Ion microscopy
&
Ion beam lithography

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Outline

Ion beam microscopy

- Experimental features
  - → **focused ion beams**
  - → **acquisition systems**
- I-beam microscopy techniques
  - → **STIM, IBIL**
  - → **other μ**-scopy techniques

Ion beam lithography

- MeV ion beam lithography
  - → **resists**
  - → **silicon**
  - → **other materials**
  - → **single ion tracks**
  - → **single ion doping**

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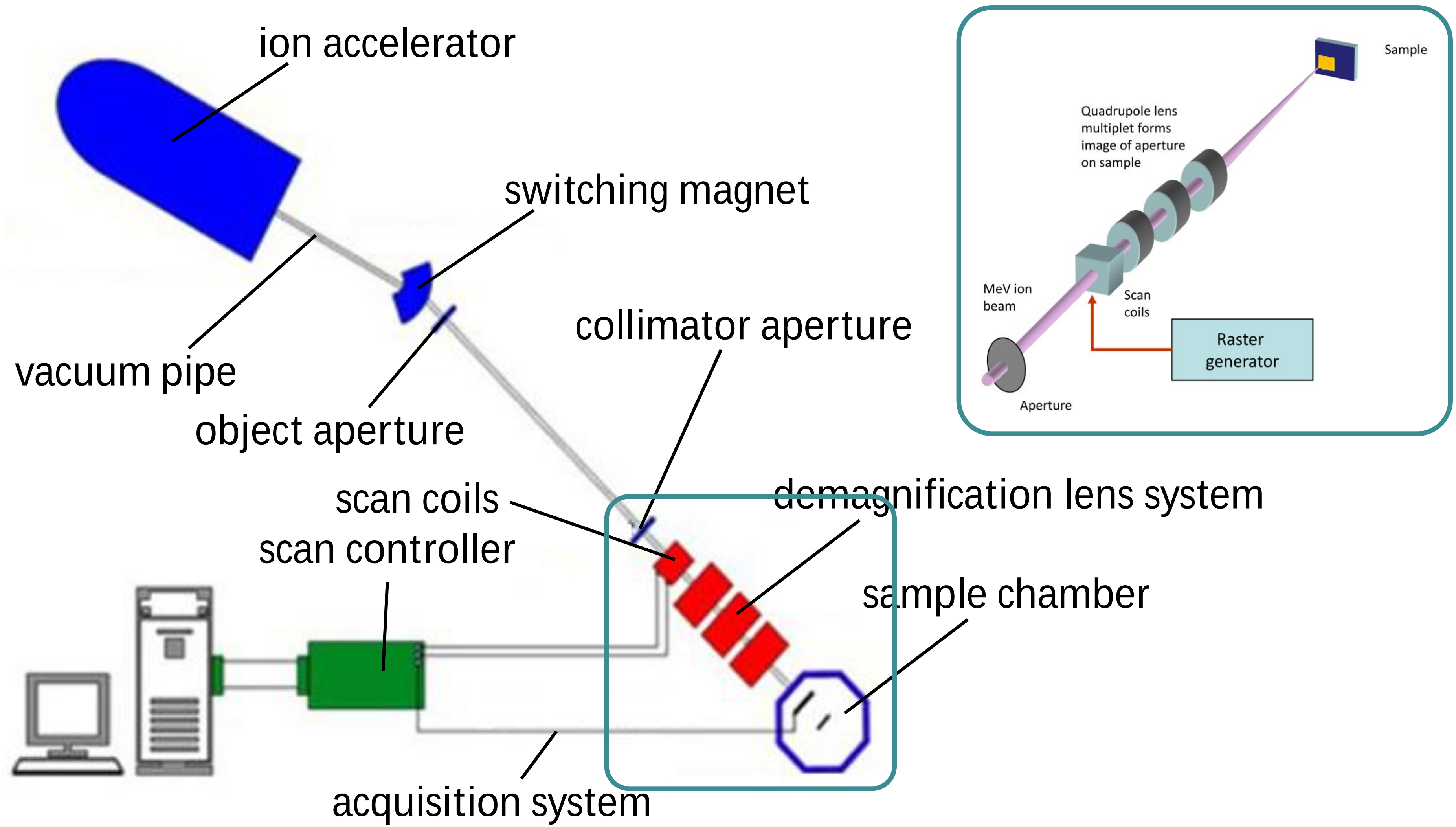
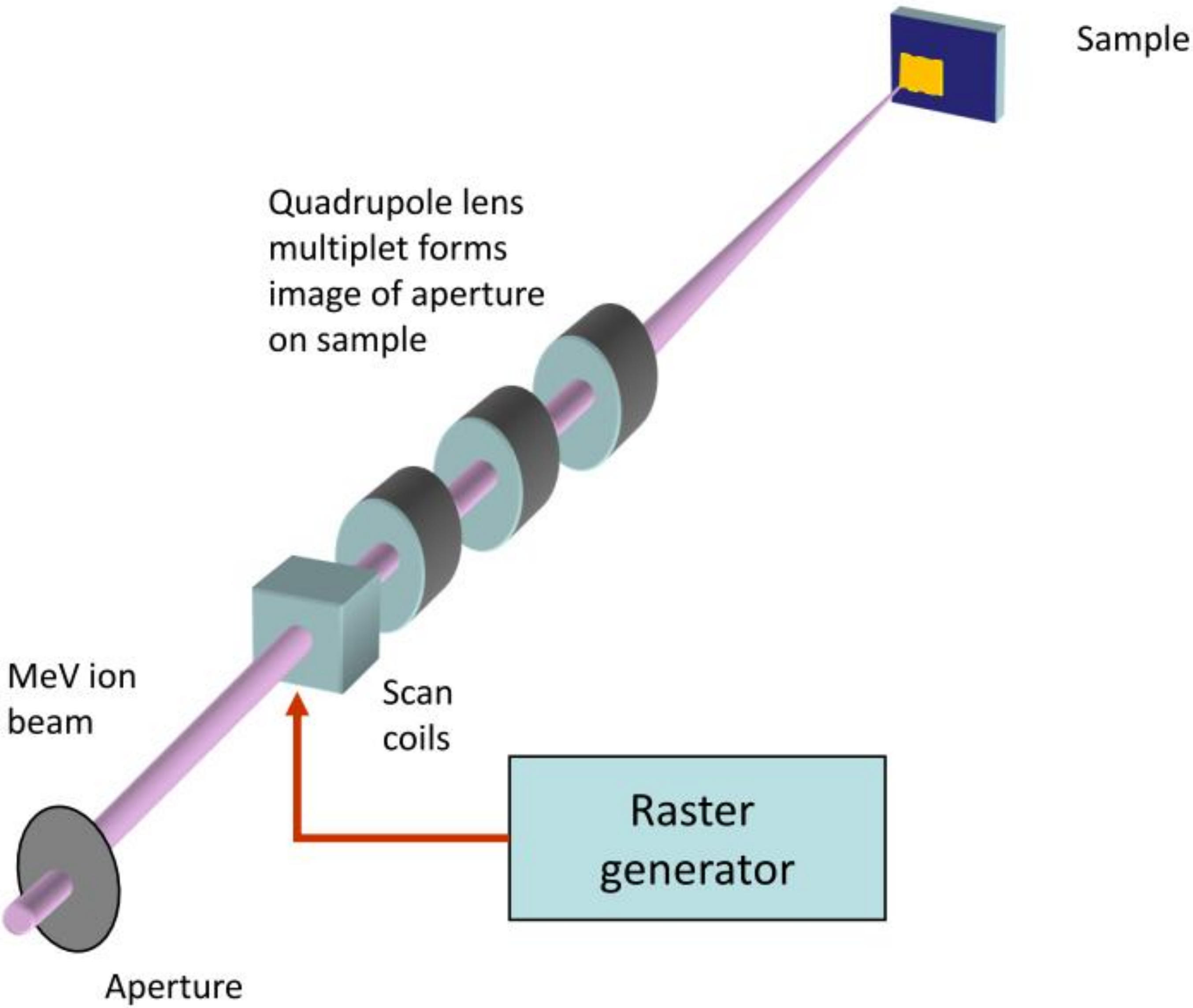
Focused ion beams



Focused ion beams



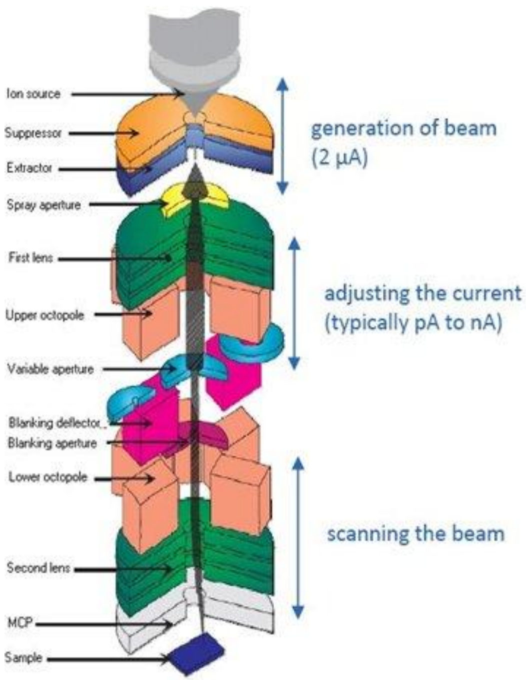
Focusing system

$M_{proton} >> M_{electron}$ → **conventional electron microscopy lenses are** not suitable:

cylindrical electrostatic lenses would require applied voltages of ~MV

to focus a 1 MeV H beam, magnetic lenses would require ~200 times higher magnetic fields with respect to a standard 30 keV electron probe ($\propto \sqrt{[\text{mass} \times \text{energy}]}$)

