

## 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

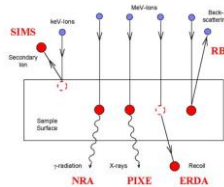
### Part II Treatment of experimental data

#### 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

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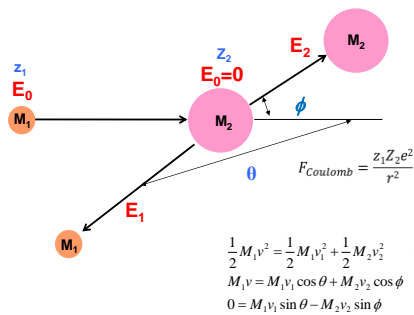
## Part I: Ion-solid interactions



- (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

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## Elastic Collisions



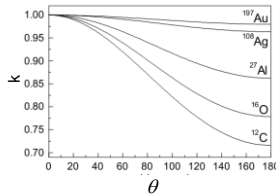
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## Kinematic Factor, k

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:

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

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



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

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## Advantages of Ion Beams

- Can be used for material modification and analysis

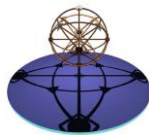
- Mass Specific  
- Kinematic factor

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

- Cross sections are very well known

- Good depth resolution
- Penetrating (can access buried interfaces)

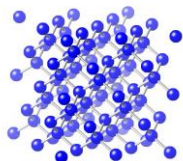
- What about substrate?  
- can use channeling and blocking effects



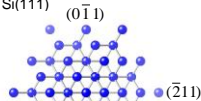
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## Ion channeling and blocking

Si (diamond structure)



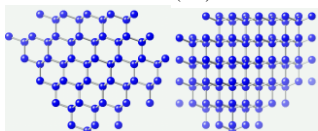
- Si(111) (011)



(011)

- Si(111) - side view

(211)



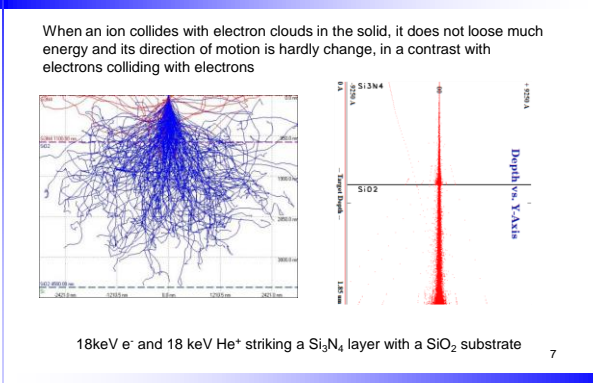
## Electrons vs Ions

When an ion collides with electron clouds in the solid, it does not lose 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

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



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

http://www.srim.org/ =>Download SRIM-2013

SRIM Main Menu

Calculation: 0

Log: 1

**The Stopping and Range of Ions in Matter**

**Stopping / Range Tables** ?

**TRIM Calculation** ?

**Experimental Stopping Powers**

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SRIM Version 5988-2008.03

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*Contributions by H. Paul, D. J. Matwick, G. A. Cuomo, W. A. Porter, S. A. Harrison  
(c) 1984, 1993, 1998, 2003, 2006 by J. F. Ziegler, M. D. Ziegler, J. P. Biersack (SRIM.com)*

