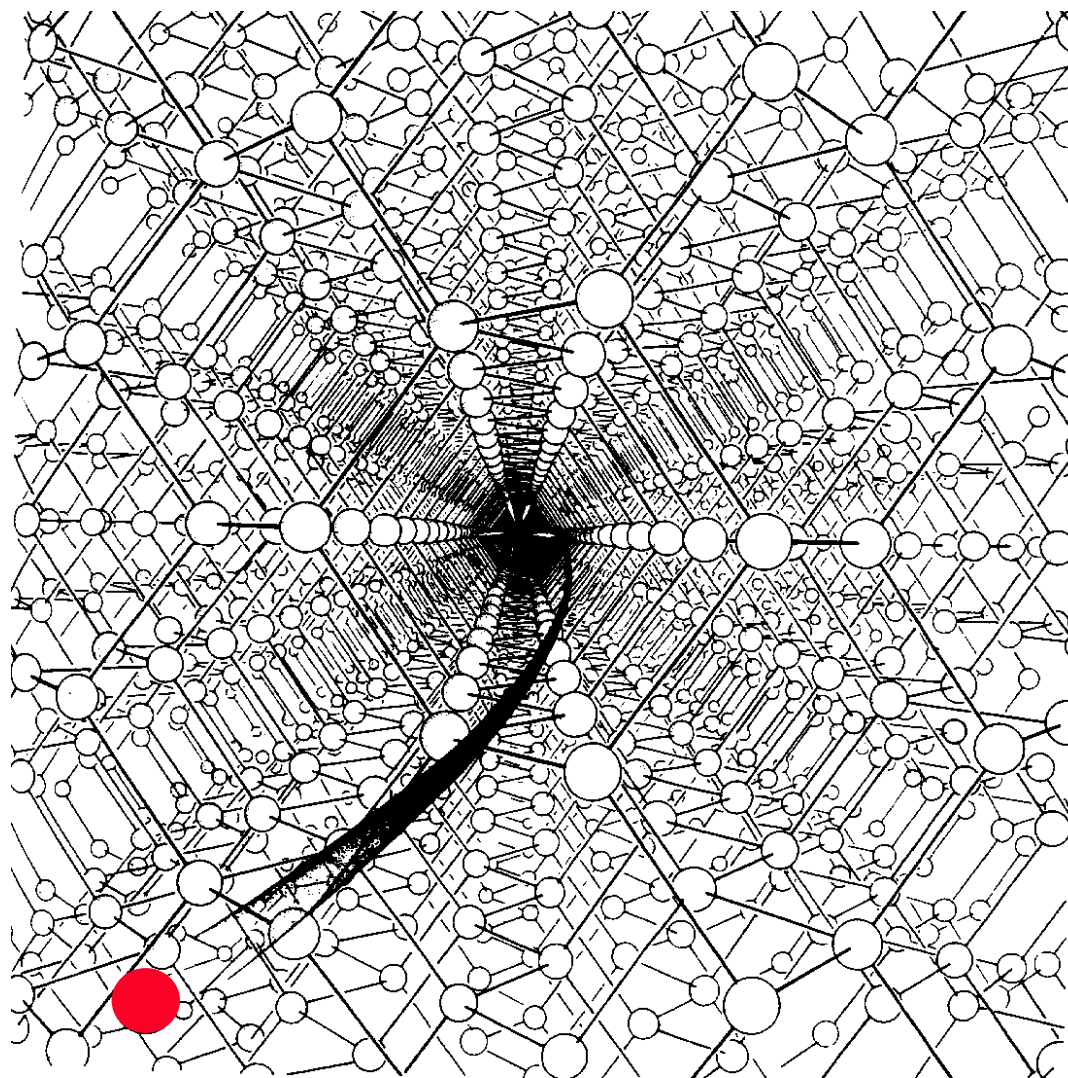
T. Ahlgren et al., Appl. Phys. Lett. **80**, 2314 (2002).



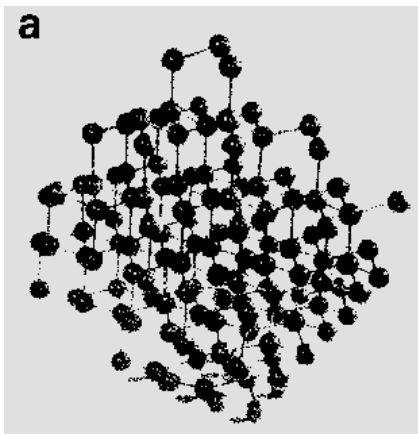
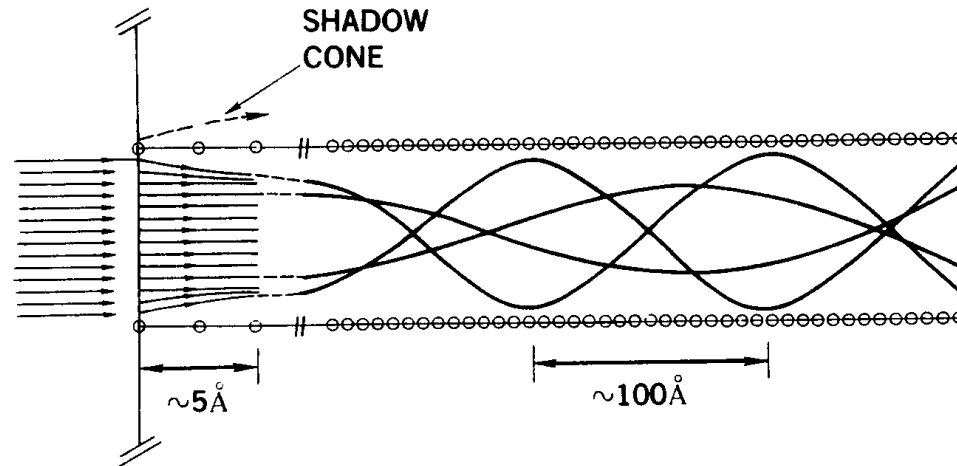


