# Rutherford Backscattering Spectrometry

**Part I** Why Ions? Ion-solid interactions

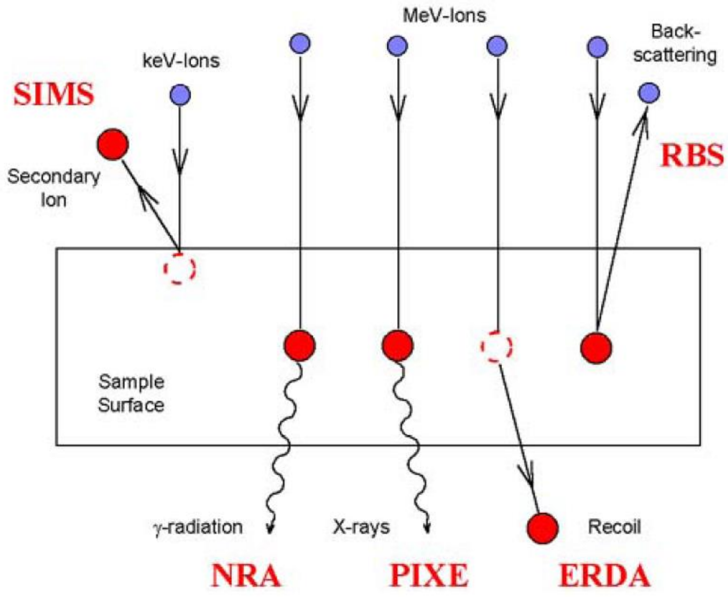
- Stopping and Range of Ions in Matter (SRIM)
- Rutherford backscattering spectrometry (RBS)
  - Introduction
  - Scattering geometry and kinematics
  - Rutherford cross section and limitations (non-Rutherford)
  - RBS spectra from thin and thick films
  - Stopping power and energy loss
  - Detector resolution
  - Energy straggling
  - Non-Rutherford cross sections

**Part II** Treatment of experimental data

References:

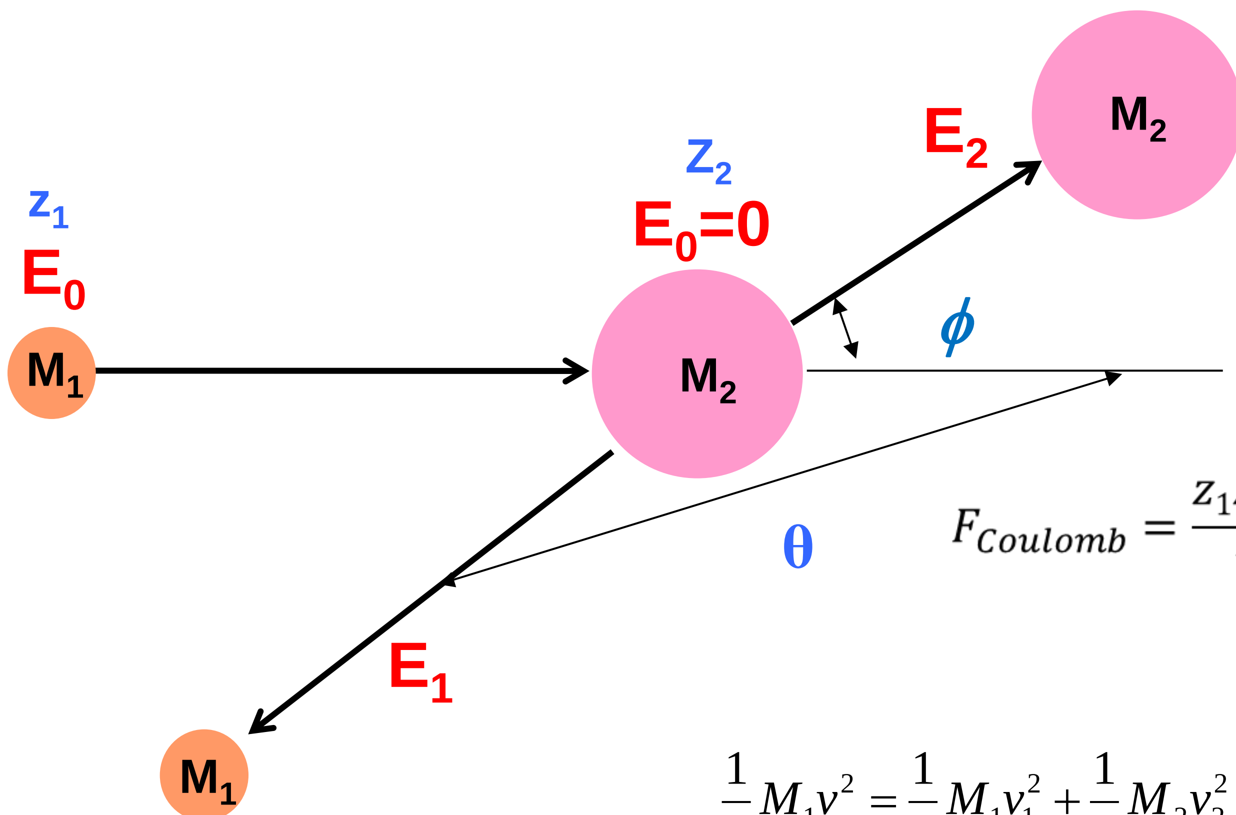
1) L.C. Feldman, J.W. Mayer (1986) Fundamentals of Surface and Thin Film Analysis.
2) Y. Wang, M. Nastasi (2010, or previous edition) Handbook of Modern Ion Beam Materials Analysis.
3) The Stopping and Range of Ions in Matter (SRIM), http://www.srim.org/
4) SIMNRA, User's Guide, Matej Mayer, Max-Planck-Institut für Plasmaphysik

# Part I: Ion-solid interactions




(1) elastic scattering
(2) fast recoils arising from elastic scattering
(3) steering effects due to the crystalline structure of target atoms
(4) inelastic processes: energy loss as a function of depth
(5) nuclear reactions
(6) interference of elastic scattering and nuclear interaction amplitudes, which leads to so-called resonant scattering

# Elastic Collisions




$$F_{Coulomb} = \frac{z_1 Z_2 e^2}{r^2}$$

$$\frac{1}{2}M_1v^2 = \frac{1}{2}M_1v_1^2 + \frac{1}{2}M_2v_2^2 \qquad \text{(Eq.1)}$$

$$M_1v = M_1v_1\cos\theta + M_2v_2\cos\phi \qquad \text{(Eq.2)}$$

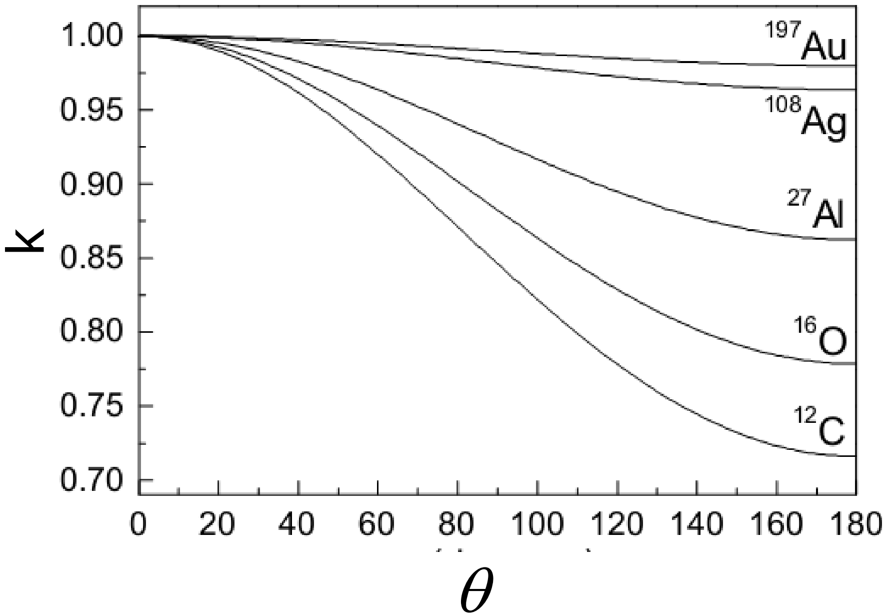
$$0 = M_1v_1\sin\theta - M_2v_2\sin\phi \qquad \text{(Eq.3)}$$

# Kinematic Factor, k

From Eq. 2 and 3, eliminating $\phi$ first, then $v_2$, one finds the ratio of particle velocities, and we can show that the energy of projectile ($M_1$) after collision can be found by the following relationship:

$$E_1 = E_0 \left[ \frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_2 + M_1} \right]^2$$

Ratio of $E_1$ and $E_0$ is called **kinematic factor**: $k = \frac{E_1}{E_o} = \left[ \frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_2 + M_1} \right]^2$



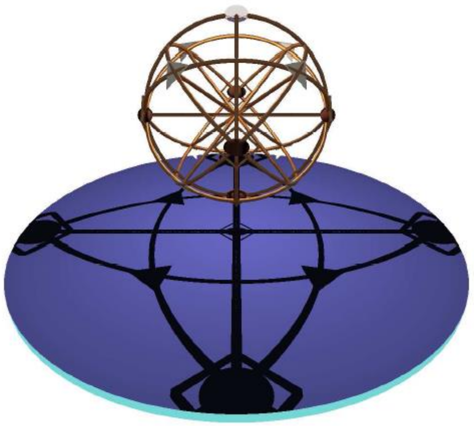

Plot of the kinematic factor, k, vs scattering angle for $H^+$ scattering from various targets

# Advantages of Ion Beams

- Can be used for material modification and analysis
- Mass Specific
  - Kinematic factor

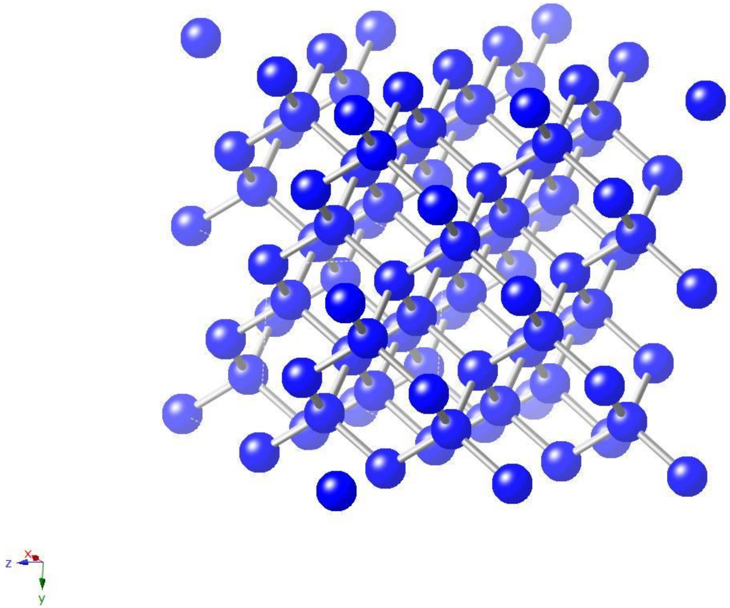
$$E_1 = E_o \left( \frac{\sqrt{M_2^2 - M_1^2 \sin^2 \theta} + M_1 \cos \theta}{M_1 + M_2} \right)^2$$

- Cross sections are very well known
- Good depth resolution
- Penetrating (can access buried interfaces)
- What about substrate?
  - can use channeling and blocking effects



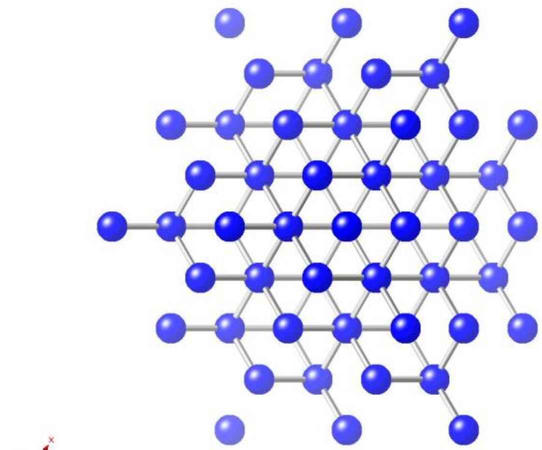
# Ion channeling and blocking

Si (diamond structure)



- Si(111)