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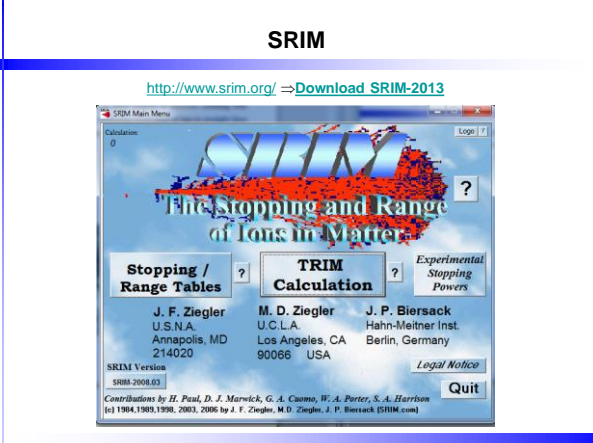
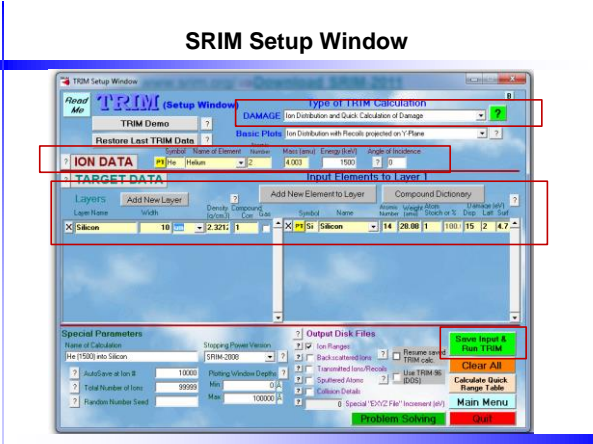
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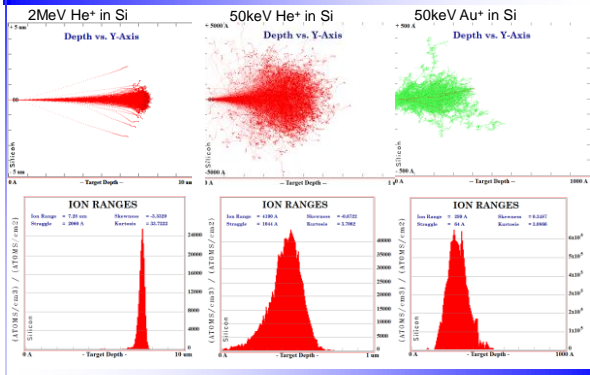
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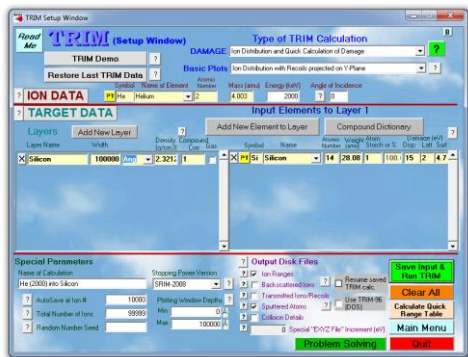
*Contributions by H. Paul, D. J. Matwick, G. A. Cuomo, W. A. Porter, S. A. Harrison  
(c) 1984, 1993, 1998, 2003, 2006 by J. F. Ziegler, M. D. Ziegler, J. P. Biersack (SRIM.com)*

[illegible]

## Calculated Ion Trajectories

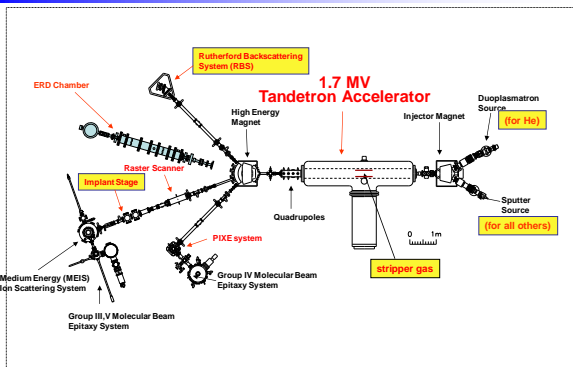


## 2MeV He<sup>+</sup> in Si



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## Tandem 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  $E_i \equiv V_t$  [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  $\mu m$
- 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

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## 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:  $\sim 2\mu m$  for He ions
- **Very sensitive** for heavy elements:  $\sim ppm$
- **Less sensitive** for light elements  $\Rightarrow$  **ERD**

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## History

Sir Ernest Rutherford (1871 - 1937)

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



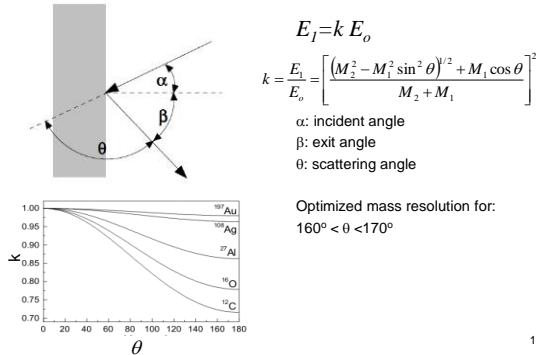
RBS as materials analysis method

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

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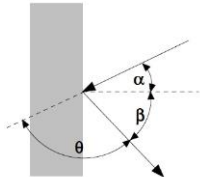
## RBS: Scattering geometry and kinematics