# Ion Channeling

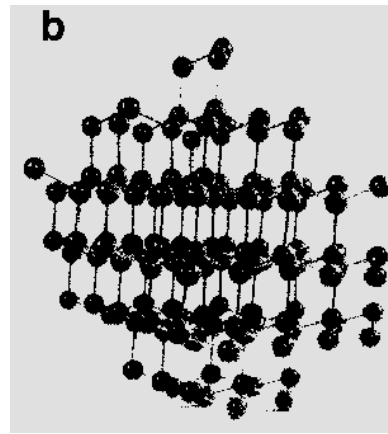
# Ion channeling



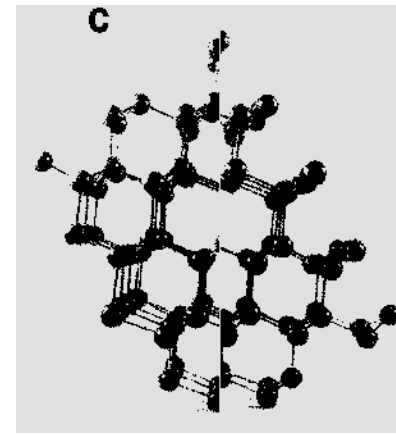
# Ion Channeling



random

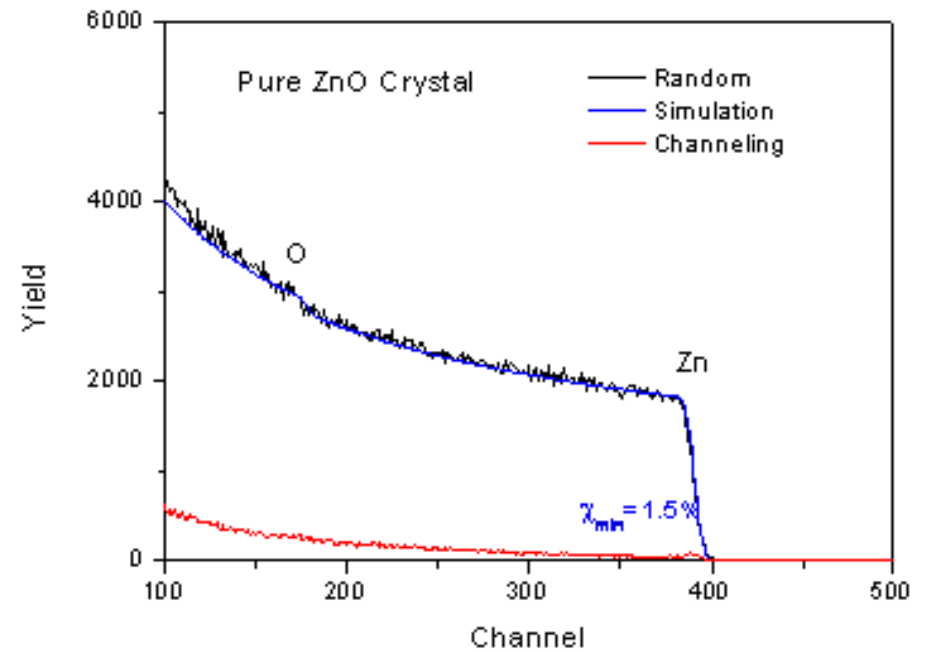
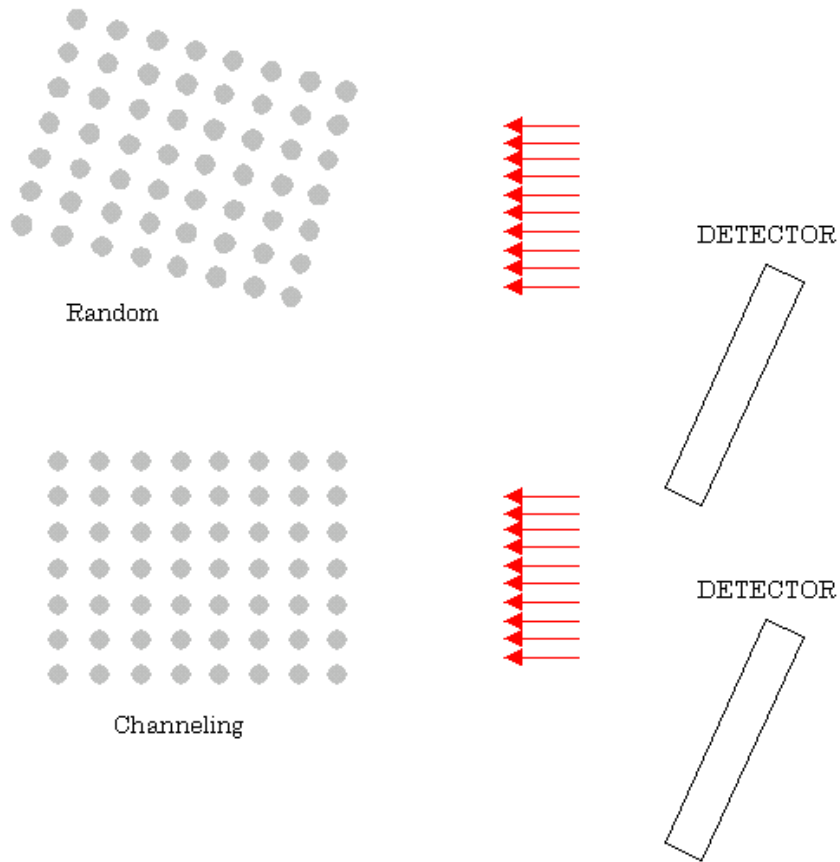