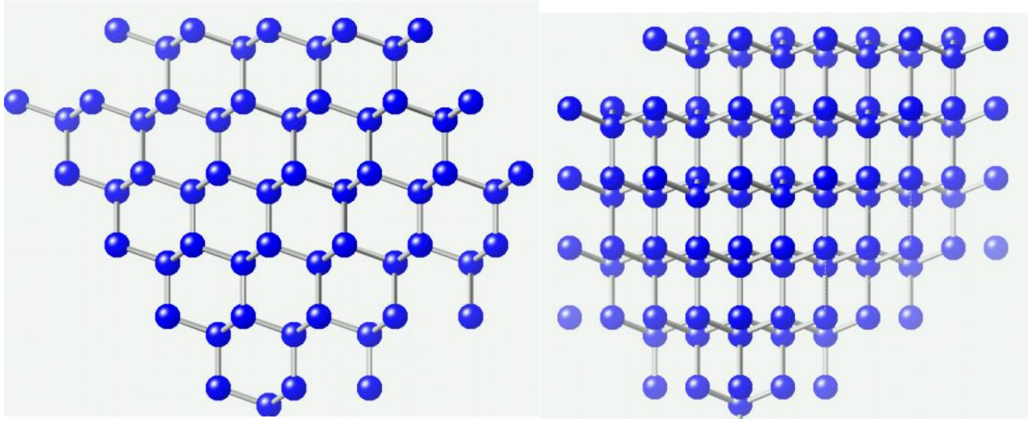
$(0\bar{1}1)$



$(\bar{2}11)$

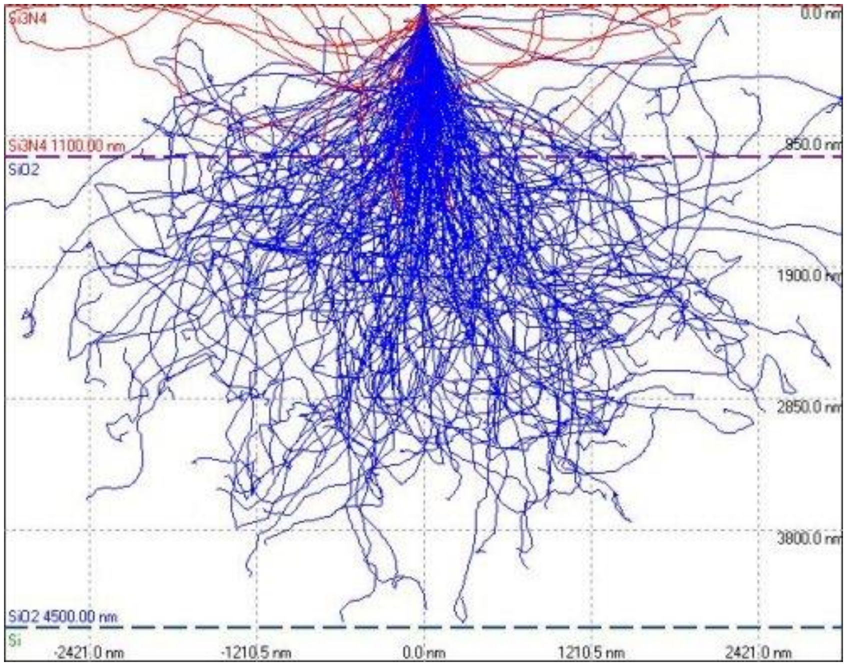
- Si(111) – side view

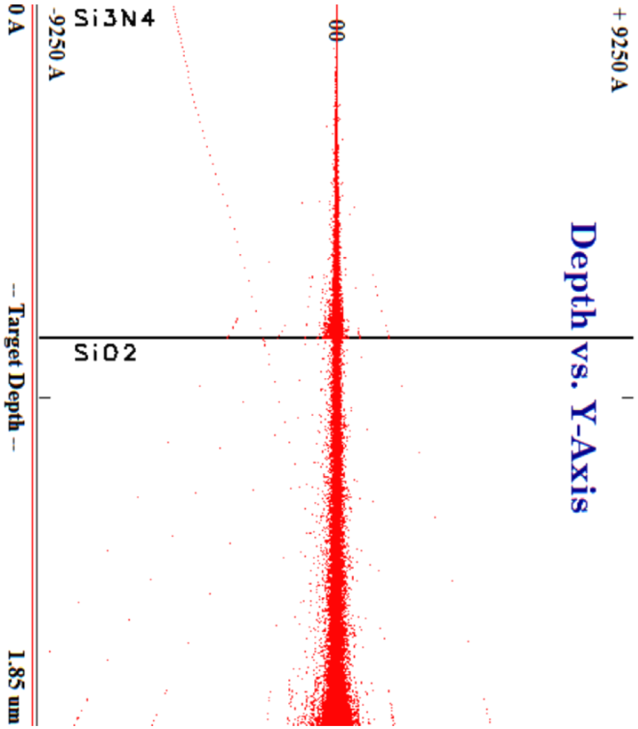
$(0\bar{1}1)$ $(\bar{2}11)$



# Electrons vs Ions

When an ion collides with electron clouds in the solid, it does not loose much energy and its direction of motion is hardly change, in a contrast with electrons colliding with electrons

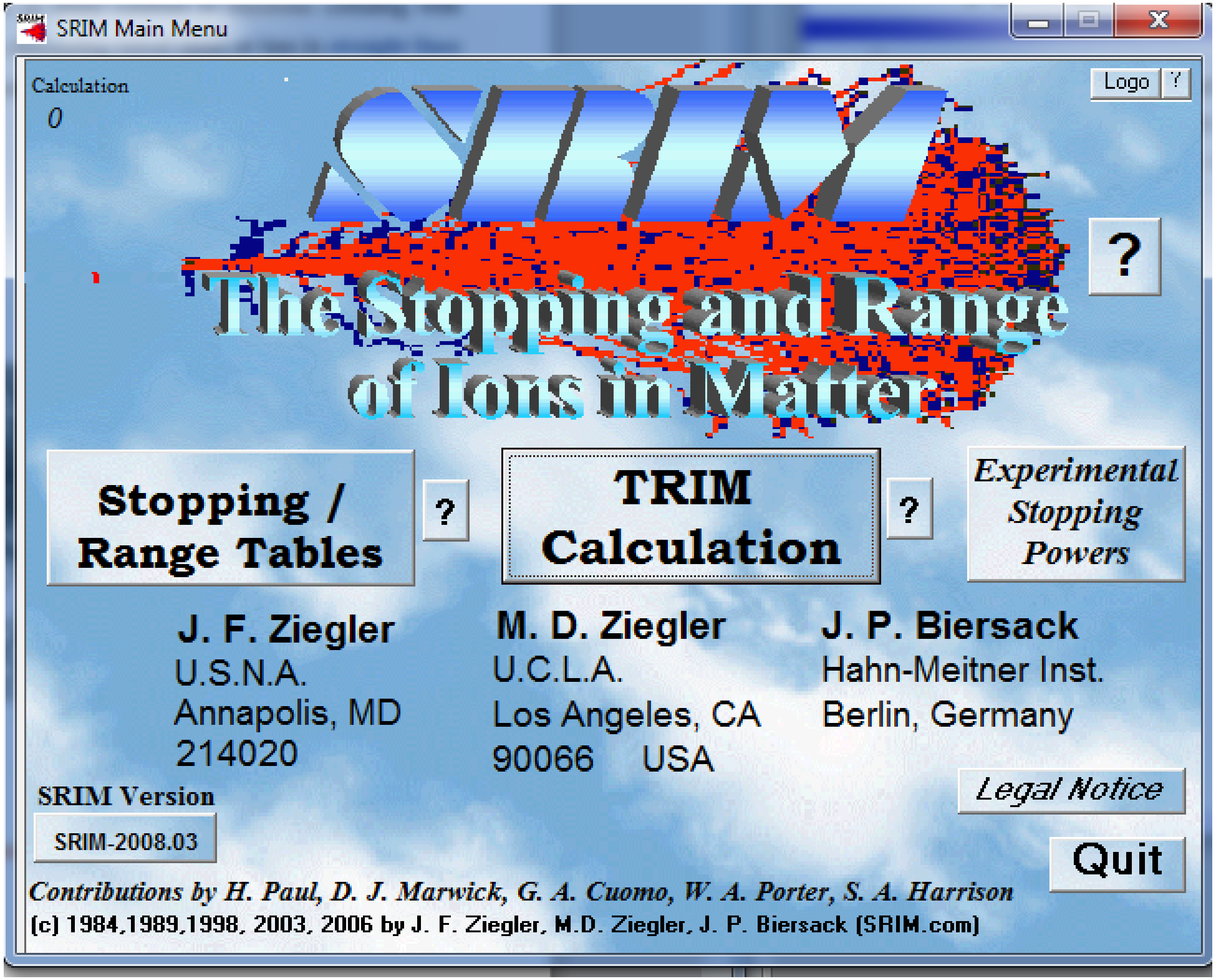


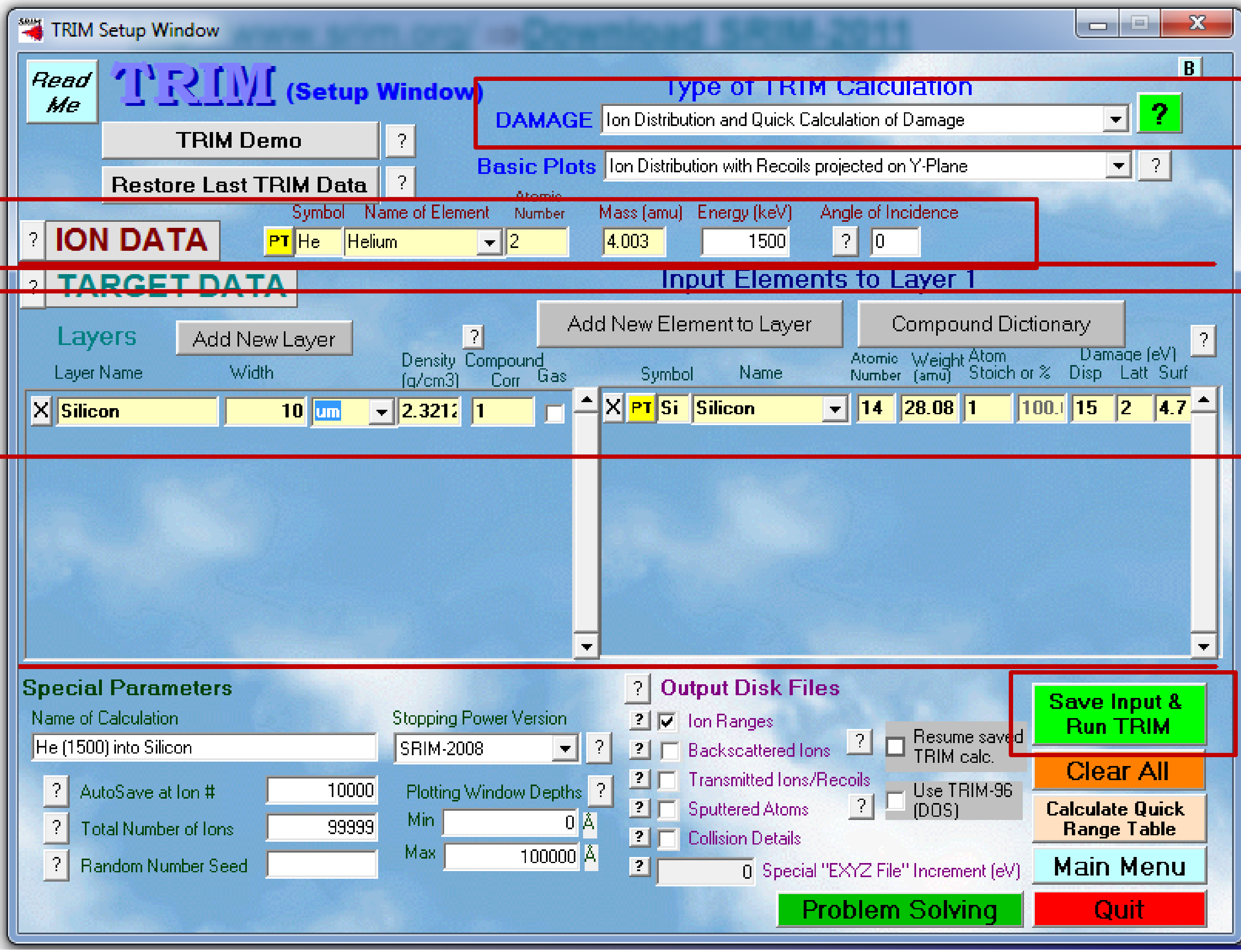

18keV $e^-$ and 18 keV $He^+$ striking a $Si_3N_4$ layer with a $SiO_2$ substrate