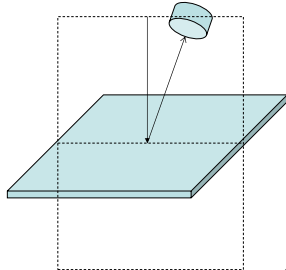
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## Scattering Geometry

IBM geometry

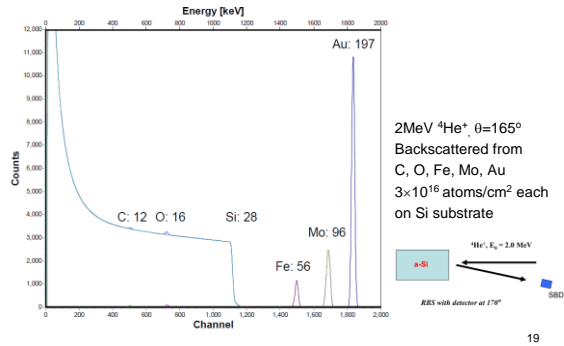


Cornell geometry → Choose this one for RBS Tandemtron experiments



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## Scattering kinematics: example 1



## Key features of RBS

Ability to quantify depth profile of buried species with a precision of  $\sim 3\%$

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

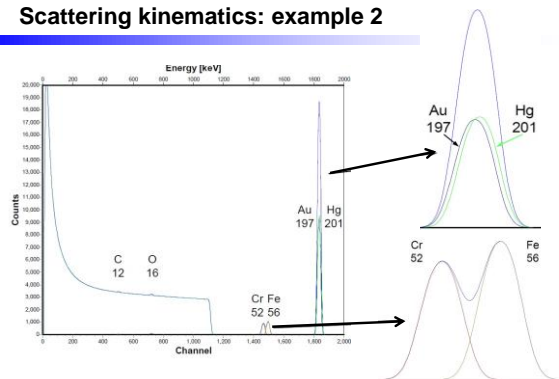
$$k = \frac{E_1}{E_0} = \left[ \frac{(M_2^2 - M_1^2 \sin^2 \theta)^{1/2} + M_1 \cos \theta}{M_2 + M_1} \right]^2$$

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

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

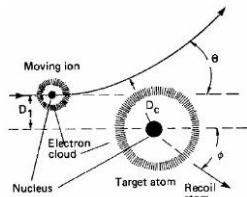
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## Scattering kinematics: example 2



⇒ 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
- ⇒ Rutherford scattering cross section

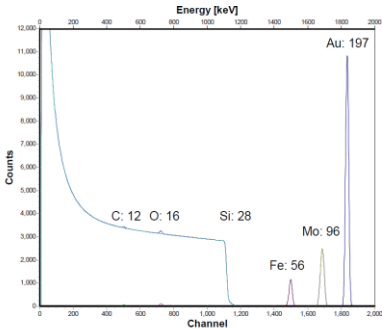
$$\frac{d\sigma}{d\Omega} = \sigma(\theta) = \left( \frac{Z_1 Z_2 e^2}{4 E \sin^2 \left( \frac{\theta}{2} \right)} \right)^2$$

Note that sensitivity increases with:

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

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Scattering kinematics: example 1



2MeV  $^4\text{He}^+$ ,  $\theta=165^\circ$   
Backscattered from  
C, O, Fe, Mo, Au  
 $3 \times 10^{16}$  atoms/cm<sup>2</sup> each  
on Si substrate

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

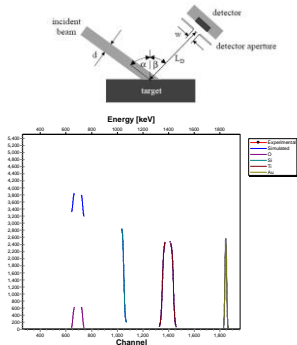
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RBS spectra from thin and thick films

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  $\text{cm}^2/\text{sr}$  of the element
  - well known (tabulated) in Rutherford cross section regime

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Areal density: note about units

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

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

➔  $\frac{N_0 \rho \ t}{M}$  [ $\text{at}/\text{cm}^2$ ]

where  $M = \text{atomic mass [amu]}$ ,  $N_0 = \text{Avogadro's number}$

In absolute numbers – close to thickness in Å

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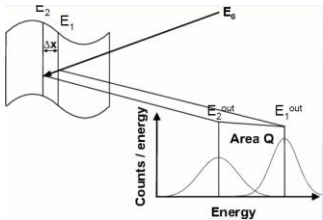
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RBS Spectrum of a thick film