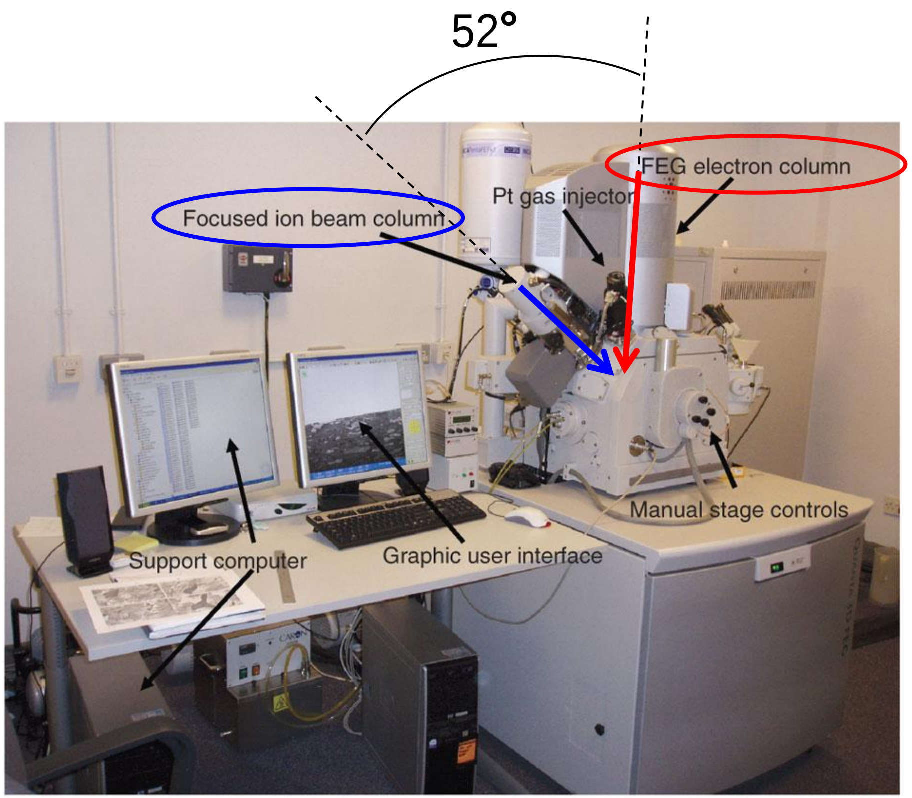
Scanning electron microscope

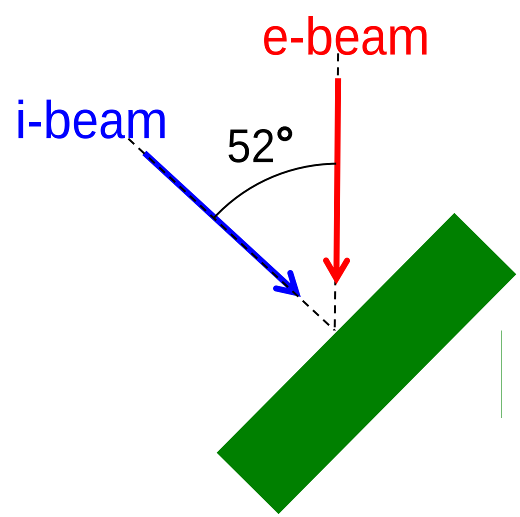


Focusing system

These limitations do not apply to ~10 keV ions → **Strong development in the last 10**-20 years of Focused Ion Beam (FIB) systems, in which the standard SEM focusing system are suitably scaled

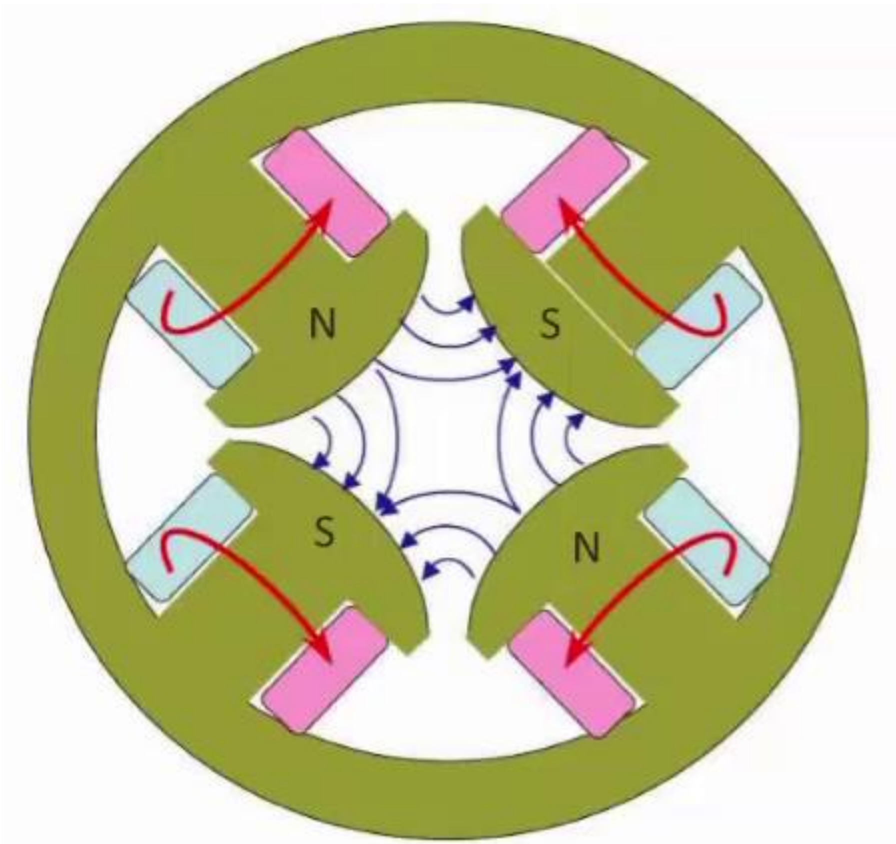
FIB (dual beam system)





Focusing system

Solution: magnetic quadrupole lenses



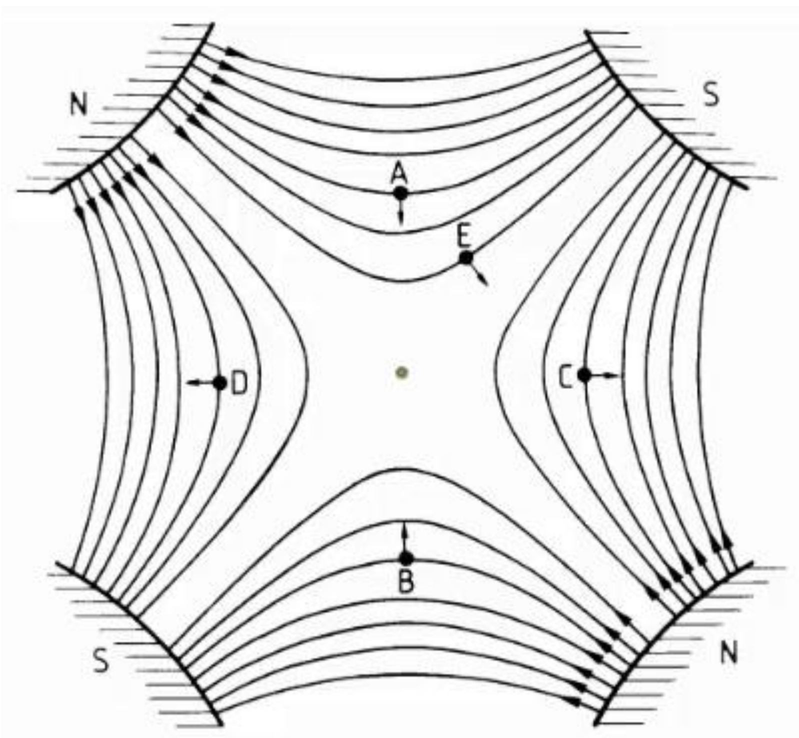
The four poles (N-S-N-S) are arranged symmetrically around the central axis to generate hyperbolic field profiles

The field strength is zero at the lens axis, and linearly increases from the axis

$B_x = - g \cdot y$
$B_y = +g \cdot x$

Focusing system

Solution: magnetic quadrupole lenses

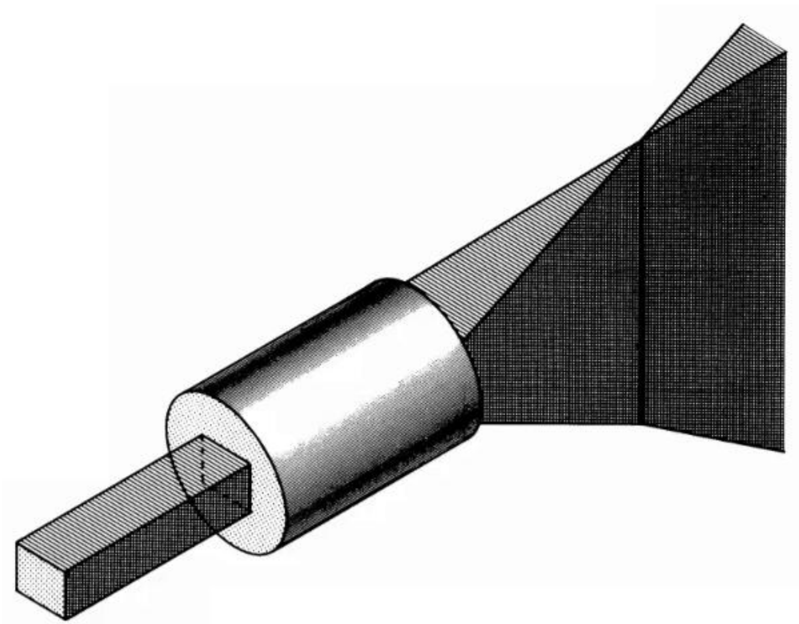


Action on a positively charged particle travelling in the direction entering perpendicularly into the plane:

→ **focusing in the Y direction**
→ **de**-focusing in the X direction

Focusing system

Solution: magnetic quadrupole lenses



Result: line focus

One single magnetic quadrupole lens is not sufficient to create a spot focus

At least two of (properly arranged) such lenses are necessary to this scope

Terminology:

C lens: lens converging ions in the X direction
D lens: lens diverging ions in the X direction

