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DEGLI STUDI
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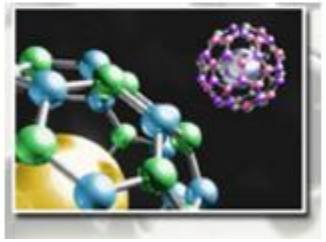
PHYSICS and
ASTROPHYSICS



CHEMICAL and
MATERIAL SCIENCES

Ion Beam Based Techniques for Materials Science

Teachers:
Paolo Olivero, Ettore Vittone
Physics Department, University of Torino
www.solid.unito.it

2 CFU : 8 h

Tuesday 2.5.2017, h. 16-18, Aula Verde, Physics Dept. - Ettore Vittone
Wednesday 3.5.2017, h. 16-18, Aula Wick, Physics Dept. - Ettore Vittone
Thursday 4.5.2017, h. 16-18, Aula D, Physics Dept. - Paolo Olivero
Friday 5.5.2017, h. 16-18, Aula Verde, Physics Dept. - Paolo Olivero

CAMPUSNET WEB SITE:
http://dott-scm.campusnet.unito.it/do/corsi.pl/Show?_id=j56m

Final exam:
Brief seminar (15 min) on a topic relevant to Ion Beam Based Techniques

Introduction

These lectures will deal with

Ions $\rightarrow$ **Charged particles**
Light Ions (^{1}H and ^{4}He)
MeV light ions
MeV light ions currents/fluxes/fluences

Ion energy (E) $\rightarrow$ E=e·V $\rightarrow$ electrostatic accelerator

Example:
12 MV accelerator voltage
$E(^{1}H^{+}) = 12$ MeV
$E(^{4}He^{++}) = 24$ MeV

Ion current: I=Q/t; Q=charge=N_{Ion}·e·n; (n=Ion charge state)
Ion flux: Φ=N/t·A; A=unit area
Ion Fluence: $F=\int_0^t \Phi dt$

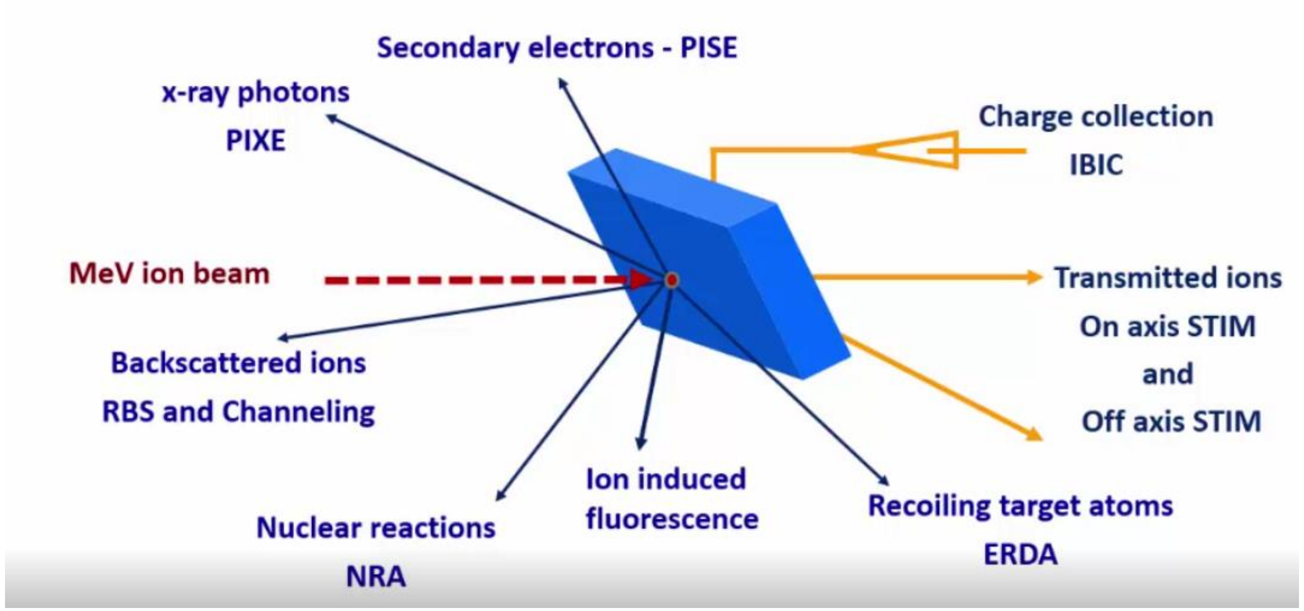
Example:
I=1 pA
If $^{1}H^{+}$: n=1; $10^{-12}/1.6\cdot10^{-19}$ ions/s = $6\cdot10^{6}$ ions/s
If $^{4}He^{++}$: n=2; $2\cdot10^{-12}/1.6\cdot10^{-19}$ ions/s = $12\cdot10^{6}$ ions/s

MeV Ion Beam Techniques in Material Science

From the many interactions that MeV ion beams create in a sample

we can extract information on the structure/composition/functionality of the material

Ion Beam Analysis



http://www.ciba.nus.edu.sg/pages/gallery.html

we can modify the structure/composition/functionality of the material

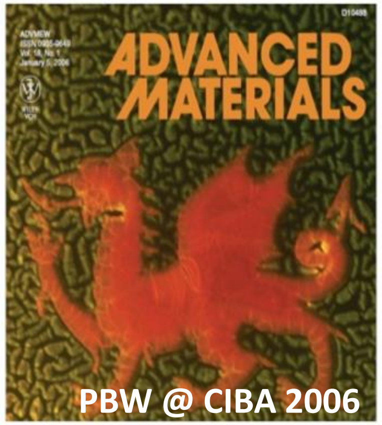
Ion Beam Material Modification

Ion micromachining

Defect Engineering

Deep Ion Beam Lithography

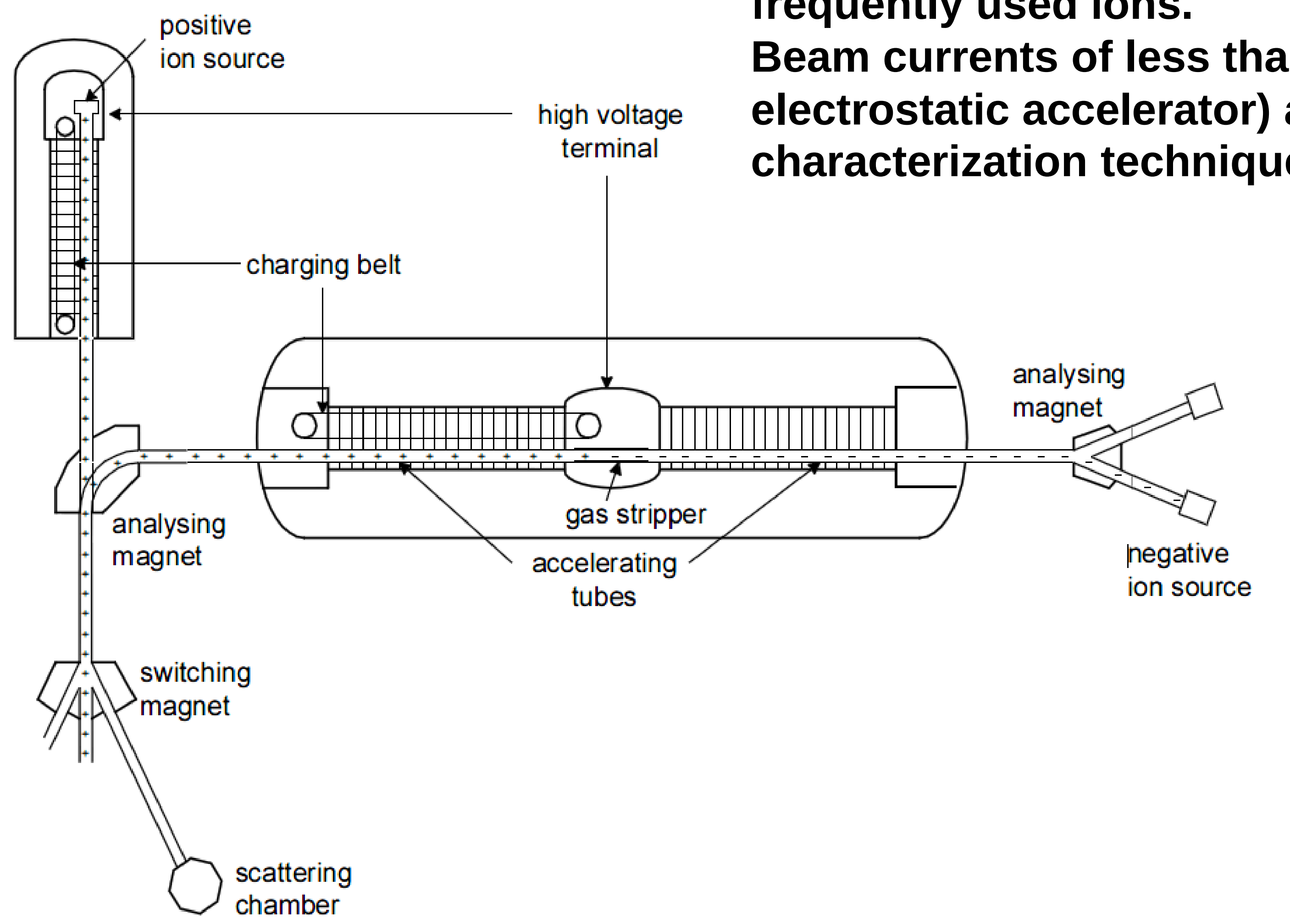
Proton Beam Writing



- Introduction
- Instrumentation
  - Accelerators
  - Ion sources
  - Detectors
- Ion-Solid interaction
  - Electronic energy loss
  - Nuclear energy loss
- IBA techniques
  - Compositional analysis
  - Structural analysis
  - Functional analysis

Instrumentation

The most common type of accelerators used for the application of ion beam analysis Techniques are the electrostatic accelerators. Protons and alpha in the energy range 1–3 MeV are the most frequently used ions.
Beam currents of less than 1 μA (easily delivered by the typical electrostatic accelerator) are sufficient for all existing characterization techniques.



Instrumentation for PIXE and RBS; IAEA-TECDOC-1190
http://www-pub.iaea.org/books/iaeabooks/5966/Instrumentation-for-PIXE-and-RBS

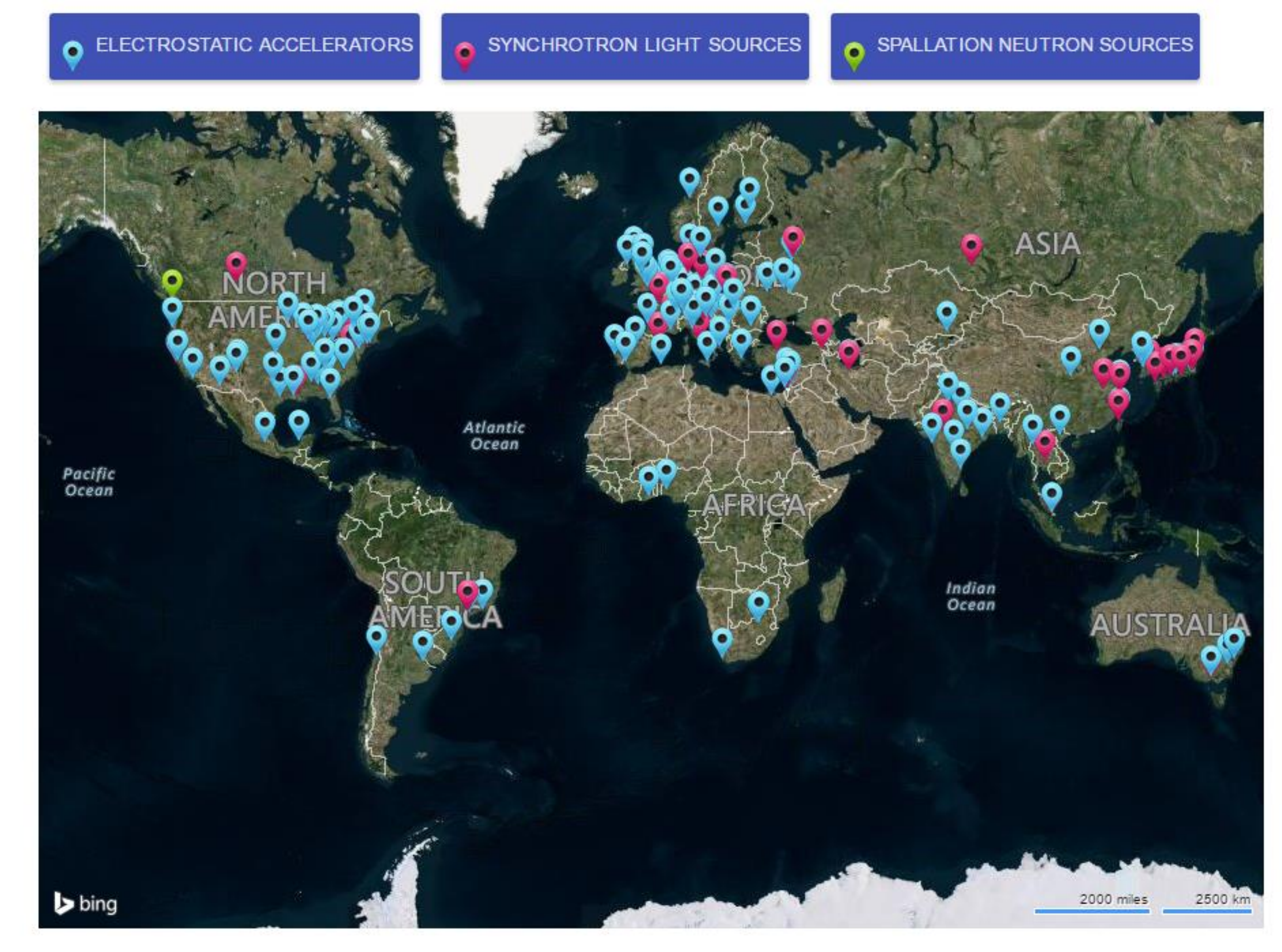
Instrumentation

IAEA.org NUCLEUS

IAEA Accelerator Knowledge Portal

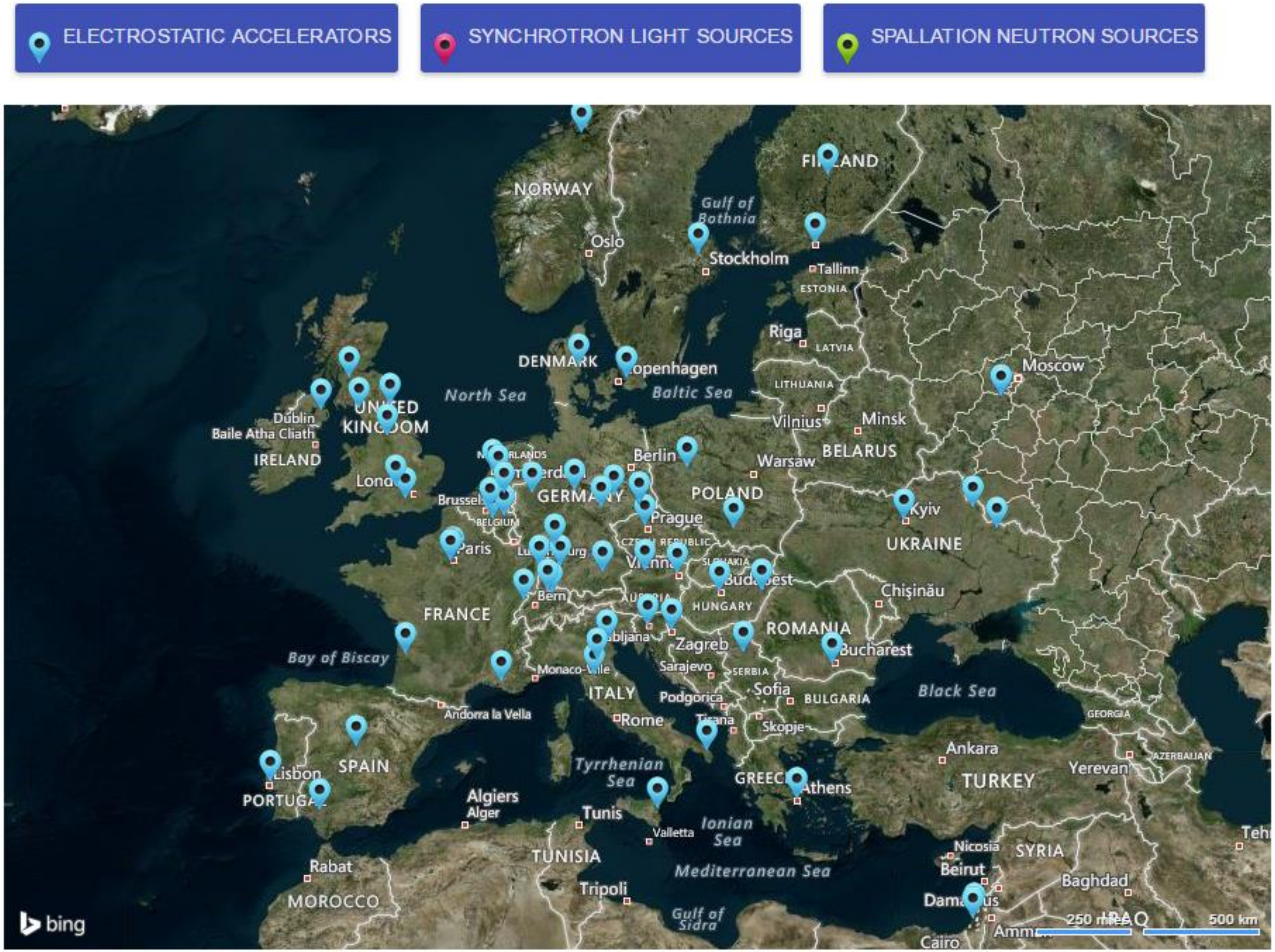
https://nucleus.iaea.org/sites/accelerators/Pages/default.aspx

