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#### **SCHOOL ON ION BEAM ANALYSIS** AND ACCELERATOR APPLICATIONS

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Microprobes and beam optics

**Geoffrey GRIME** University of Surrey, Guildford, U.K.

### **Microprobes and Beam Optics**

## Focusing MeV ions to micron and sub-micron dimensions

**Geoff Grime** 

University of Surrey, Guildford, U.K.

### Outline of talk

 Charged particle beams -Forces on particles -Focusing devices Theoretical methods The present generation o microbeams **Spot size limits** Nanometre and single ion systems

# **Nuclear Microscopy**



# We want to achieve:

- Micron or nanometre spatial resolution
  - Biological cells
  - Features on semiconductor devices
  - Aerosol/sediment particles
  - Zoning and inclusion in rocks
- 100pA beam current (or more)
  - for good detection limits in PIXE/RBS
    OR
- Precisely Positioned (and counted) Single Ions (PPSI)
  - For probing or modifying materials on a nanometre scale

# Collimated ion microbeams

Use an aperture to mask the beam:

- ✓ Simple
- Current falls rapidly with diameter
- Scattered particles and background signals from aperture affect the analysis
- \* The beam cannot be scanned rapidly



# Focused ion microbeams

Use a lens to focus the beam:

- ✓ Form a spot smaller than the collimating aperture
- ✓ Reduce the effect of slit scattering
- ✓ Rapid scanning (electromagnetic)
- > Difficult to make lenses for MeV ions



Some basics ...

# What is a beam?

- An ensemble of particles moving in such a way that the velocity in one direction is much greater than in the other directions.
- Each particle has position and velocity and can be specified by a point in 6dimensional phase space  $\{x, v_x, y, v_y, z, v_z\}$
- We will assume:
  - The particles do not interact with each other
  - The external forces acting on the particles are conservative (the beam is reversible)
  - $v_z >> v_x$  or  $v_y$  (i.e. the *z* axis is the beam direction)



# A simple example of phase space



Particles occupy an area in phase space. Conventionally this is assumed to be elliptical The area bounding all the points in a beam is called the EMITTANCE. For a real beam (two planes) the emittance is a volume in four-dimensional space.

$$\boldsymbol{\varepsilon}_{\mathrm{x}} = \pi \, \boldsymbol{v}_{x \, max} \, \boldsymbol{x}_{max} \qquad \boldsymbol{\varepsilon}_{\mathrm{y}} = \pi \, \boldsymbol{v}_{y \, max} \, \boldsymbol{y}_{max}$$

# Normalised emittance



We can express transverse velocities in terms of a SMALL angle (divergence) relative to the beam direction:  $v_x = \theta v_z$   $v_y = \varphi v_z$  (Gaussian or paraxial approximation)

Since  $v_z$  is proportional to  $\sqrt{E}$ , emittance can now be written as:  $\mathbf{\mathcal{E}}_x = \pi \, \theta_f \, x_f \, \sqrt{E} \, / \, \mathbf{\mathcal{E}}_y = \pi \, \varphi_f \, y_f \, \sqrt{E} \, / \, \mathbf{4}$  (units: mm.mr.MeV<sup>1/2</sup>) ( $x_f, \, y_f$  are the full diameter of the beam,  $\theta_f$ ,  $\varphi_f$  are the full divergence)

Or in four dimensions:  $\mathcal{E} = \pi \theta_f x_f \phi_f y_f E / 16 \text{ (mm}^2.\text{mr}^2.\text{MeV)}$ This is a constant of the beam

# Liouville's theorem

### **"For an ensemble of non-interacting particles moving under the action of adiabatic forces the density of points in normalised phase space remains constant"**

This is a form of 'uncertainty principle' for ion beams – if you confine the beam in space you make it more divergent and if you try to make the beam more parallel, the diameter has to be bigger.

#### Some exceptions:

- Scattering in a gas or foil: Non-adiabatic forces, emittance increases
- Cooling ring: Non-stochastic effects reduce emittance
- Highly aberrated lens: Aberrations "curl" the phase space ellipse round on itself. The actual area remains constant, but the "bounding" area required to contain all the beam increases.