Planar channel



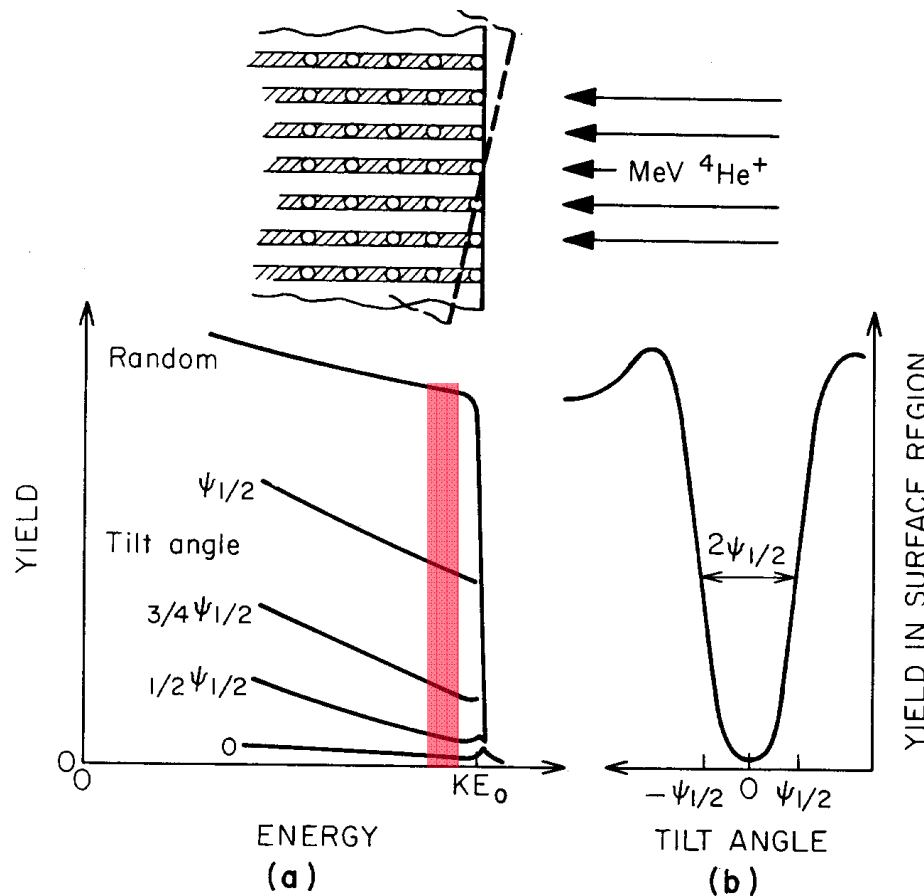
Axial channel

# Ion Channeling



Kobelco Steel Group

# Ion Channeling: minimum yield and critical angle



Two important parameters to characterize channeling results:

1. Minimum yield:

$$\chi_{\min} = \frac{Y_{\text{channeled}}}{Y_{\text{random}}} \sim 0.02-0.06$$

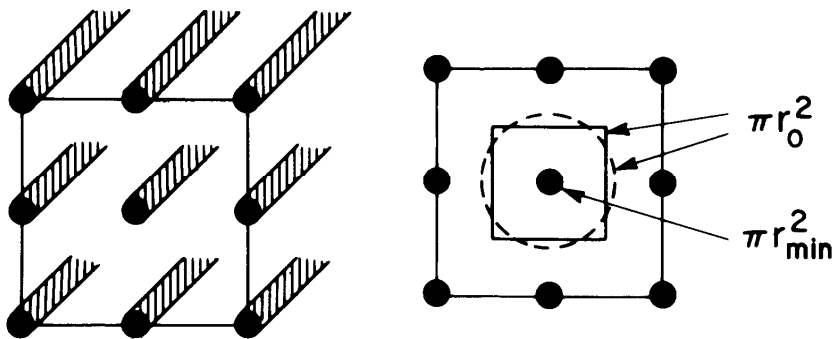
2. Critical half-angle,  $\psi_{1/2}$

indicates presence of defects responsible for beam dechanneling

# Ion Channeling: minimum yield and critical angle



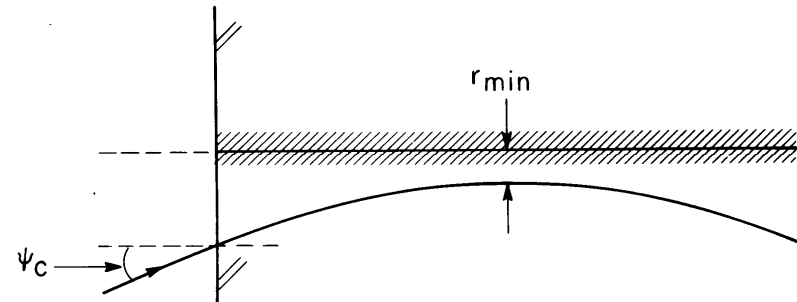
## Minimum Yield, $\chi_{\min}$



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

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

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