Focusing system

Solution: magnetic quadrupole lenses

Configuration	Notes
C D C D	Quadruplet arranged in the Russian configuration. The inner two quads and the outer two quads are electrically coupled. C = converging quadrupole D = diverging quadrupole.
C D C D	Quadruplet arranged as two separated doublets with an intermediate focus
C D C D	Unsymmetric quadruplet
C D C	Spaced triplet
C D C	Triplet
C D C	Coupled triplet
C D C	Coupled triplet
C D	Doublet

C = converging
D = diverging

Many different combinations of quadrupole lenses (from doublets to quadruplets) can have a spot-focusing action on fast ions

Most successful combination: "Oxford triplet**"**

Refs:

"Beam Optics of Quadrupole Probe Forming System**", G. W. Grime and F. Watt (Adam** Hilger, 1984)

"Materials Analysis using a Nuclear Microprobe", M. B. H. Breese, D. N. Jamieson, P. J. C. King (Wiley-Interscience, 1996), chapters 2-3

Focusing system

Solution: magnetic quadrupole lenses

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Most successful combination: "Oxford triplet**"**

Software (ray tracing methods):

WinTRAX
PBO
PRAM
SIMION
Geant4

Possible to model the beam paths and aberrations

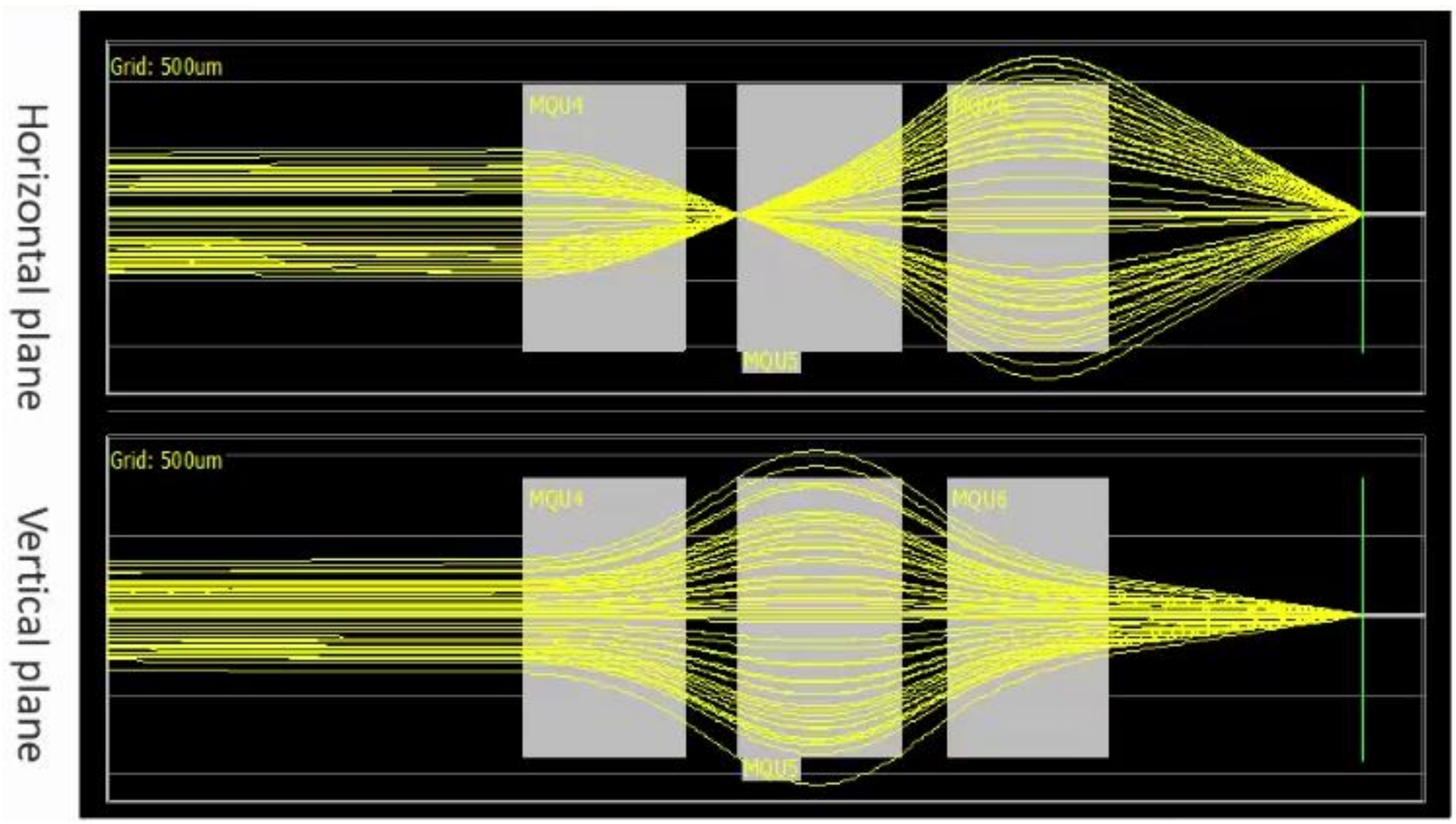
Focusing system

Example: WinTRAX simulation of the action of the Oxford triplet

C D C focal plane

X

Y



Higher demagnification factor on the X axis

Focusing system

Beam spot size: demagnification parameters + system aberrations

$$X_{image} = \underbrace{x \,/\, D_x}_{\text{demagnification}} + \underbrace{C_1 \cdot \boldsymbol{\delta} + C_2 \cdot \boldsymbol{\theta} + C_3 \cdot \boldsymbol{\phi} + C_4 \cdot \boldsymbol{\theta} \cdot \boldsymbol{\delta} + \ldots + \mathbf{C_n} \cdot \boldsymbol{\theta}^3 + \ldots}_{\text{aberrations}}$$

where

- $D_{x/y}$: demagnification of the lens system (note: in general, $D_{x/y} \neq D_{x/y}$)
- $\boldsymbol{\delta}$: energy spread of the beam
- $\boldsymbol{\theta}$, $\boldsymbol{\phi}$: initial divergences of the beam
- C_i: aberration coefficients

Aberrations: intrinsic (due to beam optics) or parasitic (external factors)

Ion beam current → Beam divergence → Beam spot

Focusing system

Intrinsic aberrations

Parameter	Cause / effect	Remedy, notes
1. System demagnification	High demagnification systems are more efficient at producing small spot sizes	Re-arrange lens configuration to produce high demagnification Increase object/image distance ratio Reduce lens-to-image distance
2. Astigmatism	X and Y planes not coincident	Adopt better focusing procedures
3. Chromatic aberration	Beam broadening due to beam energy spread	Improve accelerator energy stability Reduce beam divergence at the expense of beam current
4. Spherical aberration	Beam spot broadening due to intrinsic beam optics aberration	Reduce beam divergence (and thus the beam current) with collimation slits

Ref.: Nucl. Instr. Meth. Phys. Res. B 104, 101 (1995)

Focusing system

Parasitic aberrations

Parameter	Cause / effect	Remedy, notes
5. Rotational misalignment	Rotational misalignment between quadrupoles → major effect on beam broadening	Rotational adjustment
6. Translational misalignment	Relatively minor effects	Aligning quadrupoles
7. Sextupole / octupole contaminations of quadrupole field	Poor construction / design of lens, inferior quality of magnet iron	Use high quality lenses Reduce beam divergence at the expense of beam current
8. Fluctuations / long-term variations of quadrupole field	Unstable lens power supplies	Use high quality lens power supplies
9. Unwanted beam spot movement due to instabilities in scan system	Noise, ripple, non-linearity or instability in scan drive current/voltage	Improve quality of scan drivers
10. Unwanted beam spot movement due to external AC magnetic fields	External magnetic fields from motors, transformers, etc.	Apply magnetic screens to the most critical parts of the beam line

Ref.: Nucl. Instr. Meth. Phys. Res. B 104, 101 (1995)

Focusing system

Parasitic aberrations

Parameter	Cause / effect	Remedy, notes
11. Mechanical movement of the target	Poor temperature/mechanical stability of the target stage/lenses	Improve design of the target chamber and vibration isolation Temperature control of the target chamber and lenses
12. Slit scattering	Slit surface cause multiple scatterning and ion energy loss resulting in beam halo	Use heavy metal slits with finely polished surfaces Use higher demagnification systems → larger apertures possible
13. Poor vacuum	Residual molecules in the vacuum system cause ion scattering and energy loss	Use high vacuum components and pumps
14. Movement of target or lenses due to cooling fan	Mechanical fluctuations due to cooling fan rurbulence	Use laminar flow fans

Ref.: Nucl. Instr. Meth. Phys. Res. B 104, 101 (1995)

Focusing system

“State of the art” beam spot sizes:

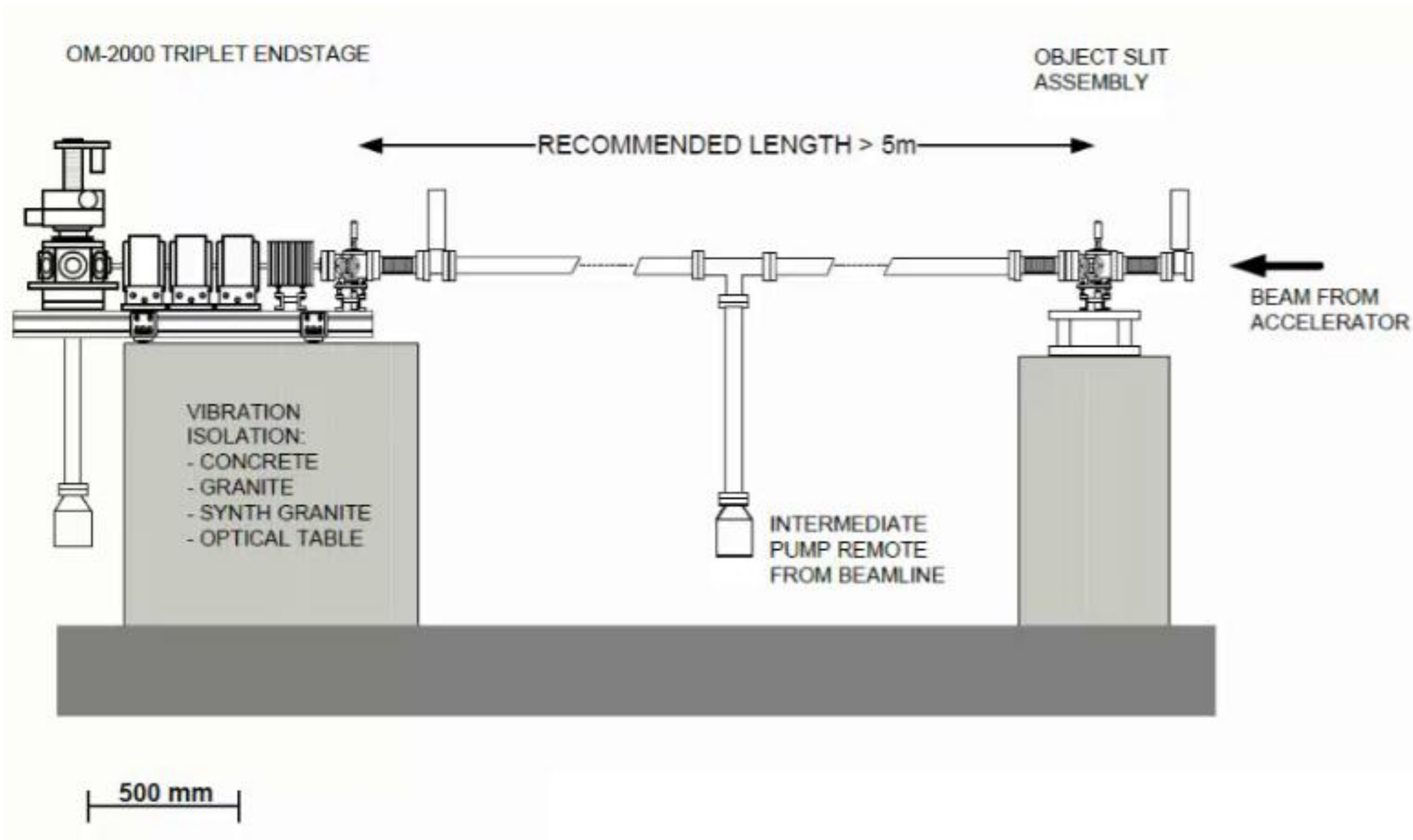
~10 nm (protons), ~20 nm (alpha)

@ Centre for Ion Beam Applications
Department of Physics
National University of Singapore

More common values are ranging
between ~10^2 nm and 10 µm

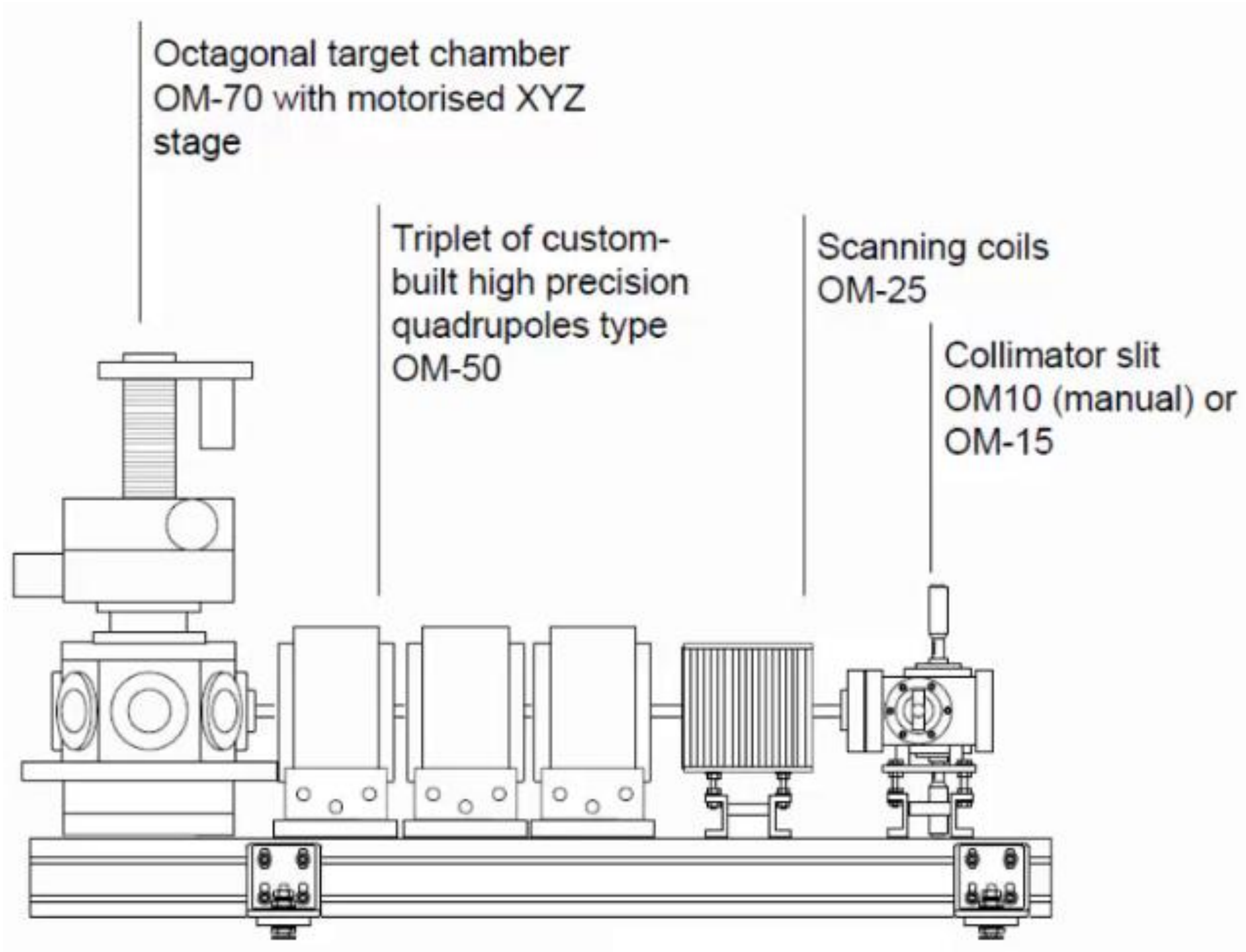
@ different facilities around the world

Ion microbeam line



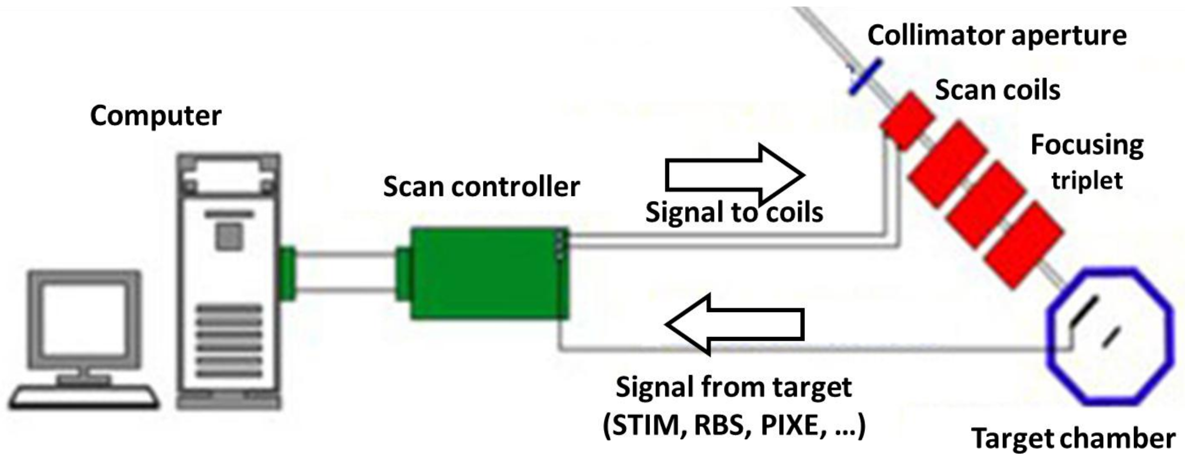
Source: Oxfod Microbeams Ltd.

Ion microbeam line



Source: Oxfod Microbeams Ltd.

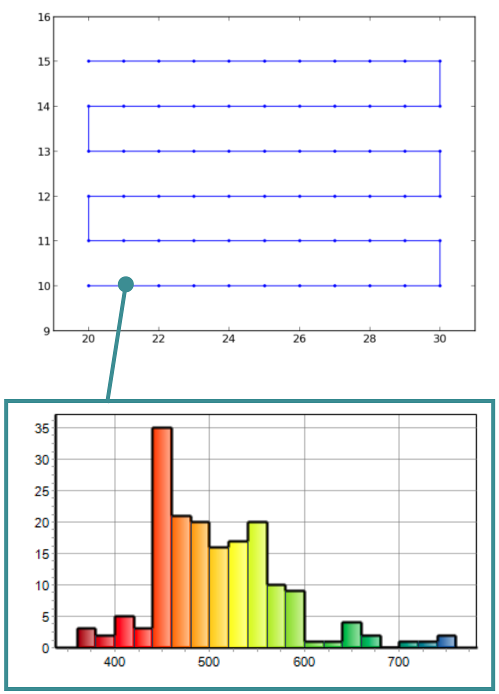
Acquisition systems



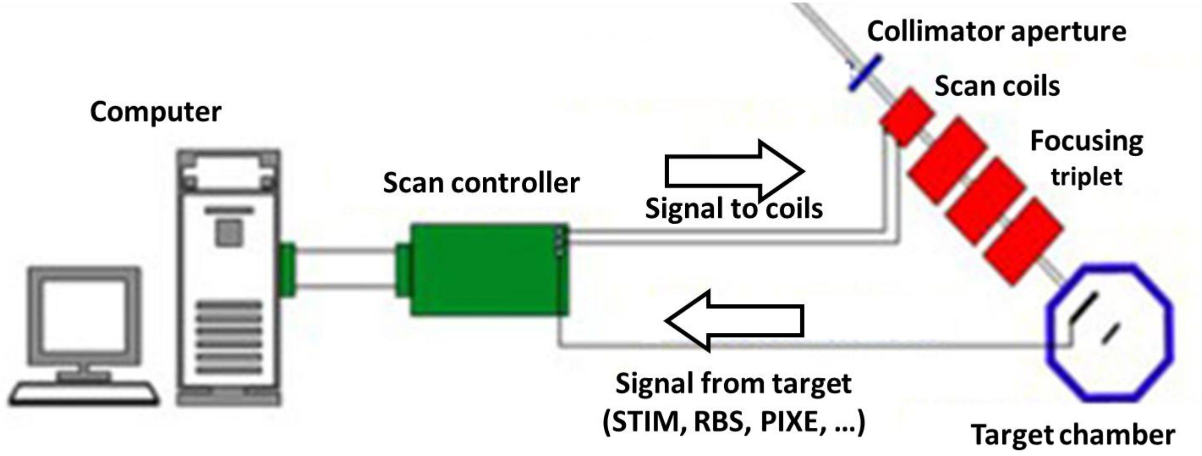
The micrograph is acquired on a "pixel by pixel" basis from the raster-scanned area

For each XY spot position, the acquisition system typically records a spectrum of a given number of signals of variable amplitudes, which codify different types of physical events (number and energy of transmitted/backscattered ions, number and energy of emitted X-rays, etc.)