More than 200 ion beam facilities in the world

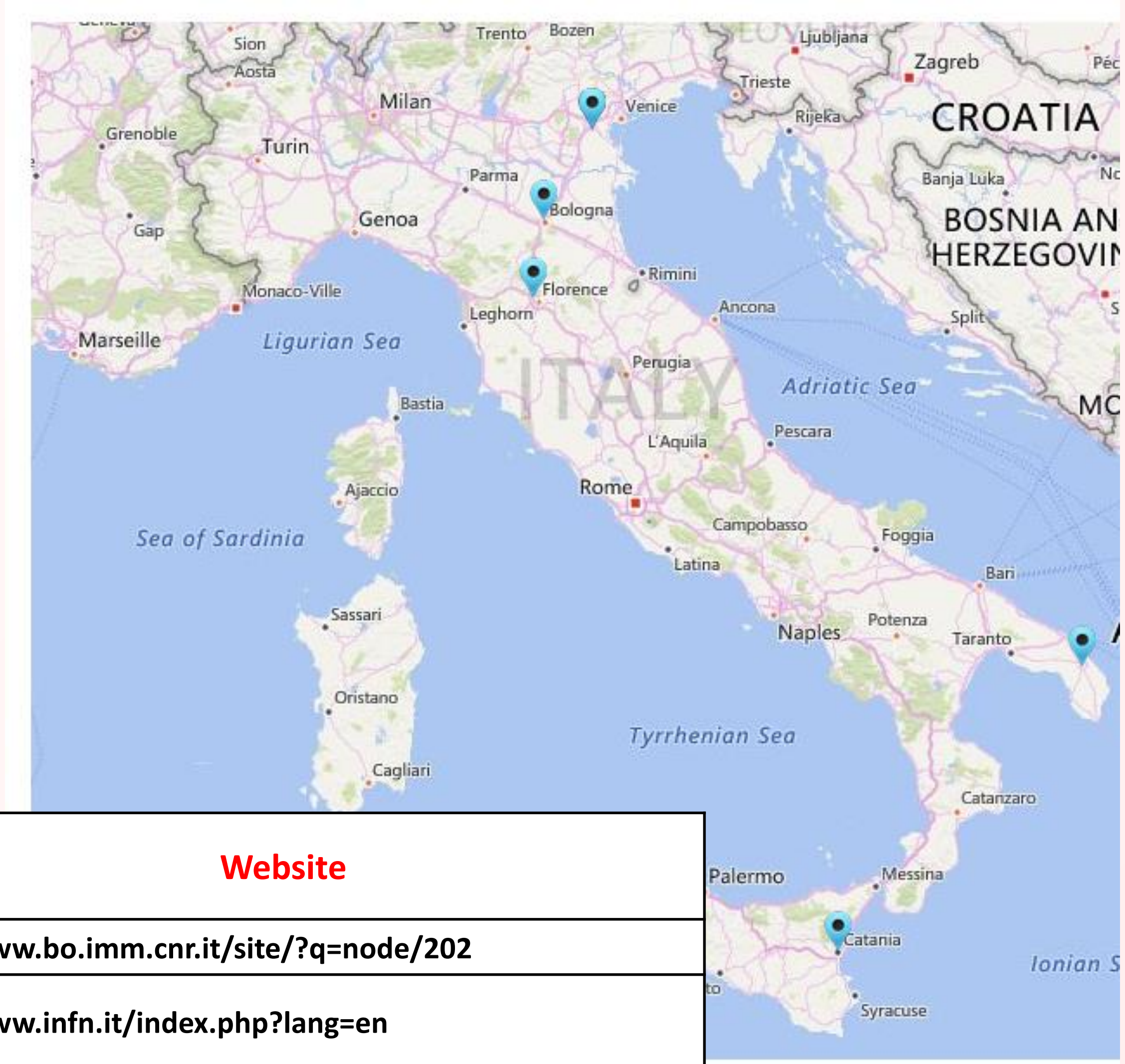


Instrumentation

92 ion beam facilities in Europe-Ukraine-Russia-Israel

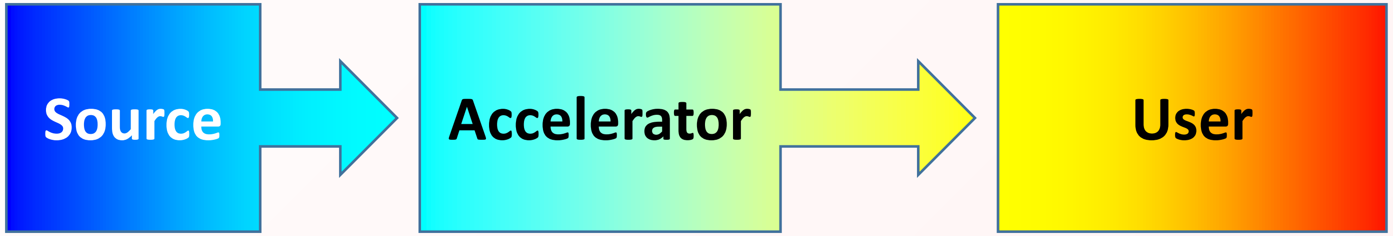


5 ion beam facilities in Italy



Facility Name	City	Accelerator Type	Accelerator Description	Terminal Voltage (kV)	Website
CNR-IMM	Bologna	Tandem	Other	1700	http://www.bo.imm.cnr.it/site/?q=node/202
INFN-Instituto Nazionale di Fisica Nucleare	Florence	Tandem	Dynamitron	3000	http://www.infn.it/index.php?lang=en
INFN.Laboratori Nazionali di Legnaro	Legnaro	Single-ended Single-ended Tandem	Van de Graaff Van de Graaff Other	7000 2500 15000	http://www.lnl.infn.it/accelerators/accelerators.html
University of Catania	Catania	Single-ended	Dynamitron	3100	http://www.unict.it/en/
University of Lecce	Lecce	Tandem	Dynamitron	3000	http://www.unisalento.it/web/guest/home_page

Schematic View of the principal component of an ion accelerator system



A **source of charged particles**

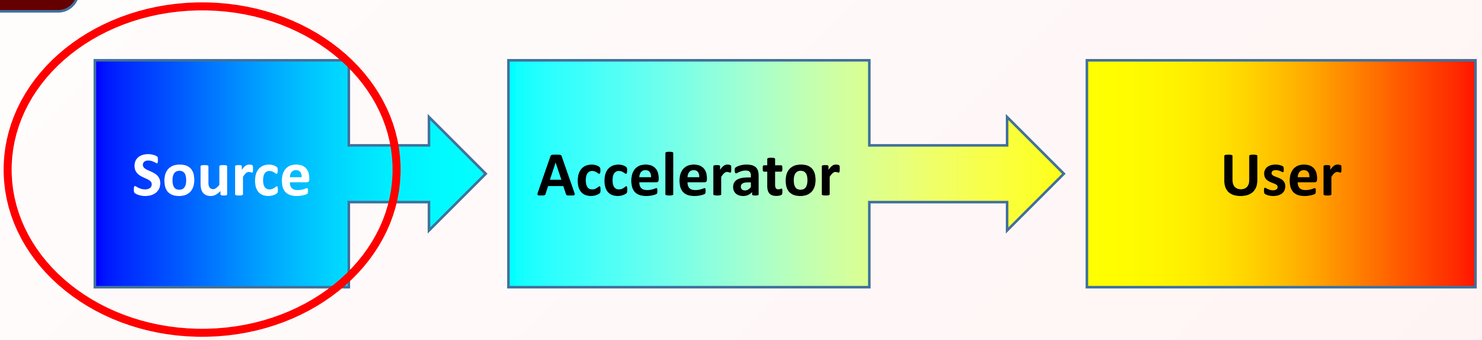
Accelerating elements
Electrostatic column which provide the electric fields giving the energy to the ions

Beam guiding elements, mainly magnetic, in order to maintain (focus) the beam on the wanted trajectory

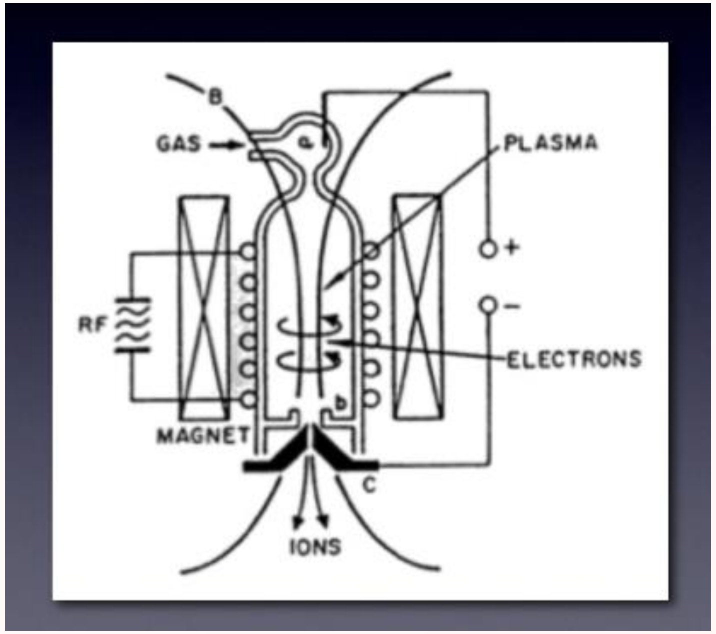
Ancillary systems:
High vacuum is needed to avoid perturbation of the beam by collisions with residual gas
Beam diagnostics assure the monitoring of the beam trajectories

User installation
Experimental set-ups including
Targets, spectrometers, detectors

Schematic View of the principal component of an ion accelerator system



Positive ion: RF ion source

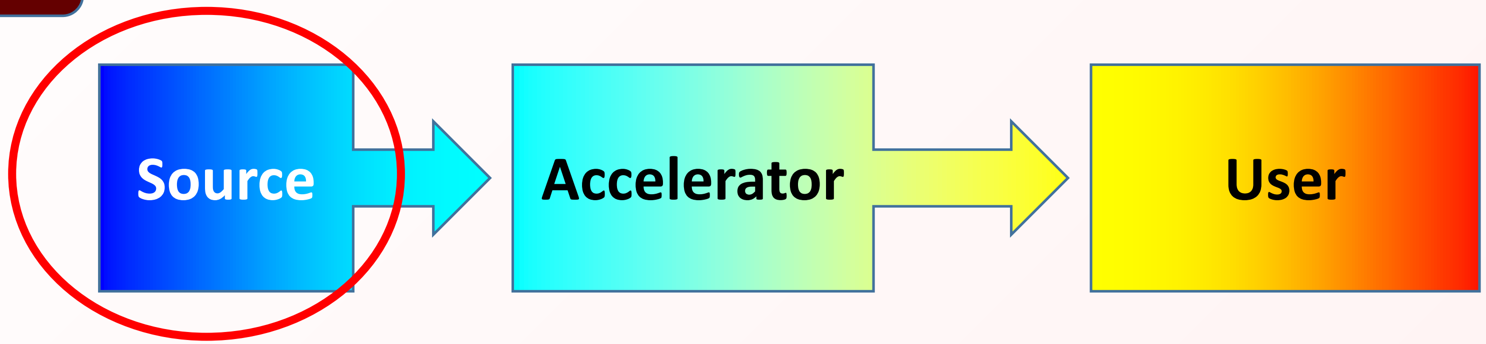


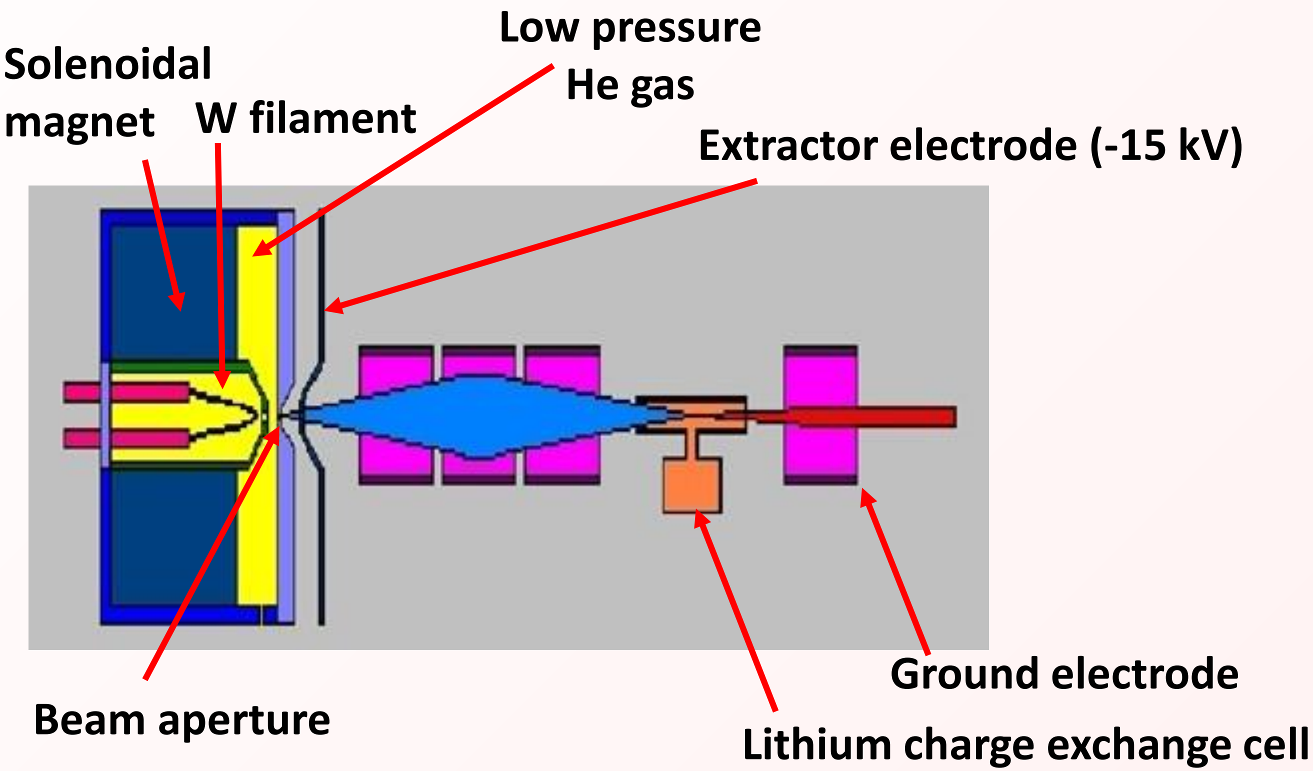
- The RF (Radio Frequency) ion source is a bottle which contains a gas (typically $^{1}H_2$ or ^{4}He), which is excited by an RF oscillator capacitive coupled to the bottle.
- The plasma is confined and positioned by an axial magnetic field.
- A small positive bias voltage across the ion source bottle propels the positive ions out through the canal.
- Typically 10 μA beam current
- Longevity of the source used in single ended machine

High Voltage Engineering Europa B.V

Instrumentation

Schematic View of the principal component of an ion accelerator system

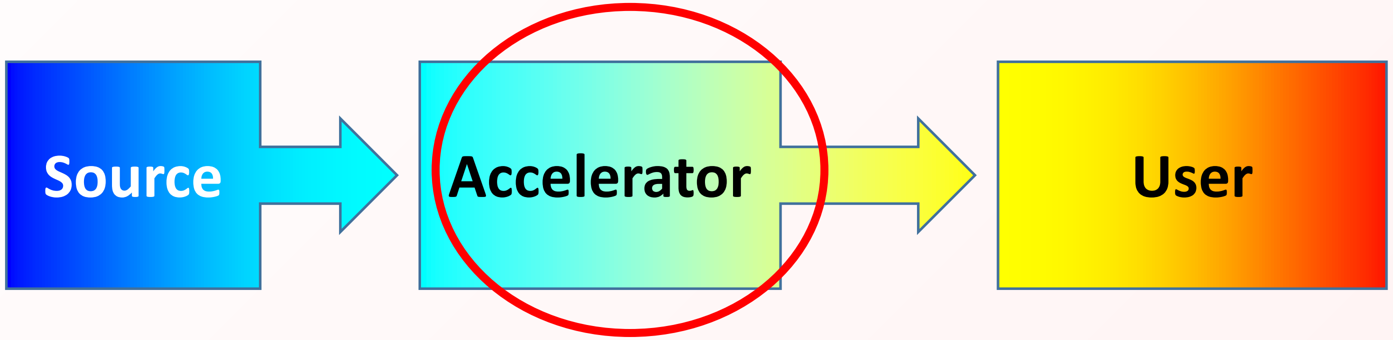




Negative ion: Duoplasmatron ion source

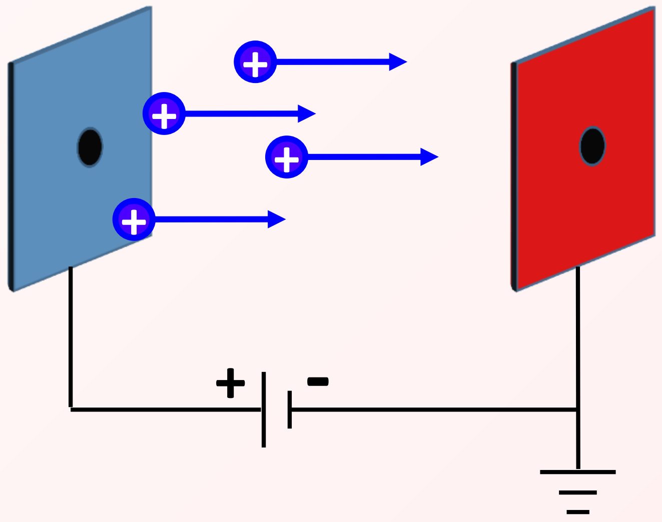
- Positive ions are generated by ionization induced by electrons produced by thermoionic emission from hot W filaments. A plasma is then generated and confined by a solenoidal magnetic field.
- The positive ion are accelerated by the extractor electrode.
- The positive ions are focused into a cell where metal Li (or Rb) is heated to form a vapor.
- The Li vapor charge exchanges with the positive ions, giving up electrons to the beam particles.
- Negative ions are accelerated out of the charge exchange cell.
- Positive ion current: 100 μA→Negative ion production efficiency: 2%

Schematic View of the principal component of an ion accelerator system



Accelerating elements

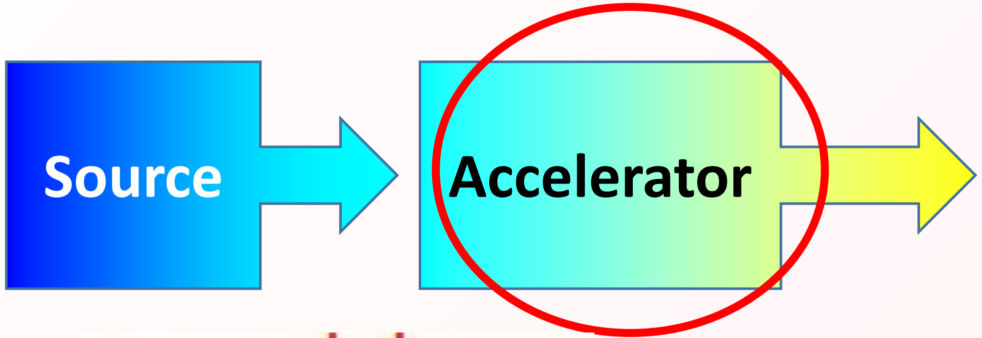
- ✓ Electrostatic column which provide the electric fields giving the energy to the ions
- ✓ An ion source injects the charged particles
- ✓ Between the entry and the exit, a continuous high voltage is applied, mediated by intermediate electrodes for a smooth and regular increase of the electric field



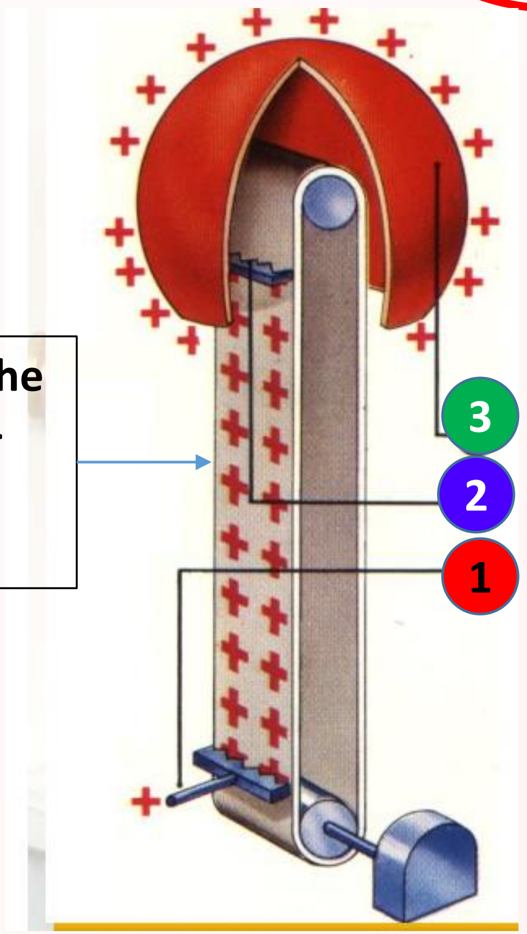
A.C. Mueller, IAEA School on ADS, Trieste, 19-30/10/2007

Instrumentation

Schematic View of the principal component of an ion accelerator system



High voltage for electrostatic acceleration
(Van de Graaff method)



The conveyor transports the charges inside the sphere-shaped terminal, which forms a Faraday cage

3 The charges accumulate on the outside of the sphere and the inside get charge free, ready to accept further charging

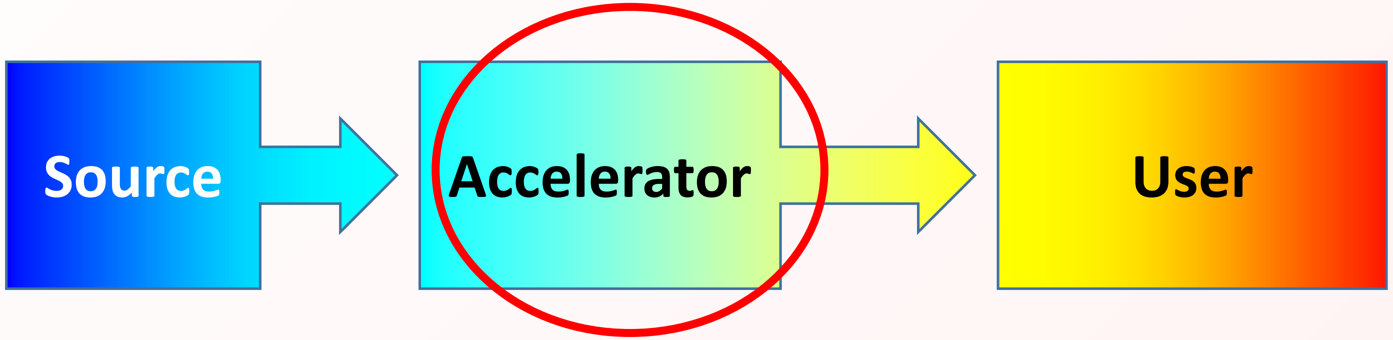
2 The charges are collected by a second, comb-like electrode, which is connected to the sphere