# **Evolution of phase space**



Shape of phase space changes, but AREA REMAINS CONSTANT

# Brightness

The density of points in transverse phase space is called the BRIGHTNESS, and for ion beams it is defined as the ratio of beam current to emittance.

 $B = i / \mathcal{E}$  (typical units:  $\mu A \text{ mm}^{-2} \text{ mrad}^{-2} \text{ MeV}^{-1}$ )

From Liouville's theorem, this is a constant parameter of the beam, but in practice, the brightness may not be constant over the whole emittance volume:



## Acceptance

Acceptance is a property of a beam optical component or system.

It is the largest beam emittance that can be transmitted through the component without loss of flux. Units are the same as un-normalised emittance (e.g. mm<sup>2</sup>.mrad<sup>2</sup>). The *shape* of the acceptance volume is also important.



# Transmitted beam current

Now we can see how to calculate how much of a given beam will be transmitted through a system



Emittance of beam transmitted through system is intersection of beam emittance and system acceptance:  $\mathcal{E}_{T}$ .

Current = B.  $\mathcal{E}_{T}$ 

Beam optics is the art of matching emittances to acceptances

# Forces on charged particles

Charged particles are accelerated, steered and focused using combinations of electrostatic and magnetic fields B

$$\overrightarrow{P} = qE$$

Electrostatic

#### In a uniform electrostatic field:

- Particles are accelerated (energy increases)
- Paths of particles are straight lines
- Field required for a given path is proportional to (*Energy*/q) – independent of mass

 $\begin{array}{c} \mathbf{B} \\ \mathbf{P} \\ \mathbf{Q} \\ \mathbf{P} \\ \mathbf{$ 

#### In a uniform magnetic field:

- Particle energy stays constant (force is always normal to direction)
- Paths of particles are circles
- Field required is proportional to  $\sqrt{(mass \times energy/q^2)}$  or momentum/charge

Calculating the path of a particle through an optical system. 1: Numerical raytracing



- Set up a mathematical model for the distribution of fields and solve the relativistic equation of motion numerically
- Accurate method, but time consuming and complex. Useful for specific applications (e.g. microbeam).

# Raytracing programs

- TRAX / OXRAY
  - Specifically for microbeams. VMS software
- GEANT4
  - CERN simulation software under development to treat microbeam systems
- ZGOUBI
  - From CEA Saclay, France

Incerti, S., R.W. Smith, M. Merchant, G.W. Grime, et al., *A comparison of ray-tracing software for the design of quadrupole microbeam systems.* NIM **B231** 76-85 (2005).

Calculating the path of a particle through an optical system. 2: Matrix methods



- Derive an analytical expression for a transfer matrix for the system and use this to calculate the emerging beam parameters
- This method is not so accurate (some assumptions are made) but is "quick and dirty" and can be used to get a very fast idea of the design of an optical system

# Some properties of the first order transfer matrix



1.  $Det(\mathbf{M}) = 1$  (*ad-bc* = 1) This is a consequence of Liouville's theorem

2. At a focus, x does not vary with  $\theta$ and so b = 0. In this case d = 1/a. a is the magnification, d is the demagnification.

3. Alternative notation (useful for higher order effects:

$$a = \langle x | x \rangle; b = \langle x | \theta \rangle; c = \langle \theta | x \rangle etc...$$

### Combining optical elements



Overall transfer matrix is  $\mathbf{M}_T = \mathbf{M}_N \mathbf{M}_{N-1} \dots \mathbf{M}_4 \mathbf{M}_3 \mathbf{M}_2 \mathbf{M}_1$ 

# Matrix programs available

### • TRANSPORT

– Available in PBOlab

- MULE
  - Free copy on CD
- Many others...