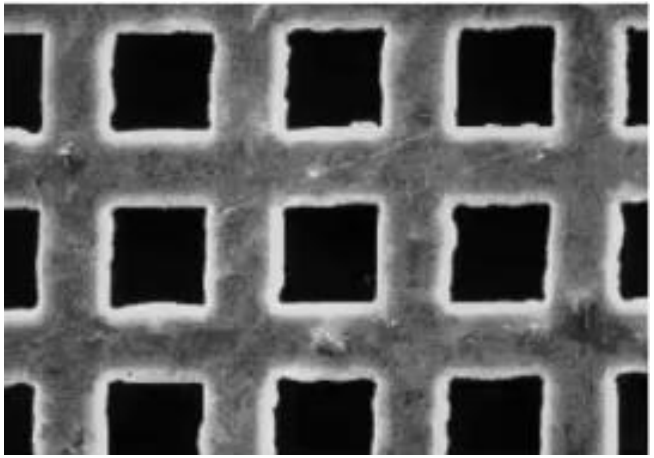
In this way, over multiple scans, a map is reconstructed which can codify different types of information extracted from the acquired spectra (overall number of signals, number of signals within a given channel window, etc)



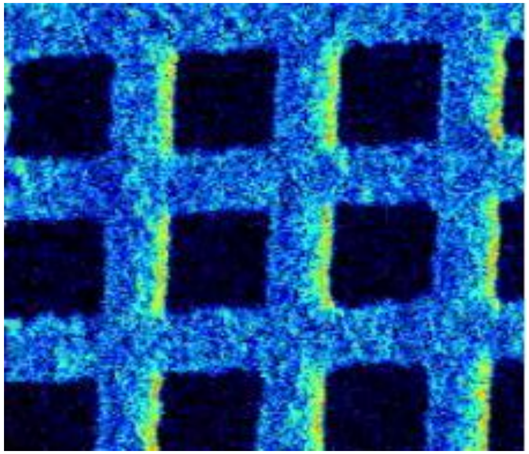
Acquisition systems



Example: secondary electron emission map from a calibration 2000-mesh metal grid



SEM micrograph

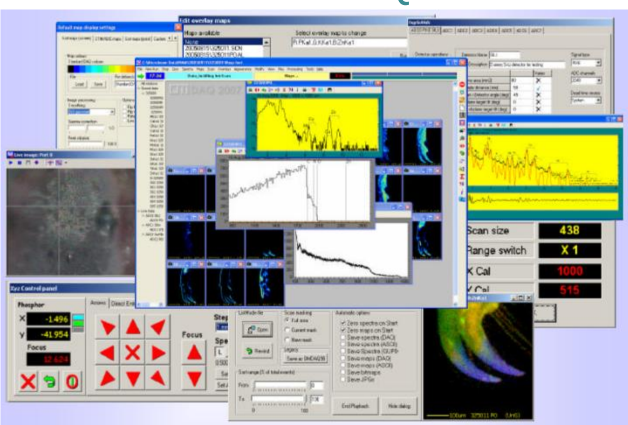


Scanning-ion-µbeam micrograph

Acquisition systems

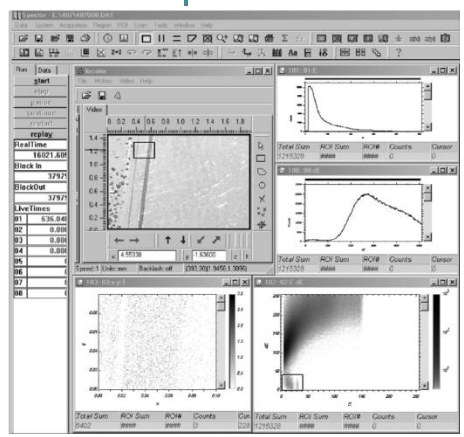
Different acquisition systems have been developed

OMDAQ



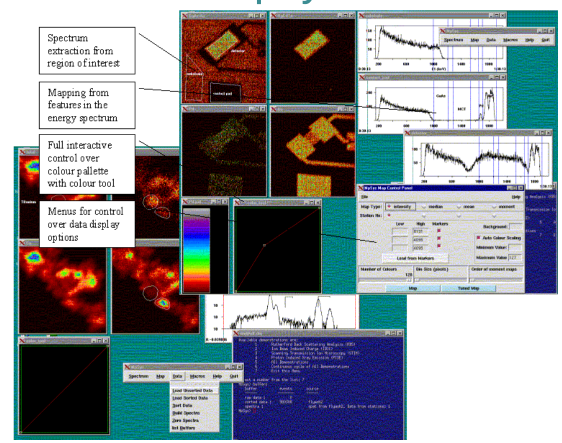
(Oxfod Microbeams Ltd.)

Spector



(**Ruđer Bošković Institute**)

MpSys



(Melbourne University)

… plus several other home-developed systems

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Ion-beam microscopy techniques

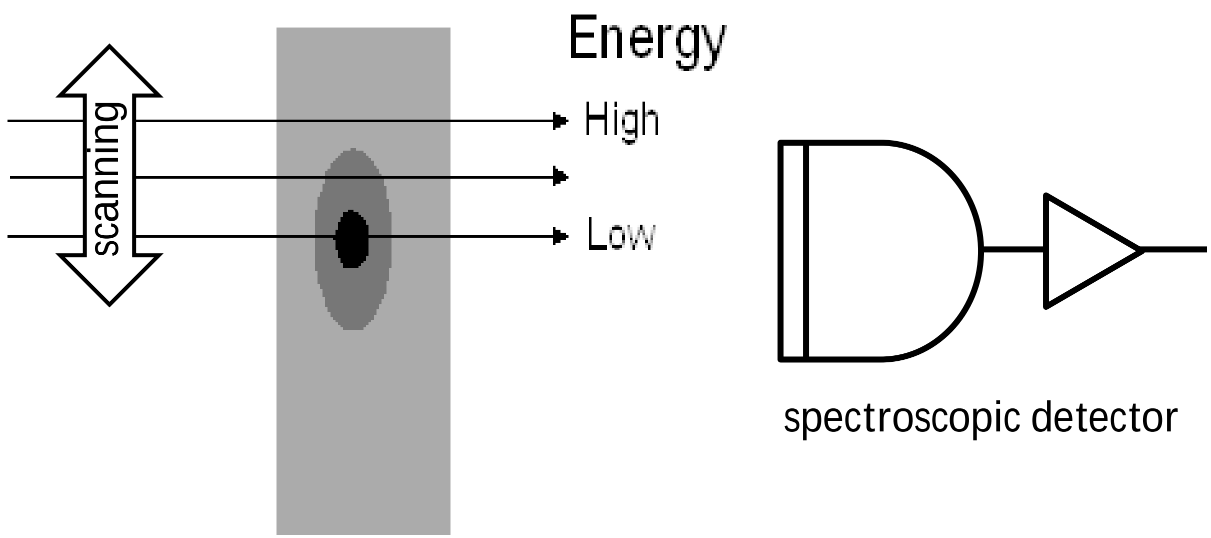
Scanning transmission ion microscopy (STIM)

Acquired signal (pixel by pixel): energy spectrum of transmitted ions

Typically, light ions (H, He) are employed because they are more penetrating → **Possibility of** mapping (relatively) thick samples

H: higher penetration depth → thicker samples (up to few tens of µm)
He: higher energy loss → higher contrast

Ion energy loss **(mainly electronic)→ Local information on the** structure and density variations within the sample

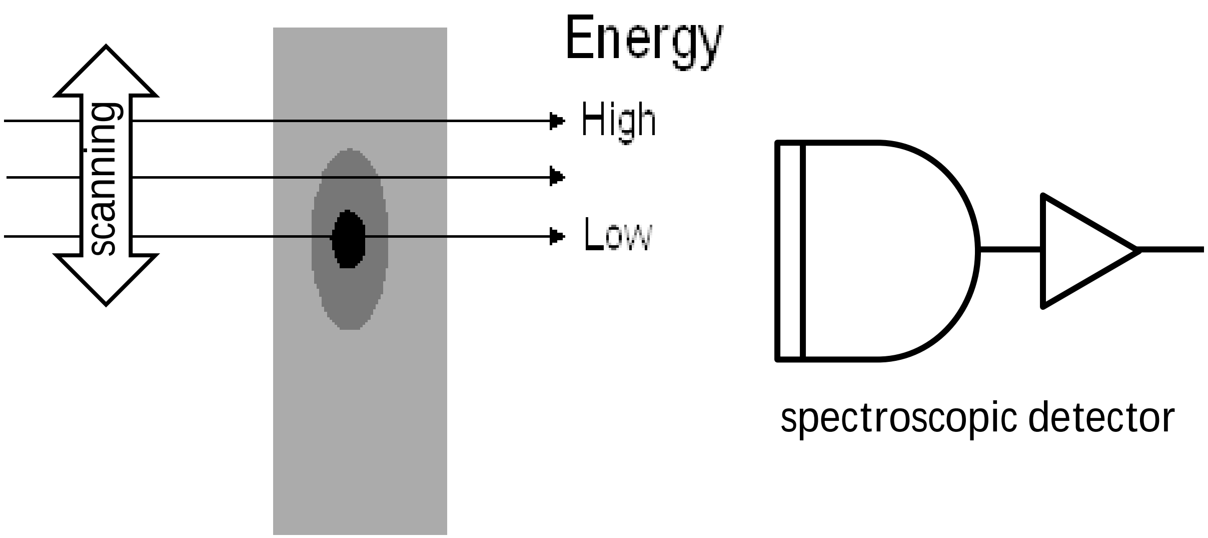


Ion-beam microscopy techniques

Scanning transmission ion microscopy (STIM)