- 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



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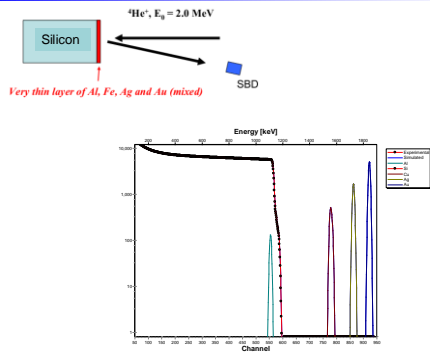
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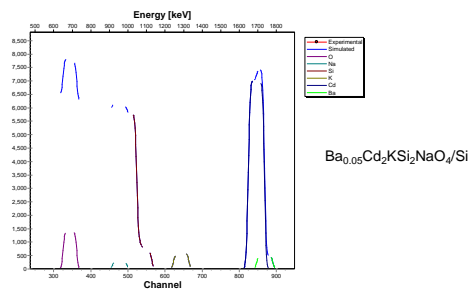
## RBS spectrum: bad choice of the substrate



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## RBS Spectrum: Stoichiometry

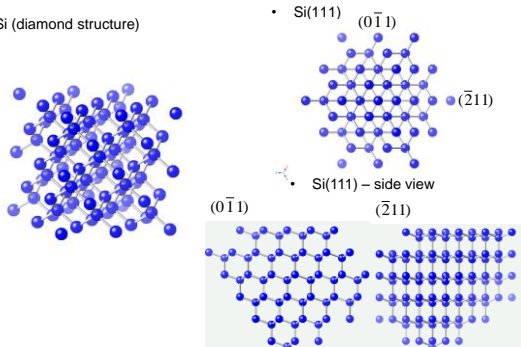
2 MeV  $^4\text{He}^+$ , backscattered from ceramic films on Si substrate



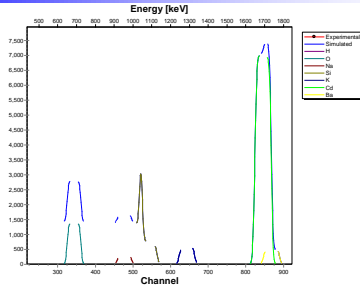
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## Ion channeling and blocking

Si (diamond structure)



## Use crystal structure of the substrate



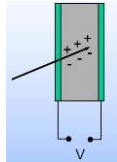
- Substrate can be aligned to a major crystallographic direction to minimize background signal in some cases

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## Silicon detector resolution

### Principle of operation:

- Creation of electron-hole pairs by charged particles
- Separation of electron-hole pairs by high voltage  $V$   
 $\Rightarrow$  Number of electron-hole pairs  $\propto$  Particle energy  
 $\Rightarrow$  Charge pulse  $\propto$  Particle energy



### Limited energy resolution ( $\sim 1\text{keV}/100\text{\AA}$ ) 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

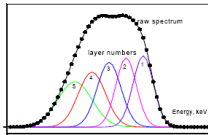
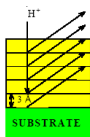
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## Depth resolution and concentration profiling

Depth resolution for  $\approx 100\text{ keV}$  protons (resolution of the detector  $\approx 1\text{keV}$ )

- Stopping power  $\text{SiO}_2 \approx 12\text{ eV/\AA}$ ;  $\text{Si}_3\text{N}_4 \approx 20\text{ eV/\AA}$ ;  $\text{Ta}_2\text{O}_5 \approx 18\text{ eV/\AA}$
- "Near surface" depth resolution  $\approx 50\text{ \AA}$ ; worse for deeper layers due to energy straggling

### Layer model:



- Areas under each peak corresponds to the concentration of the element in a  $50\text{ \AA}$  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.