1 A comb-like electrode sprays charges on an insulating conveyor belt

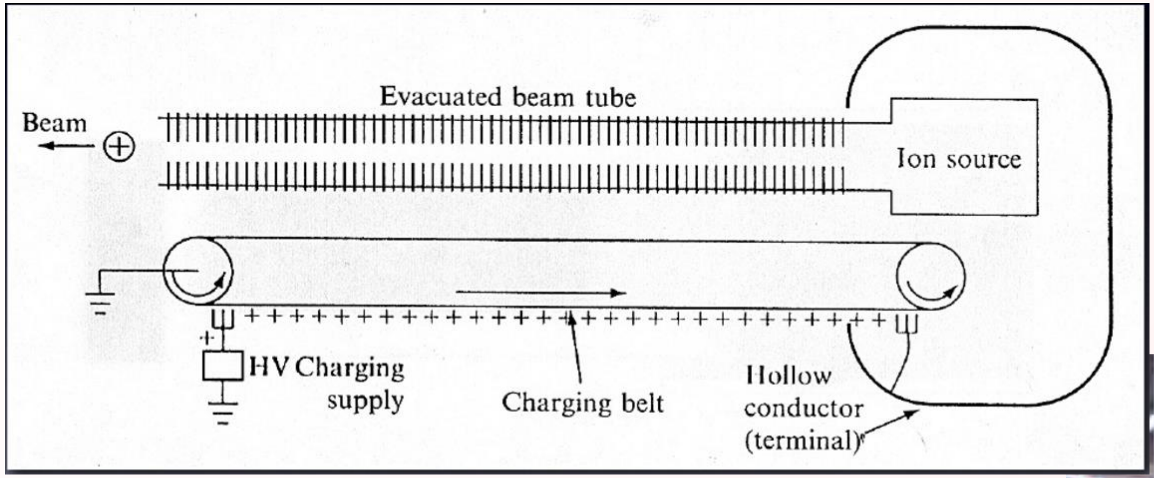
In practice, one can reach up to 25 MV, provided one uses SF6 gas for limiting breakdowns

Instrumentation

Schematic View of the principal component of an ion accelerator system



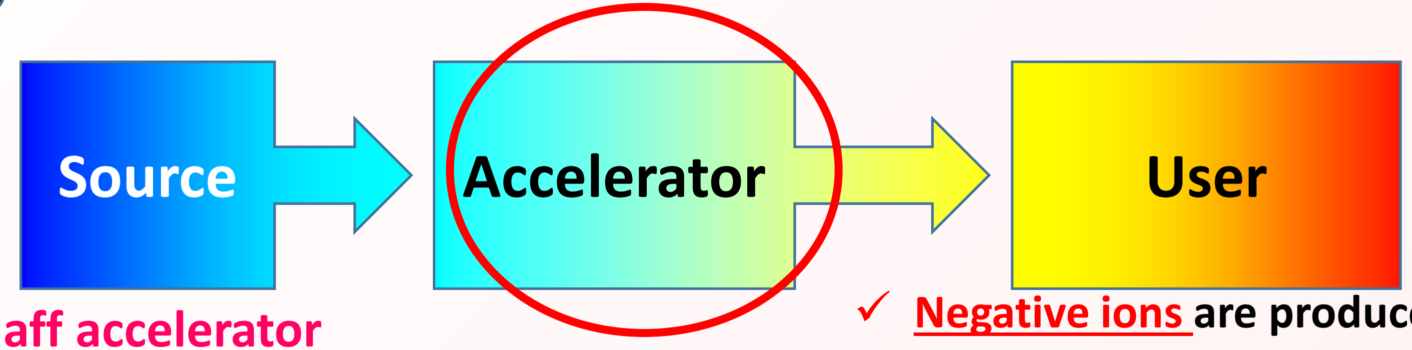
Single ended Van de Graaff accelerator



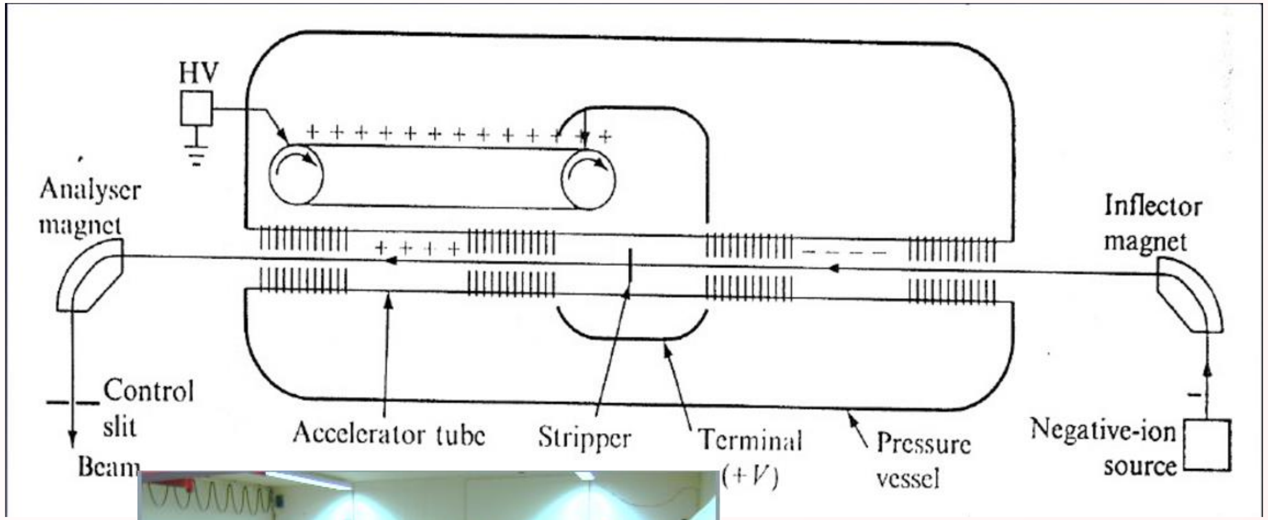
The positive ion source is inside the high voltage terminal
The voltage is distributed uniformly between the terminal and the ground by a chain of high voltage resistors.
The chain operates inside a vessel, pressurized with an insulating gas (SF6) to aid in reducing ionization and arc-over.

Instrumentation

Schematic View of the principal component of an ion accelerator system



Tandem Van de Graaff accelerator



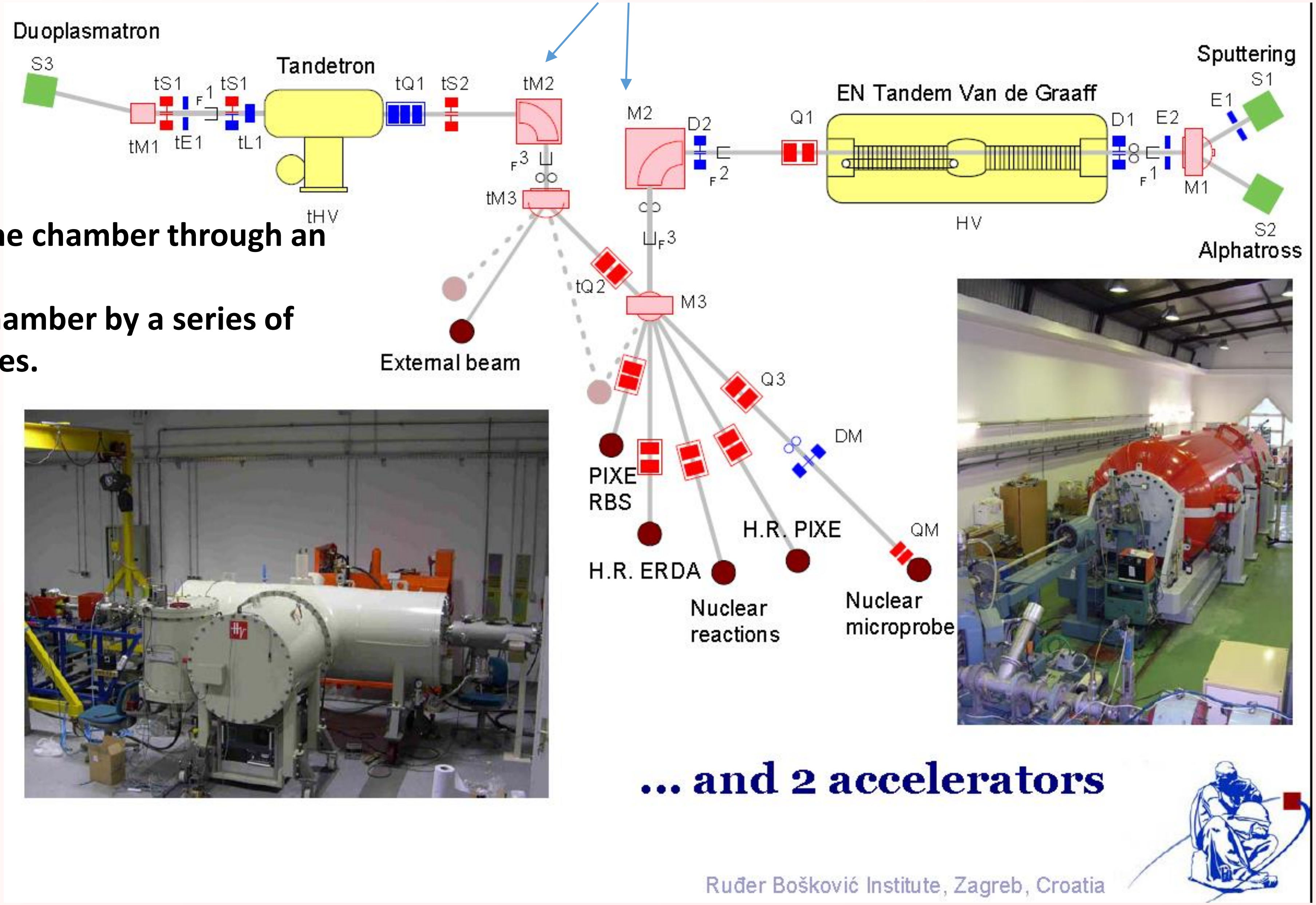
Bochum

- ✓ Negative ions are produced at ground potential and accelerated to the positive terminal located midway along the tube structure.
  - ✓ Source not inside the terminal
  - ✓ Limited range of ion available
- ✓ Inside the terminal, ions encounter either athin carbon foil or tenuous gas (typically N2).
- ✓ Ions are stripped of electrons through collisions within the stripper region and emerge with positive charge.
- ✓ Because the terminal is positive, they are further accelerated.
- ✓ The total energy they acquire is (n+1)V, where n is the final charge state of the ions and V is the terminal voltage.
- ✓ Tandem accelerators can produce ion beams that are far more energetic than those available from single ended accelerators.

Instrumentation

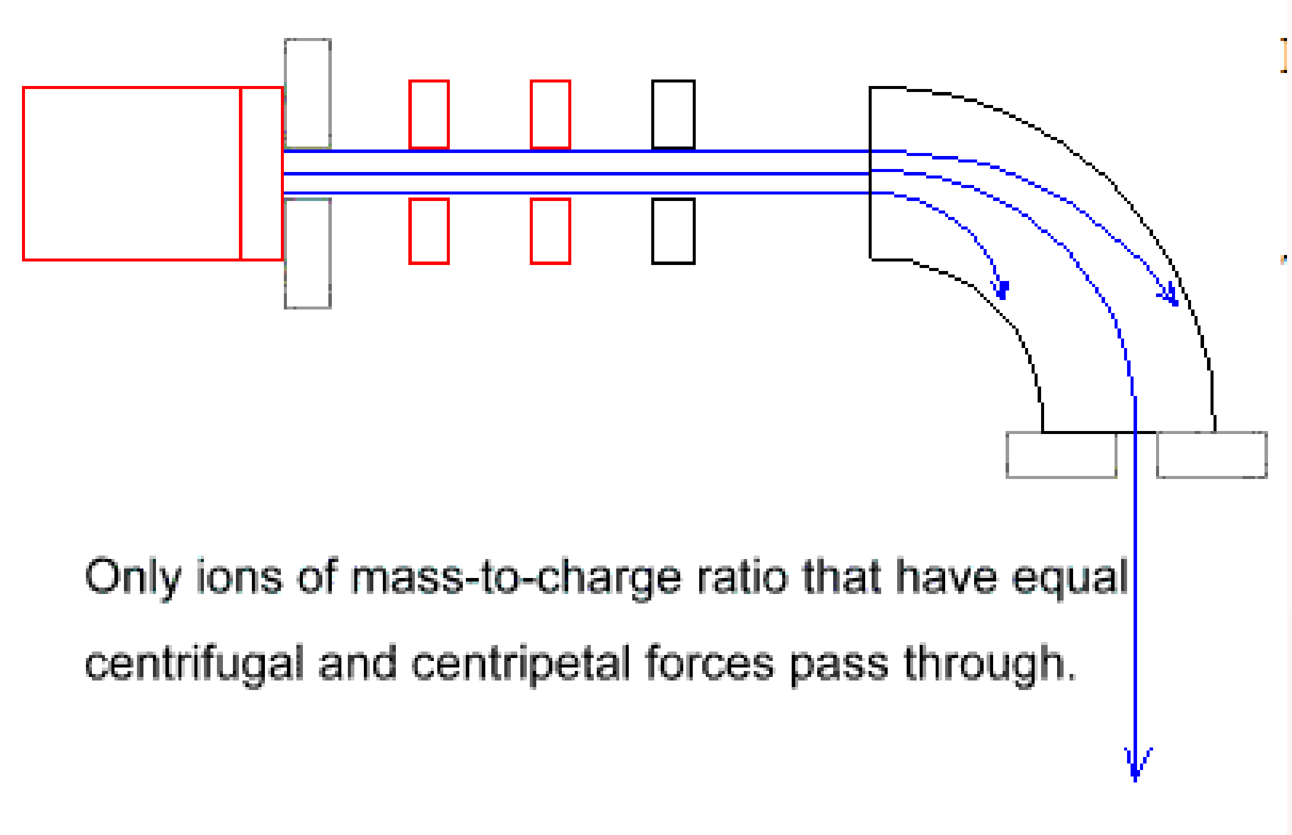
Analyzing magnets

The beam is transported to the chamber through an evacuated pipe
The beam is steered to the chamber by a series of electric and/or magnetic lenses.



Instrumentation

The analyzing magnet



http://www.casetechnology.com/implanter/magnet.html

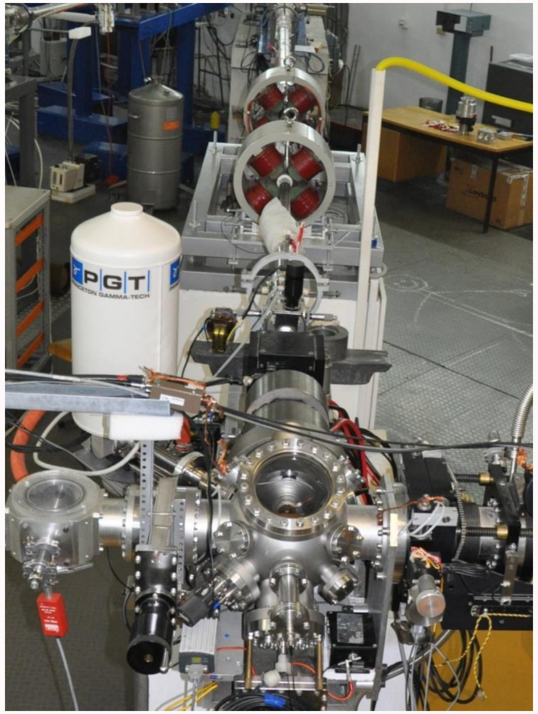
Filters the ions by the momentum per unit charge or, equivalently, by the magnetic rigidity

B = magnetic induction
M = particle mass
E = particle energy
e = particle charge
R = magnet radius of curvature

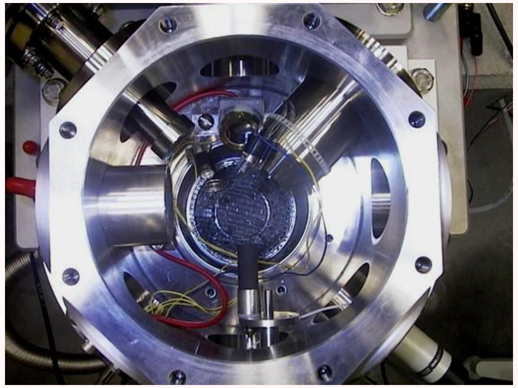
$$\mathbf{B \cdot R} = \sqrt{\frac{\mathbf{2ME}}{\mathbf{e^2}}}$$

Schematic View of the principal component of an ion accelerator system

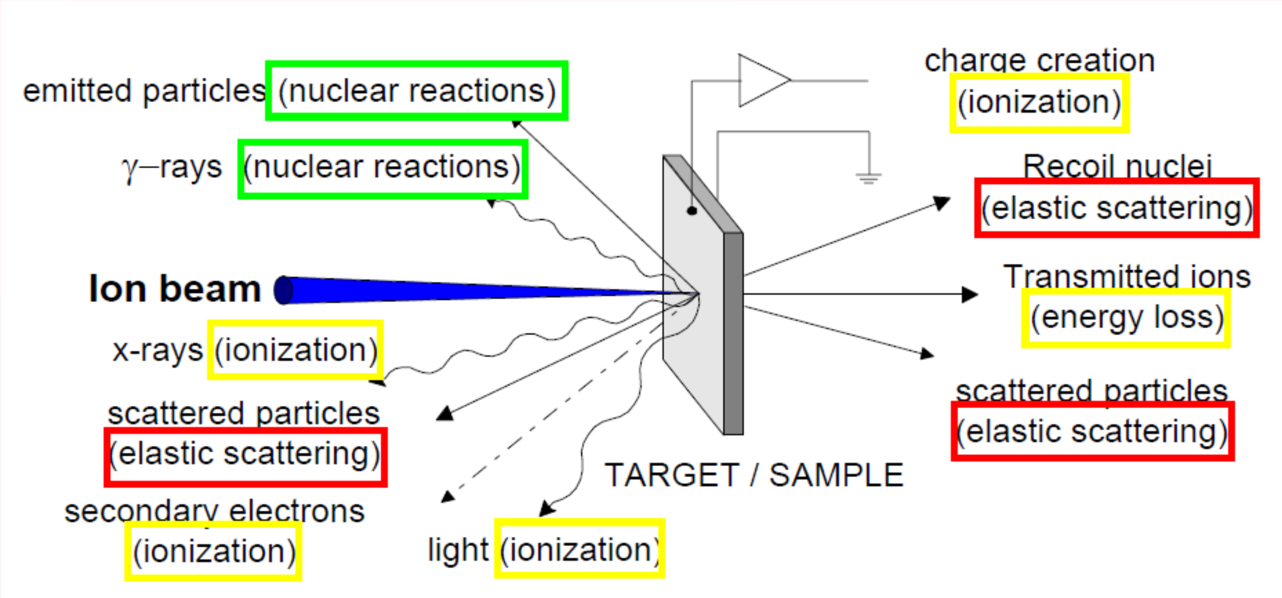
Source → **Accelerator** → **User**



Ruder Boskovic Institute, Zagreb (HR)



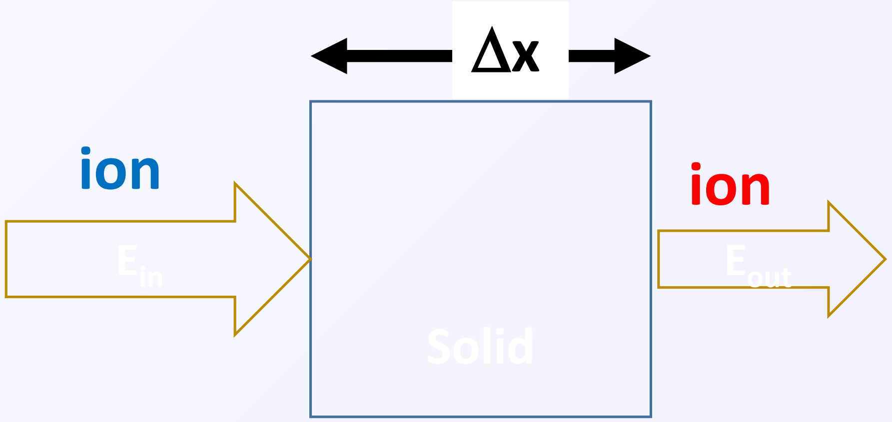
Centro Nacional de Aceleradores, Sevilla (E)



Many interactions → different detection techniques and instrumentation

Ion-solid interaction

Basic concepts



The incoming ion interacts with the atoms in the target and loses energy via collisions with atomic electrons and nuclei
Energy transfer -> ion slowing until they come to rest at some depth in the material

Energy loss per distance Δx traversed: $\Delta E = E_{in} - E_{out}$

Stopping Power or Specific Energy Loss: $S = \lim_{\Delta x \to 0}\left(\frac{\Delta E}{\Delta x}\right) = \frac{dE}{dx}$ **Units:** $\left(\frac{keV}{\mu m}\right)$

Mass Stopping Power or Linear Energy Transfer: $LET = \frac{1}{\varrho}\frac{dE}{dx}$ (ϱ = mass density) **Units:** $\left(\frac{MeV \cdot cm^2}{g}\right)$

Stopping Cross section : $\epsilon = \frac{1}{N}\frac{dE}{dx}$ (N = atomic density) **Units:** $\left(\frac{eV}{10^{15} atoms/cm^2}\right)$

Example: Si; M_m=28 g/mol; ρ=2.3 g/cm^3; $N = N_A \cdot \rho / M_m = 5 \cdot 10^{22}$ atoms/cm^3
3 MeV He ions:
$S = 200\,\frac{keV}{\mu m}$; $S_M = 858\,\frac{MeV \cdot cm^2}{g}$; $\varepsilon = 40\,\frac{eV}{10^{15} atoms/cm^2}$

Ion-solid interaction

- ✓ The fundamental physics of the interactions between incident ions and individual atoms in the solid provides the underlying science for Ion Beam Analysis.
- ✓ Focus on 1-10 MeV ^{1}H and ^{4}He ions, which are the ions most commonly used for analytical methods.
- ✓ The most relevant features are the ways in which MeV ions lose their energy in collisions (energy loss mechanisms).