Applications:

→ **scanning system** calibration (quick & simple mapping process)
→ **structural / density characterizaton**
→ **no compositional information**
→ correlation with compositional maps (RBS, PIXE)
→ **samples: both materials and biological (cells, …)**

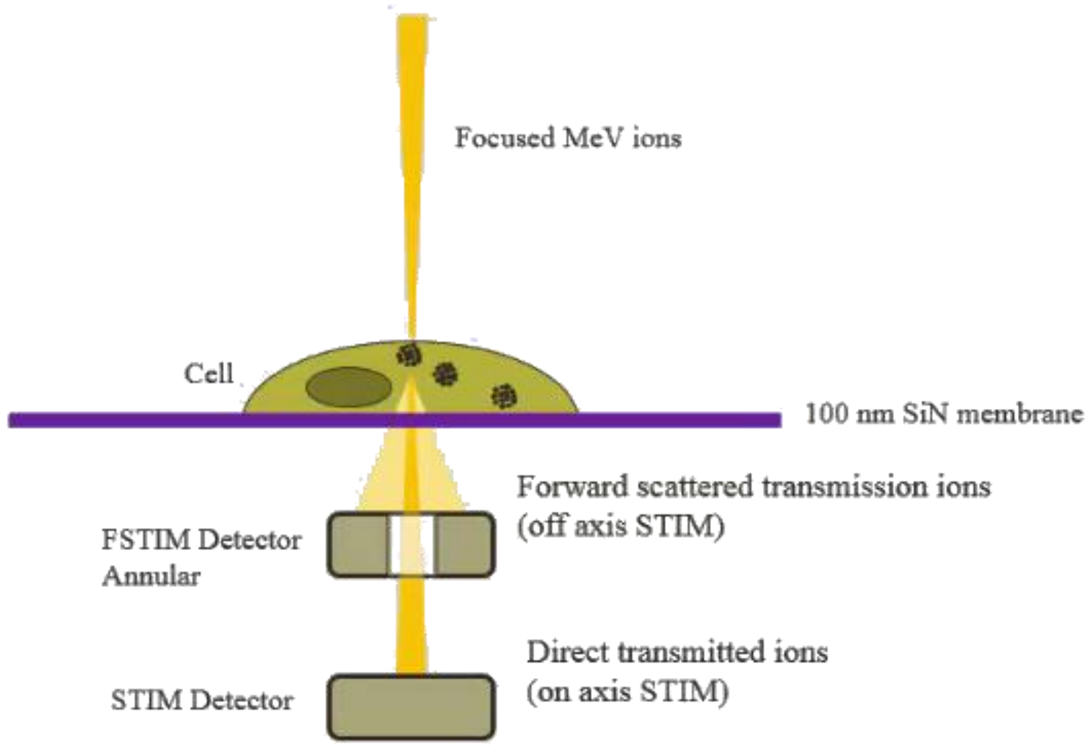


Ion-beam microscopy techniques

On-axis and Off-axis STIM

Off-axis: annular detector, forward scattered ions, nuclear energy loss
($\boldsymbol{\sigma} \propto Z^2$ → **discrimination between** light and heavy elements)

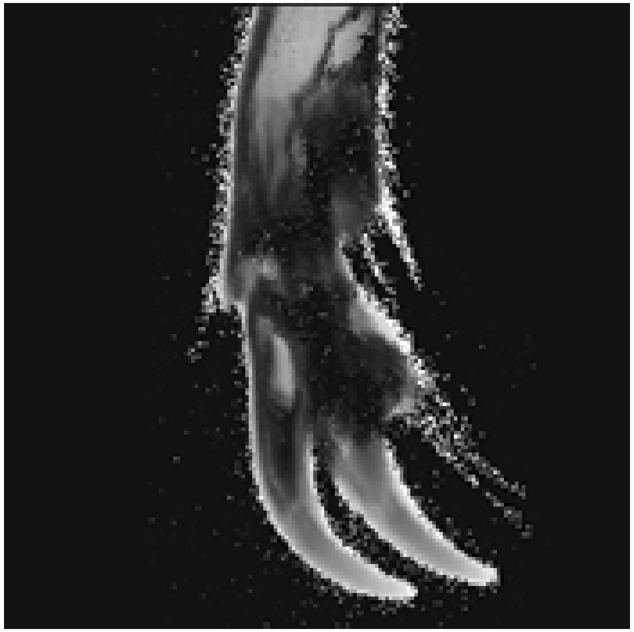
On-axis: planar detector, direct transmitted ions, electronic energy loss
(→ electronic density)



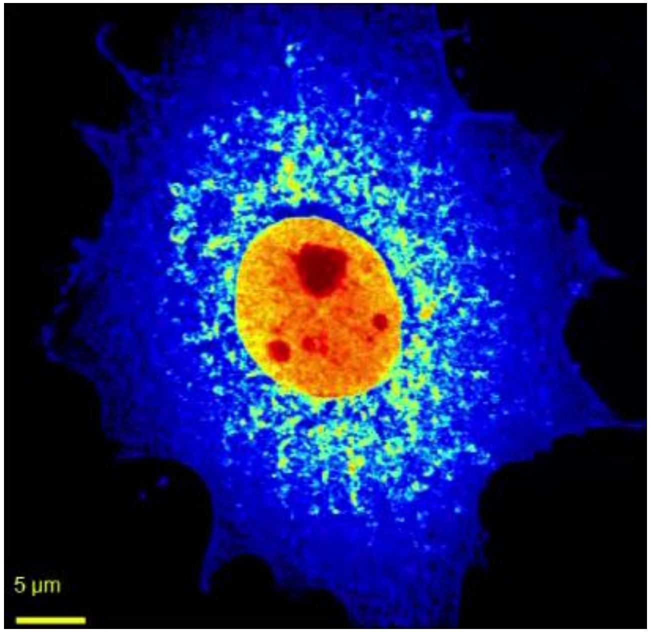
Ion-beam microscopy techniques

Scanning transmission ion microscopy (STIM)

Examples: biological samples



Leg & claw of a wasp
showing internal details



Human liver cell

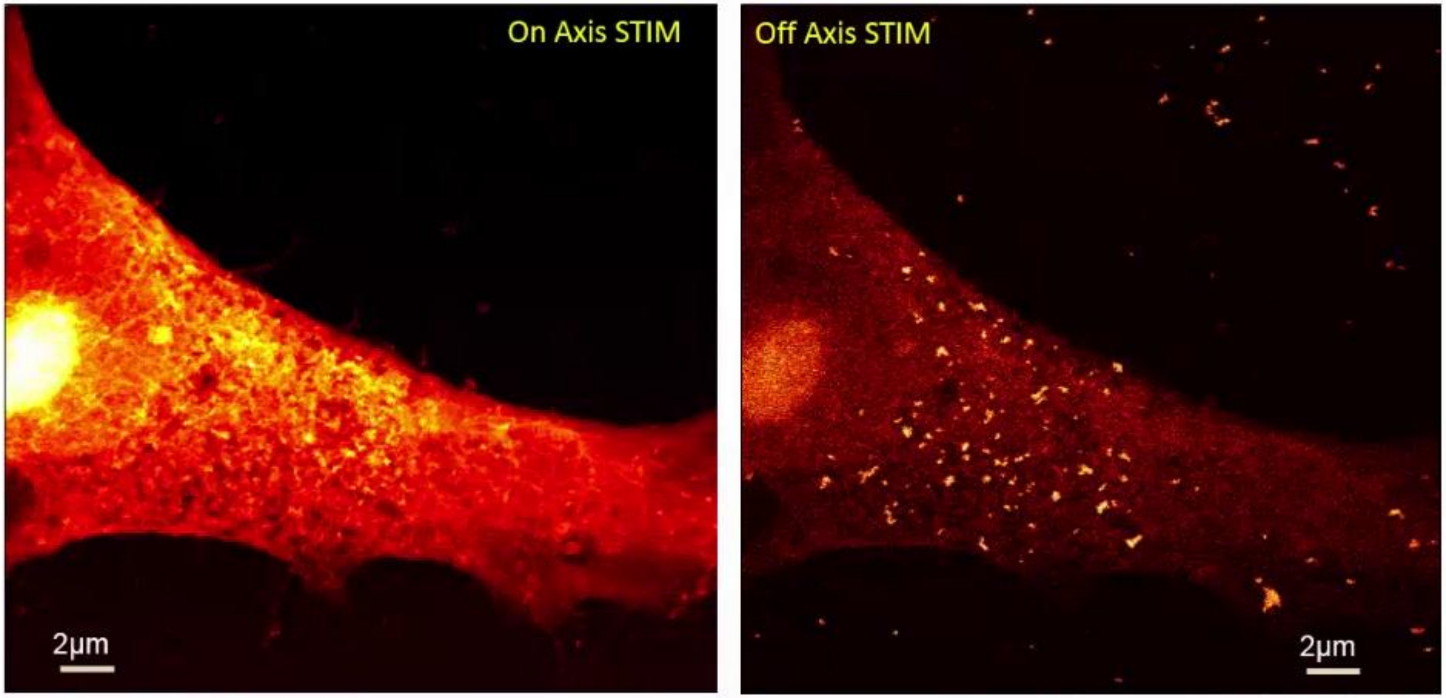
Source: Porto Alegre Ion Beam Centre

Ref.: Biophys. J. 101 (7), 1788 (2011)