# SRIM

http://www.srim.org/ ⇒**Download SRIM-2013**

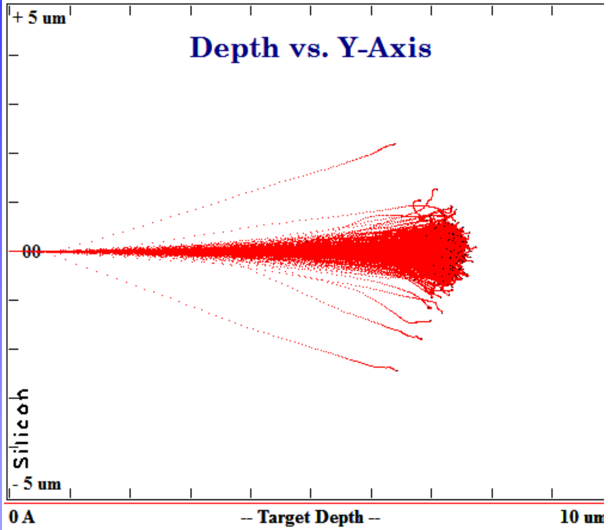


# SRIM Setup Window

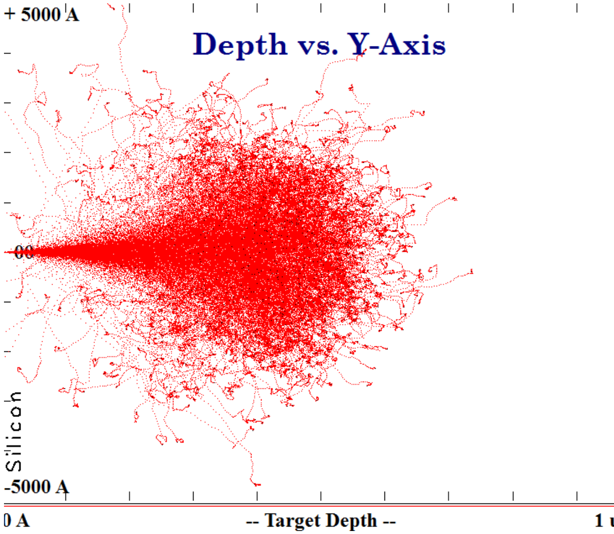


# Calculated Ion Trajectories

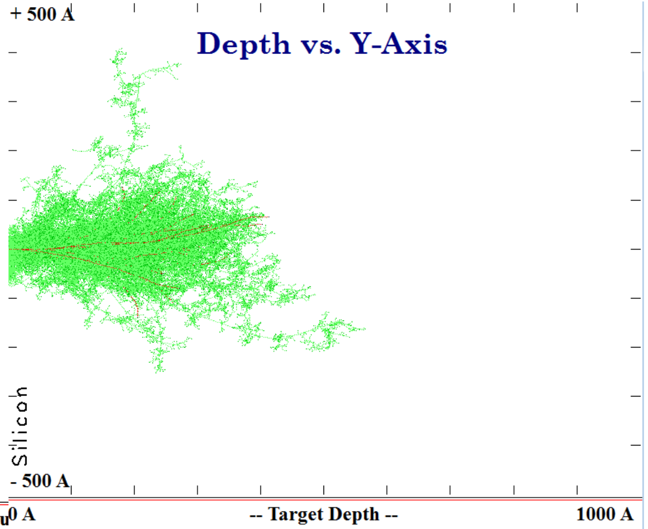
2MeV He$^+$ in Si

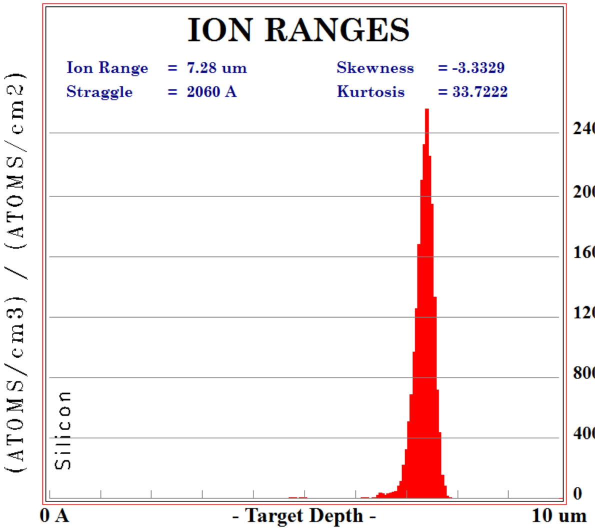


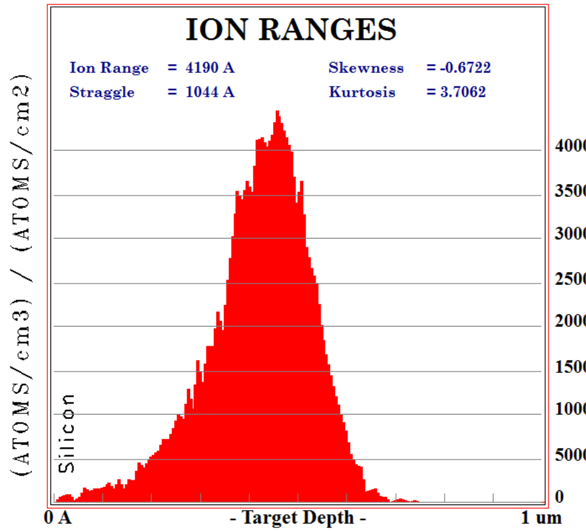

50keV He$^+$ in Si

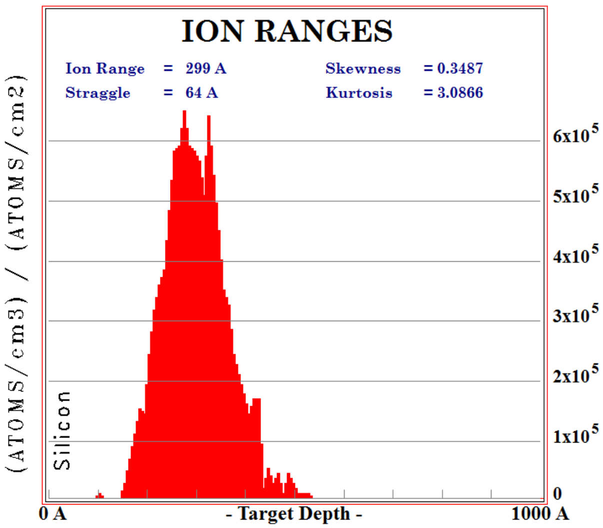



50keV Au$^+$ in Si

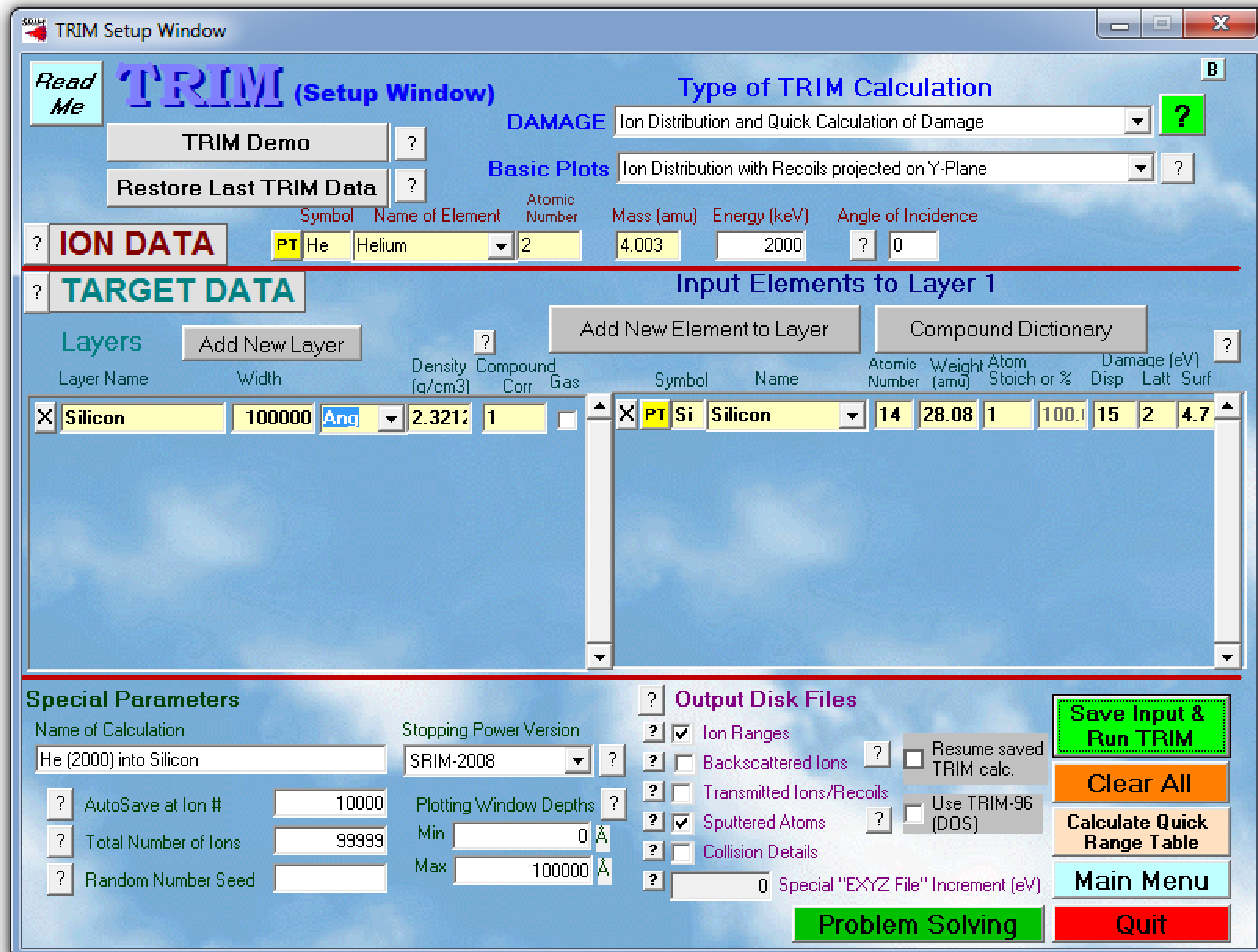


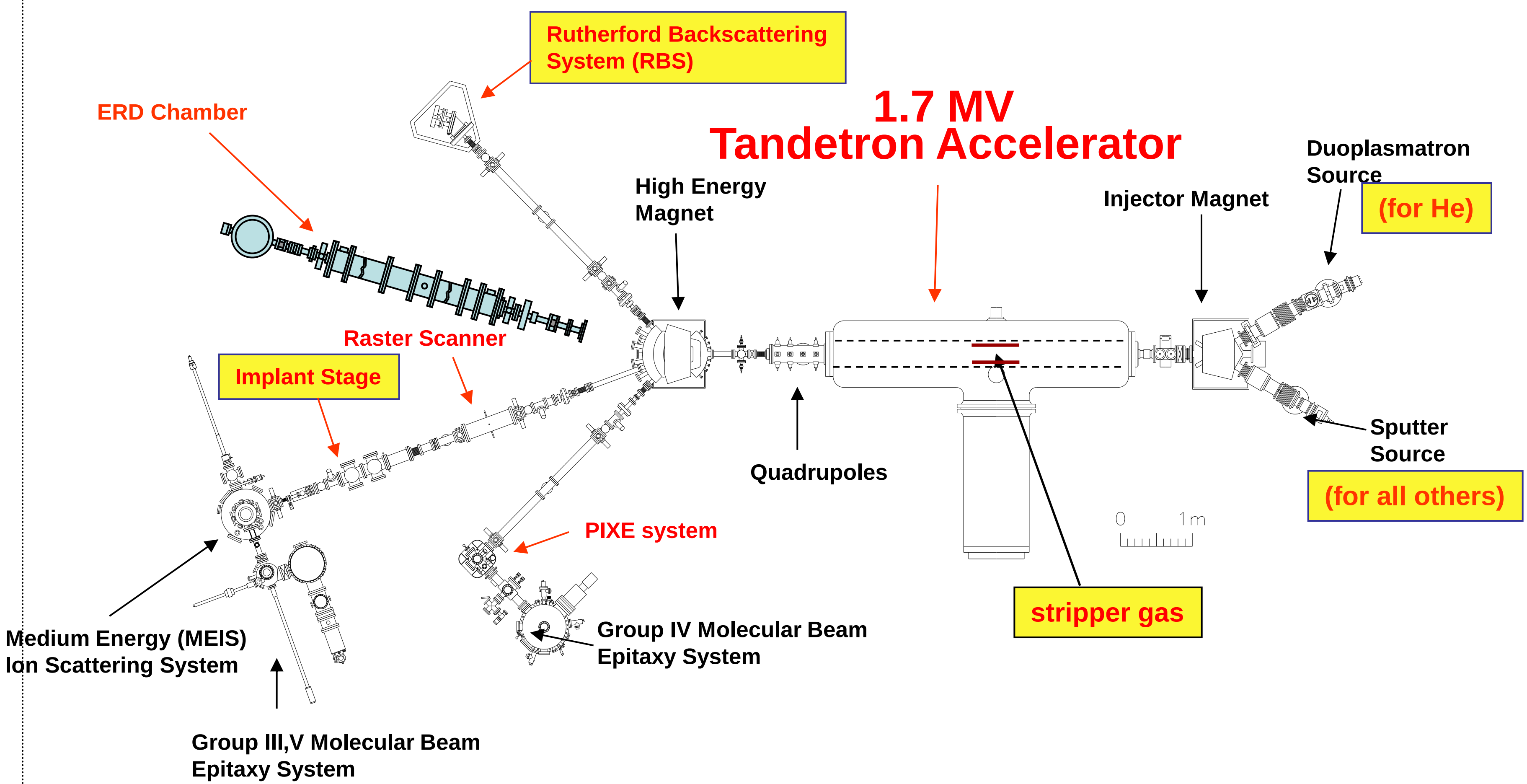



# 2MeV He$^+$ in Si


TRIM Setup Window
Read Me
TRIM (Setup Window)
TRIM Demo
Restore Last TRIM Data
Type of TRIM Calculation
DAMAGE
Ion Distribution and Quick Calculation of Damage
Basic Plots
Ion Distribution with Recoils projected on Y-Plane
ION DATA
Symbol
Name of Element
Atomic Number
Mass (amu)
Energy (keV)
Angle of Incidence
PT He
Helium
2
4.003
2000
0
TARGET DATA
Input Elements to Layer 1
Layers
Add New Layer
Add New Element to Layer
Compound Dictionary
Layer Name
Width
Density (g/cm3)
Compound Corr
Gas
Silicon
100000
Ang
2.3212
1
Symbol
Name
Atomic Number
Weight (amu)
Atom Stoich or %
Damage (eV)
Disp
Latt
Surf
PT Si
Silicon
14
28.08
1
100.
15
2
4.7
Special Parameters
Name of Calculation
He (2000) into Silicon
Stopping Power Version
SRIM-2008
AutoSave at Ion #
10000
Total Number of Ions
99999
Random Number Seed
Plotting Window Depths
Min
0
Å
Max
100000
Å
Output Disk Files
Ion Ranges
Backscattered Ions
Transmitted Ions/Recoils
Sputtered Atoms
Collision Details
Resume saved TRIM calc.
Use TRIM-96 (DOS)
0
Special "EXYZ File" Increment (eV)
Save Input & Run TRIM
Clear All
Calculate Quick Range Table
Main Menu
Problem Solving
Quit

# Tandetron Accelerator Laboratory (WSC G49)



# Tandetron Operating Principle

(1) Begin with negative ions via sputtering for most species, or He ($H_2$) gas

(2) Accelerate to kinetic energy = $qV_t$ where $V_t$ = terminal voltage (MV) and $q_i$ = -1 so that $E_t \equiv V_t$ [MeV]

(3) Ions traverse a stripper gas at the high voltage terminal to produce
a charge state distribution of positive ions

(4) Accel/decel mode is available when the stripper gas is ***OFF***: used for $E_{ion} \leq 100$ keV and the incident ions then have $q_i$ = -1

# Rutherford Backscattering Spectroscopy

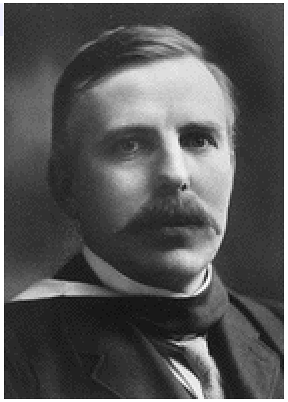
- Typical parameters: 500keV-4MeV $H^+$, $He^+$
- Ion range: 1.5-14 μm
- Ion beam is close to surface normal
- Small fraction of incident ions will scatter back

- Introduction
- 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

# History



Sir Ernest Rutherford (1871 - 1937)

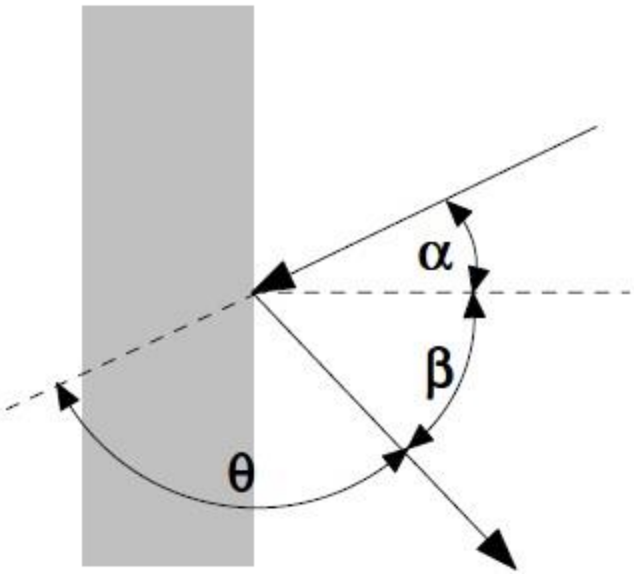
- 1911: Rutherford's scattering experiments: $^4$He on Au
  - Atomic nucleus, nature of the atom

RBS as materials analysis method

- 1957: S. Rubin, T.O. Passell, E. Bailey, "Chemical Analysis of Surfaces by Nuclear Methods", *Analytical Chemistry* 29 (1957) 736

"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 μm, with sensitivities in the range of $10^{-8}$ to $10^{-6}$ g/cm$^2$."