# Problems focusing MeV ions

- MeV ions are much more rigid than keV electrons so we cannot use solenoidal electron lenses.
- Alternatives?
  - Quadrupoles (electrostatic or magnetic)
  - Superconducting solenoid
  - Plasma lens
  - Other electrostatic devices

# Quadrupole lenses

# Magnetic Quadrupoles



- Four poles arranged symmetrically about the beam axis
- Hyperbolic field profiles:

$$B_x = -gy, \quad B_y = gx$$

Field is normal to motion so strong force towards the axis:

$$F_x = -kx, \quad F_y = ky$$

This is the condition for focusing

but note that the lens CONVERGES in one plane and DIVERGES in the other.

# Quadrupole multiplets

• We must use at least two quadrupoles to achieve a point focus:



#### Object

An aperture which defines the beam shape to be demagnified onto the sample. Typical dimensions  $< 100 \mu m$ 

#### Collimator

An aperture which defines the maximum divergence of the beam entering the lenses. Typical dimensions < 1mm.

#### Typical length of system ~5m.

This is determined by the length of the quadrupole required to give sufficient focusing strength

# More accurate treatment of particle motion in real quadrupoles



# Aberration coefficients

Action of lens (from object to image) can be expressed as a series of polynomial terms involving  $x, y, \theta, \phi$  and  $\delta$  as well as the misalignment terms:

$$x_i = \sum C_{ijklmP} x^i y^j \theta^k \phi^l \delta^m P$$

In this expression P represents parameters such as quadrupole misalignments, the amplitude of non-quadrupole fields, quadrupole field errors etc.

*C* coefficients are aberration coefficients. Coefficients involving P are called PARASITIC aberrations. Coefficients without P are called INTRINSIC aberrations and occur even in a perfect system.

Aberration coefficients can be calculated using RAYTRACING programs. High numerical accuracy is required

### Aberrations

 In probe forming systems only a few aberrations are important (x and θ are small):



For a given spot size, the relative amounts of each component can be chosen to maximise the beam transmission

### Typical aberration coefficients

Aberration coefficients (and other parameters calculated for the standard Oxford triplet

Parameter	Standard "Oxford" triplet
Quadrupole length (mechanical)	100mm
Quadrupole bore radius	7.5mm
Coil turns per pole	12
Location of lens centres (metres from object slit)	6.75, 6.91, 7.07
Overall length (object to sample)	7.29m
Working distance (m from pole face)	0.17
Pole tip fields for 3MeV protons (T)	0.182, -0.182, 0.194
Coil currents for 3MeV protons (A)	45.3, -45.3, 48.3
D <sub>x,</sub> D <sub>y</sub>	83, -24
<b>&lt;</b> <i>x</i> <b> </b> θδ>	-347
< <b>y</b>  φδ>	897
< <i>x</i>  θ <sup>3</sup> >	262
< <i>y</i>  θ <sup>2</sup> φ>	-1590
$\langle x \theta\phi^2 \rangle$	436
< <i>y</i>  φ <sup>3</sup> >	-1400
< <b>x</b>  φρ <sub>1</sub> >, < <b>x</b>  φρ <sub>2</sub> >, < <b>x</b>  φρ <sub>3</sub> >	11.2, 18.3, -29.7
< <b>y</b>  θρ <sub>1</sub> >, < <b>y</b>  θρ <sub>2</sub> >, < <b>y</b>  θρ <sub>3</sub> >	-40.2, -65.7, 106
$\langle x \theta \varepsilon_1 \rangle$ , $\langle x \theta \varepsilon_2 \rangle$ , $\langle x \theta \varepsilon_3 \rangle$	38.3, -29.4, 344
< <b>y</b>  φε <sub>1</sub> >, < <b>x</b>  φε <sub>2</sub> >, < <b>x</b>  φε <sub>3</sub> >	360.1, -1530, 256

### Acceptance and beam current

The transmitted beam current is:  $i = B x y \theta \phi$ 

(current = **brightness** of beam x **acceptance** of lens)

So we can try to choose any combination of (x, y), $(\theta, \phi)$  to give the required spot size **and** maximise acceptance.