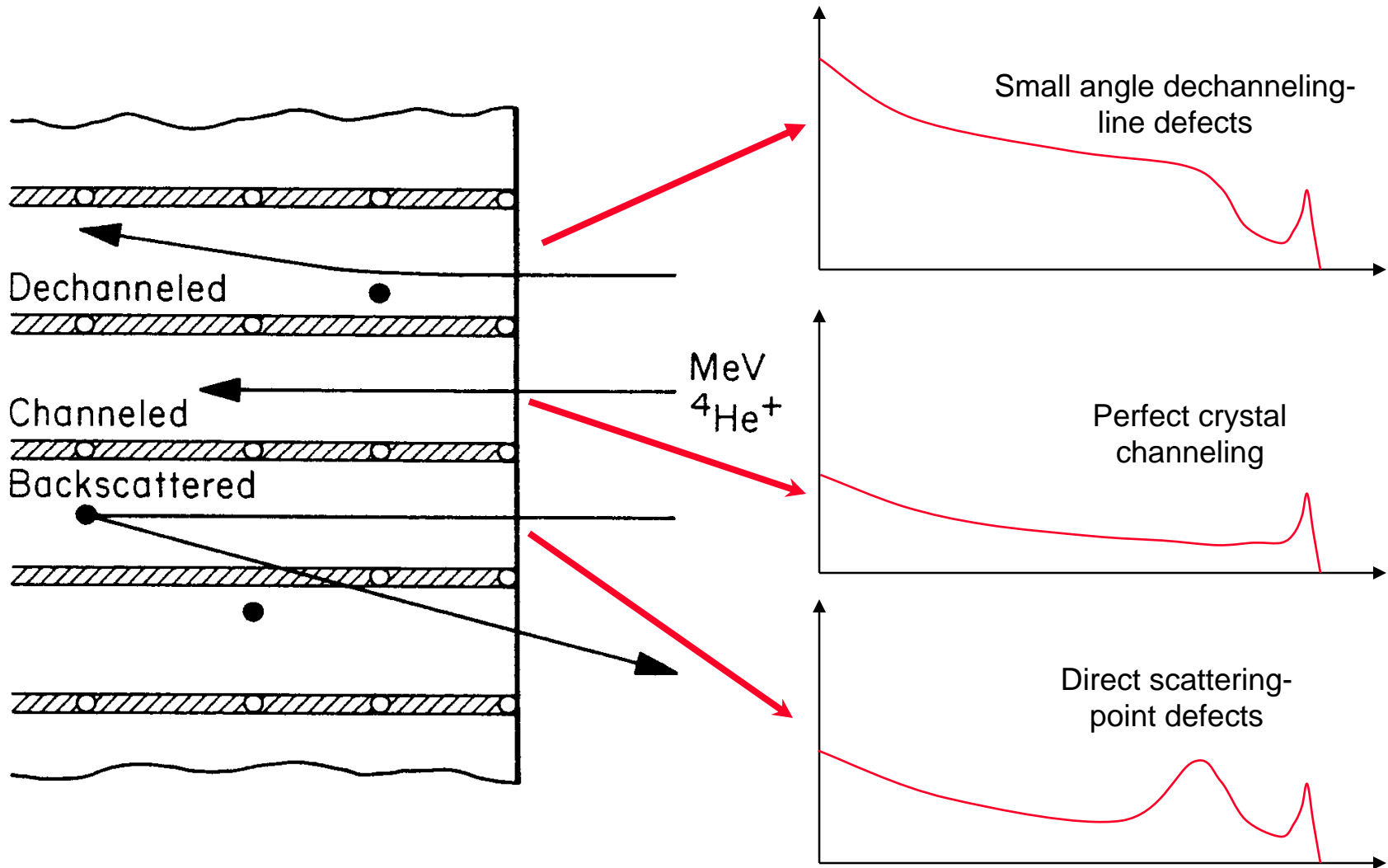


$$\Psi_c = \frac{1}{\sqrt{2}} \left( \frac{2Z_1 Z_2 e^2}{Ed} \right)^{1/2} \left\{ \ln \left[ \left( \frac{Ca}{\rho} \right)^2 + 1 \right] \right\}^{1/2}$$

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

$$\Psi_{1/2} \sim \Psi_c \sim \left( \frac{2Z_1 Z_2}{E} \right)^2 \sim 0.5 - 1^\circ$$

# Dechanneling by defects



# Homo- and Heteroepitaxy

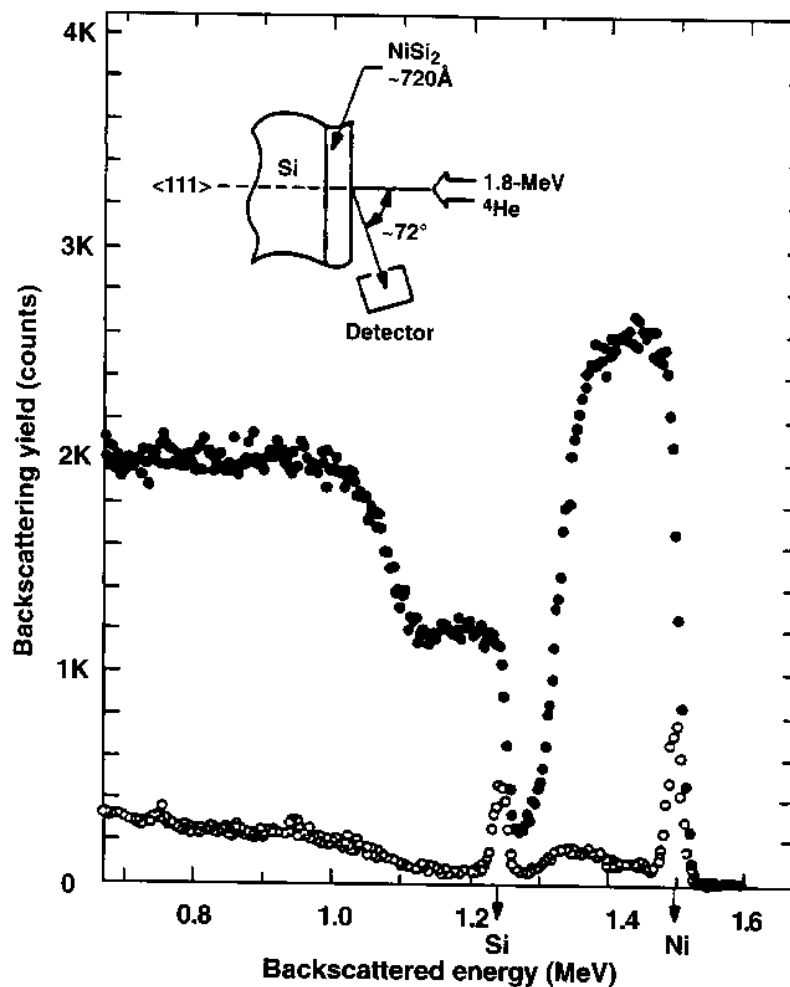
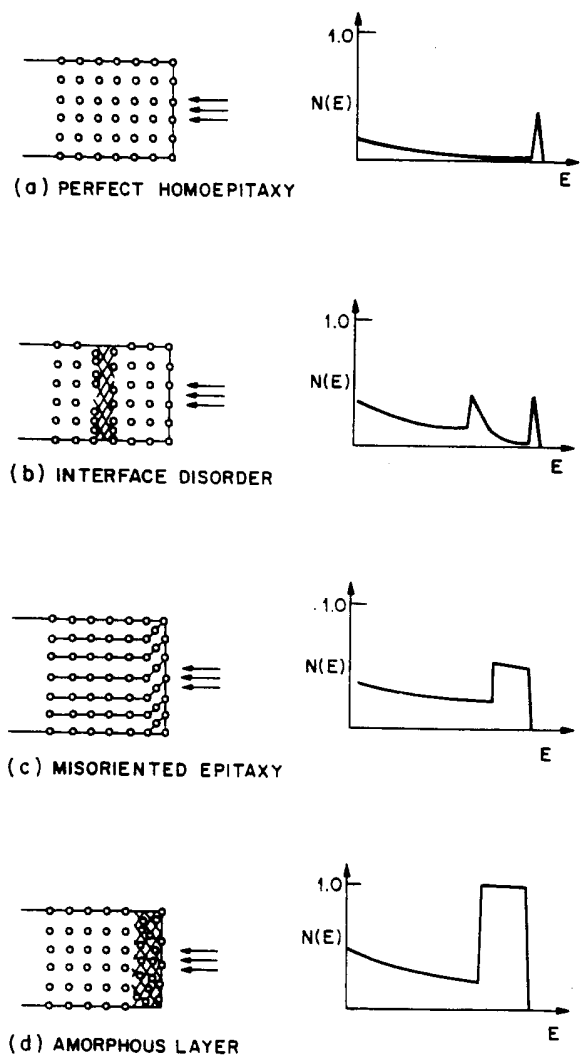
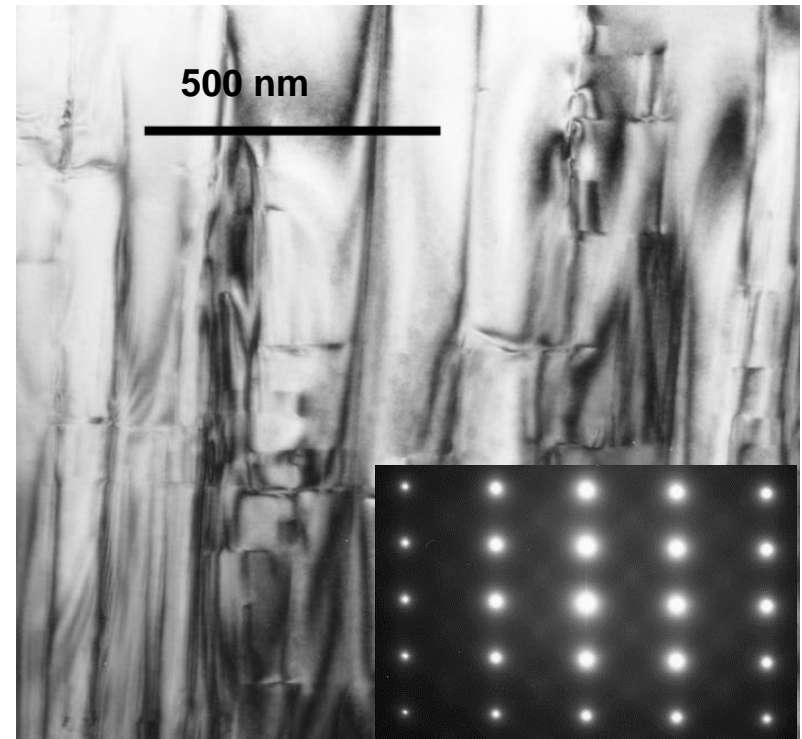
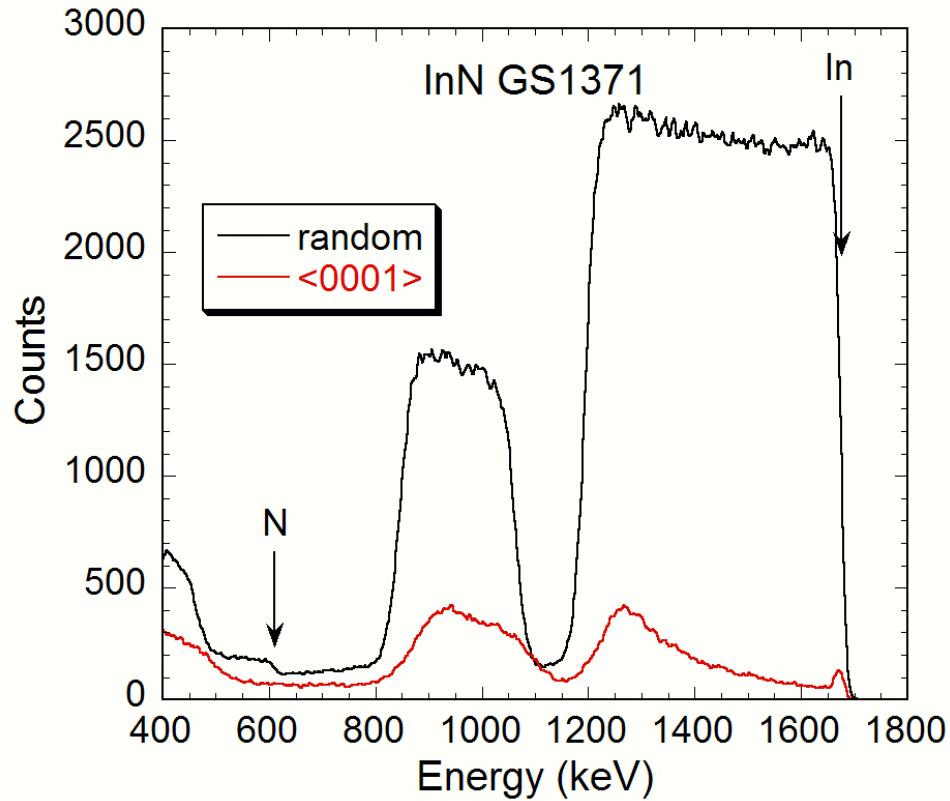