# RBS: Scattering geometry and kinematics



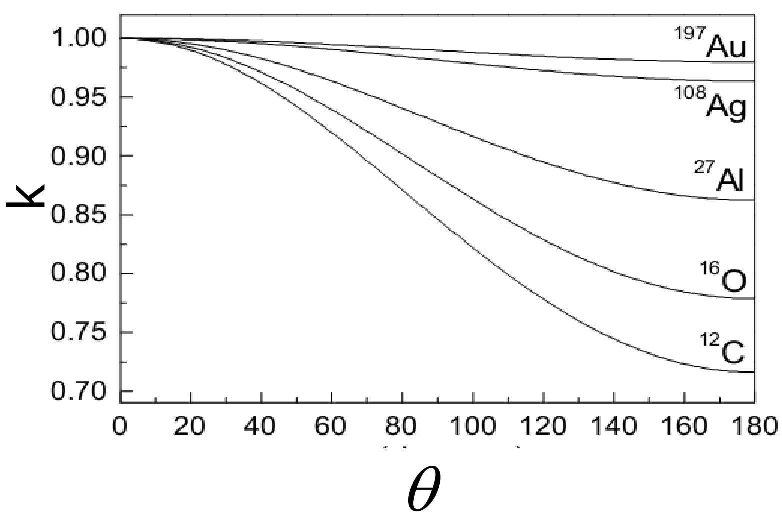

$$E_1 = k\,E_o$$

$$k = \frac{E_1}{E_o} = \left[ \frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_2 + M_1} \right]^2$$

$\alpha$: incident angle

$\beta$: exit angle

$\theta$: scattering angle

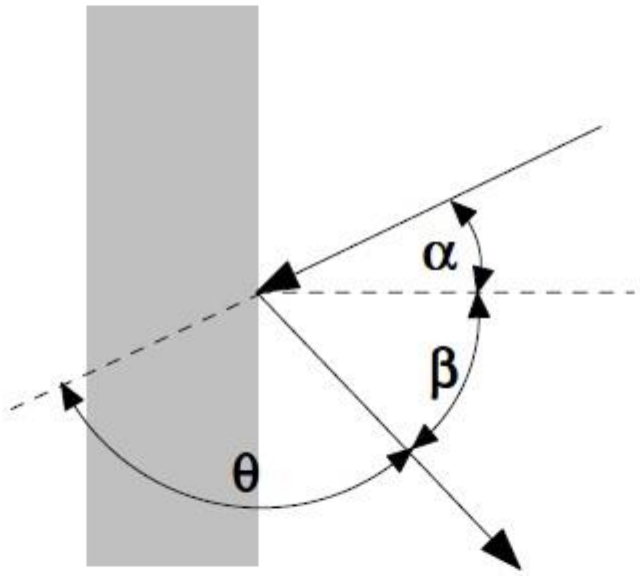



Optimized mass resolution for:

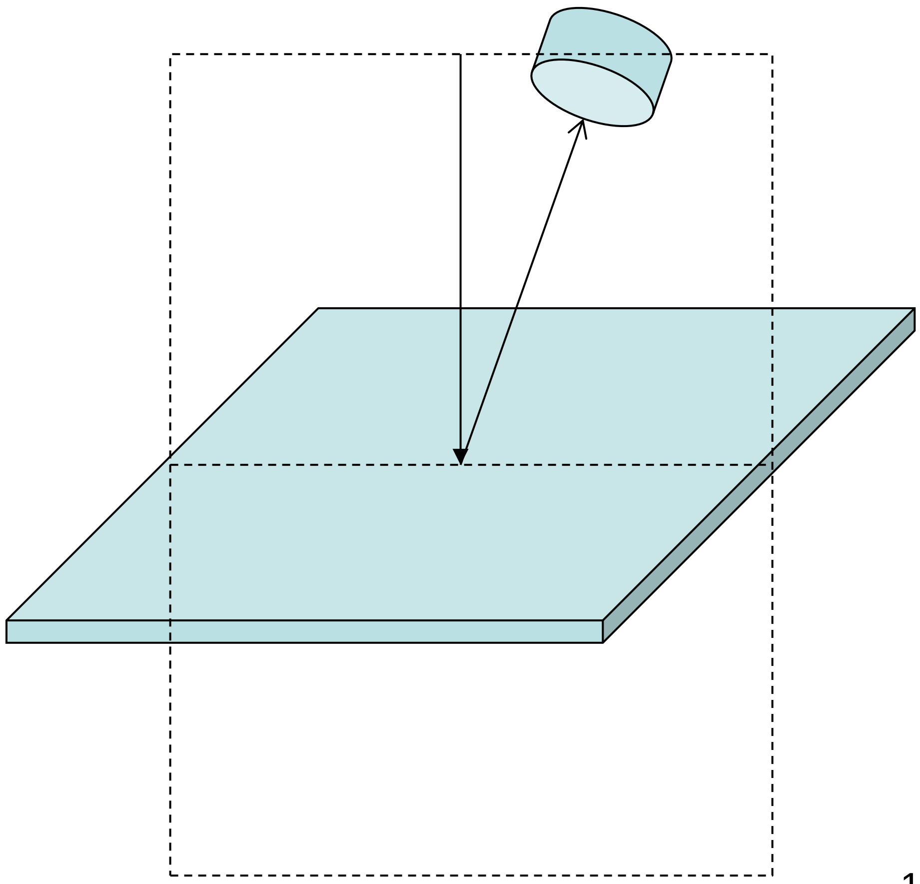
$160^o < \theta < 170^o$

# Scattering Geometry

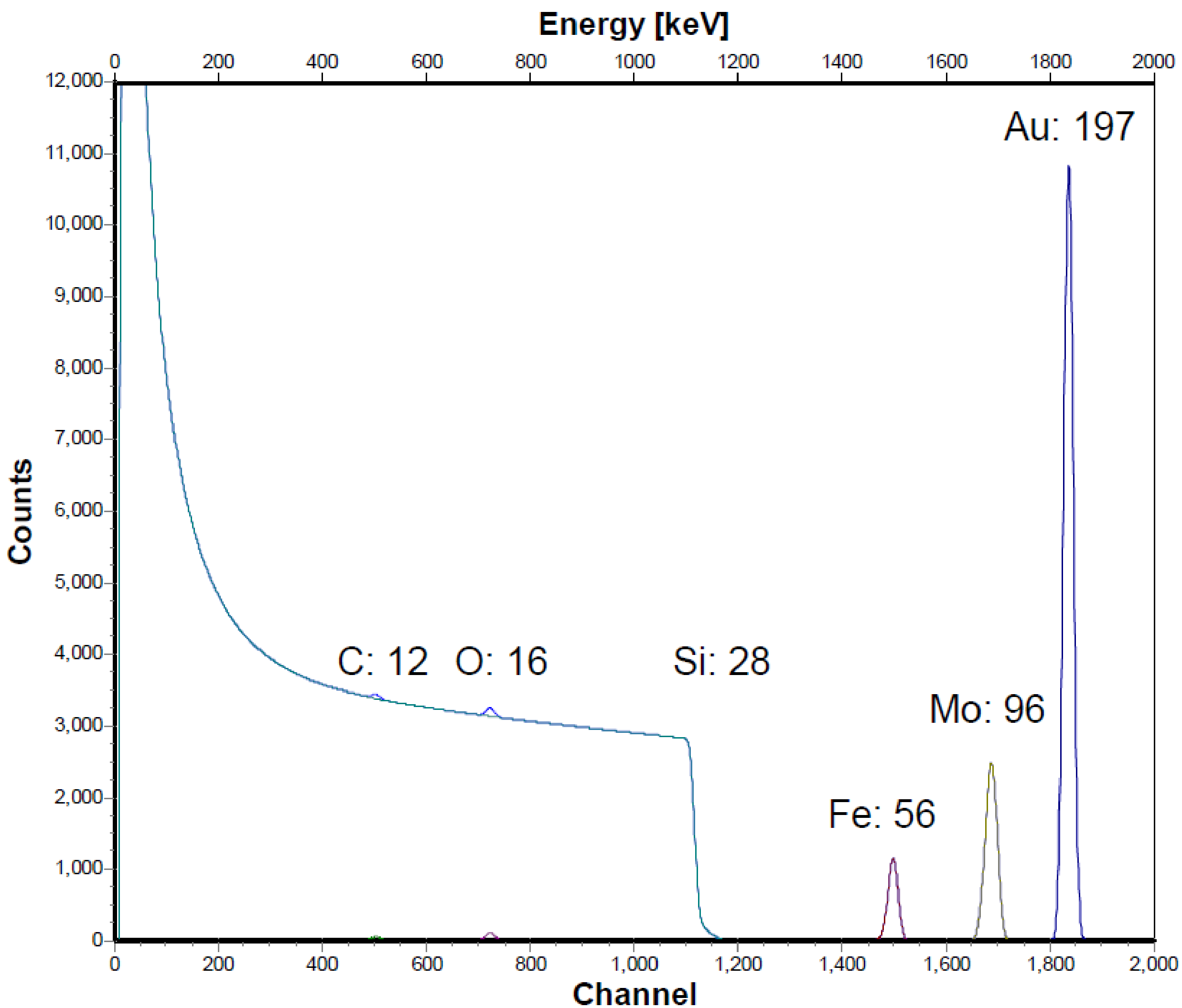
IBM geometry



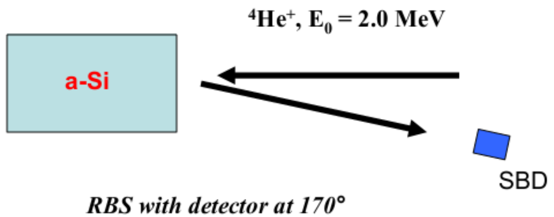

Cornell geometry → Choose this one for RBS Tandetron experiments



# Scattering kinematics: example 1




2MeV $^4He^+$, $\theta$=165°
Backscattered from
C, O, Fe, Mo, Au
$3\times10^{16}$ atoms/cm$^2$ each
on Si substrate



# Key features of RBS

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

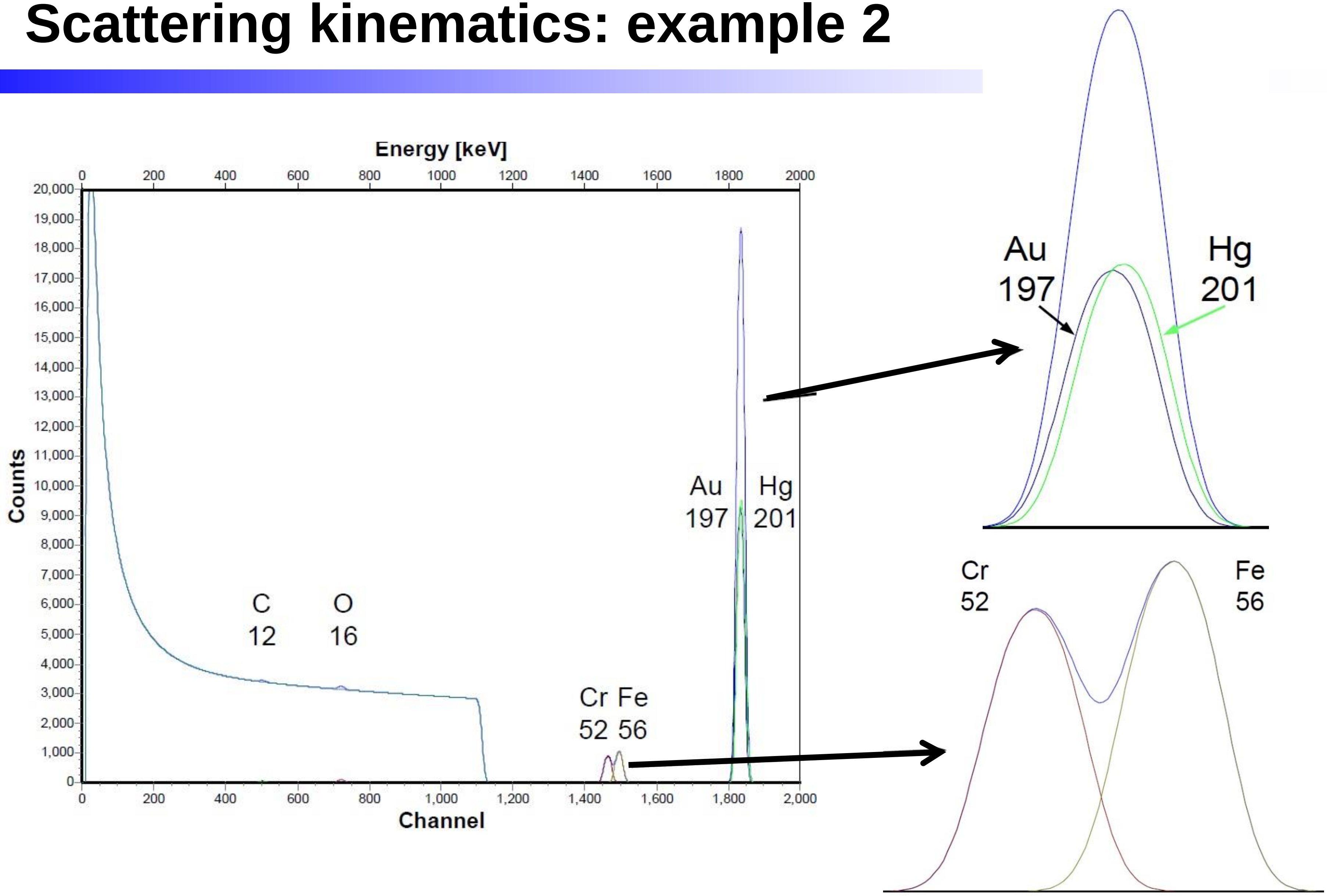
Qualitative information: **kinematic factor,** $k$

$$k = \frac{E_1}{E_o} = \left[ \frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_2 + M_1} \right]^2$$

Quantitative: **scattering cross section**, $\sigma$

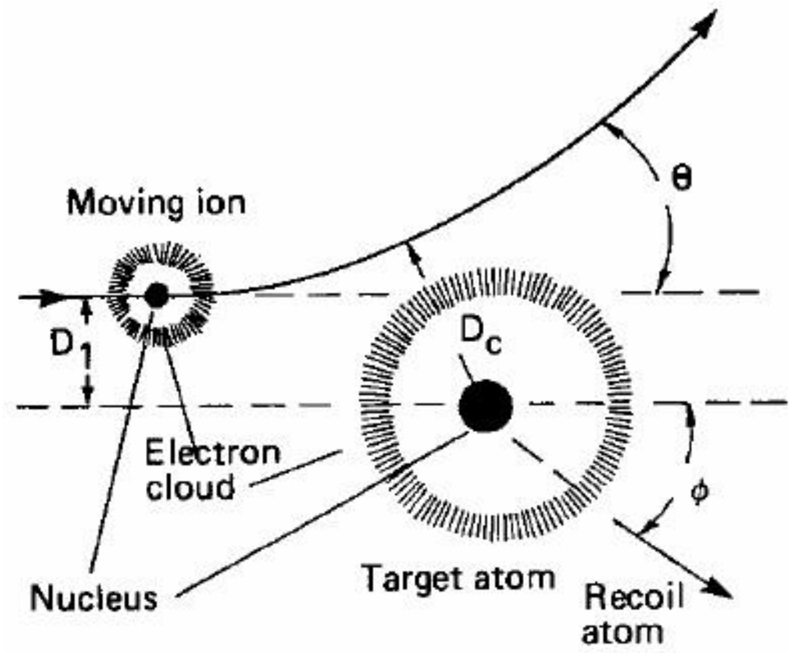
$$\frac{d\sigma}{d\Omega} \equiv \sigma(\theta) = \left( \frac{Z_1 Z_2 e^2}{4E \ \sin^2\left(\dfrac{\theta}{2}\right)} \right)^2$$