Radius of atomic nucleus: $r \sim r_0 \cdot A^{1/3}$; $r_0 \sim 1.4\ fm$
Example: $r_{Si} = 4.3\ fm$

Bohr radius: $a_0 = 52910\ fm$
Si atomic radius: $a_{Si} = 111000\ fm$
Si lattice constant: $d_{Si} = 543100\ fm$

Electronic stopping power

Slowing down of a projectile ion due to the inelastic collisions between bound electrons in the medium and the ion moving through it

Nuclear stopping power

Due to elastic collistions between the projectile ion and atomic nuclei in the sample. The interaction is the electrostatic repulsion between the incoming charged ion and the positive nucleus (not the strong nuclear force).

- ✓ Infrequent collision between ion and nucleus
- ✓ Most of the energy of the MeV ion is lost in collision with electrons
- ✓ Electron energy loss is the process that determines the distance ions travel before they come to rest

Ion-solid interaction

Electronic Energy Loss Regime

$$\mathrm{E} = \text{Ion energy}$$

$$\text{v= Ion velocity} = \sqrt{\frac{2 \cdot E}{M_{Ion}}}$$

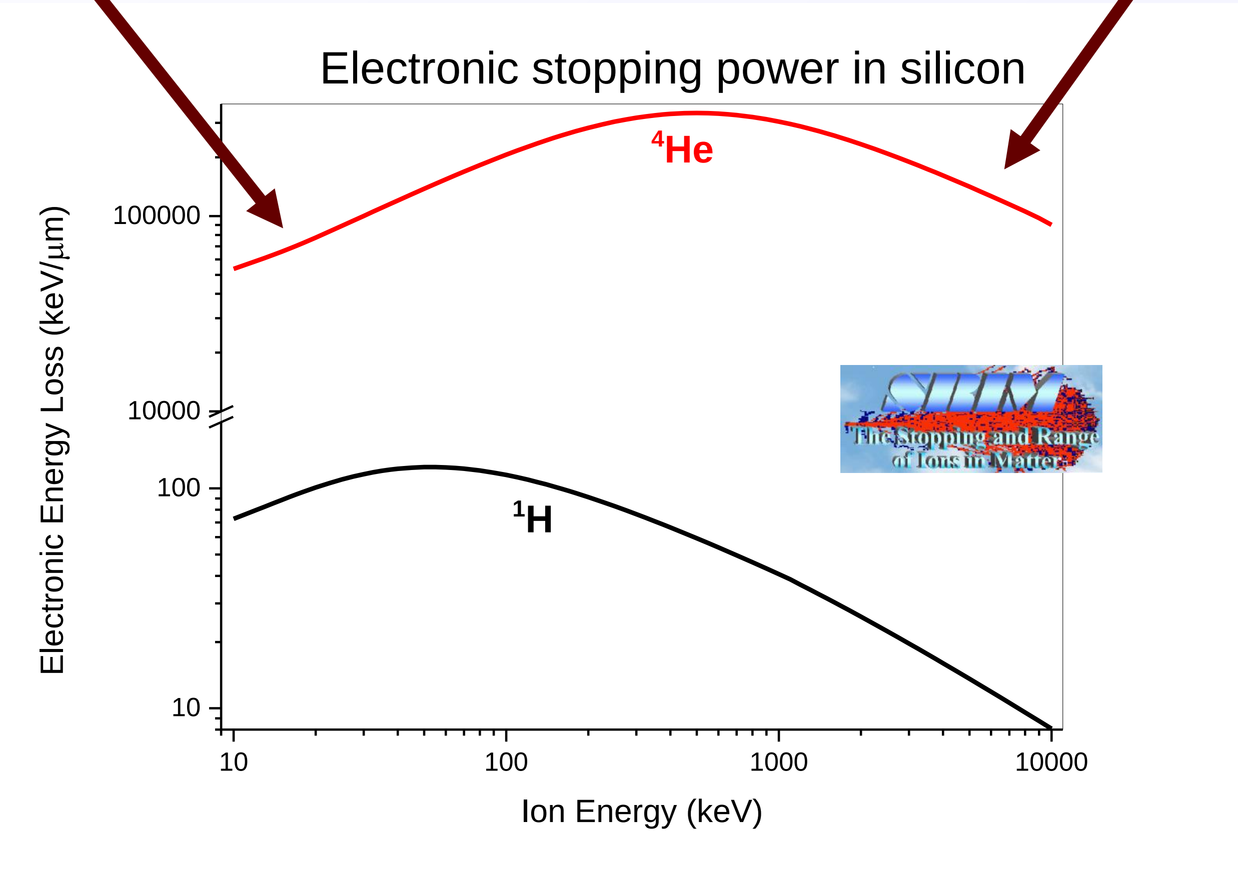
$$v_{low} < v_{Bohr} = \frac{e^2}{4\pi\varepsilon_0\hbar} = \alpha \cdot c \sim 2.2 \cdot 10^6 \frac{m}{s} < v_{High}$$

$$E_{low}(\,^1H) < \frac{1}{2} M(\,^1H) \cdot (v_{Bohr})^2 = 25keV < E_{High}(\,^1H)$$

$$E_{low}(\,^4He) < \frac{1}{2} M(\,^4He) \cdot (v_{Bohr})^2 = 100keV < E_{High}(\,^4He)$$

Low energy regime:

High energy regime



Ion-solid interaction

Electronic Stopping Power: High Energy regime

$v_{Ion} > v_{Bohr} \sim$ electron velocities in their atomic shells → Atoms appear static to the ions

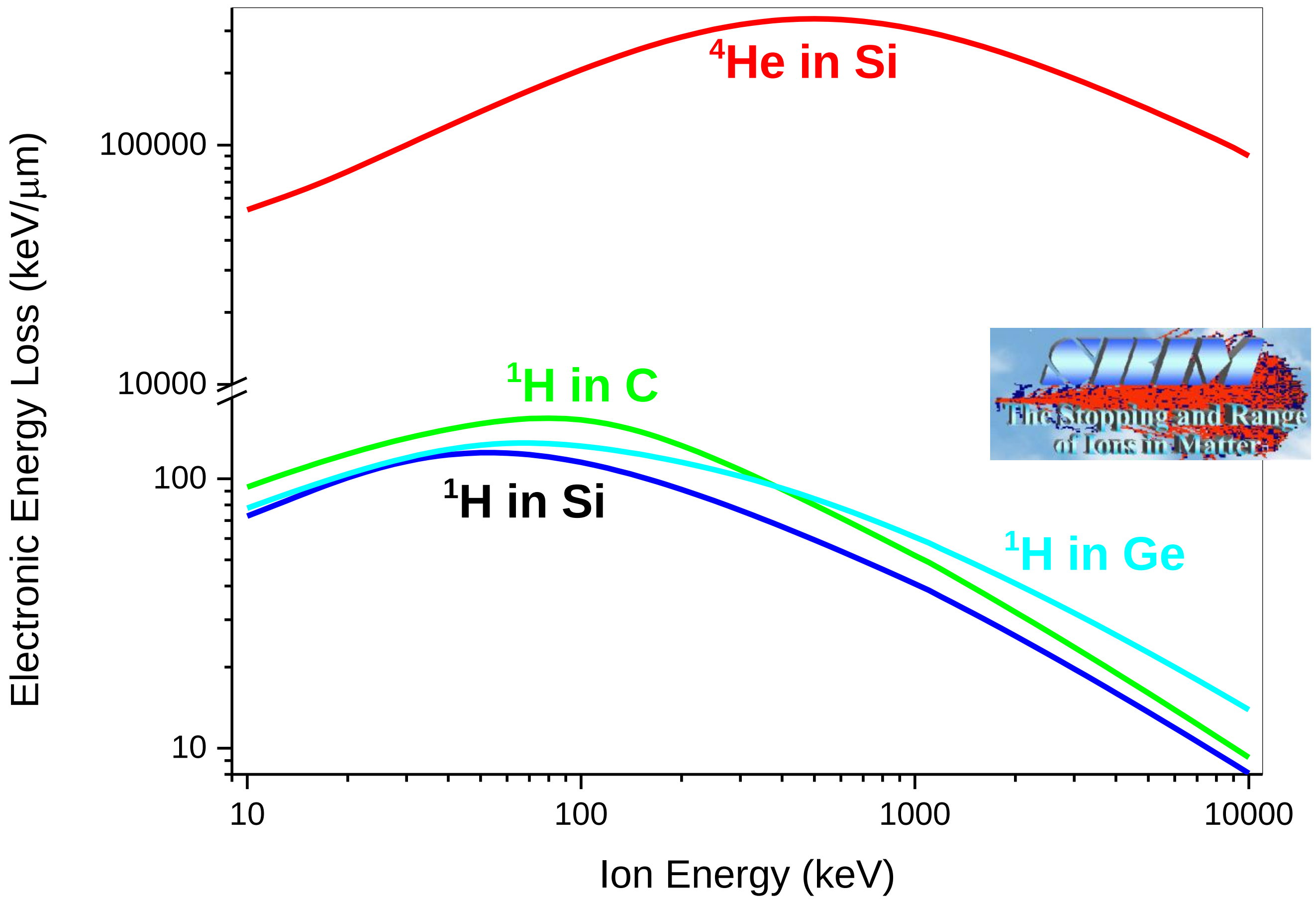
↓

Stripping of electrons

↓

Fully ionized particle with a positive charge

The rate of energy loss decreases with increasing energy because the ion pass through the orbiting electron clouds faster and have less chance of interacting with them



Electronic stopping power is:
Proportional to electron density
Inversely proportional to ion kinetic energy

Ion-solid interaction

Electronic Stopping Power: Low Energy regime

$v_{Ion} < v_{Bohr} \sim$ electron velocities in their atomic shells → Atoms appear no more static to the ions

Incomplete stripping of electrons

Partially ionized particle

Inner shell electrons of the target have a declining role in energy loss

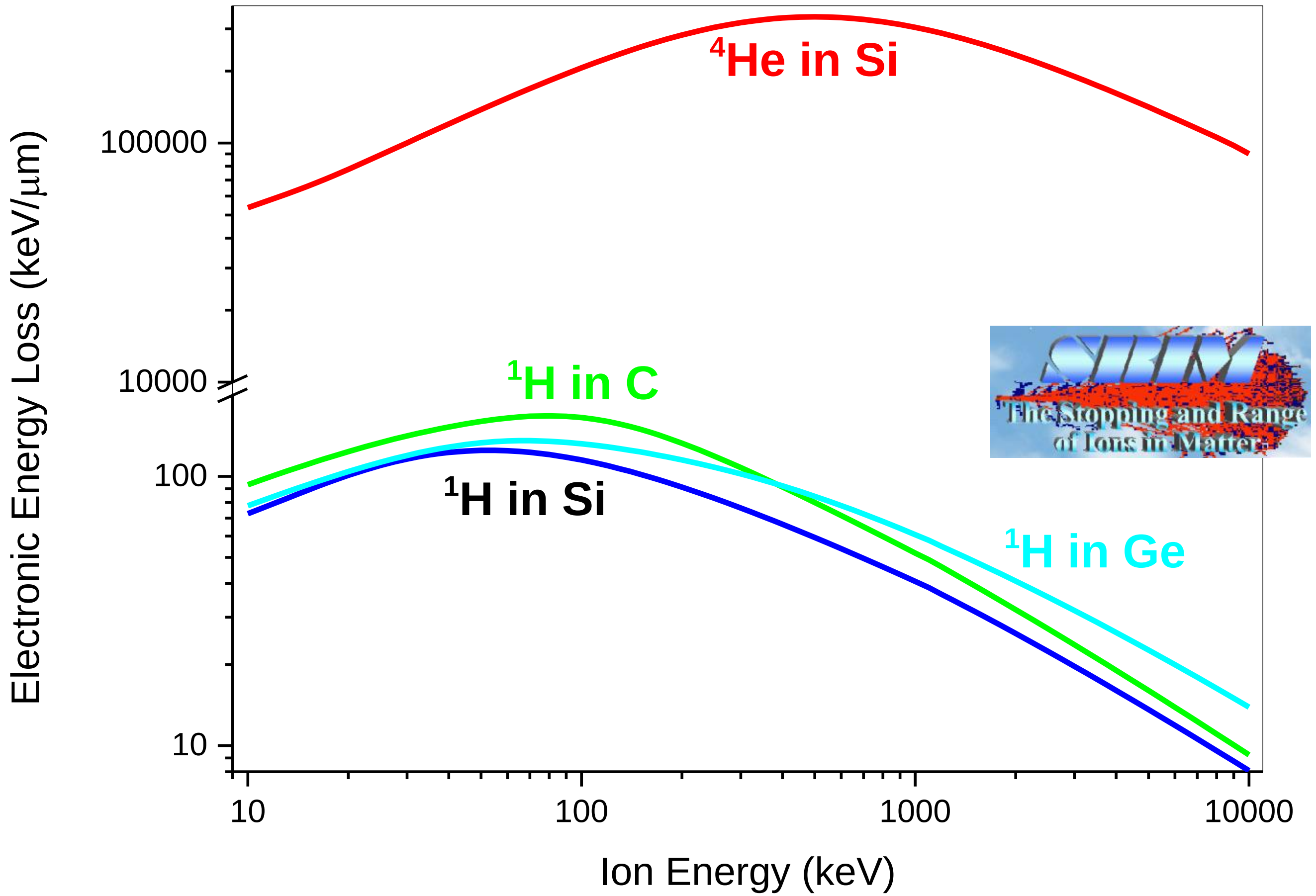
The rate of energy loss **increases with increasing energy**

The type of interaction and the rate of energy loss **depends on the chemical nature of the target**

The maximum rate of ion energy loss occurs at the Thomas-Fermi ion velocity

$v_{TF}=v_{Bohr}\cdot Z^{2/3}$

$v_{TF}(^4He)=250$ keV



Ion-solid interaction

Bethe-Bloch theory

$$S = \frac{e^4}{4\pi\epsilon_0^2 m_e} \cdot \left(\frac{Z_{Ion}}{v_{Ion}}\right)^2 Z_{Target} \cdot N_{at} \cdot L(\beta)$$

$Z_{target} \cdot N_{at}$: Electron density of the target

m_e : electron mass;

ε_0 : vacuum dielectric constant; $\frac{e^4}{4\pi\epsilon_0^2 m_e} = 4.57\ MeV \cdot \mu m^2$

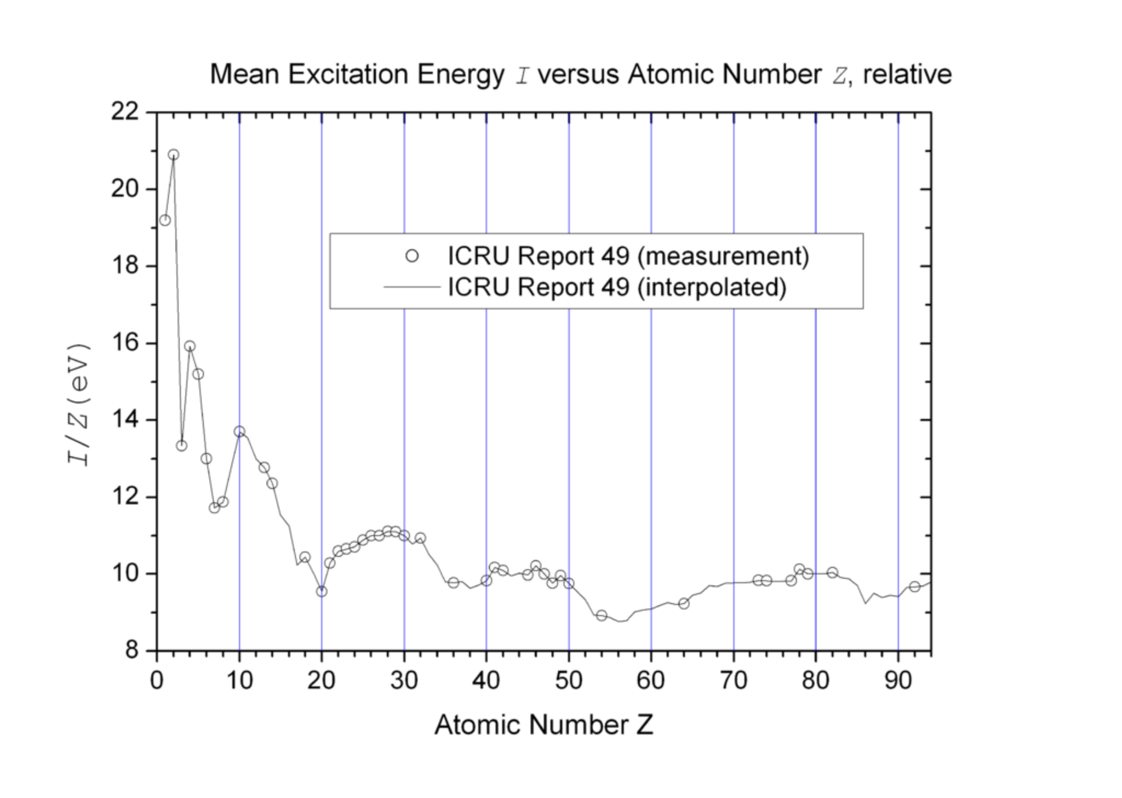
e :elementary charge

The factors preceding *L* take into account the gross features and *L* takes into account fine details.