FIG. 10.24. Random and  $\langle 111 \rangle$  channeling spectra for a 72 nm  $\text{NiSi}_2$  epitaxial film on a  $\langle 111 \rangle$  Si substrate (from Chiu *et al.*, 1980).



# Channeling: Heteroepitaxy

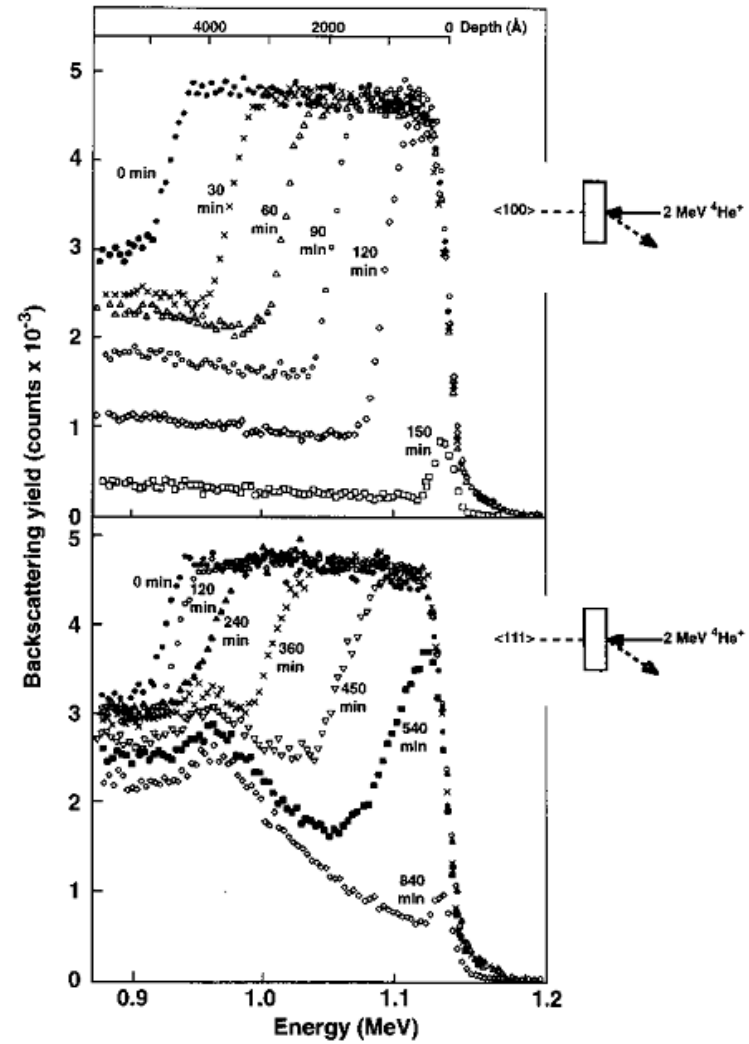
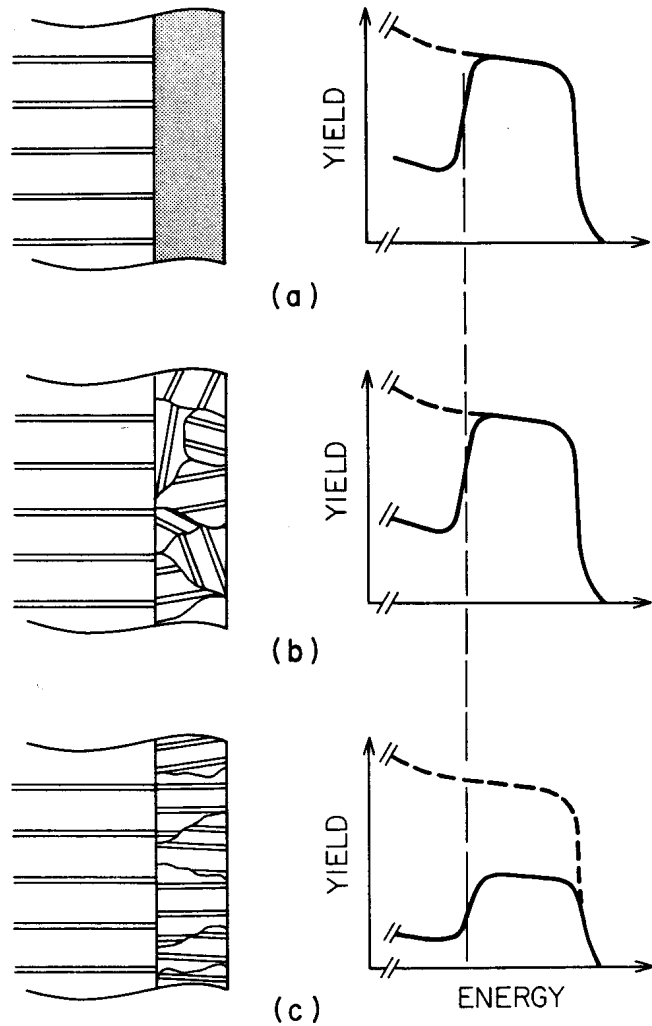


~530nm InN on 210 nm GaN

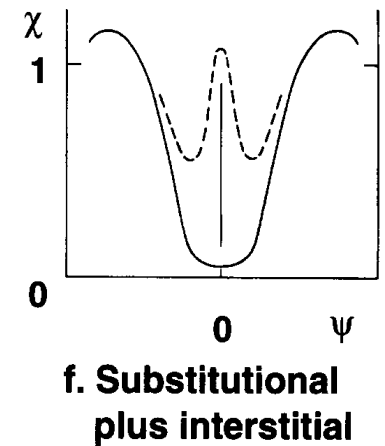
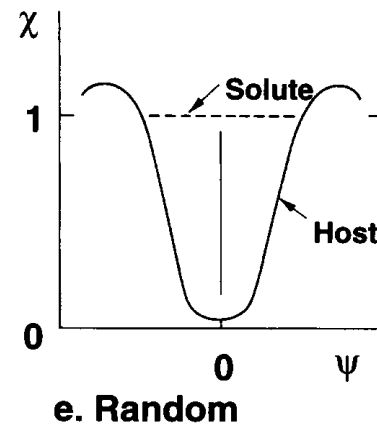
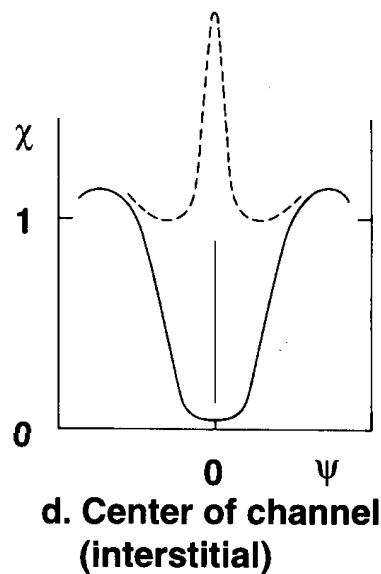
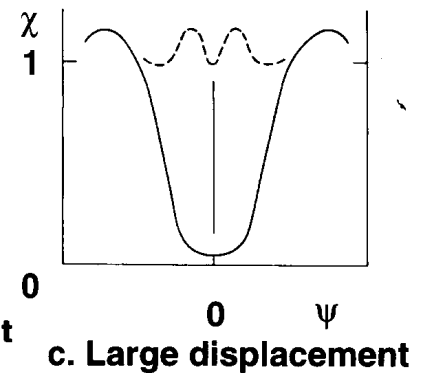
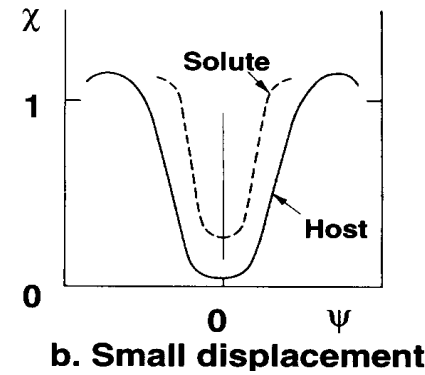
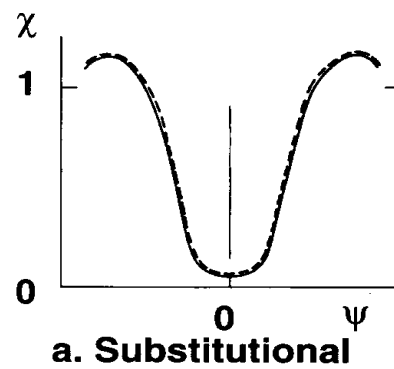
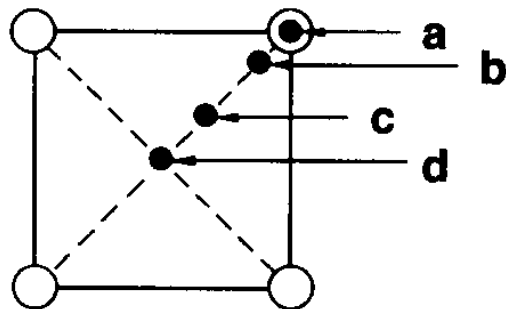
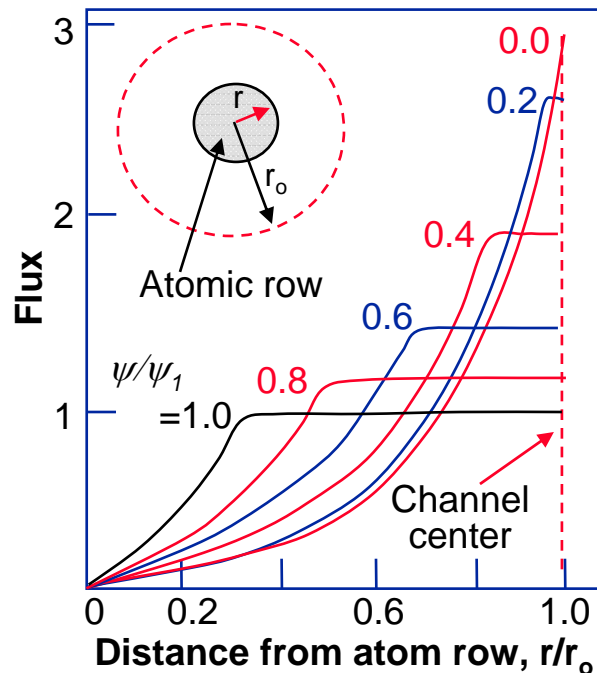
*K. M. Yu et al., LBNL 2004.*