# 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

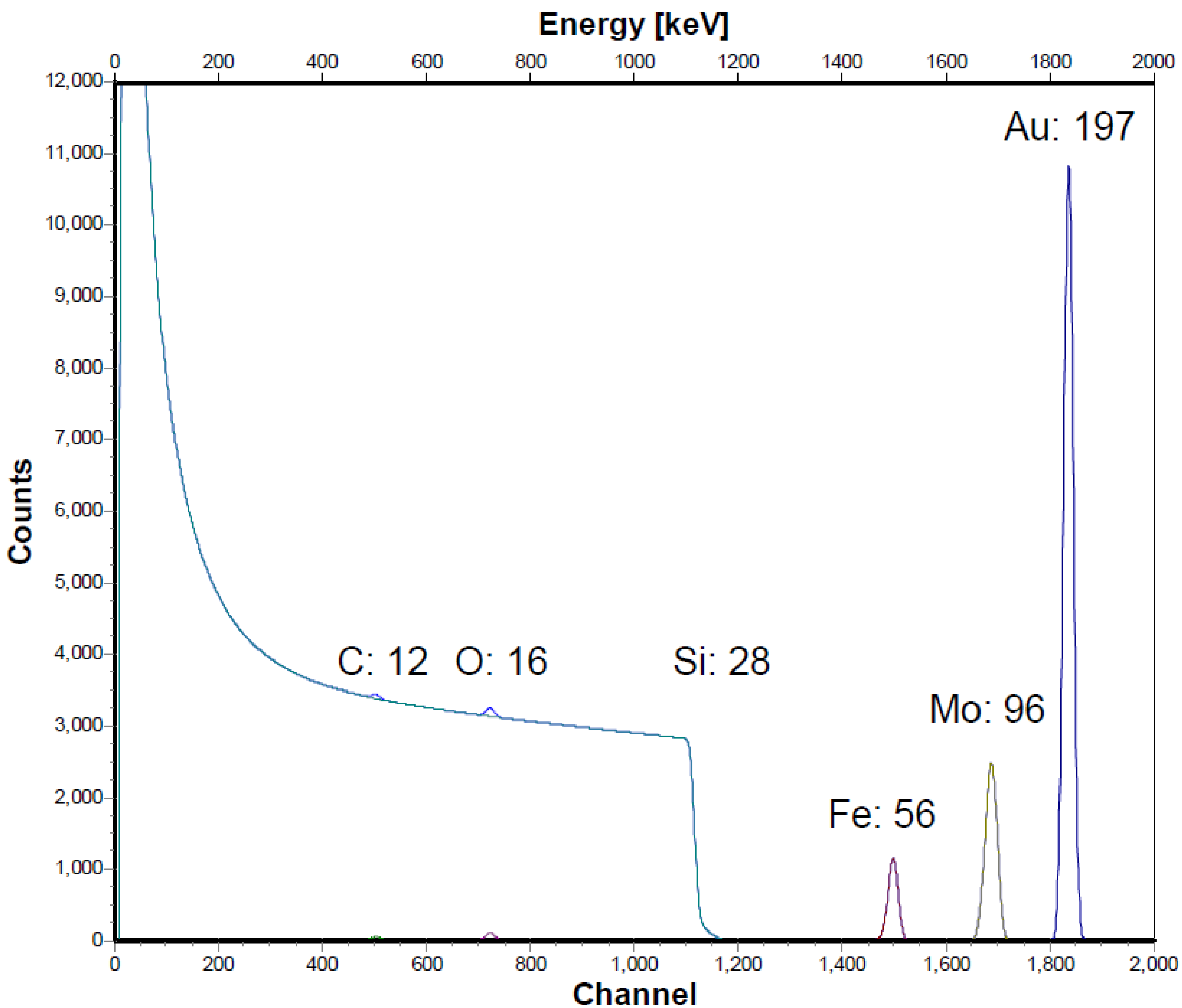
$\Rightarrow$ Rutherford scattering cross section

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

Note that sensitivity increases with:

- Increasing $Z_1$
- Increasing $Z_2$
- Decreasing $E$

# Scattering kinematics: example 1




2MeV $^4He^+$, $\theta=165^o$

Backscattered from

C, O, Fe, Mo, Au

$3\times10^{16}$ atoms/cm$^2$ each

on Si substrate

- Increased sensitivity for heavier elements
- Good for heavier elements on lighter substrates
- Bad for light elements on heavier substrates

# 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:

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

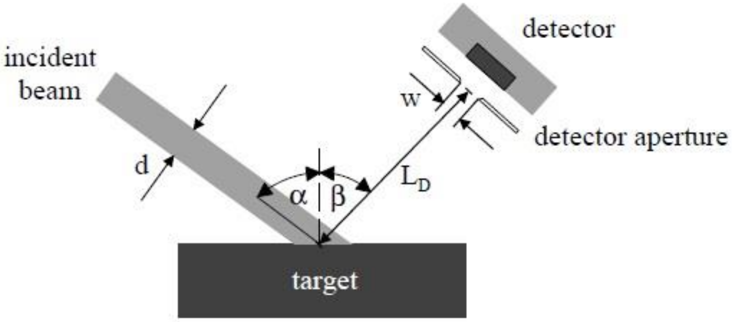
where

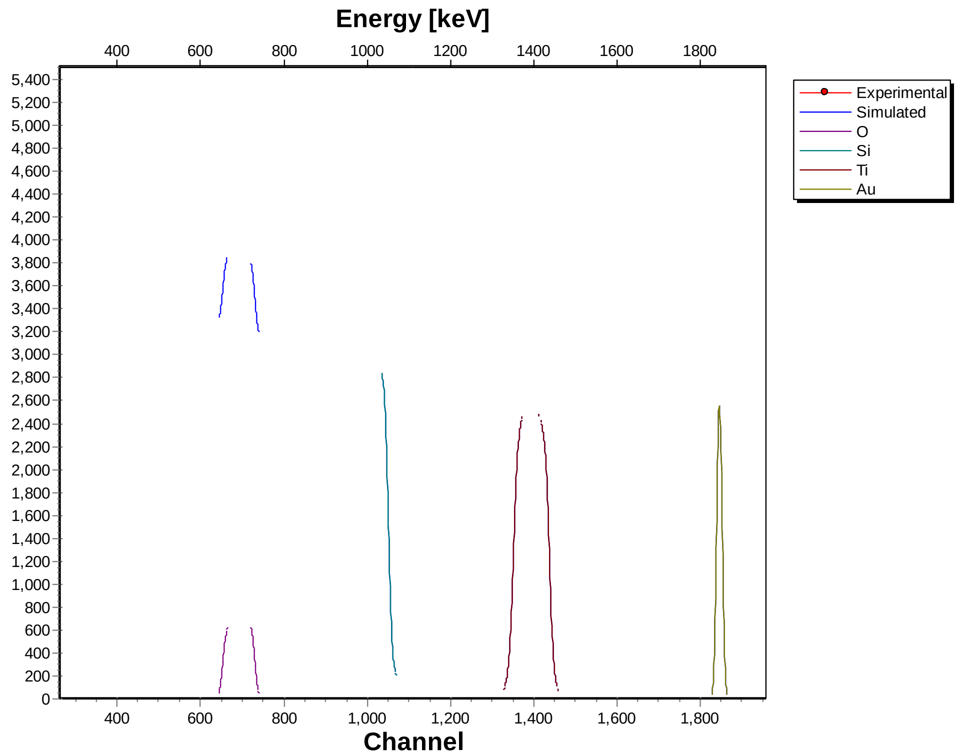
$(Nt)_i$ is areal density, atoms per unit area;

$Q$ – ion beam fluency;

$\Omega$ – solid angle of the detector;

$\sigma(E, \theta)/cos\,\theta$ – cross section of an element



# Ion dose (fluency), solid angle, cross section

- **Ion dose (fluency), the number of incident particles (collected charge)**
  - measured by Faradey 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

# Areal density: note about units

Areal density = $\rho\, t$ [g/cm$^2$],

where $\rho$ = g/cm$^3$, $t$ = cm

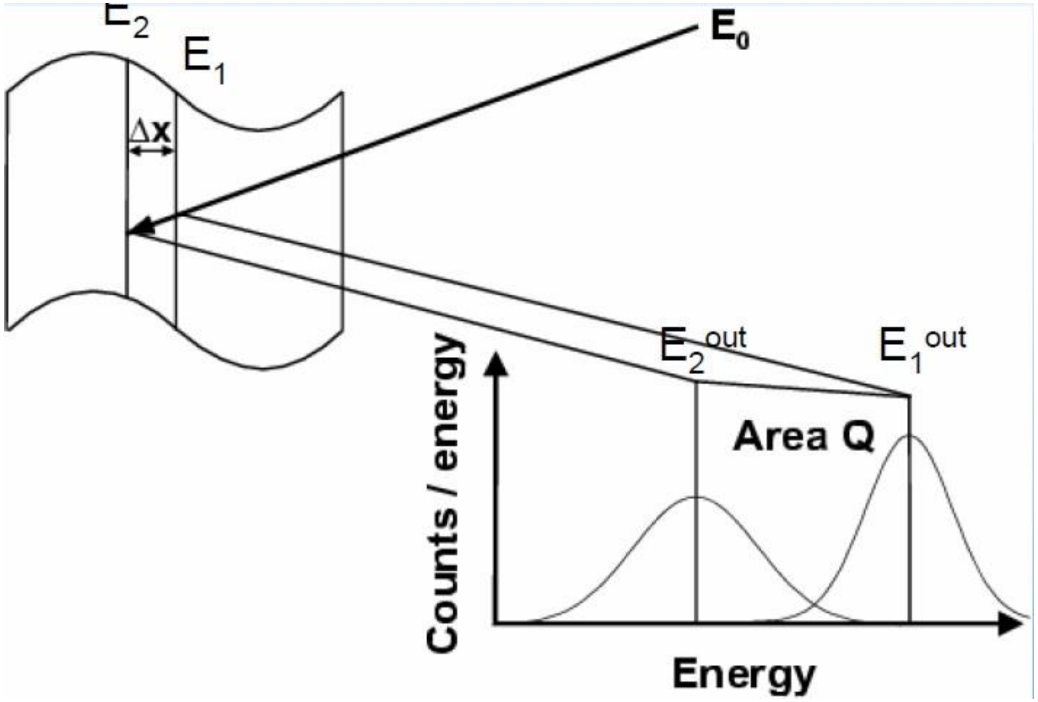
$$\rightarrow \quad \frac{N_0\, \rho\, t}{M} \quad [\text{at./cm}^2]$$

where $M$ = atomic mass [amu], $N_0$ = Avogadro's number

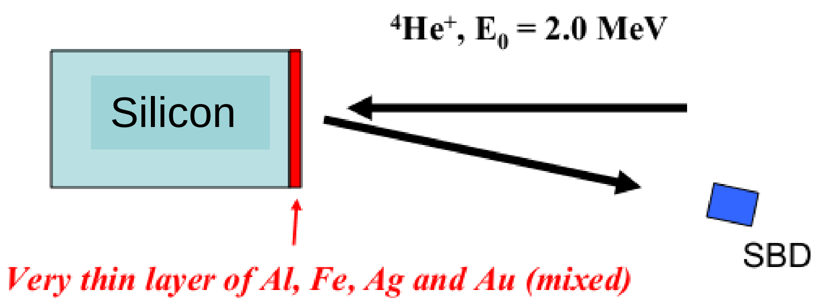
In absolute numbers – close to thickness in Å

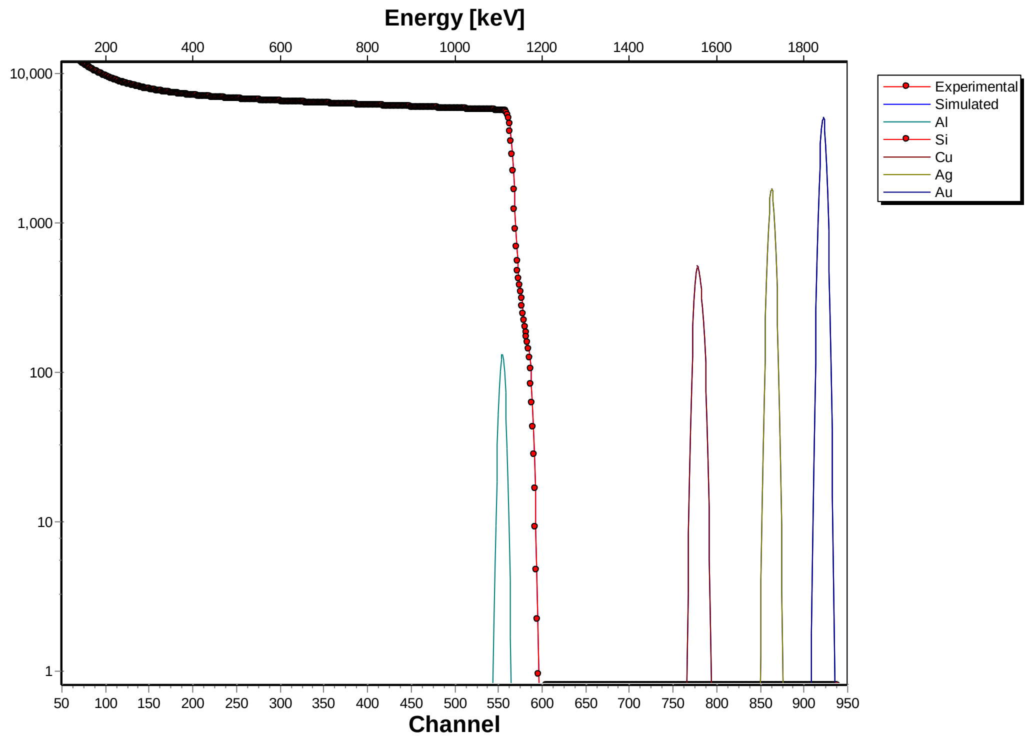
# RBS Spectrum of a thick film

- 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



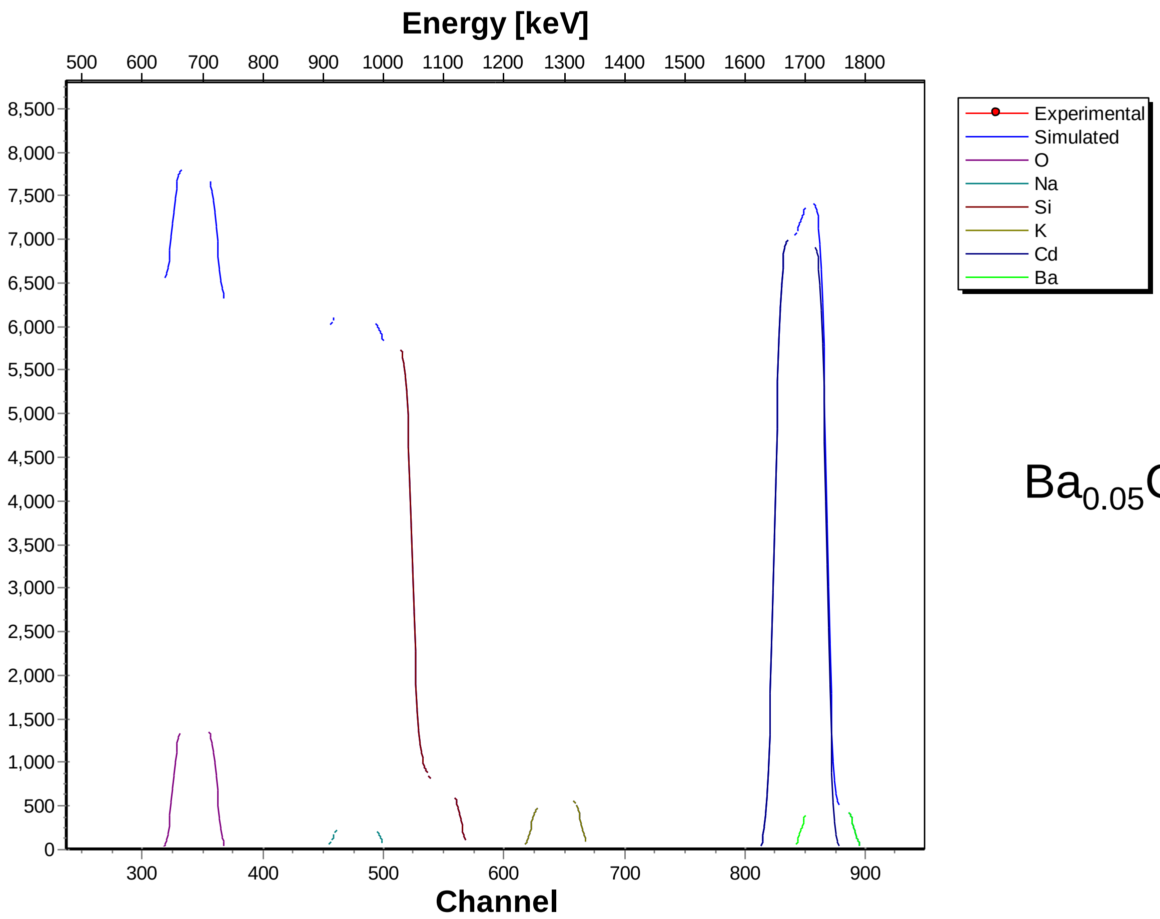
# RBS spectrum: bad choice of the substrate