First approximation: $L(\beta) = ln\left(\frac{2m_e v_{Ion}^2}{I}\right)$

I $\approx 10 \cdot Z_{Target}$: average electron excitation energy

Other corrections for relativistic terms at high ion energy and for strongly bound inner electrons



Bethe-Bloch theory

Example of the validity of the Bethe-Bloch theory:

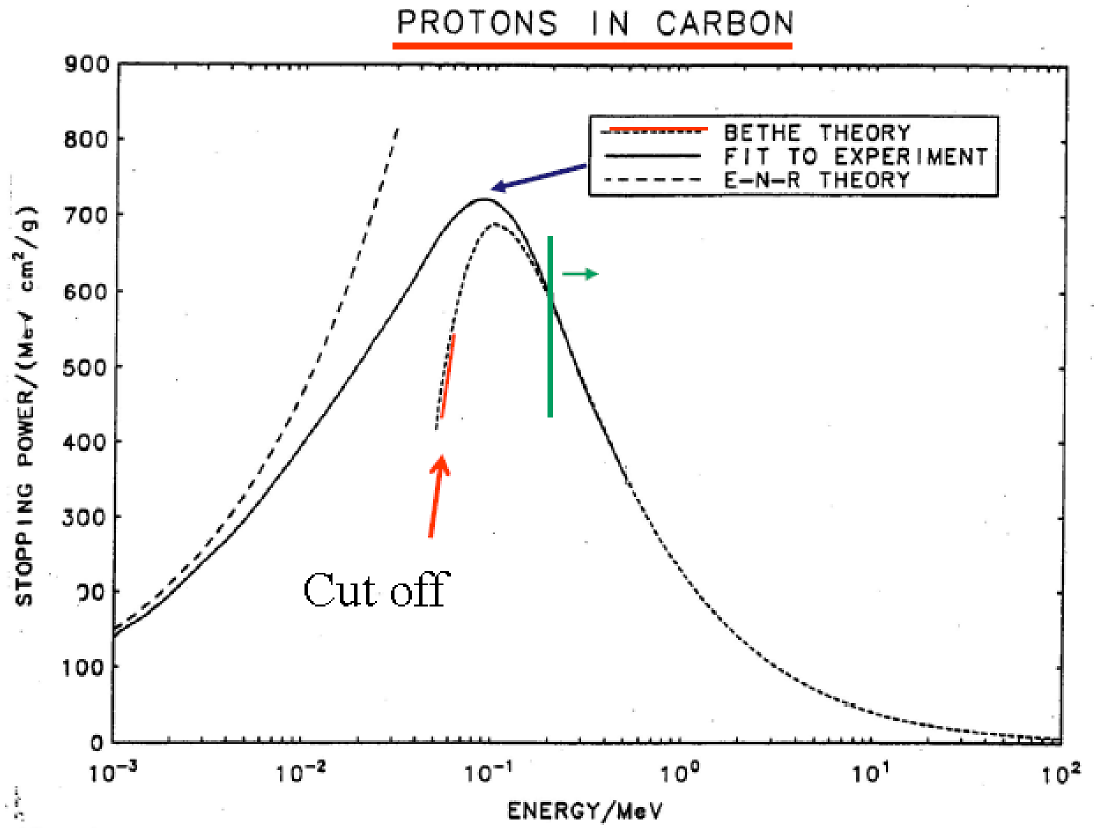


Fig. 3.10. Stopping power of amorphous carbon for protons. The short-dashed curve is from Bethe's theory, and the long-dashed curve (ENR) from the theory of Echenique *et al.* (1986). The solid curve is that adopted in the present work.

The large scatter in experimental results is a problem.

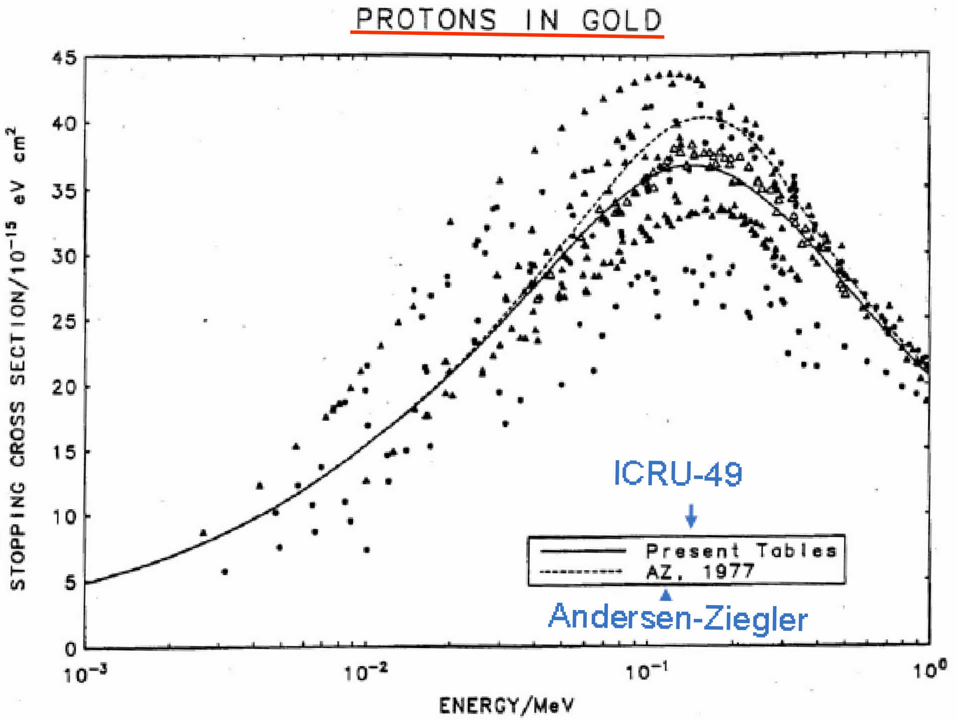


Fig. 3.2. Electronic stopping cross section of gold for protons. Experimental values are indicated by circles and triangles. Circles represent values from papers published before 1978, and triangles from papers published in 1978 or later. Hollow triangles are experimental results of the Linz-Berlin collaboration (Semrad *et al.* 1986a). Experimental values were obtained from a compilation currently in preparation by Paul *et al.* (1991). Dashed curve represents cross section from fitting formula of Andersen and Ziegler (1977), and solid curve represents stopping cross section adopted in present work.

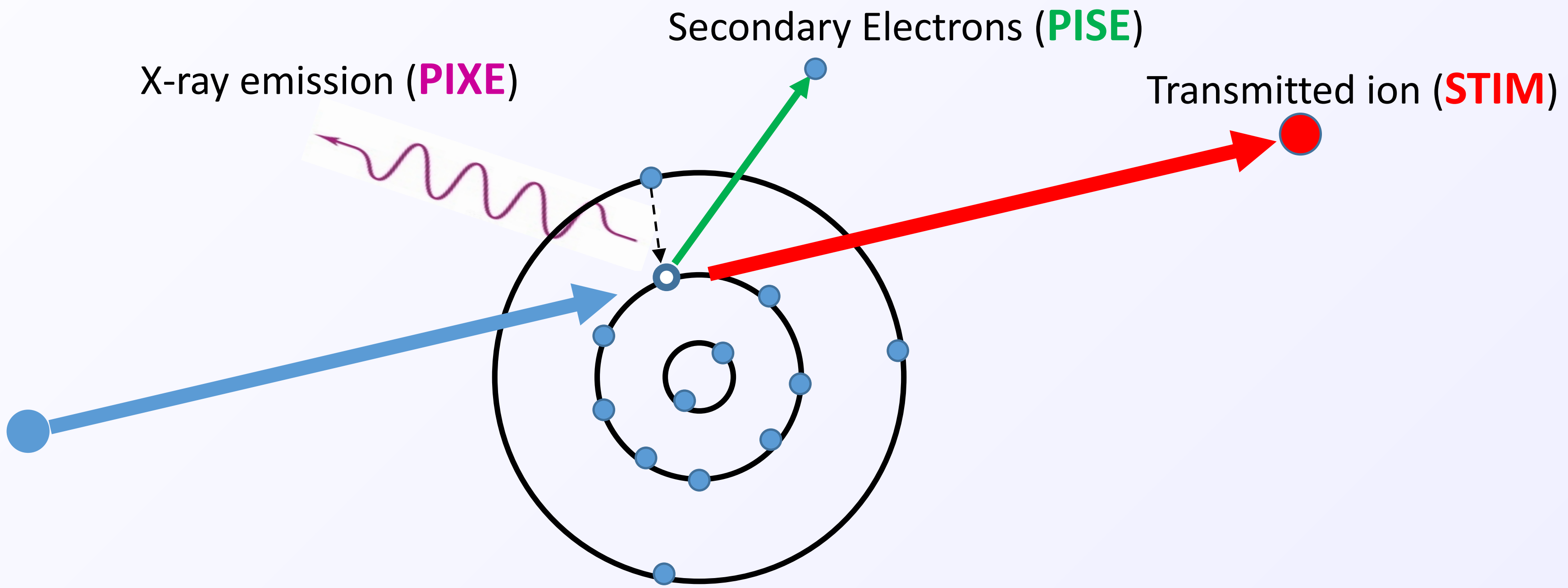
Ion-solid interaction

For Ion Beam Analysis, interactions between the ion probe and atomic electrons are much more probable than with atomic nuclei

Electronic energy loss provides a higher rate of signals

Different electronic processes

For Example

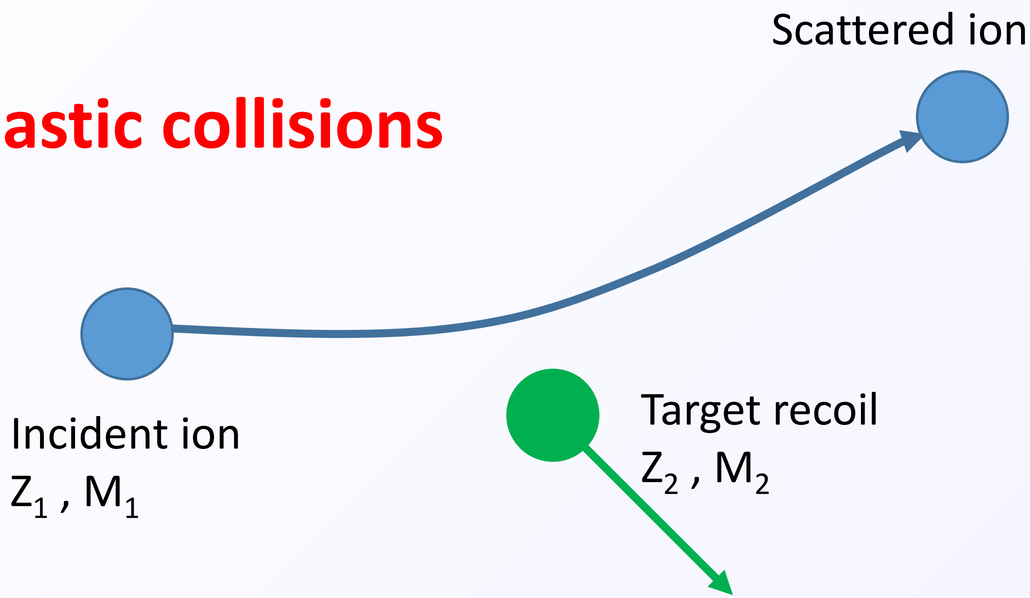


Ion-solid interaction

Nuclear Energy Loss

Typically, the energy lost by ^1H and ^4He MeV ions in collisions with atomic nuclei is less than 10 keV.

Elastic collisions



Screened Coulomb Potential

$$\mathbf{V(r) = Z_1 \cdot Z_2 \frac{e^2}{4\pi\epsilon_0 r}\chi(r)}$$

First terms: Coulomb potential for two bare nuclei with charges eZ_1 and eZ_2.

χ : screening function

(for high energy ions, i.e. Rutherford model, : χ=1)

Ziegler-Biersack-Littmark (ZBL) Universal potential

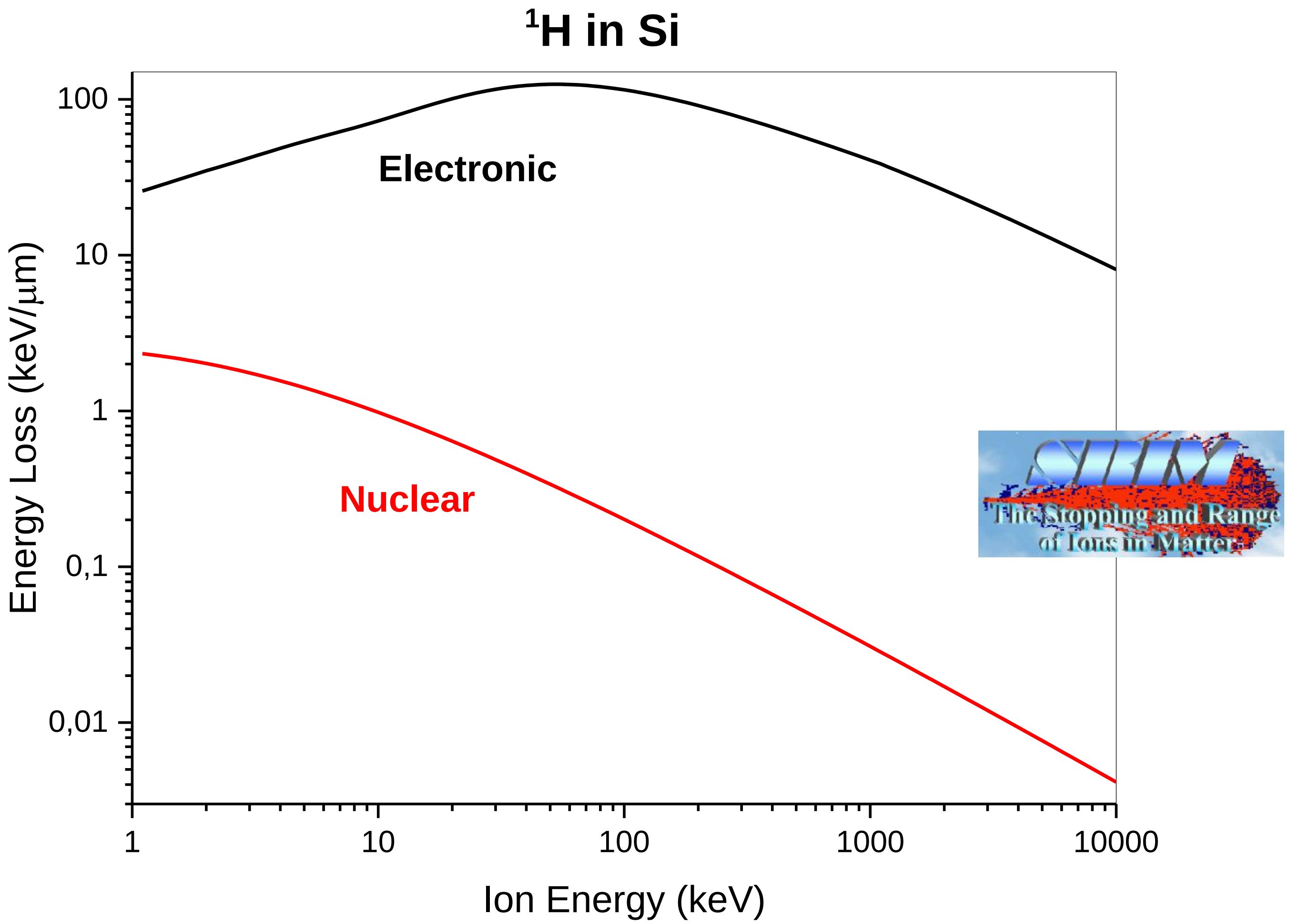
χ Can be determined from fitting simulations of interatomic potentials for different Z_1 and Z_2 combinations

$$\boxed{\chi(x) = \sum_{i=1}^{4} a_i e^{-b_i x}}$$

$$\boxed{with\ x = \frac{r_{12}}{a_u} \quad and \quad a_U = \frac{0.885 \cdot a_{Bohr}}{Z_1^{0.23} + Z_2^{0.23}}}$$

Thomas-Fermi Screening radius

Ion-solid interaction



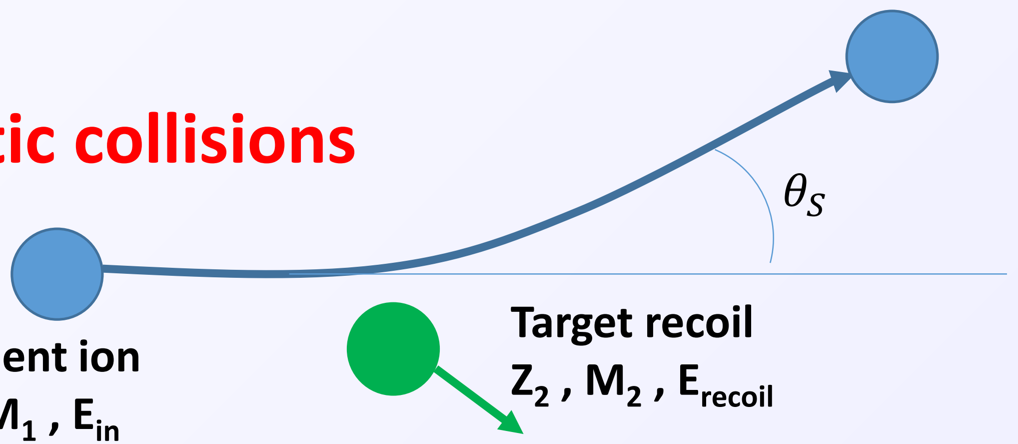
An ion can approach closer to the atomic nucleus with increasing energy and eventually it can approach to within a distance comparable with the nuclear radius

No pure Coulomb potential -> nuclear forces affect the collision

Classical Scattering theory

Fraction of the ion energy which remains with the ion after the collision; $E_{out} = E_{in} \cdot K(\theta_S)$
Kinematic factor $K(\theta_S)$; θ_S scattering angle.
Energy of the recoil nucleus: $E_{recoil} = (1 - K(\theta_S)) \cdot E_{in}$

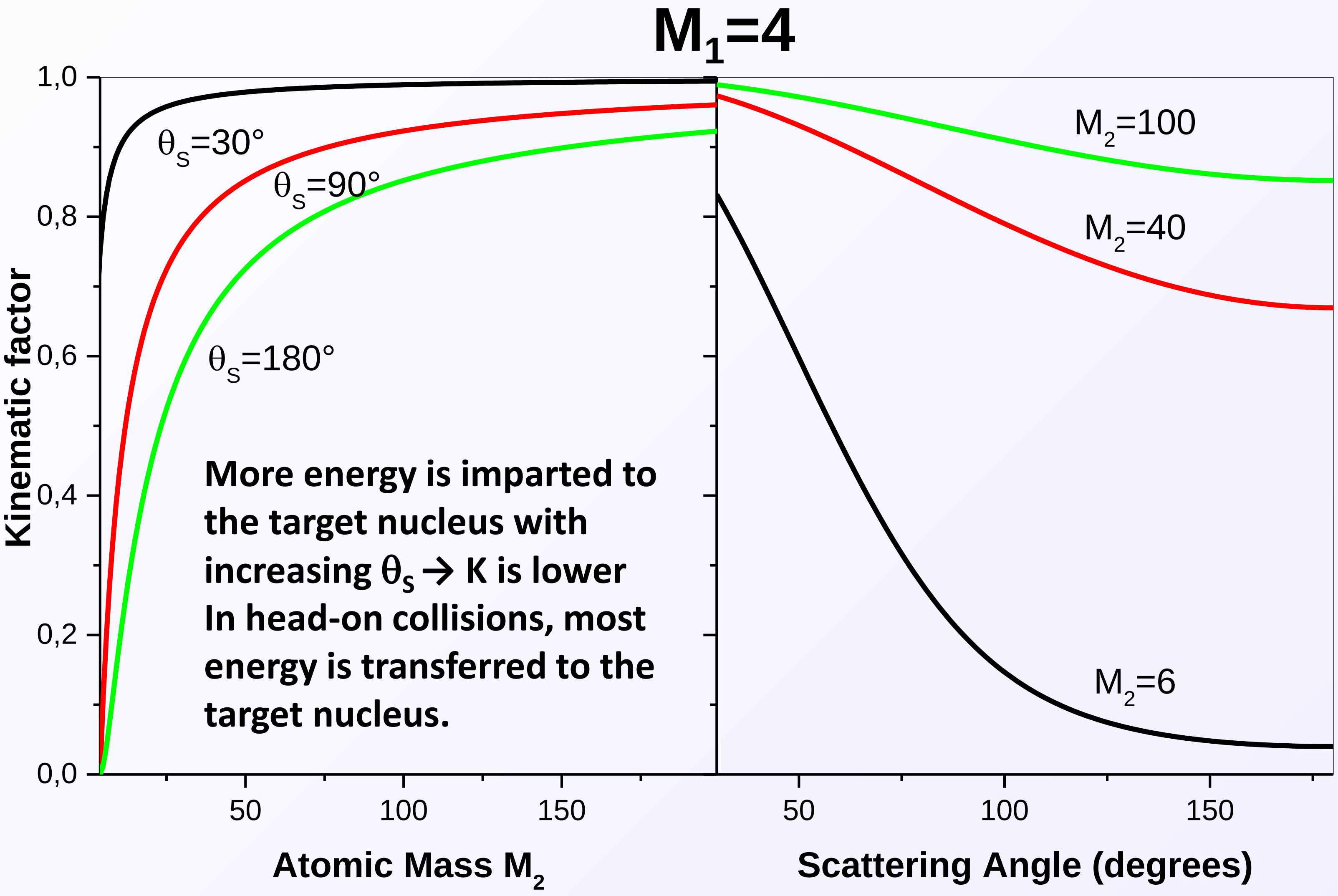
Elastic collisions



From the conservation of energy and momentum, in non-relativistic form and in the laboratory reference frame:

$$\mathbf{K}(\boldsymbol{\theta}_S) = \left\{ \frac{\sqrt{1 - \left(\frac{\mathbf{M}_1}{\mathbf{M}_2}\right)^2 \cdot \sin^2(\boldsymbol{\theta}_S)} + \left(\frac{\mathbf{M}_1}{\mathbf{M}_2}\right) \cdot \cos(\boldsymbol{\theta}_S)}{1 + \frac{\mathbf{M}_1}{\mathbf{M}_2}} \right\}^2$$