Ion-beam microscopy techniques

Scanning transmission ion microscopy (STIM)

Examples: on-axis vs off-axis STIM



Gold nanoparticles in HeLa cell

Source: Centre for Ion Beam Applications, National University of Singapore

Ion-beam microscopy techniques

Ion Beam Induced Luminescence (IBIL)

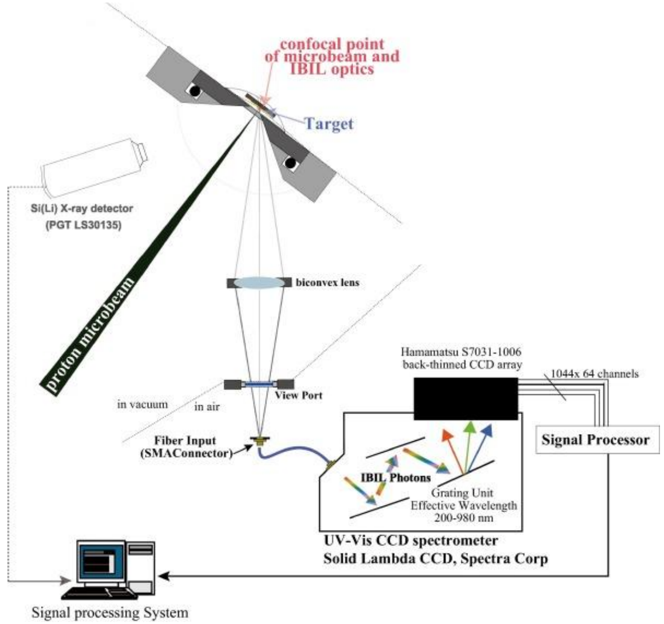
Acquired signal (pixel by pixel): energy spectrum of ion-induced photon emission

Luminescence excitation: via electronic energy loss

Information:

→ **mapping of structural phases characterized by different fluorophores**

→ **mapping of fluorescent labelling particles in biological samples**

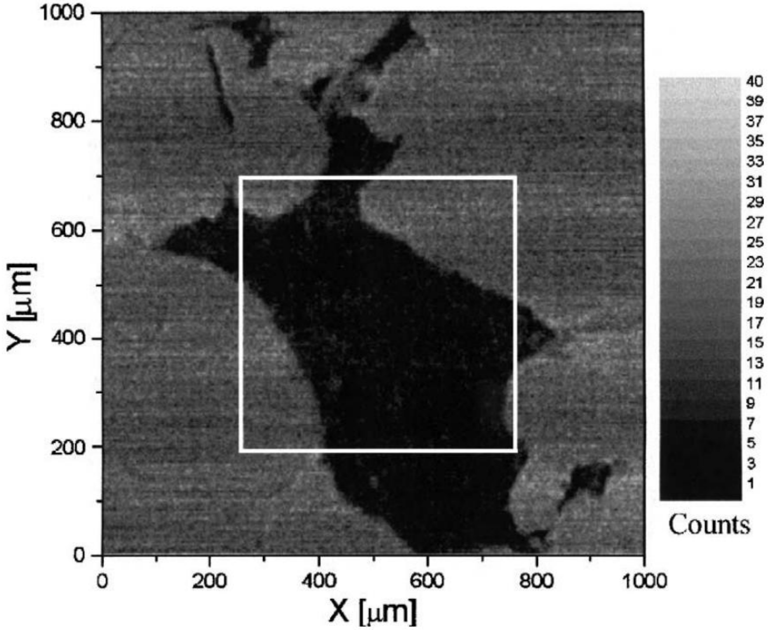


Ref.: Nucl. Instr. Meth. Phys. Res. B, in press, DOI: 10.1016/j.nimb.2017.01.025 (2017)

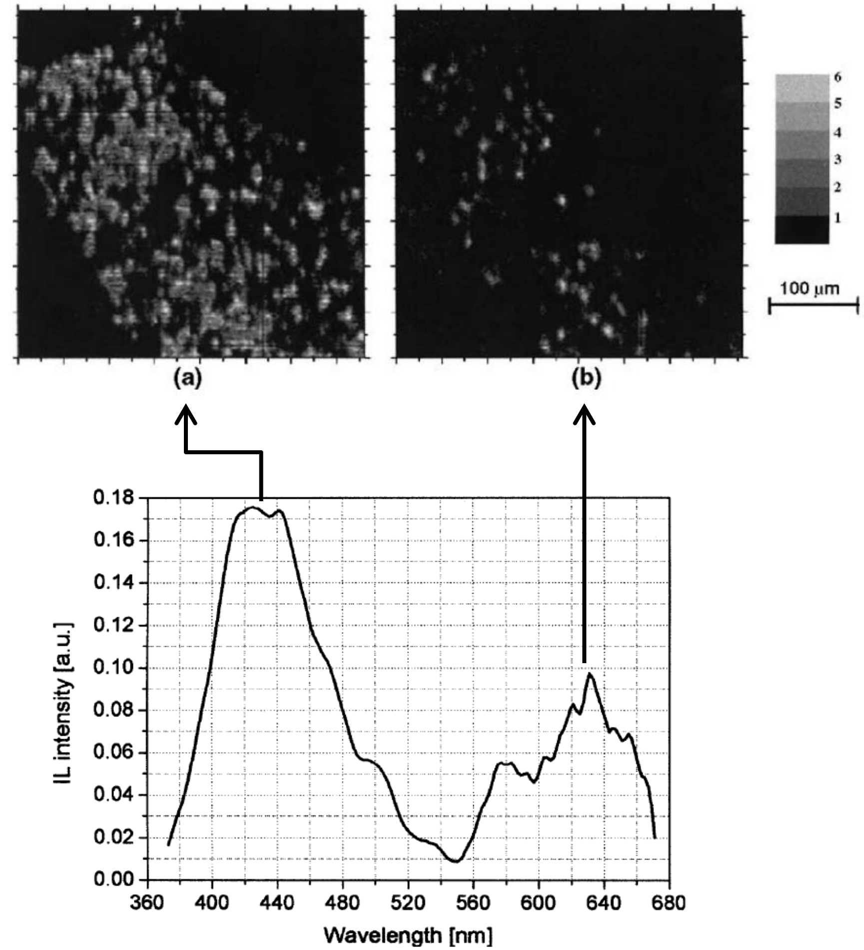
Ion-beam microscopy techniques

Ion Beam Induced Luminescence (IBIL)

Examples: geological samples with diamond inclusions



PIXE map of a C inclusion in meteorite



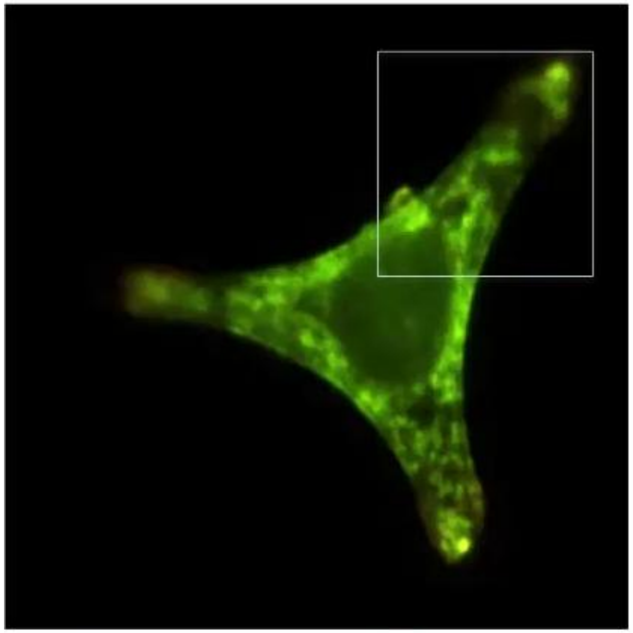
IBIL maps and spectra

Ref.: Nucl. Instr. Meth. Phys. Res. B 210, 429 (2003)

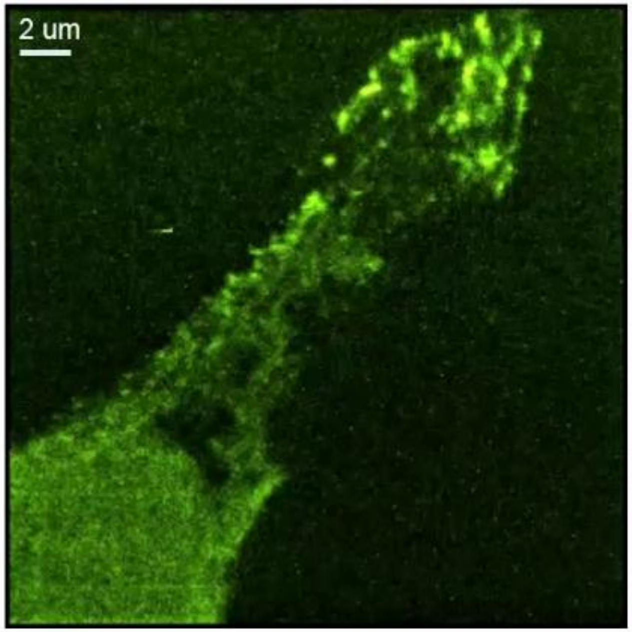
Ion-beam microscopy techniques

Ion Beam Induced Luminescence (IBIL)

Examples: biological samples
(luminescent markers in HeLa cells)