# RBS Spectrum: Stoichiometry

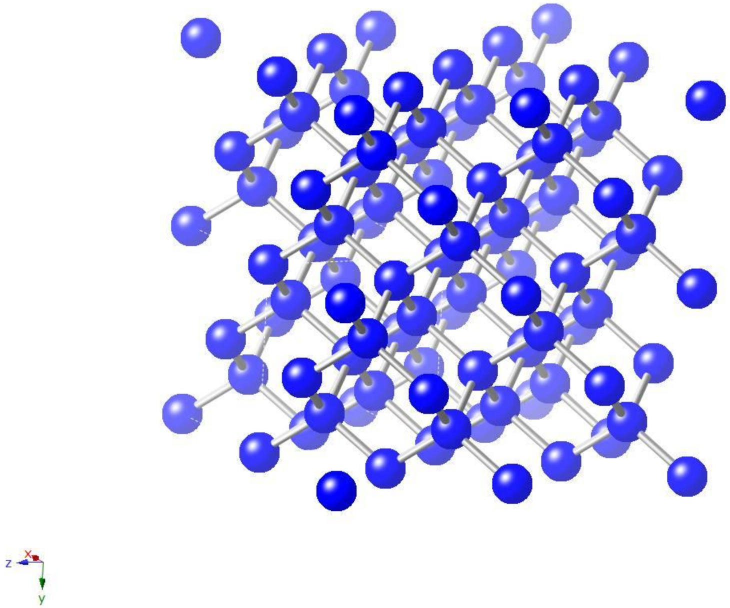
2MeV $^4He^+$, backscattered from ceramic films on Si substrate




$Ba_{0.05}Cd_2KSi_2NaO_4/Si$

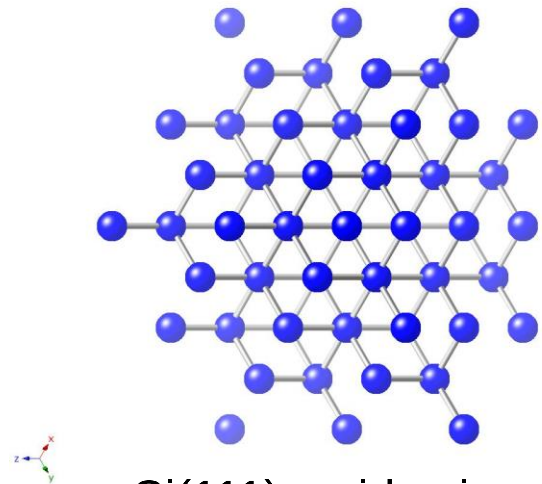
# Ion channeling and blocking

Si (diamond structure)



- Si(111)

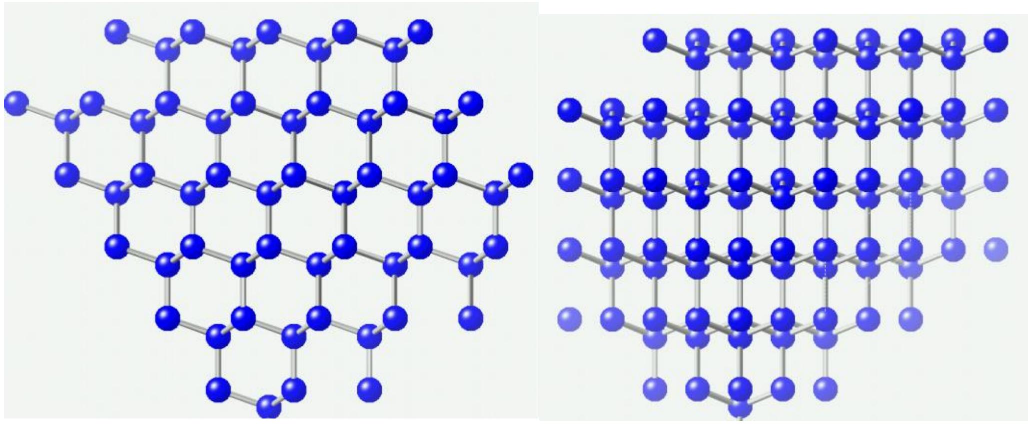
$(0\bar{1}1)$



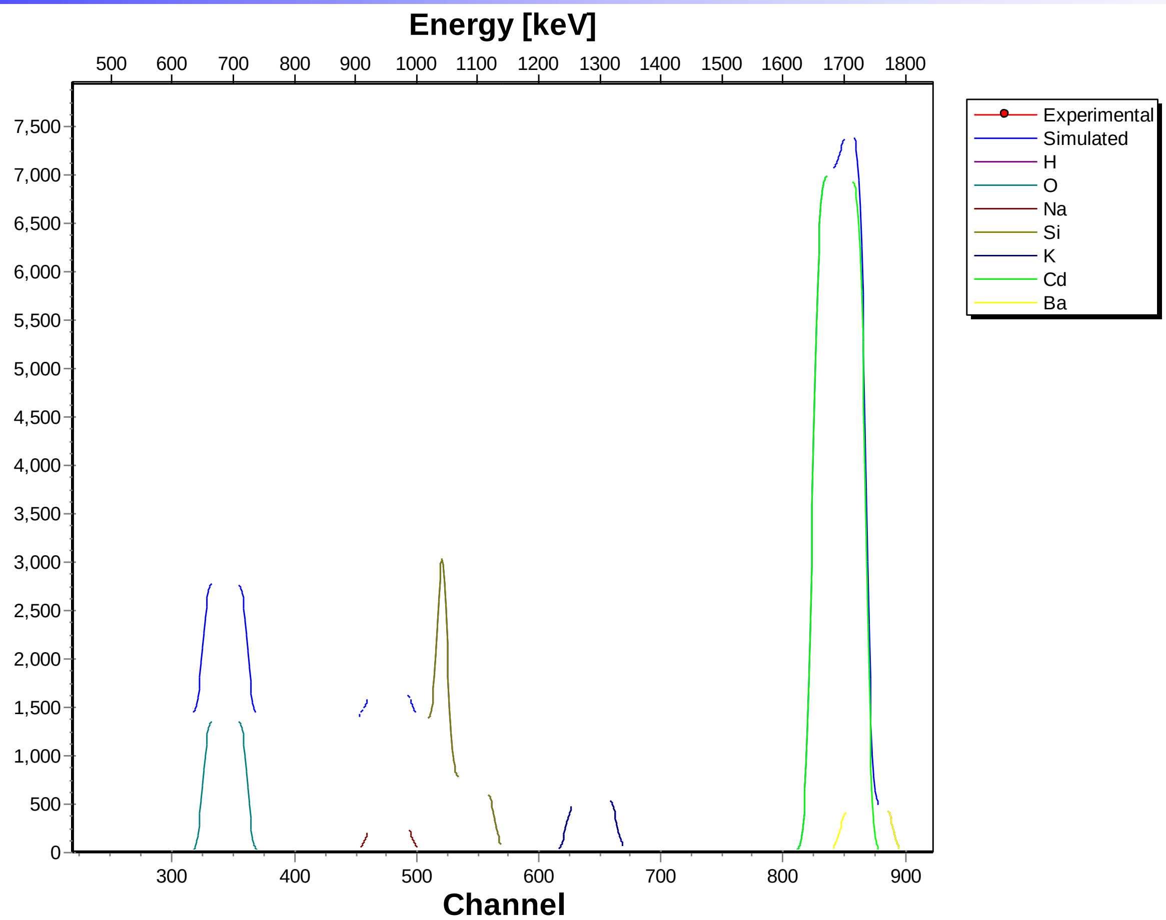
$(\bar{2}11)$

- Si(111) – side view

$(0\bar{1}1)$ $(\bar{2}11)$

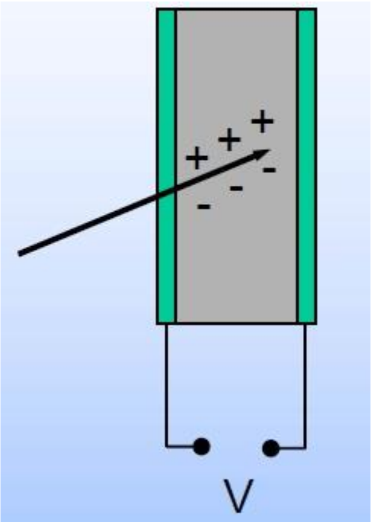


# Use crystal structure of the substrate




- 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

⇒ Number of electron-hole pairs ∝ Particle energy

⇒ Charge pulse ∝ Particle energy

**Limited energy resolution (~ 1keV/100Å ) due to:**

• Statistical fluctuations in energy transfer to electrons and phonons

• Statistical fluctuations in annihilation of electron-hole pairs

**Additional energy broadening due to:**

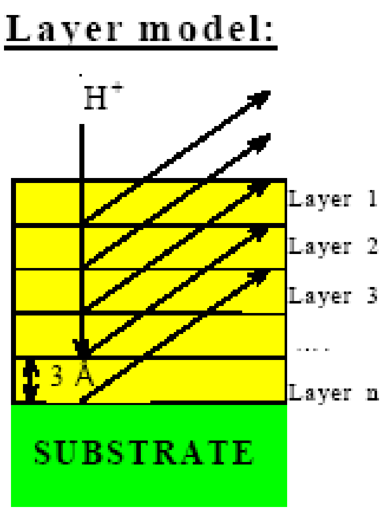
• Preamplifier noise

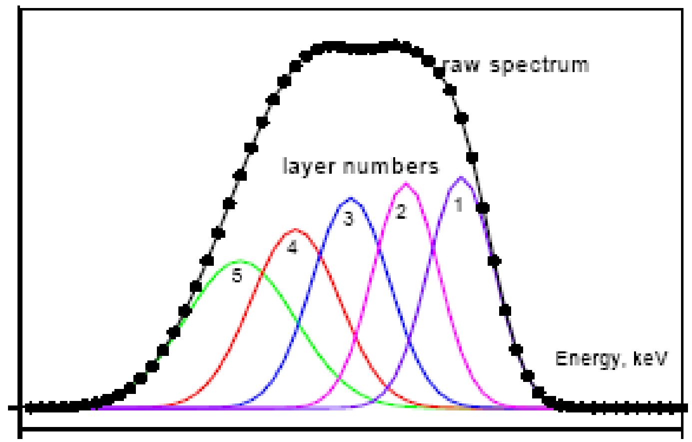
• Other electronic noise

# Depth resolution and concentration profiling

Depth resolution for ≈100 keV protons (resolution of the detector≈1keV)

- Stopping power $SiO_2$ ≈ 12 eV/Å; $Si_3N_4$ ≈ 20 eV/Å; $Ta_2O_5$ ≈ 18 eV/Å
- "Near surface" depth resolution ≈ 50 Å; worse for deeper layers due to energy straggling




- Areas under each peak corresponds to the concentration of the element in a 50Å slab
- Peak shapes and positions come from energy loss, energy straggling and instrumental resolution
- The sum of the contributions of the different layers describes the depth profile.