This gives a acceptance dependence like:

$$A = xy \theta \phi = \frac{d^{n}}{f(C)}$$
$$2 \le n \le 4$$

(f(C) is some function of the aberration coefficients {smaller is better} and ndepends on the dominant aberration)

### Acceptance and spot size

Example: first order chromatic aberration in one plane only:

$$\frac{d}{2} = \frac{x}{D} + C\theta\delta$$
$$A = x\theta = D\theta\left(\frac{d}{2} - C\theta\delta\right)$$
$$\frac{dA}{d\theta} = 0 \text{ for maximum acceptance}$$
$$A_{\text{max}} = d^2 \frac{D}{16C\delta}$$

In two planes this would lead to a  $d^4$  dependence on spot size.

In real systems the situation is much more complex and only numerical methods can give results

### Theoretical acceptance as a function of spot size for two Oxford triplets

(Spaced triplet has 5 quadrupole lengths between first two lenses)



### Microbeam performance

 $R = Y \Omega i$  (count rate=yield x detector solid angle x current)

$$i = BA = Bxy\theta\phi$$
$$A = \frac{d^{n}}{f(C)}$$



#### i.e. for small spot size we need:

- Accept a low count rate

- Low aberration lens

# The Oxford lenses

- The dominant parasitic aberrations were found to be:
  - rotational misalignment
  - contaminant 6-pole fields due to errors in four-fold symmetry of pole position
  - power supply ripple and drift
- Oxford lenses were built with:
  - single piece construction of yoke and poles to minimise 6-pole fields
  - Mounting base allowing micrometer adjustment of lens rotation
  - power supplies with <10ppm long term stability</li>
- First 1µm beam in December 1980
### OM-50 quadrupole lens



www.microbeams.co.uk



- Iron yoke cut from a single piece of high quality iron with an accuracy better than 2µm
- Accurate hyperbolic pole tips
- Undetectable 6-pole field contamination

#### The Oxford Microbeams OM-2000 endstage



### OM-52 quadrupole lens



 Miniature version for nanobeam applications (bore diameter = 7.5 mm)

Breese, M. B. H., G. W. Grime, et al. (1999). Nucl. Instr. & Meths. B158: pp48-52.

#### OM-52 triplet for vertical beam applications



#### 2MV Tandetron accelerator at University of Surrey



## Surrey microbeam line



Best microbeam performance at present: National University of Singapore using OM-2000 lenses and modern HVEE accelerator



F. Watt et al., Nucl. Instr. and Meths. B: Vol210, 2003, pp 14-20

## Applications of Nuclear Microscopy

#### **Life Sciences**

- Metal uptake by cells, plants etc.
- Trace element distribution in tissues
- Metal toxicology

#### **Materials Science**

- Composition of new materials
- Mapping crystal quality using channelling
- Studies of microelectronic devices using IBIC
- Composition of magnetic multilayers

#### **Environmental Science**

- Analysis of single aerosol, fly ash and sediment particles
- Environmental history from trace element zoning in shells

#### **Earth Sciences**

- Trace element zoning in rocks
- Trace elements in solid and fluid inclusions
- Surface weathering studies

#### **Archaeological Science**

- Fingerprinting artefacts and remains for provenance studies
- Investigation of ancient technologies (e.g. glass, metallurgy)
- Investigation of ancient lifestyles from human remains

#### Industrial/Technological

- Contamination in processes
- Polymer studies (diffusion, inclusions etc.)
- Catalyst studies
- Micromachining (MEMS)

### **Trace element geochemistry**



Partitioning of trace elements in garnet in volcanic rock from Santorini

- 1. Make elemental map to locate regions of interest
- 2. Point analysis at selected points

Scan area 2500 x 2500 μm

(Dr Alba Santo, Universita di Firenze)

### **Trace element geochemistry**



Scan area 2500 x 2500 μm

(Dr Alba Santo, Universita di Firenze)

# Metals in pupating ants



*left:* Secondary electron image of mature mandible.