*Z. Liliental-Weber*

# Amorphous layer analysis



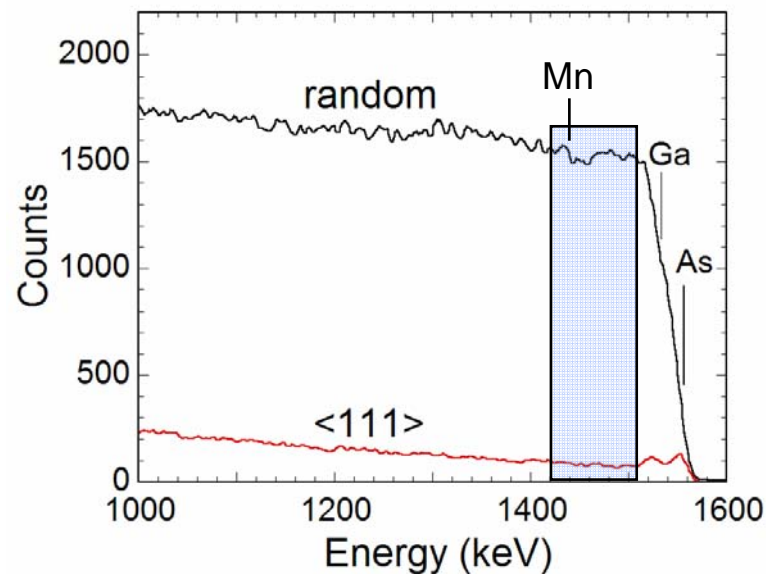
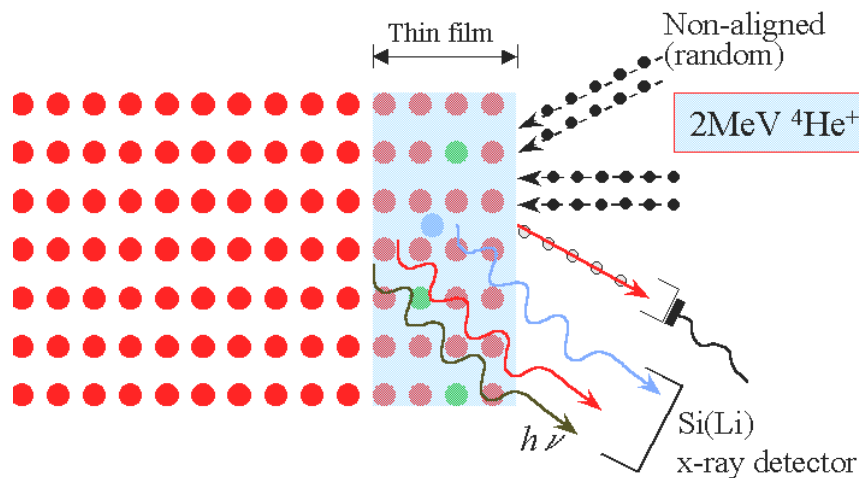
# Channeling: Impurity Lattice Location



## Channel cross section

# Experimental techniques : combined channeling RBS/PIXE

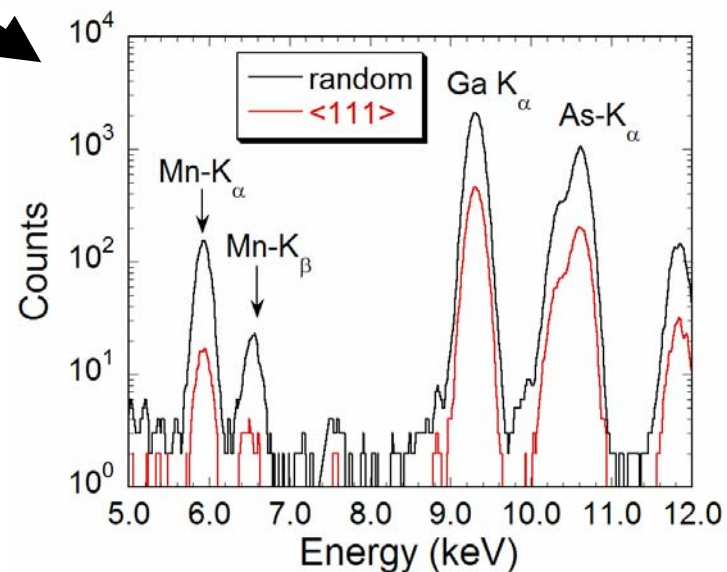
## Rutherford backscattering (RBS)