# Energy Straggling

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

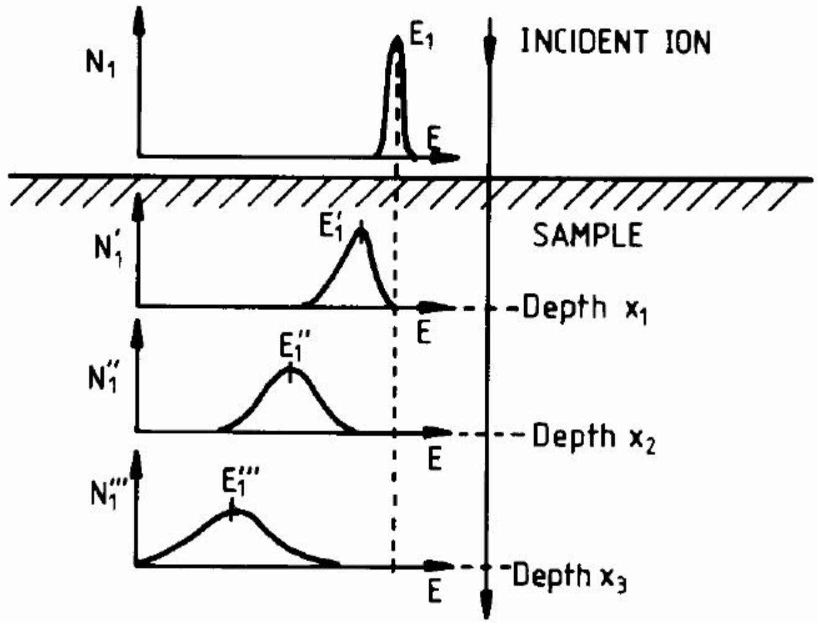
⇒ **energy straggling**

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 $\Delta x$: $\langle E \rangle = E_0 - S\,\Delta x$

$\langle E \rangle$ - mean energy; s - stopping power

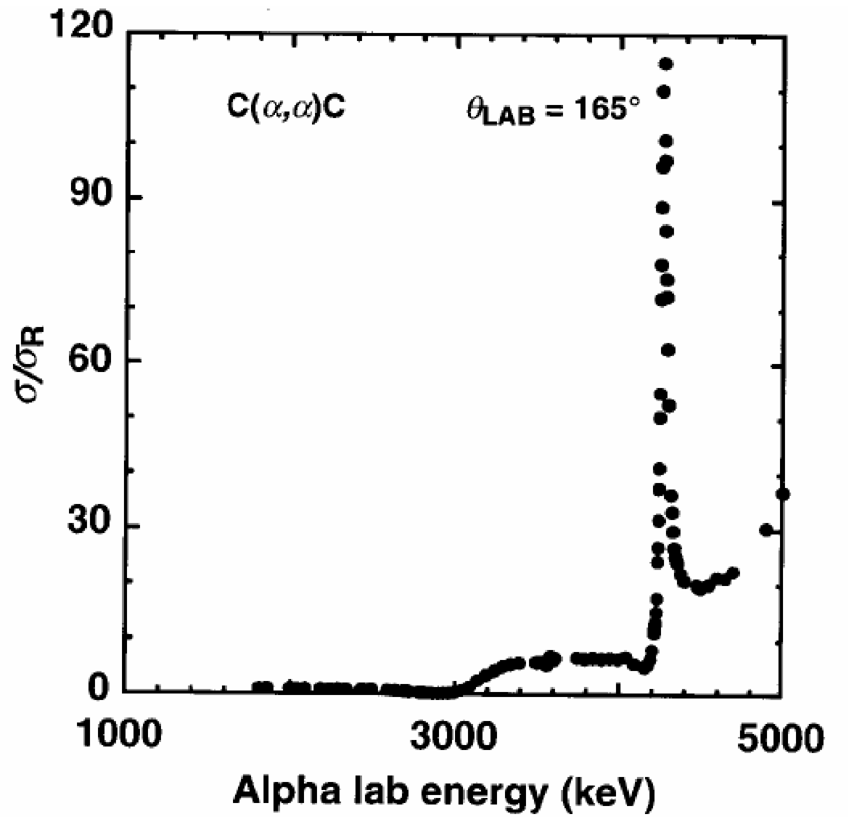
**⇒ only applicable for mean energy of many particles**

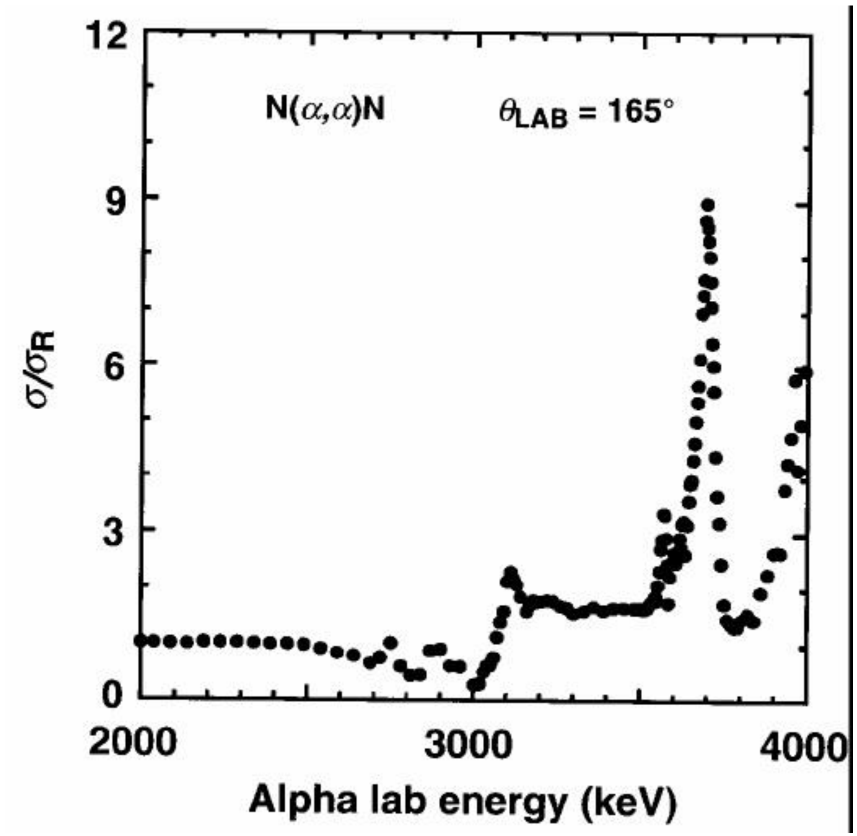


# Non-Rutherford cross sections

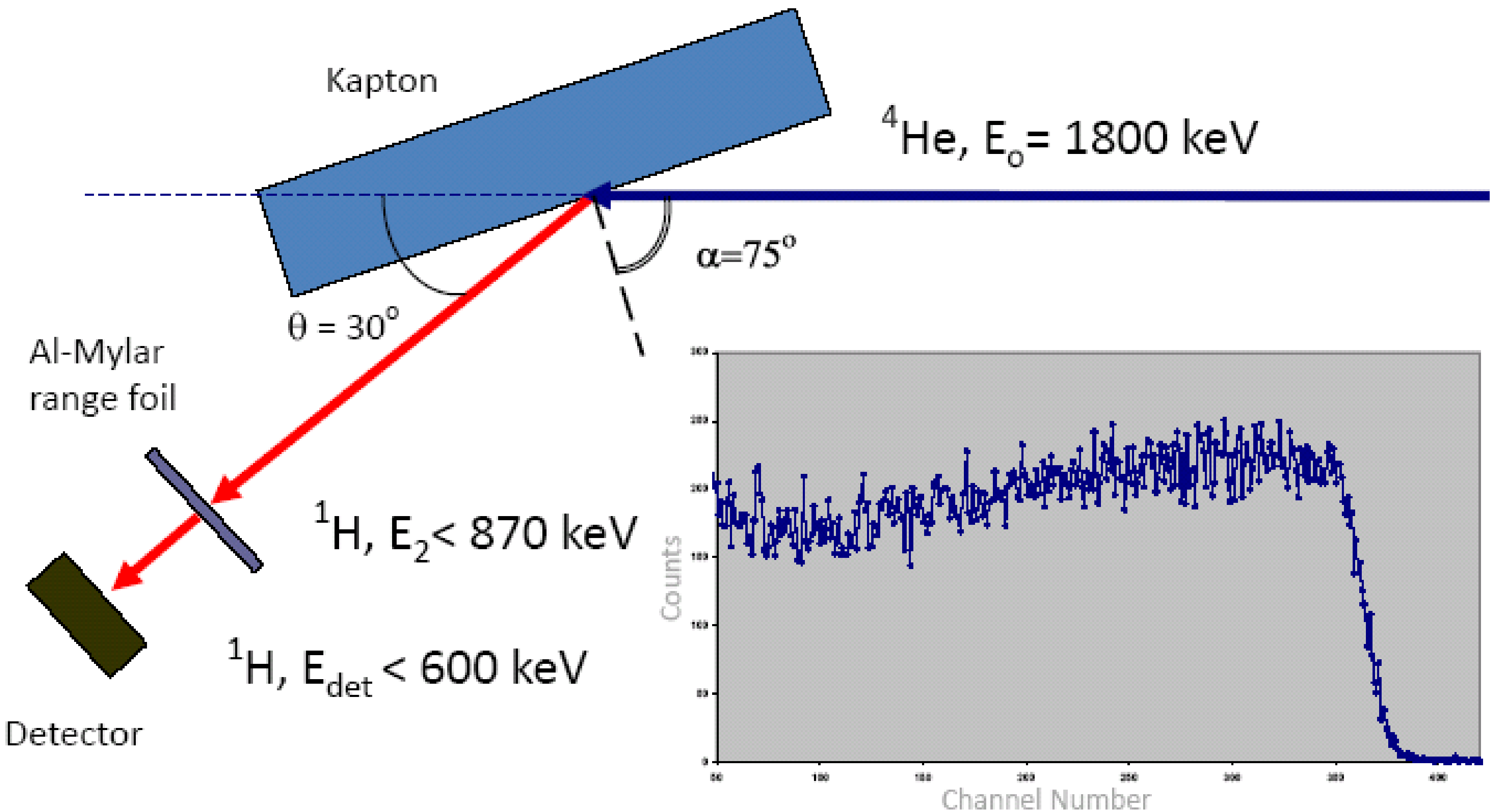
Typical problem for light elements: overlap with thick layers of heavier elements

⇒ **High cross sections wishful!!!**



# Elastic Recoil Detection (ERD)



# Part II: Treatment of experimental data

**To do list:**

- Download and install SIMNRA (use 26100 to register)
- File: “follow.xls”
- Data file for SiSb standard
- Data file for unknown sample
- Data files for your know samples

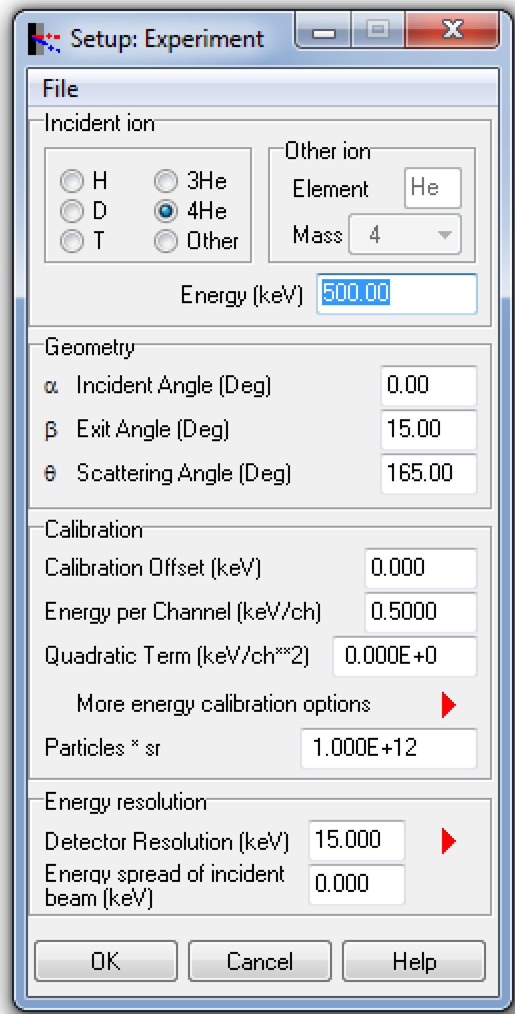
1. Download SIMNRA 6.0 software from

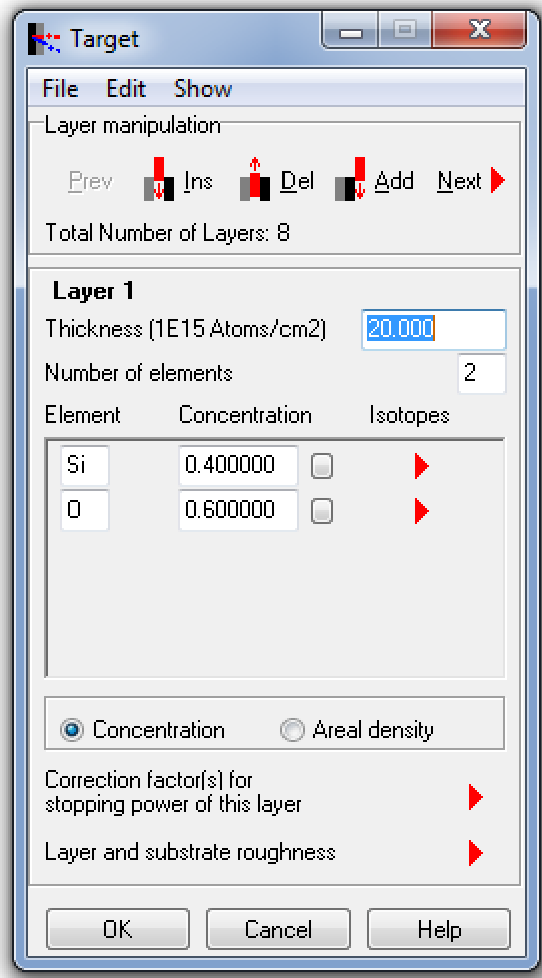
http://home.rzg.mpg.de/~mam/Download.html

2. Install it (as  a Demo version) on your laptop.

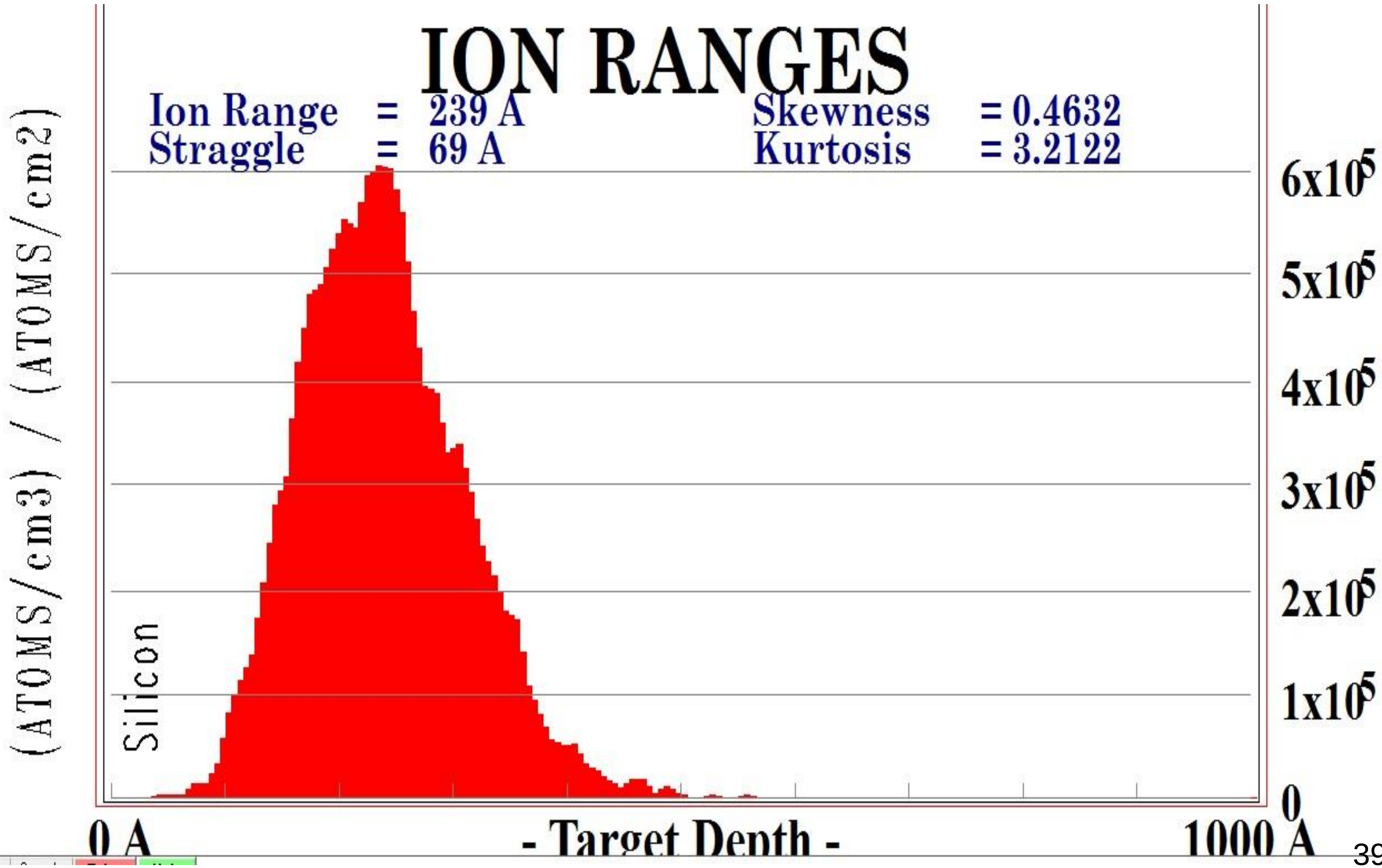
3. After installation, run the program, go to Help -> Register, and enter "26100" as a registration code.