*right:* simultaneous PIXE image of zinc distribution



- Mandibles of leaf cutting ants are hardened by the presence of high levels of zinc in the chitin matrix.
- Can we understand this process and exploit it to develop new materials?
- Zinc incorporation takes place during pupation. Can we use the long penetration of MeV protons to identify the sites of Zn storage and observe the mobilisation of zinc?

## Head of late stage pupa

Head of late stage pupa shows high manganese in mandibles, but no zinc.

Zinc seems to be concentrated in tip of abdomen

Manganese is associated with enzymes involved in forming bio-polymers



## **Enlargement of mandibles**



We see Mn at the cutting edge, but no Zn

# So where is the zinc?

Side view of whole pupa shows a feature in the abdomen rich in Mn, Zn, Ca.

Is this the site of metal storage? But how and when does the Zn go into the mandibles?

Note that this analysis was carried out with no sectioning or other preparation other than freeze drying.







### Can we identify this feature?



# Aerosol particles in the human respiratory tract

**Aim:** to try to understand the toxic impact of various types of aerosol particles

**Microscopic analysis:** Can we observe aerosol particles directly in the walls of the trachea and bronchi, and if so, are there differences?

**Samples:** Post-mortem tissue removed at autopsy. Fast frozen, cryosectioned at  $5\mu$ m thickness and air dried.

T. Pinheiro et al., Nucl. Instr. and Meth. B, Vol 158, 1999, pp 499-504

#### Particles in bronchus mucous membrane



#### 500x500µm

#### Bronchus: Particle agglomeration in macrophage



50x50µm

### Particles in trachea epithelium



50x50µm

## Summary:

- Isolated particles or agglomerations were observed with diameter 2 to 10  $\,\mu\text{m}$
- Particles in upper respiratory tract are mainly earth crust elements (AI, Si, Ca, Fe)
- Particles in bronchi have more varied composition (e.g., V, Cr, Mn, Fe, Cu, Zn, Ti and Ba) - technological origin
- Sub-micron particles have much greater toxicity

   could we characterise these if we had better
   spatial resolution?

## Proton beam writing (MeV Ion Beam Lithography)

# MeV ions have very low scattering in matter



# Compare this with electrons and low energy ions (FIB)



- Positive resist materials become soluble when exposed to radiation
  - Poly-methyl methacrylate (PMMA)
  - Commercial photoresists
- This can be used to make solid structures





- Positive resist materials become soluble when exposed to radiation
  - Poly-methyl methacrylate (PMMA)
  - Commercial photoresists
- This can be used to make solid structures



(Oxford 1993)



- Negative resist materials become INsoluble when exposed to radiation
  - SU-8
- Exploit the different range of ions of different energy to make three dimensional structures





- Negative resist materials become INsoluble when exposed to radiation
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# Proton beam writing

- Lateral feature size equal to beam diameter
  - Sub-100 nm possible at present
  - Smaller features with new facilities (like AIFIRA)
- Depth depending on beam energy
  - > 20µm
  - High aspect ratio
- Very smooth walls
- Features made in polymers can be transferred to other materials (e.g. by electroplating and stamping)

#### **Proton beam writing**



Matrix of 1 micron squares joined by 60 nm walls Aspect ratio >160 Use a microfocused MeV proton beam to form high aspect ratio nanostructures in photoresist.

- MEMS
- Microfluidics
- Integrated optics etc.





## Limits to the spot size?

### Practical limitations of spot size

- Mechanical stability
  - Vibration
  - Long term drift (e.g. thermal changes)
- Stray magnetic fields
- Scattering from residual gas in vacuum (halo)
- These are engineering issues, presumably capable of being solved

#### A more fundamental limitation...

Slit scattering

(can we make micron size apertures for MeV ions?)

#### Calculated optimum slit apertures for a high demagnification triplet



## Slit scattering



- Penetration of slit edges by MeV ions creates a transparency zone round the edge of the slit.
- Small angle scattering changes the direction and energy of ions
- Creates a 'halo' of scattered beam around the beam spot.