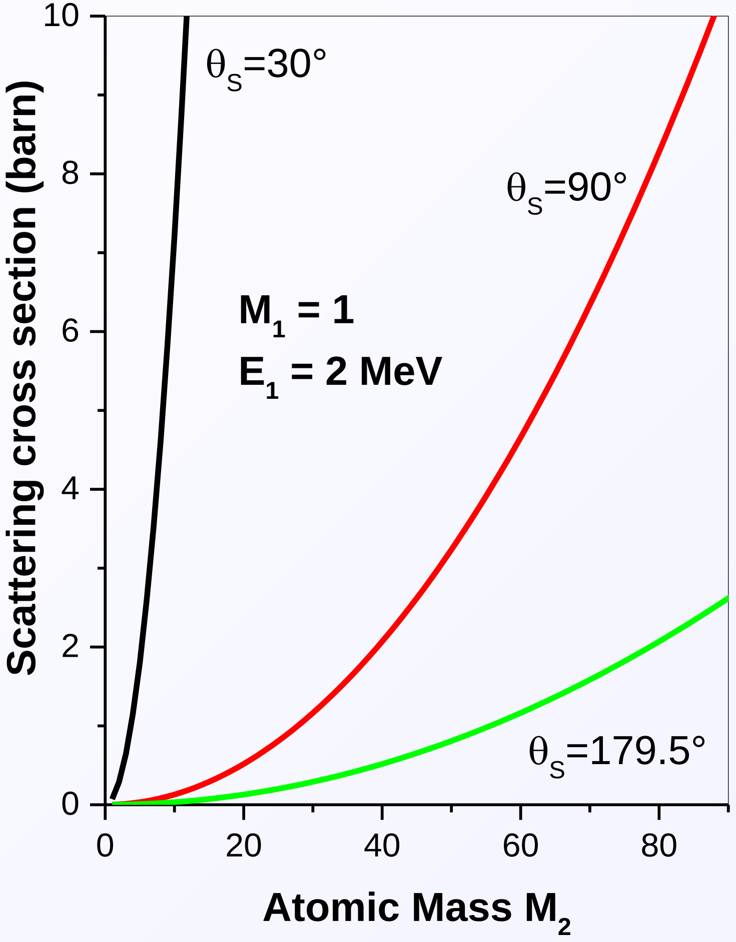
$K(\theta_S)$ depends only on the scattering angle and the ratio of masses of the ion and the atomic nucleus.
It does not depend on the ion energy

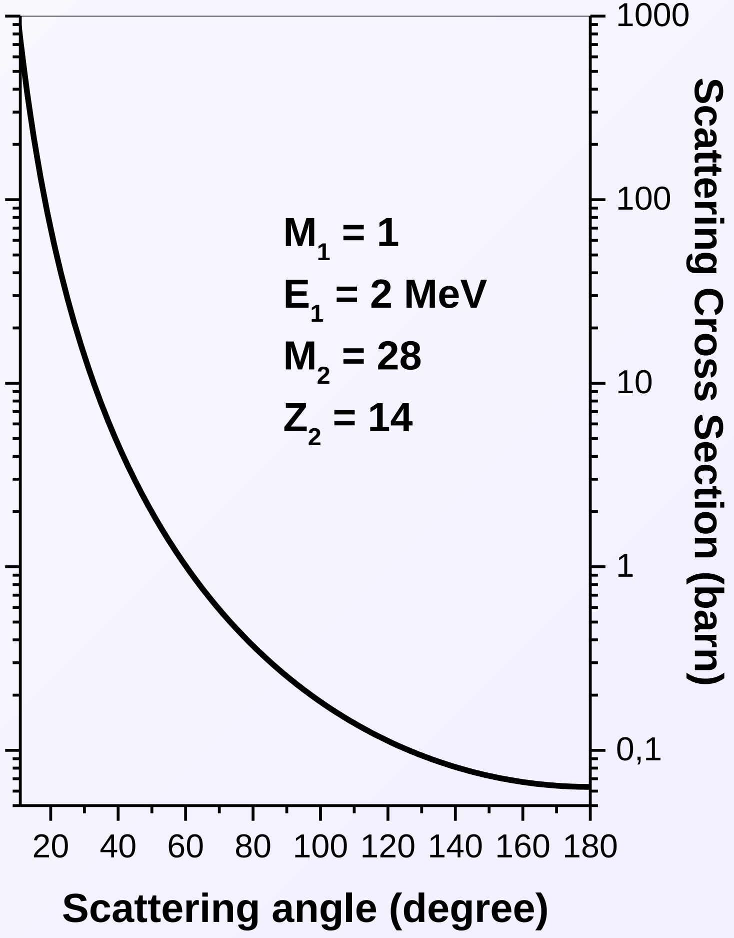

M1=4
1,0
0,8
0,6
0,4
0,2
0,0
Kinematic factor
θS=30°
θS=90°
θS=180°
More energy is imparted to the target nucleus with increasing θS → K is lower
In head-on collisions, most energy is transferred to the target nucleus.
50
100
150
Atomic Mass M2
M2=100
M2=40
M2=6
50
100
150
Scattering Angle (degrees)

Scattering cross section

Probability of ions being scattered through an angle θ_s

$$\sigma(\theta_s) = \left[\frac{e^2}{4 \cdot \epsilon_0} \cdot \frac{Z_1 \cdot Z_2}{4 \cdot E_{in}}\right]^2 \cdot \left\{\frac{1}{\sin^4\left(\frac{\theta_s}{2}\right)} - 2\left(\frac{M_1}{M_2}\right)^2\right\}$$

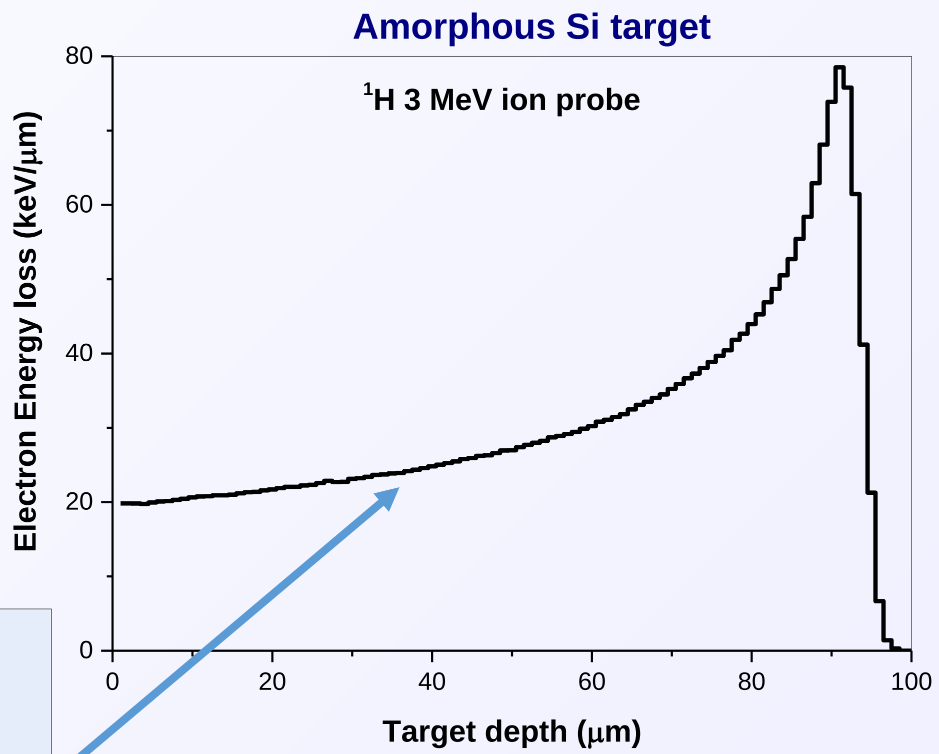


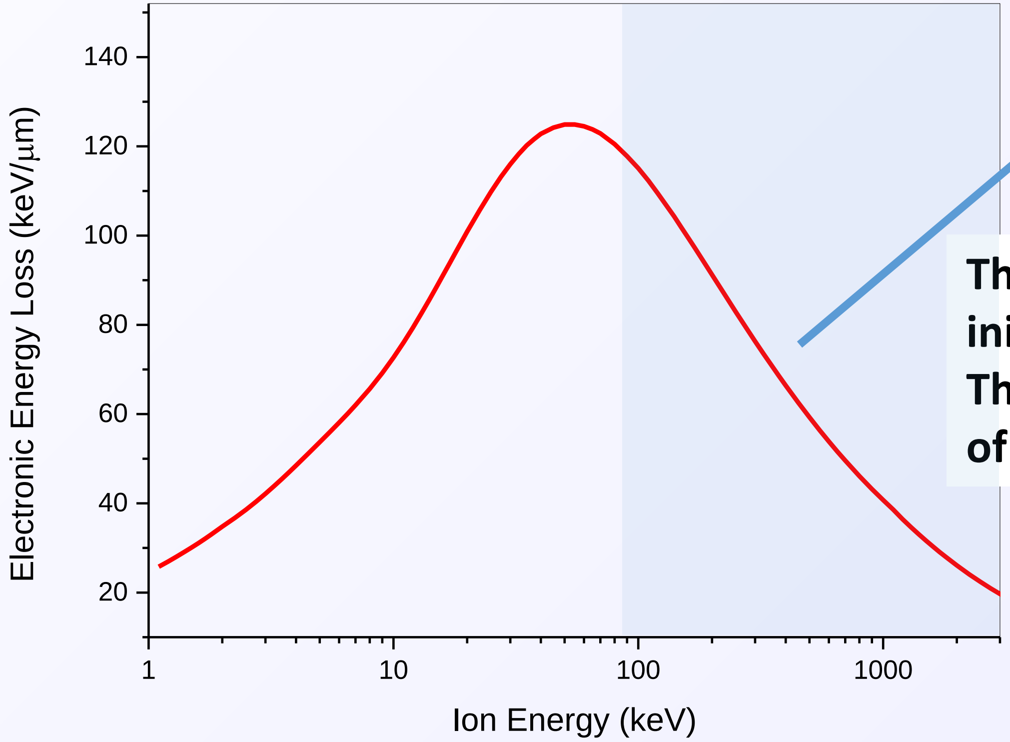


Ion-solid interaction

Ion Range

MeV ions continually lose their kinetic energy in collisions mainly with the electrons until they come to rest at some depth within the material.



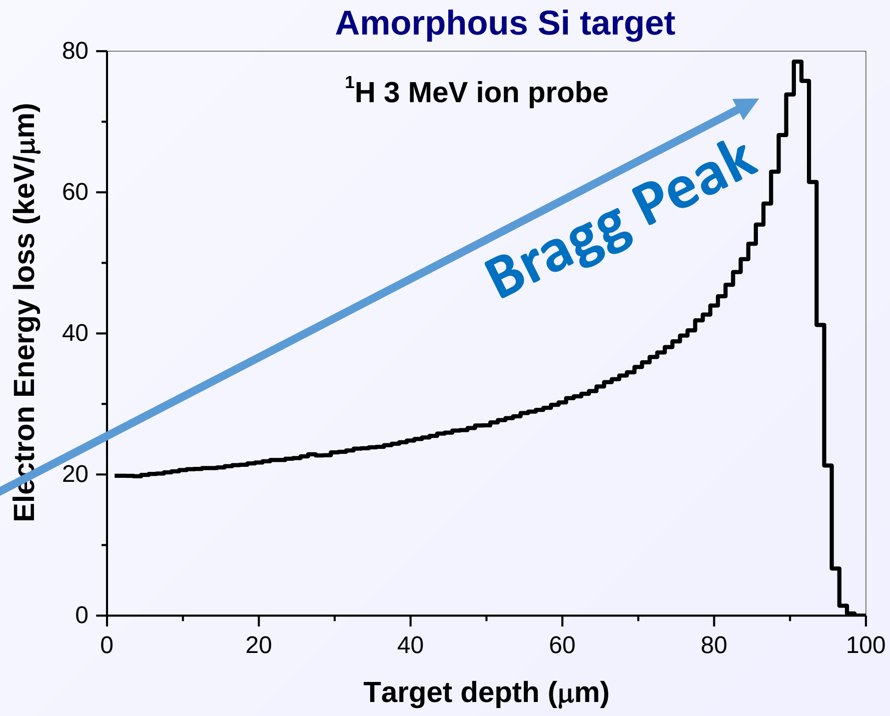


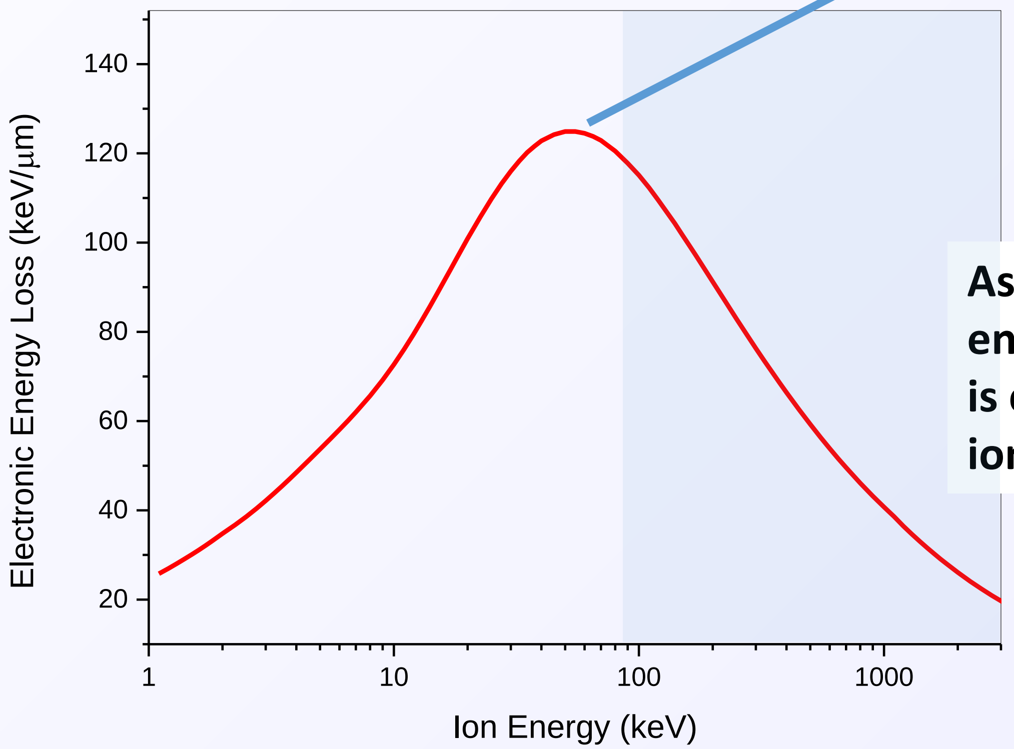
The rate of ion energy loss increases initially as the ion penetrate further
The rate of ion energy loss is to the right of the maximum value

Ion-solid interaction

Ion Range

MeV ions continually lose their kinetic energy in collisions mainly with the electrons until they come to rest at some depth within the material.



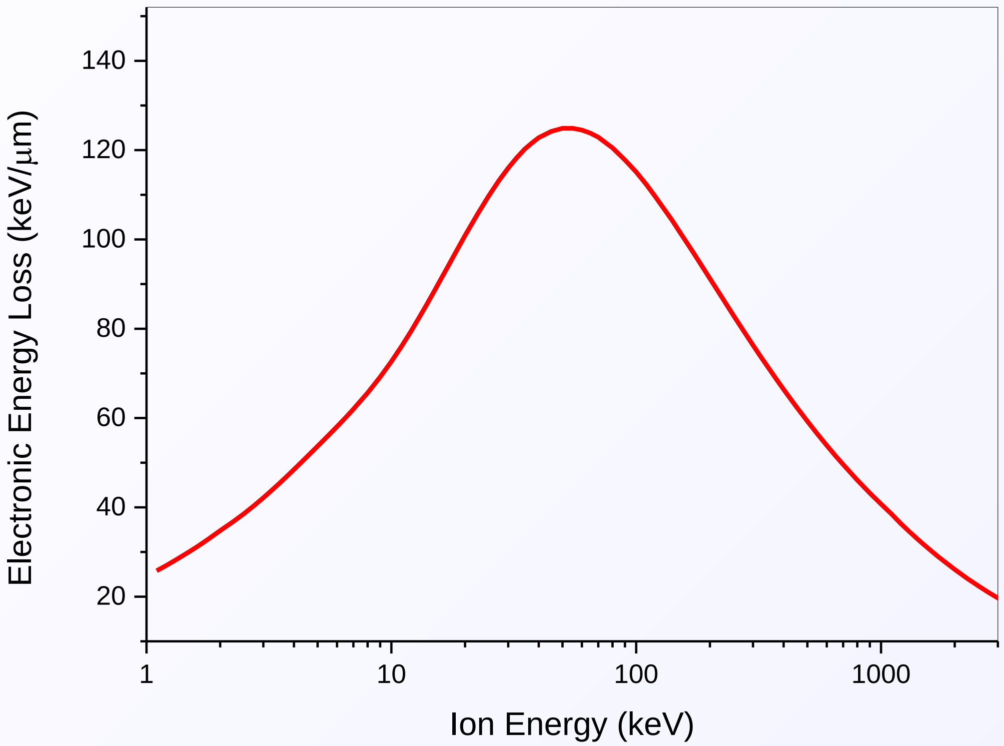


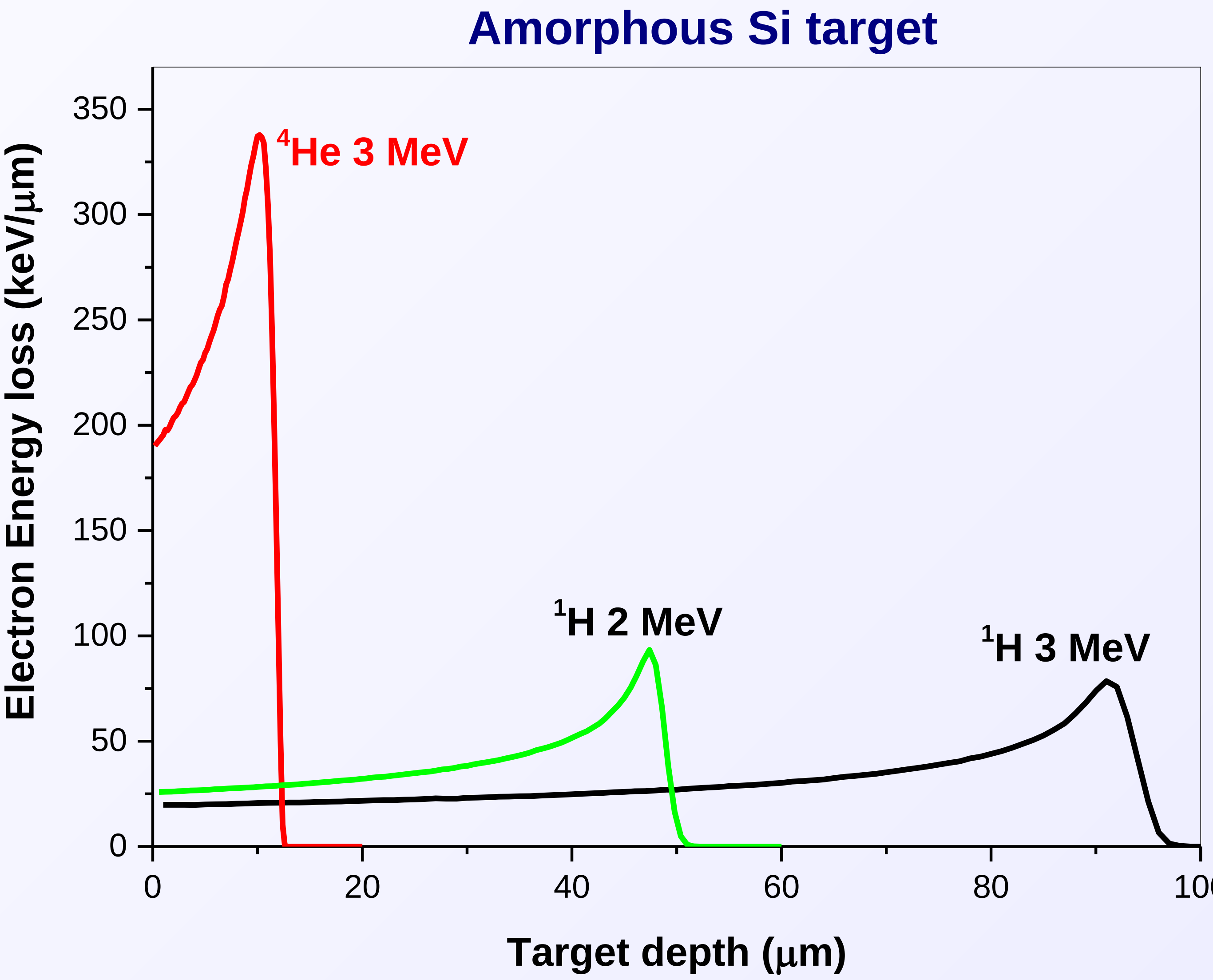
As the ion penetrate further, the rate of energy loss reaches a maximum, which is close to the end of range for MeV light ions→BRAGG PEAK

Ion-solid interaction

Ion Range

MeV ions continually lose their kinetic energy in collisions mainly with the electrons until they come to rest at some depth within the material.