# SIMNRA main menu



# SiSb standard



# Pitfalls in RBS data analysis

- Some trivial examples: common pitfalls and their origin
- Simulation codes and user knowledge
- Over-interpretation and under-interpretation
- What to do? – the best and the worse practices

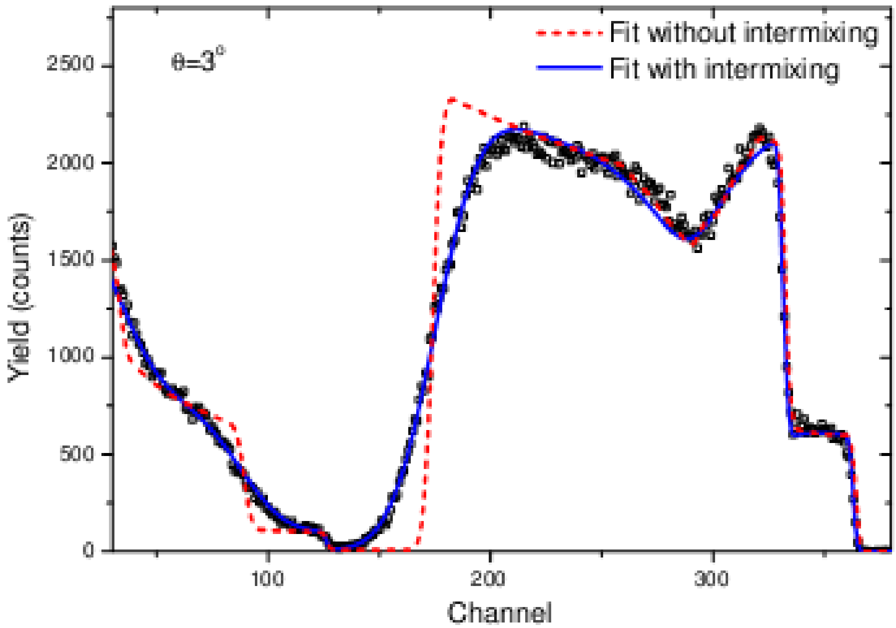
# Trivial pitfalls 1: assumptions about experimental parameters

- Scattering angle
- Angle of incidence
- Solid angle, beam fluence/flux
- Energy calibration

**How to solve/to remedy the problem:**

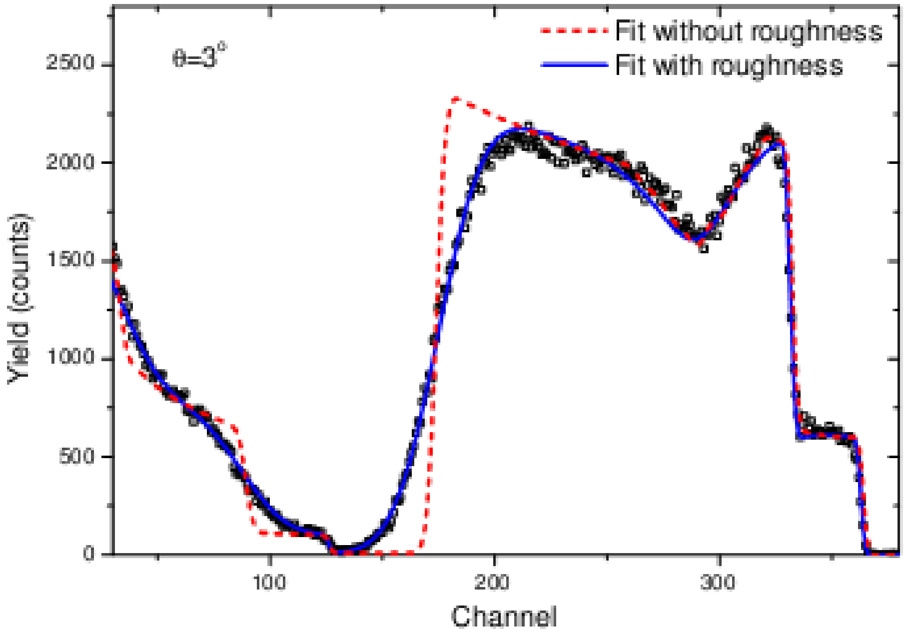
- Acquire data at 2-3 different detector scattering angles, and find consistent positions
- Treat experimental parameters as fitting parameters
- If possible – find a channeling/major crystallographic direction and relate it to the incident angle
- Collect more data with different ion doses
- Measure independent calibration standards

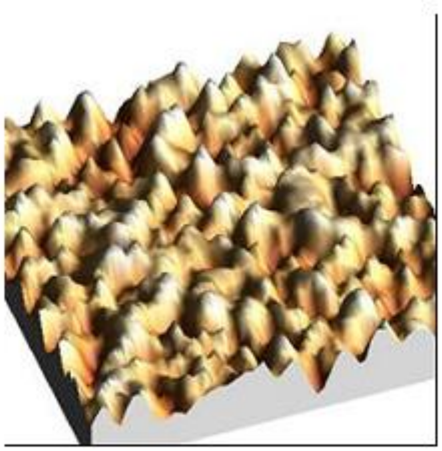
# Layer mixing: GaInN/GaN on $Al_2O_3$




- Film interacts with the substrate leading to extensive intermixing

# Roughness: GaInN/GaN on $Al_2O_3$



- Very few samples have atomically flat surface
- Very often "diffusion" or "mixing" is actually roughness
- Lateral homogeneity has to be proven with other methods than IBA

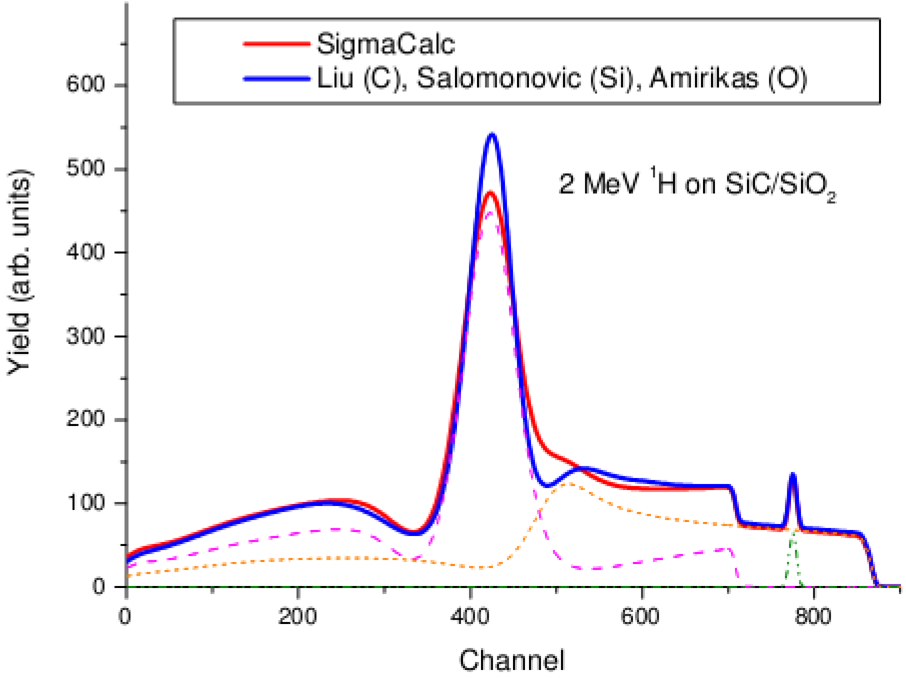
# Trivial pitfalls 2:
# Assumptions about the sample

- Roughness
- Missing element -ignored or falsely postulated
- Postulate your favorite depth profile

**How to solve/to remedy these problems**:

- Use complementary techniques
- Measure samples are different energies
- Keep an open mind?

# RBS: $SiO_2$ on SiC




- Make sure to use right cross sections in the non-Rutherford regime

# Trivial pitfalls 3: assumptions about the basic data used

- Scattering cross sections in the non-Rutherford regime
- Stopping powers (including use of Bragg rules)

**How to solve/to remedy these problems:**

- Try simulations with different cross sections
- Accept lower accuracy than achievable (often the only practical alternative)

# Less trivial data analysis pitfalls

**Over-interpretation**

- Analyst unjustifiably imposes a given model on the data, when other models would also lead to an equivalent or better solution

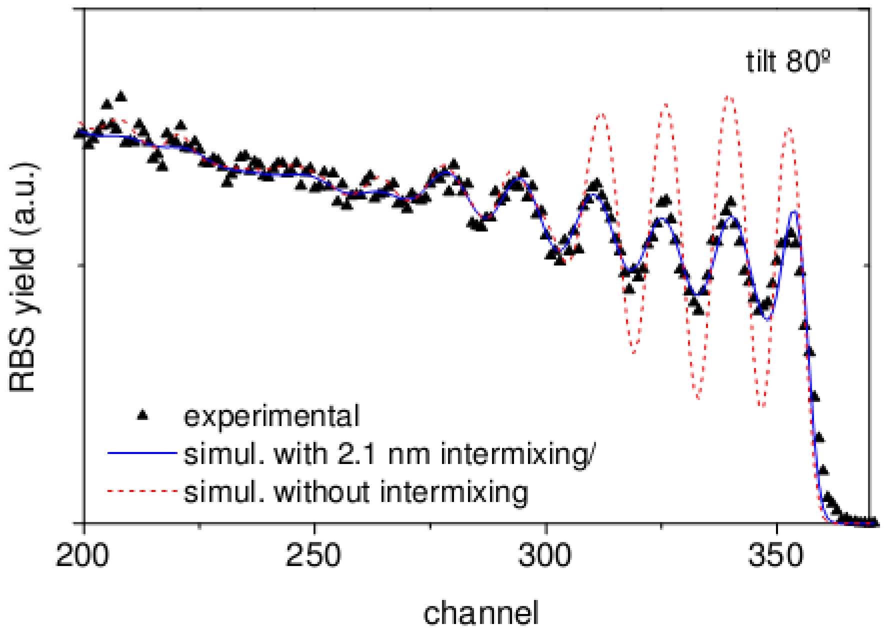
**Under-interpretation**

- Analyst does not extract all information that could be extracted, often due to lack of knowledge or lack of proper code/cross section

**In general**

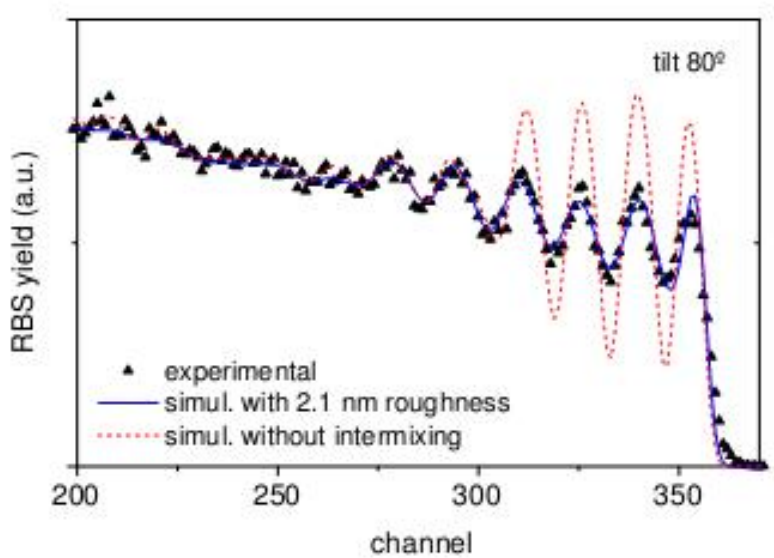
- A good fit does not mean analysis is reliable
- Good use of a suitable code requires extensive knowledge

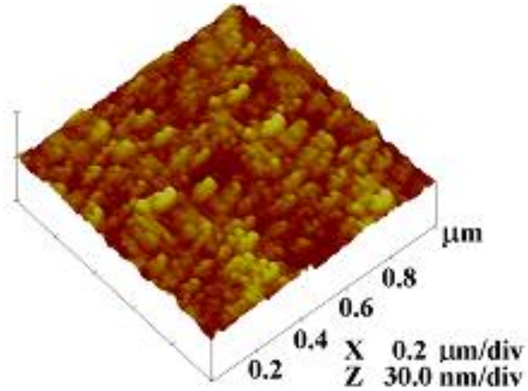
# Over-interpretation

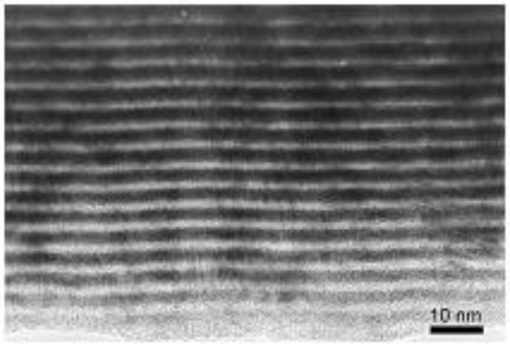



- Nominally ($Ti_{0.4}Al_{0.6}N$ 5 nm/Mo 5nm) ×50
- Simulation consistent with extended layer intermixing

# Under-interpretation: same sample




- AFM and TEM for few samples
- Reasonable report quantifies roughness for all the samples

# The best and the worse practices

- Trust a computer

 - all codes have limitations, sometimes severe

 - the more advanced features you need, the more knowledge you need to justify using them

- Take the stopping powers and cross sections for granted

 - you must be aware of the values used in your analysis

- Codes are a tool

 - you are the analyst: know your system, know your parameters