Photoluminescence map



IBIL map

Source: Centre for Ion Beam Applications, National University of Singapore

Ion-beam microscopy techniques

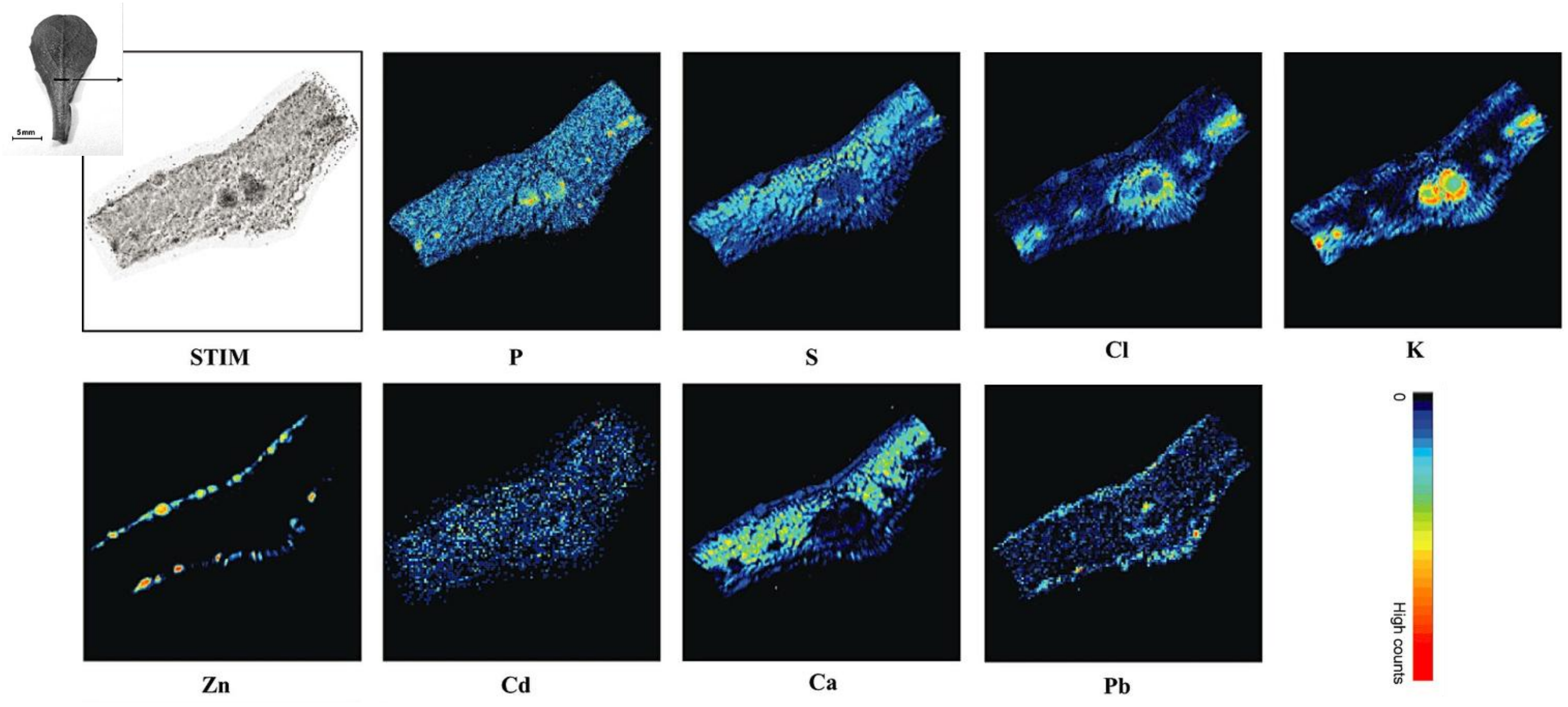
Other ion-microscopy techniques

All of the Ion Beam Analysis techniques can be "declined" as microscopy techniques provided that a scanning micro/nano-beam is employed to produce a map:

→ PIXE microscopy
→ RBS microscopy
→ IBIC microscopy
…

Ion-beam microscopy techniques

PIXE microscopy

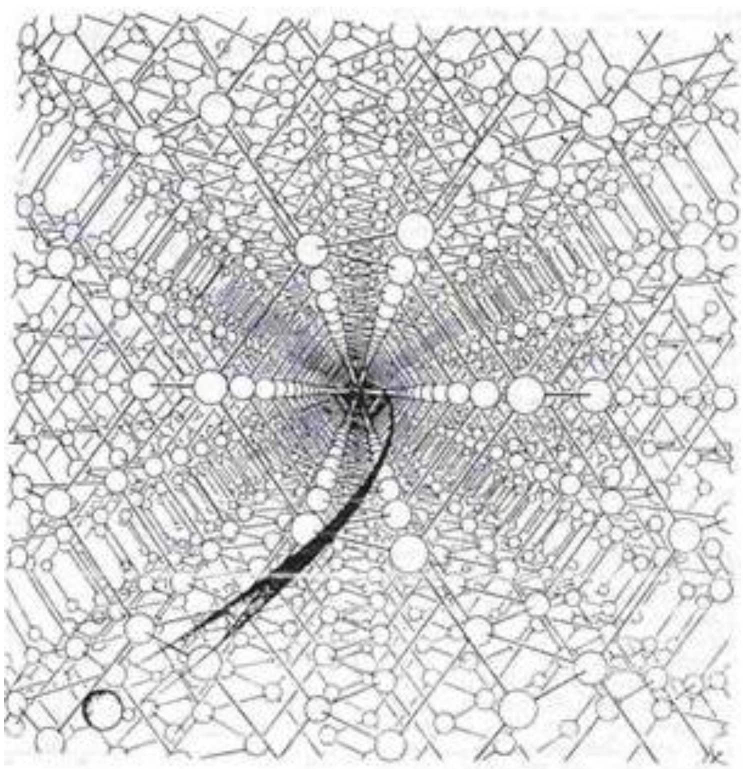


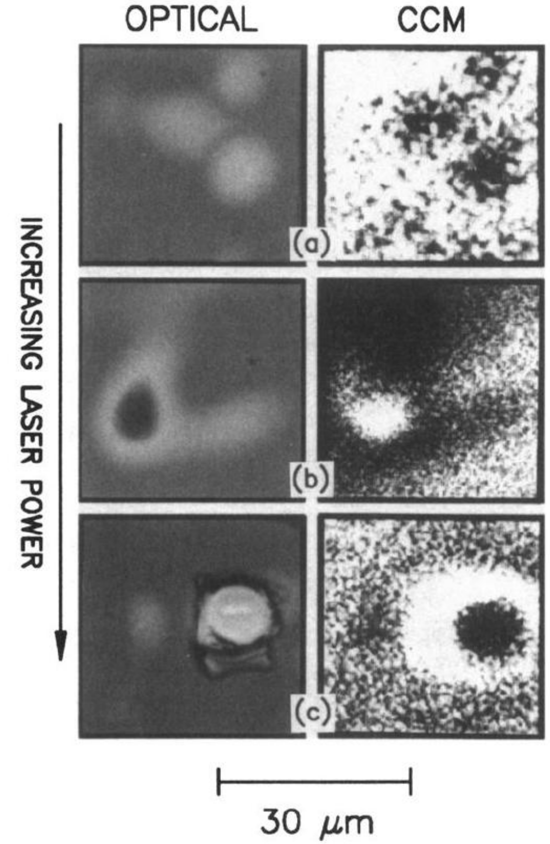
Cross section of a cryo-fixed and freeze-dried leaf of Thlaspi praecox leaf

Ref.: Plant Cell Environ. 31 (10), 1484 (2008)

Ion-beam microscopy techniques

Channeling RBS microscopy



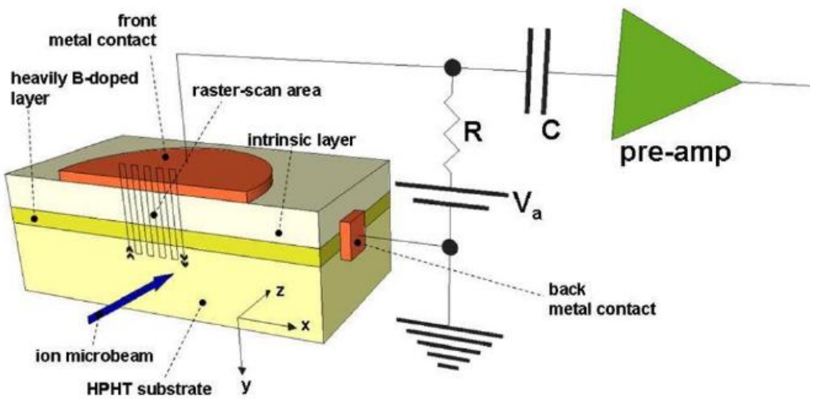


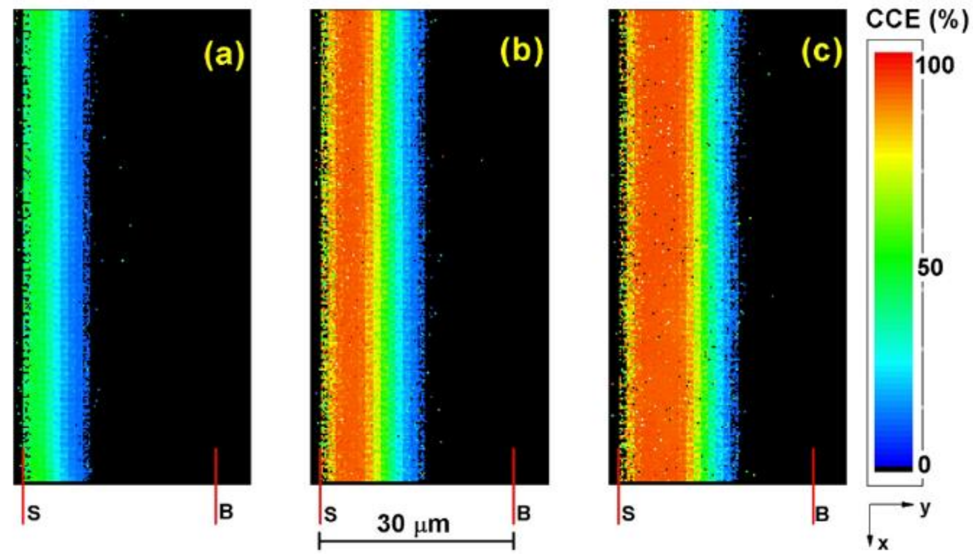
C-RBS mapping of locally laser-damaged diamond

Ref.: Phys. Rev. Lett. 69 (20), 2991 (1992)

Ion-beam microscopy techniques

IBIC microscopy





IBIC mapping of the depletion region of a diamond-metal Schottky junction

Ref.: Phys. Stat. Sol. - RRL 5 (2), 80 (2011)