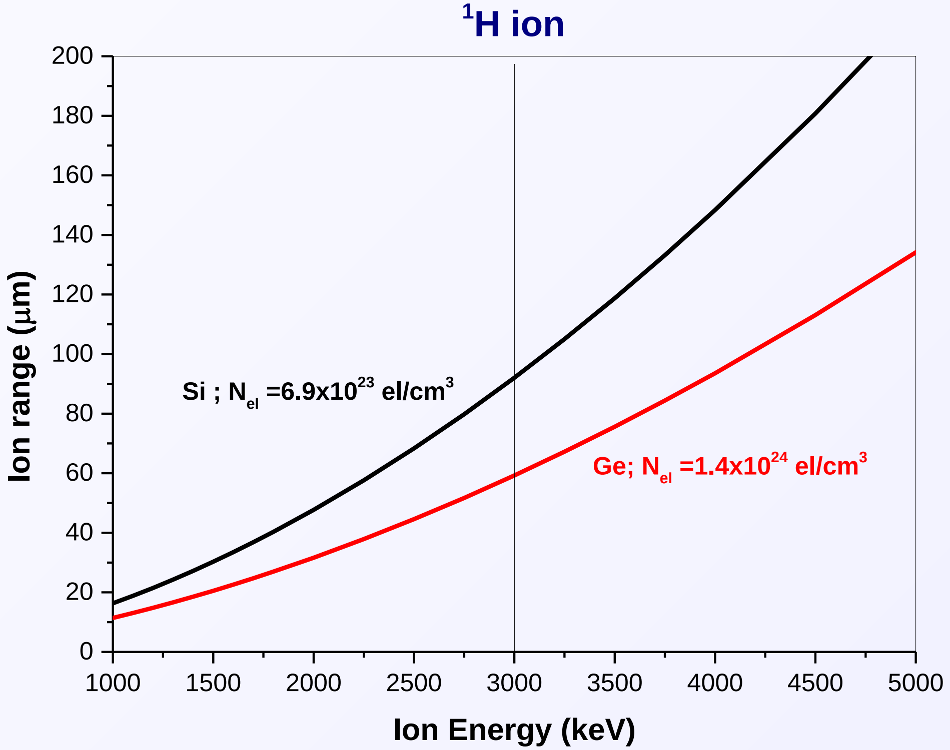
Ion-solid interaction

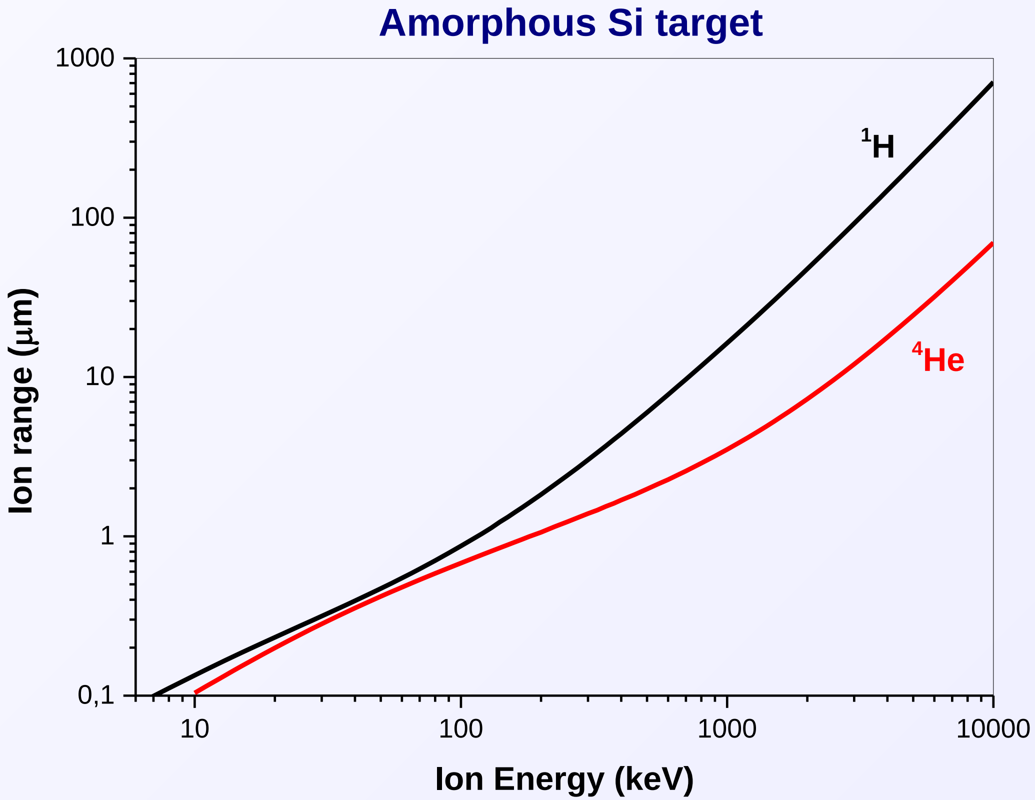
Ion Range

MeV ions continually lose their kinetic energy in collisions mainly with the electrons until they come to rest at some depth within the material.

Ion Range: average range R which MeV light ions travel through matter before coming to rest:

$$R(E_{in}) = \int_0^{E_{in}} \frac{dE}{\left(\frac{dE}{dz}\right)}$$





Ion range is function of energy

Ion range is function of the target density

Ion Straggling

In practice, MeV ions lose their energy in discrete collisions with individual atomic electrons

Fluctuations in the number and geometry of collisions

Distribution of energies of initially monoenergetic ions after traversing a depth of material

Variation in the distance to which individual ions penetrate the material

Longitudinal straggling

Distribution of the transverse momentum acquired by the ions

Alteration of the ion trajectories and different stopping distances from the beam axis

Lateral straggling

Ion-solid interaction

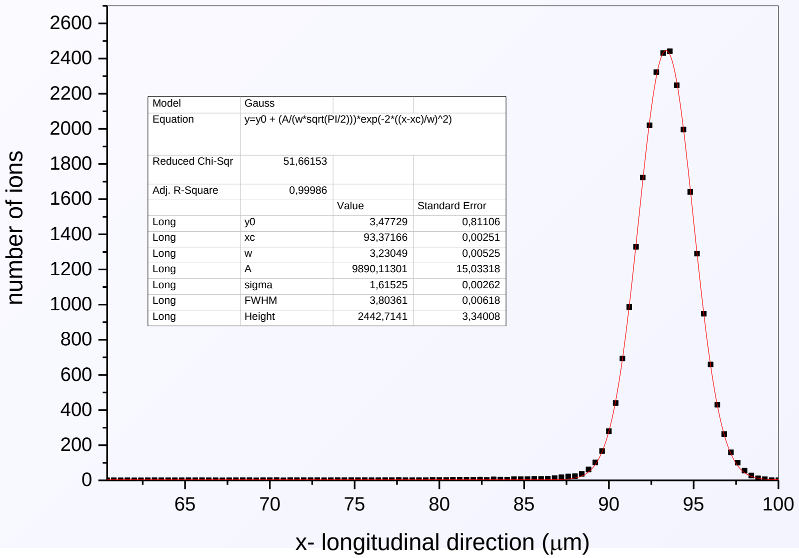
Ion Straggling

Mean projected range

$$R_P = \frac{\sum_{i=1}^{N} x_i}{N} = < x >$$

Longitudinal straggling

$$\sigma = \sqrt{< x^2 > - < x >^2}$$



Lateral projected range

$$R_P = \frac{\sum_{i=1}^{N} y_i}{N} = < y >$$

Lateral straggling

$$\sigma_y = \sqrt{< y^2 > - < y >^2}$$

- analytical models PRAL (Projected Range Algorithm)
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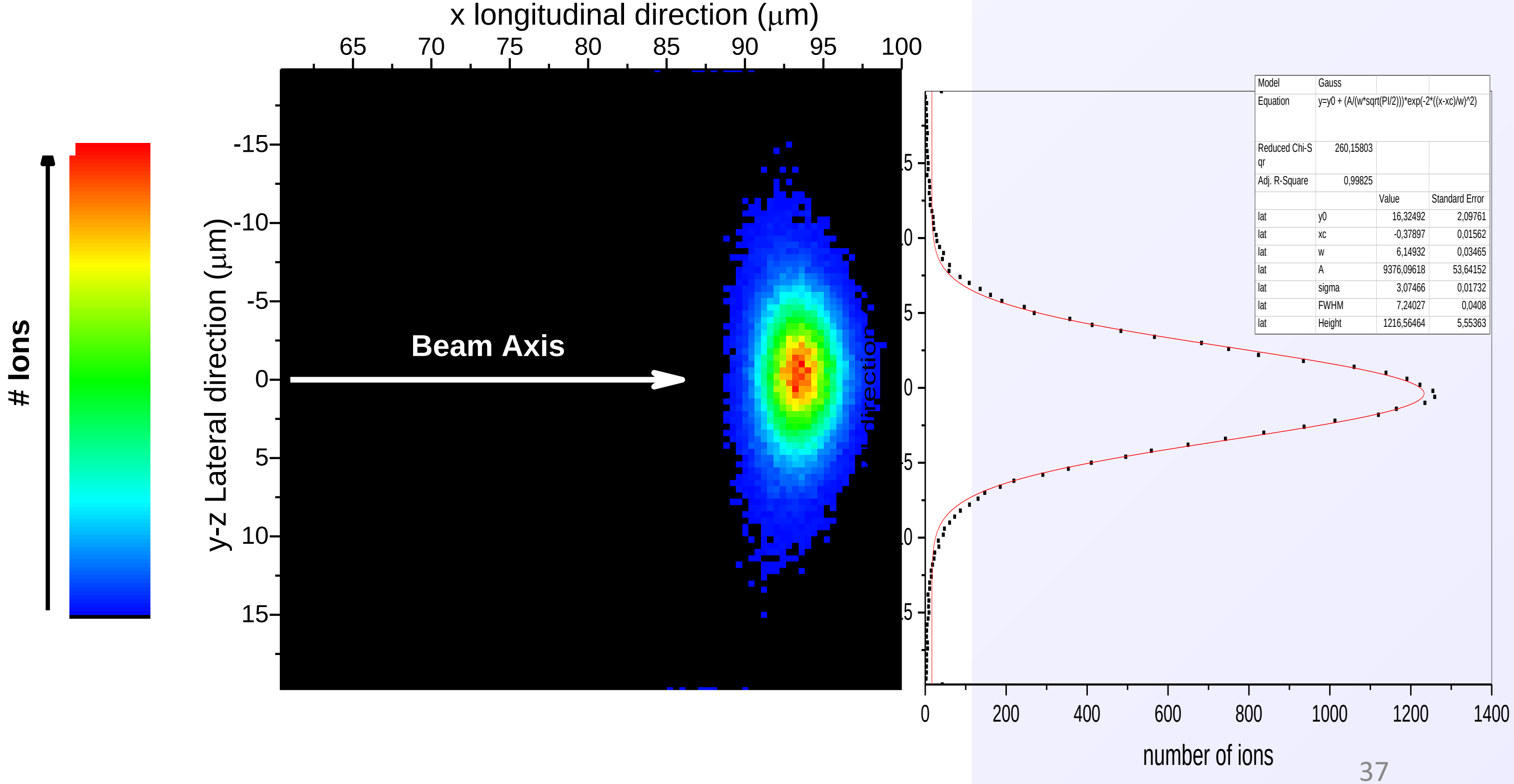
  - J.P. Biersack, Z.Phys. A305 (1982) 95
  - Book by Ziegler, Biersack, Littmark (1985)
- values fitted to experimental data
  - Gupta & Bhattachraya, Phys. Rev.B29 (1984) 2449
  - Izsack et al. NIM B15 (1986) 34
  - Kido et al. NIM B15 (1986) 42



Computer codes for stopping power:

- **PSTAR and ASTAR, from NIST, for protons and alphas**
  http://physics.nist.gov/PhysRefData/Star/Text/PSTAR.html
- **MSTAR, from IAEA, calculates electronic stopping power for heavy ions (from 3Li to ^{18}Ar) :**
  https://www-nds.iaea.org/stopping/MstarWWW/MSTARInstr.html
- **ATIMA, from GSI, calculates quantities characterizing the slowing down of protons and heavy ions in matter**
  https://web-docs.gsi.de/~weick/atima/
- **CasP, from HZB, electronic stopping power of all ions**
  https://www.helmholtz-berlin.de/people/gregor-schiwietz/casp_en.html

For a critical comparison:
«Judging the reliability of stopping power tables and programs for heavy ions»
Nucl. Instr. Meth. Phys. Res. B 209 (2003) 252

Simulation codes (Monte Carlo Tracing Techniques) to investigate the intraction and trajectories of fast ions with matter

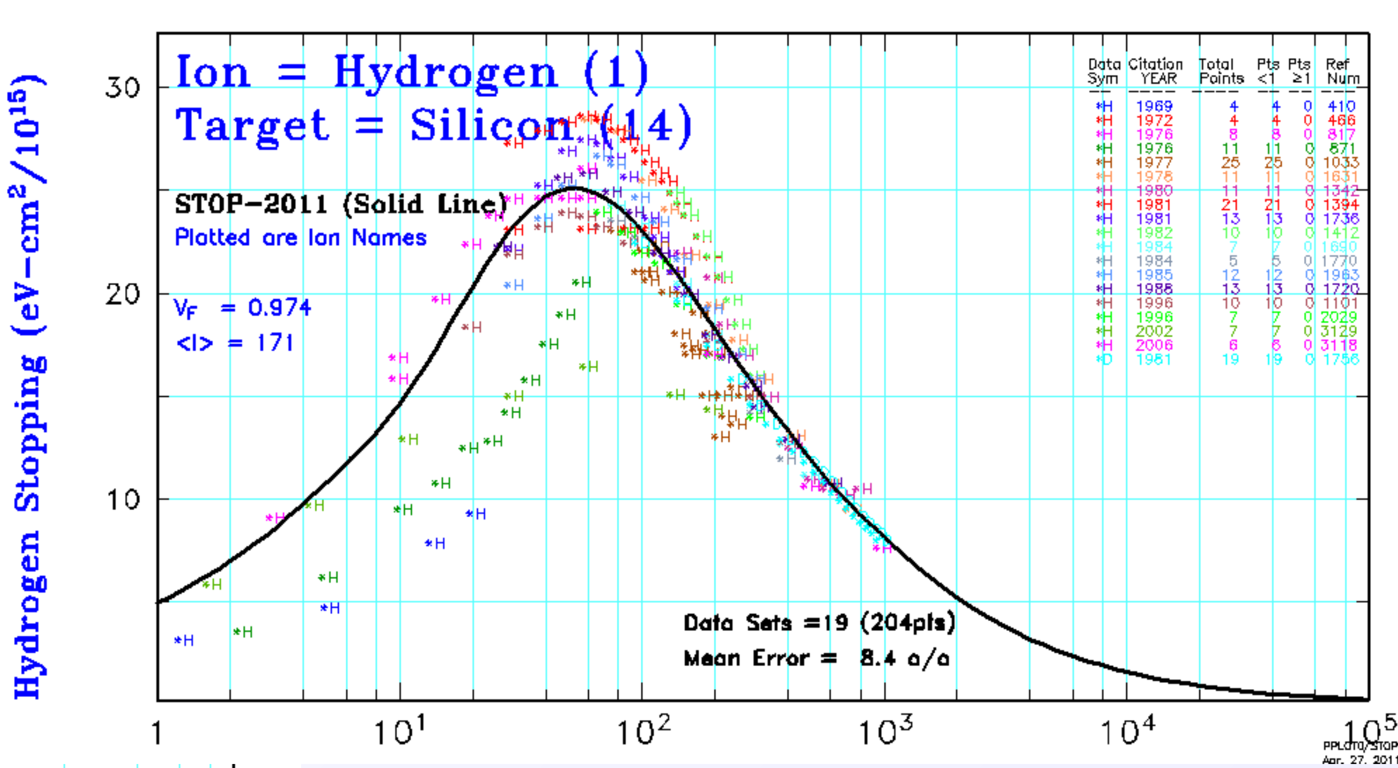
- **SRIM: The Stopping and Range of Ions in Matter**
  www.srim.org
- **CRYSTAL SRIM: For trajectories in crystal lattices**
  https://www.hzdr.de/db/Cms?pOid=21576&pNid=0
- **GEANT4: (Geometry and Tracking=) is a platform for the simulation of the passage of particles through matter using Monte Carlo methods**
  www.geant4.org/geant4
- **MARLOWE: simulates atomic collisions in crystalline targets using the binary collision approximation**
  https://www.oecd-nea.org/tools/abstract/detail/psr-0137

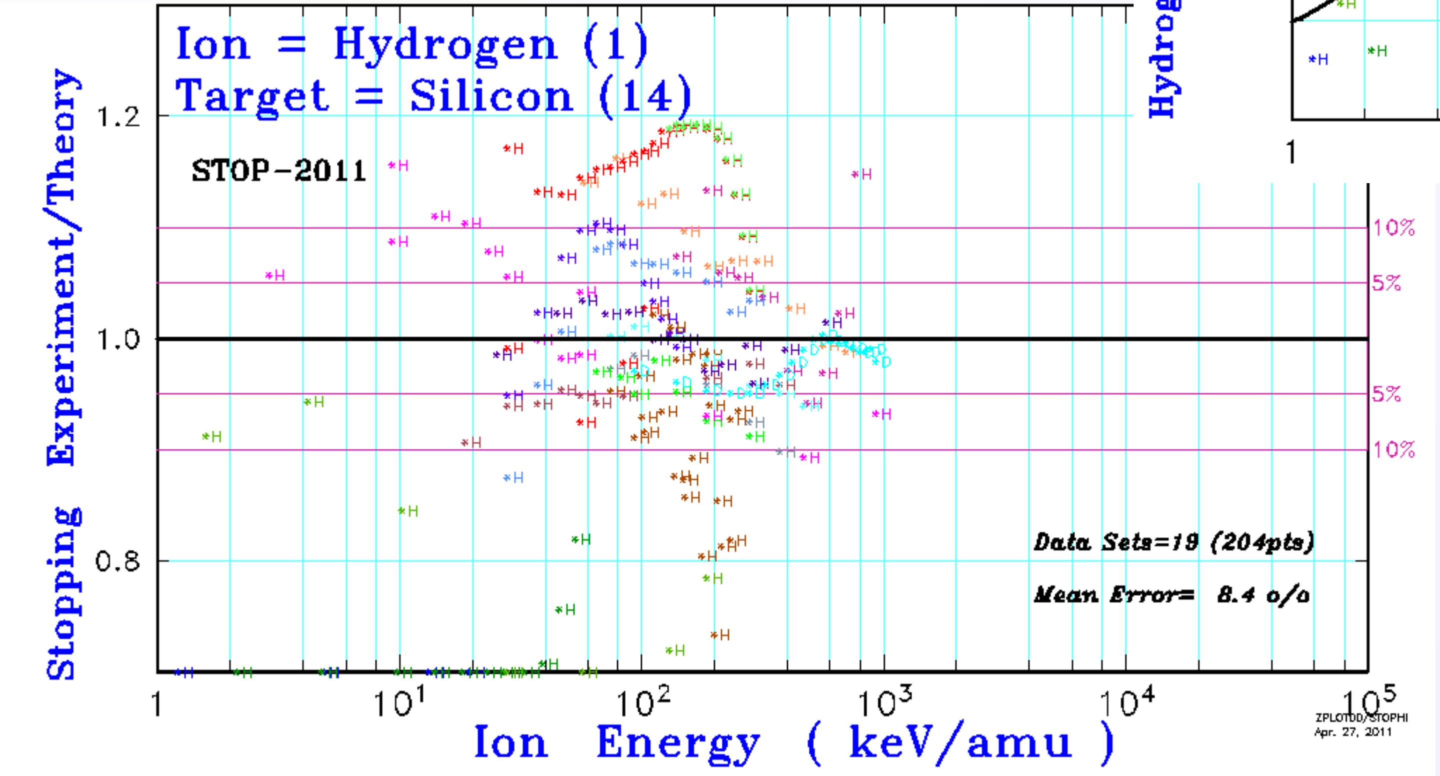
SRIM
The Stopping and Range of Ions in Matter

www.SRIM.org

SRIM is based on a Monte Carlo simulation method, namely the binary collision approximation (BCA) with a random selection of the impact parameter of the next colliding ion.
BCA treats interactions as binary collisions and one collision is calculated at a time.