## Particle-induced x-ray emission (PIXE)

**c-RBS:** crystalline quality of film  
(from GaAs backscattering yields)

**c-PIXE:** substitutionality of Mn  
atoms w.r.t. host GaAs

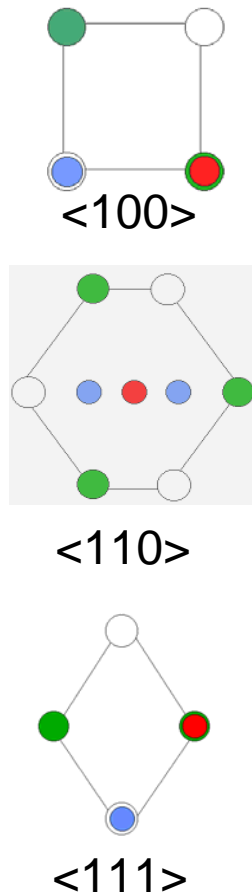


# Channeling: $\text{Ga}_{1-x-y}\text{Be}_y\text{Mn}_x\text{As}$

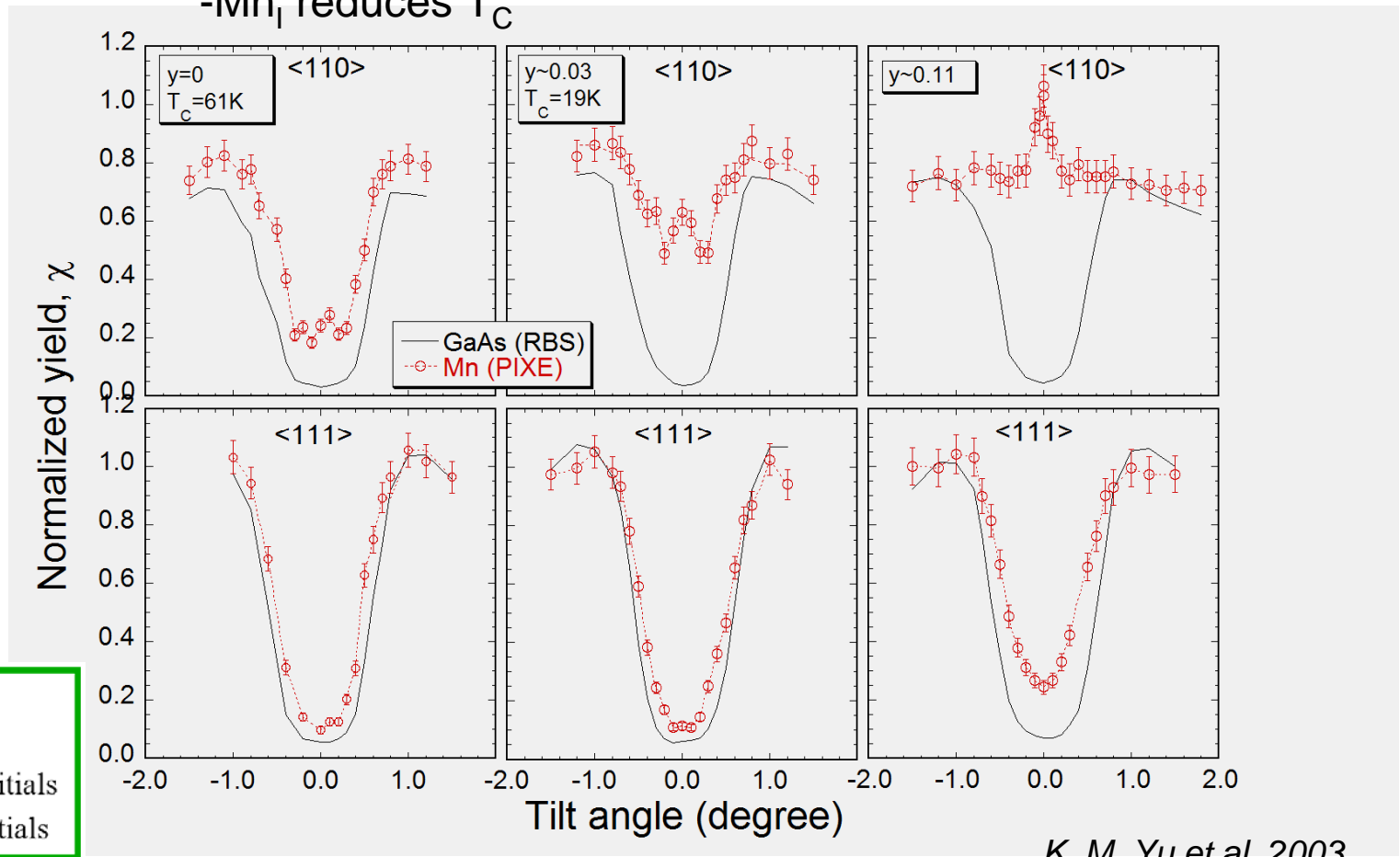


Channeling RBS/PIXE:

- presence of interstitial Mn in GaAs
- $[\text{Mn}_i]$  increases with Be doping
- $\text{Mn}_i$  reduces  $T_c$



- Cation
- Anion
- Tetrahedral interstitials
- Hexagonal interstitials



*K. M. Yu et al. 2003.*

# Examples of applications for IBA



- Thin film analysis: composition and thickness
- Multilayer analysis: identification of reaction products; obtaining reaction kinetics, activation energy, and moving species
- Composition analysis of bulk garnets
- Depth distribution of heavy ion implantation and/or diffusion in a light substrate
- Surface damage and contamination
- Providing calibration samples for other instrumentation such as secondary ion mass spectroscopy and Auger electron spectroscopy
- Defect depth distribution due to ion implantation damage or residue damage from improper annealing
- Lattice location of impurities in single crystal
- Surface atom relaxation of single crystal
- Lattice strain measurement of heteroepitaxy layers or superlattices