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## Energy Straggling

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

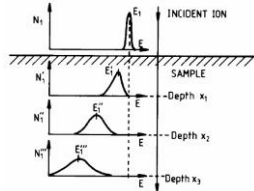
⇒ **energy straggling**

**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**

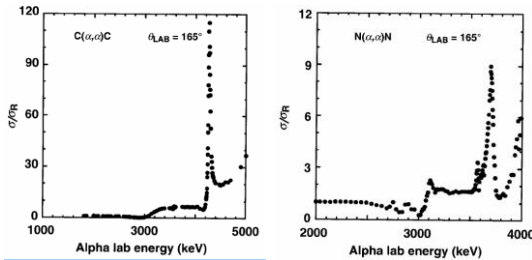


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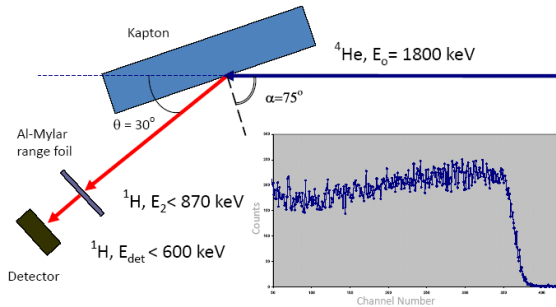
## Non-Rutherford cross sections

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

⇒ **High cross sections wishful!!!**



## Elastic Recoil Detection (ERD)



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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

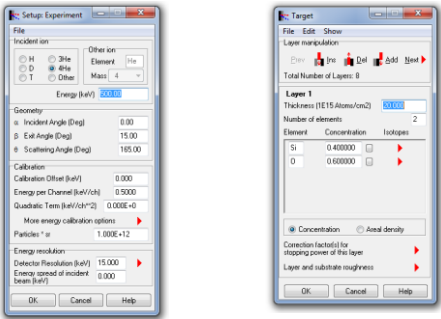
<http://home.rzg.mpg.de/~mam/Download.html>

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.

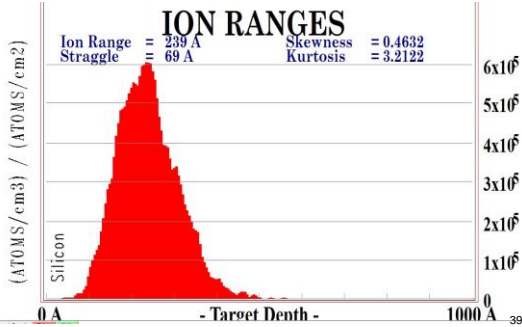
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SIMNRA main menu



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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

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### Trivial pitfalls 1: assumptions about experimental parameters

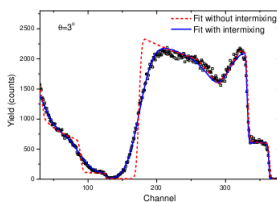
- Scattering angle
- Angle of incidence
- Solid angle, beam fluence/flux
- Energy calibration

#### 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

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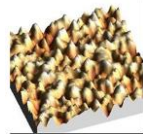
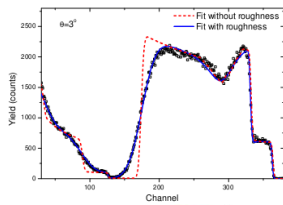
### Layer mixing: GaInN/GaN on $\text{Al}_2\text{O}_3$



- Film interacts with the substrate leading to extensive intermixing

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## Roughness: GaInN/GaN on $\text{Al}_2\text{O}_3$



- 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

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## Trivial pitfalls 2: Assumptions about the sample

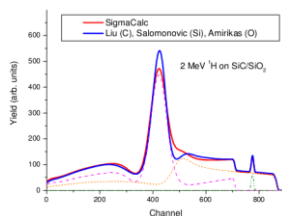
- Roughness
- Missing element - ignored or falsely postulated
- Postulate your favorite depth profile

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

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

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## RBS: $\text{SiO}_2$ on SiC



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

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### 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)

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### 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

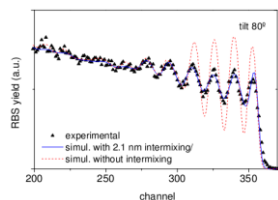
- Analyst does not extract all information that could be extracted, often due to lack of knowledge or lack of proper code/cross section

#### In general

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

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### Over-interpretation



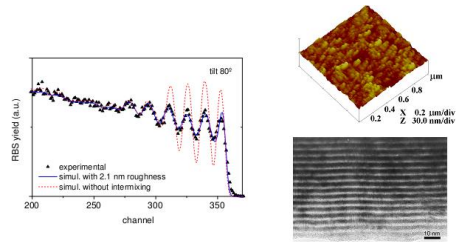
➤ Nominally ( $\text{Ti}_{0.4}\text{Al}_{0.6}\text{N}$  5 nm/Mo 5 nm) x50

➤ Simulation consistent with extended layer intermixing

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## Under-interpretation: same sample



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

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## 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

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