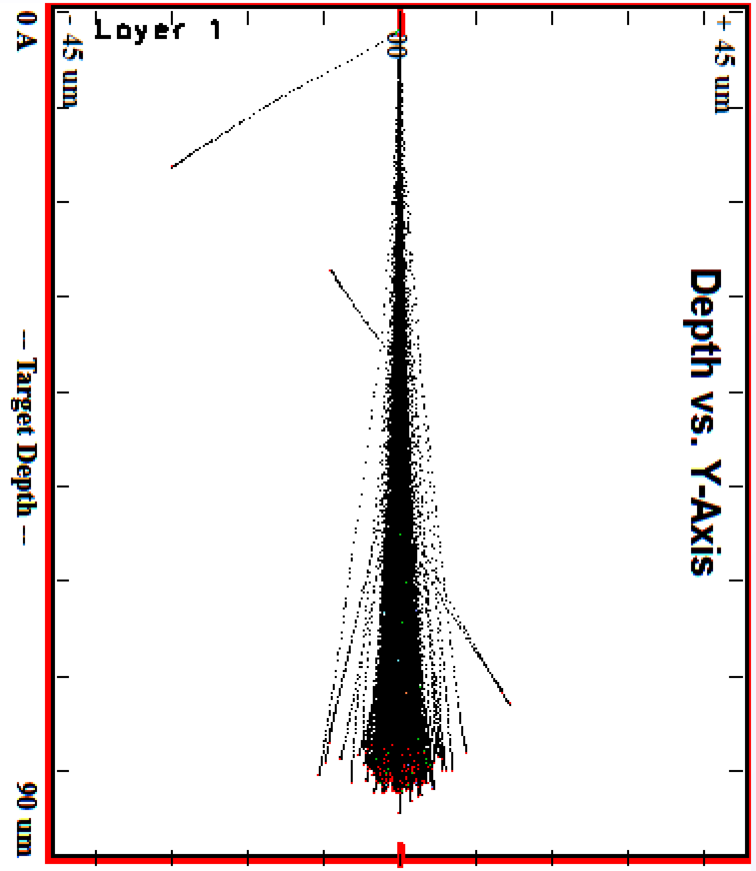
Ion-solid interaction





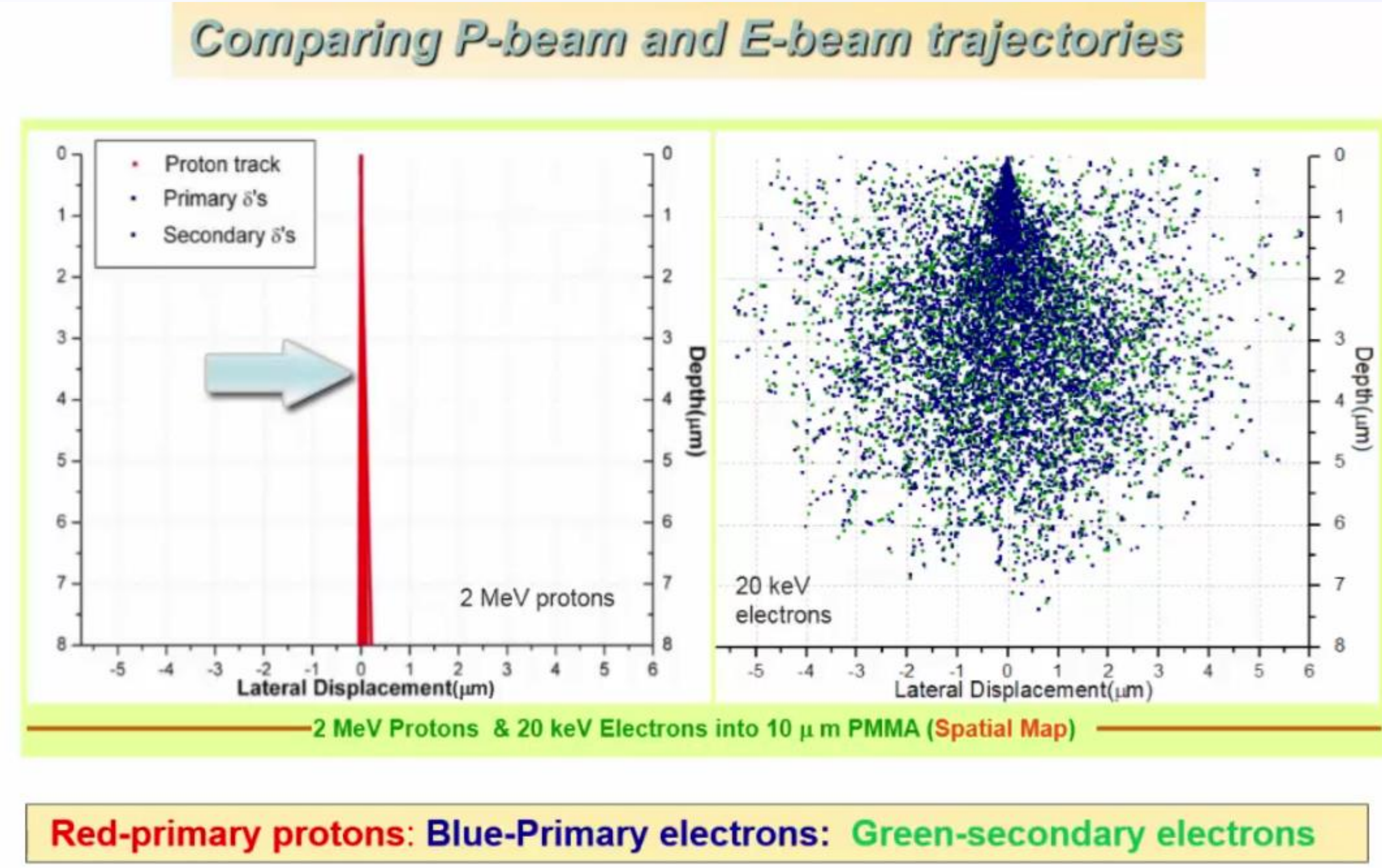
Ion-solid interaction

2 MeV ^{1}H in PMMA



ION STATS

	Range	Straggle
Longitudinal	79.3 um	2.53 um
Lateral Proj.	1.44 um	2.17 um
Radial	2.29 um	2.42 um



CIBA: Centre for Ion Beam Applications, Dept of Physics, NUS.

http://www.ciba.nus.edu.sg/pages/gallery.html

Ion-solid interaction

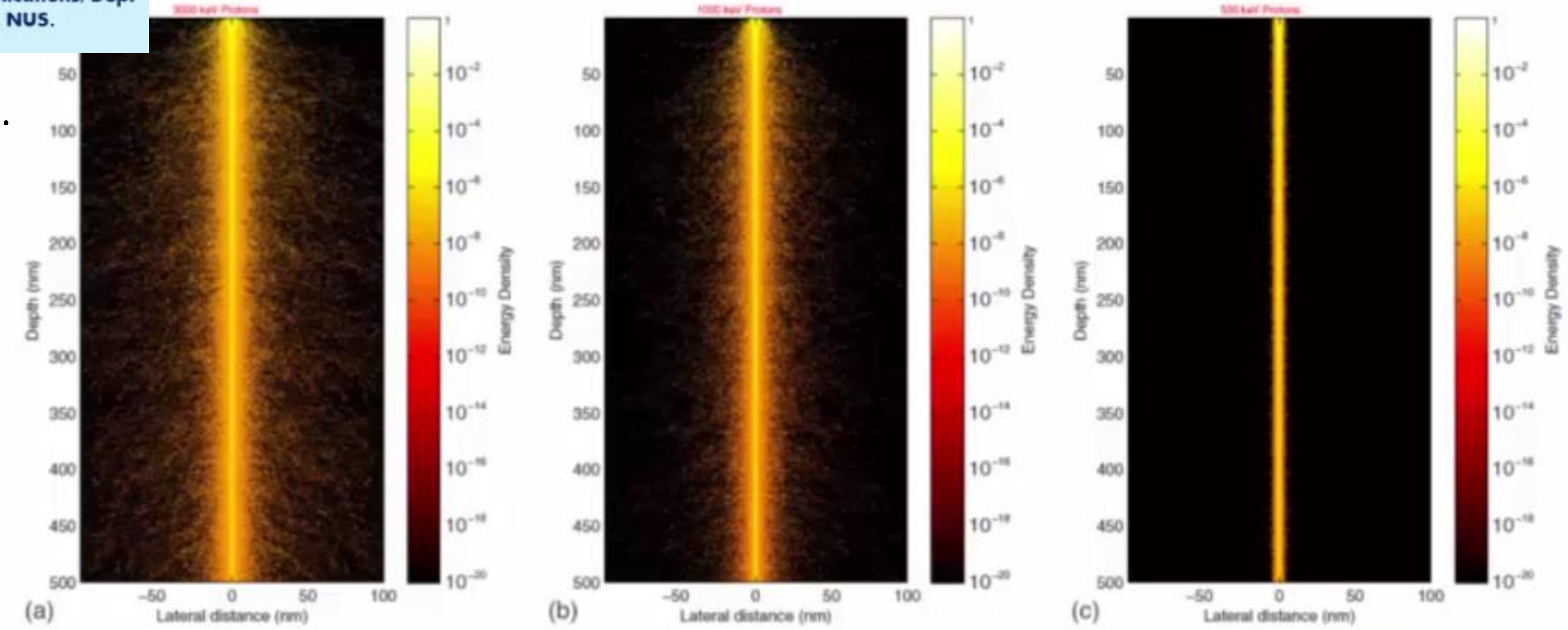
CIBA: Centre for Ion Beam Applications, Dept of Physics, NUS.

Energy density distributions around the proton tracks: (10,000 protons) calculated using 'DEEP'.

Includes secondary electron production energy loss, plasmons, atomic excitation……..

http://www.ciba.nus.edu.sg/pages/gallery.html

Udalagama et al. Phys.Rev. B80 224107 (2009)



3 MeV protons

1 MeV protons

0.5 MeV protons

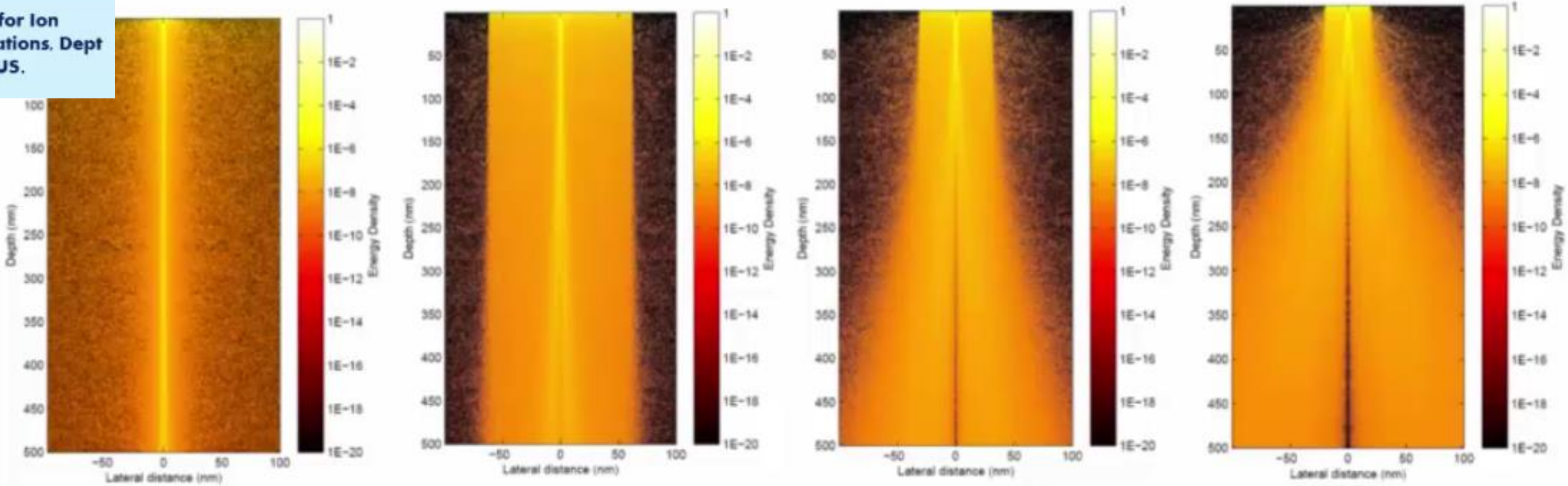
Results show not only a straight path for primary ions, but a highly confined lateral energy deposition in the initial path into organic material.

Ion-solid interaction

CIBA: Centre for Ion Beam Applications, Dept of Physics, NUS.

http://www.ciba.nus.edu.sg/pages/gallery.html

Energy density distributions (electrons) calculated using ‘DEEP’.



1 MeV electrons)*

100 keV electrons*

25 keV electrons*

10 keV electrons*

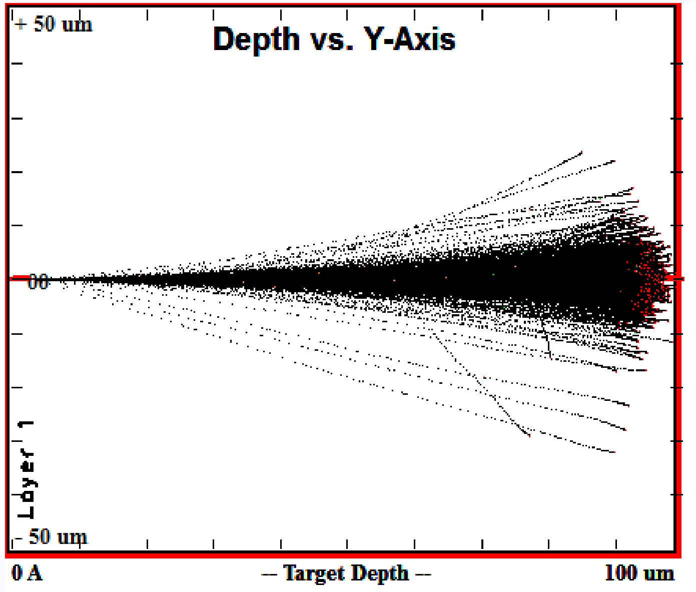
The lateral energy distribution (proximity effects) is much worse for electrons.

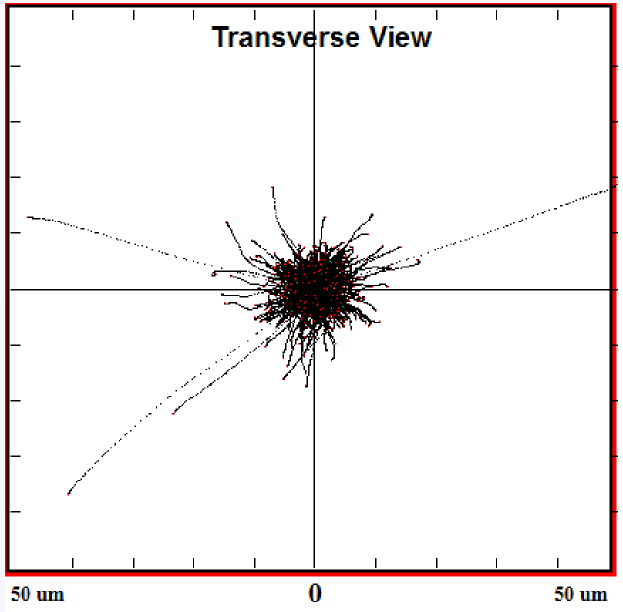
*The number of electrons have been adjusted to give the same linear energy deposition as 10,000 protons.

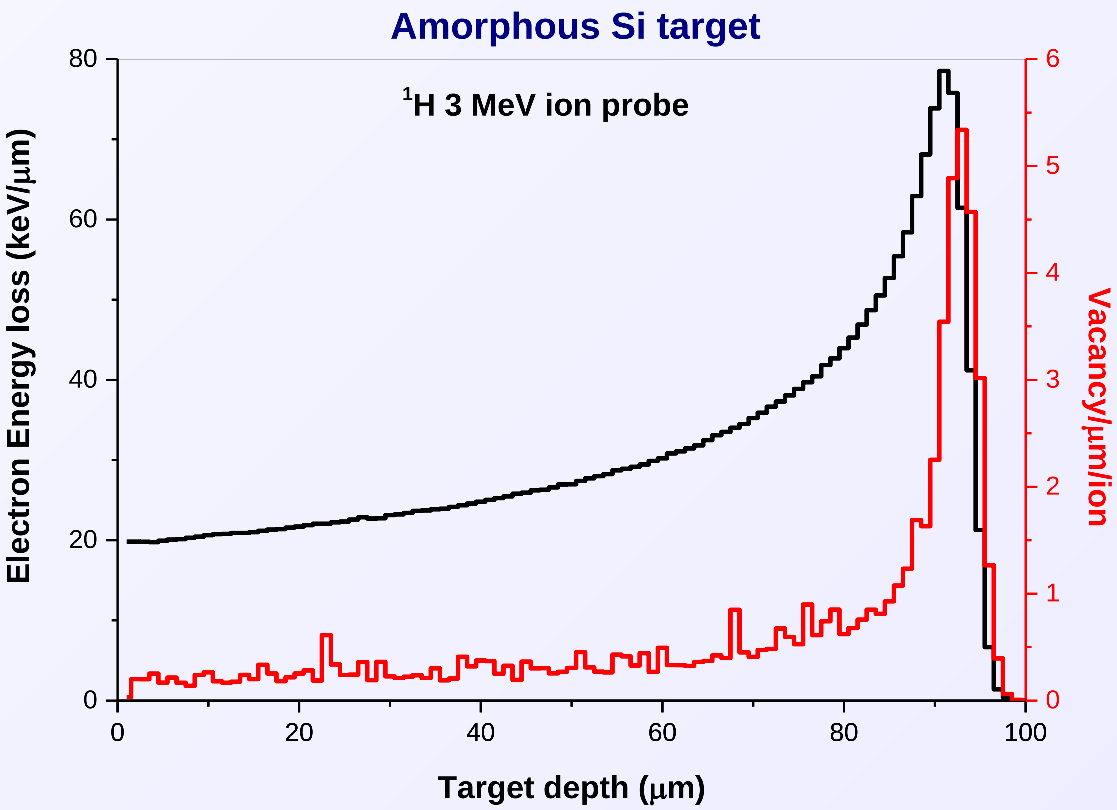
Ion-solid interaction

Characteristics of MeV ion interaction with matter

- Light MeV ions (^1H, ^4He) maintain a strainght path through the sample (unlike electrons), until they reach the end of range. Over the first few micrometers of their trajectory, the ions only diverge few nanometers.
- At the en of range, teh energy deposition increases due to nuclear collisions and the ion suffers increased scattering. This can disrupt the position of target atoms, and result in modified electronic, optical and chemical properties in the target.







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Rutherford Backscattering Spectrometry (RBS)
http://users.ictp.it/~pub_off/lectures/lns022/Mayer_1/Mayer_1.pdf