Ratio of transparency area to free aperture is: (*R*=radius of slit, d=slit aperture,  $\rho$ =range of ions)

	Slit aperture(µm)	
Range(µm)	20	1
60	0.060	1.200
10	0.002	0.033

$$\frac{2\rho^2}{Rd}$$

For 6mm jaws

Scattered beam/ Direct beam

#### Current trends in nanobeam design 1: High demagnification systems

Lich democratication allows larger object alite t

High demagnification allows larger object slits to be used (but usually means high aberration...)



#### Current trends in nanobeam design 1: High demagnification systems


#### Current trends in nanobeam design 2: Multi-stage systems



- High quality intermediate image
- Aperture at intermediate image removes halo from slit scatter
- High overall demagnification so large object aperture
- Final stage can have long working distance to help design of target chamber

#### Novel optics of Surrey nanobeam

High demagnification two stage quadrupole lens with intermediate focus



#### Nanobeam design



University of Surrey Ion Beam Centre Nanobeam facility Projected beam diameter < 20nm (low current modes) Applications in analysis and lithography Construction commencing March 2006 Will incorporate single cell irradiation facilities

## Accelerator performance

- Microbeam performance depends strongly on accelerator brightness and energy stability
- New designs of accelerators from HVEE (Amersfoort, NL) contribute strongly to performance of microbeams

- e.g., Singapore, Leipzig

• Will we see the 1nm beam in the near future?

## Single ions ...

Can we control the beam so that we can place a precise number of single ions within the beam diameter?

Precisely Positioned (and counted) Single lons

#### PPSI

B. E. Fischer et al, Nucl. Instr. and Meths. B: Vol 210, pp 285-291

#### Schematic of single ion system



- 1. Beam current is reduced to a very low level (1000s of particles / second)
- 2. Electrostatic deflection plates block the beam until START signal is applied
- 3. When the first ion hits the target the ion detector switches the deflector and blocks the beam again.

Timing of single ion system



## Critical components for PPSI

- Lens performance (spot size and HALO)
  - Determines targeting accuracy
- Beam switch
  - Must switch ~1 kV in  $<1\mu s$
- Ion detector
  - High (100%?) efficiency
  - Type depends on application: e.g.
    - Surface barrier detector
    - Secondary electron detector
    - Scintillation counter
    - Geiger-Muller detector

## **Applications of PPSI?**

- Single cell radiobiology
  - Observe the effect of single particles passing through living cells (cancer therapy, radiation safety)
- Characterisation of semiconductor devices
  - Radiation hardness testing for space applications
- Nanolithography using ion tracks
  - Tailored filter membranes
  - 3-D nanostructures
- Single ion implantation
  - Quantum dots
  - Quantum computers



A high performance connector fabricated from a bundle of nanowires created by plating through the etched tracks of single random MeV ions in photoresist

# Proposed vertical nanobeam facility

- Dedicated facility for the irradiation and analysis of cells and tissues in culture
- Collaboration between Surrey and GCI
- Funding for the tower extension obtained from the Wolfson Foundation (January 2006)
- Completion projected for Spring 2007





## Architect's views of the proposed new building





#### Further reading (books and conference proceedings)

•There are many books on charged particle beam optics. Most of the modern ones concentrate strongly on the physics of circular accelerators and are not really relevant to mircobeams. One of the earliest and probably still the clearest basic book on beam optics if you can find it is

 A P Bamford "Transport of Charged Particle Beams" (1966) Spon; ISBN: 0419103201

#### Other books with references to microbeams include

- G.W. Grime, F. Watt "Beam Optics of Quadruple Probe-forming Systems" (1983) Hilger; ISBN: 085274546X
- S.A.E. Johansson, J.L. Campbell, K.G. Malmqvist, "Particle-induced Xray Emission Spectrometry" (1995) John Wiley & Sons Inc; ISBN: 0471589446
- F Watt G W Grime (eds.), "Principles and Applications of High Energy Ion Microbeams" (1987) Publisher: Hilger; ISBN: 0852745176 (rather dated by now)
- Proceedings of the International Conferences on Nuclear Microbeam Technology and Applications