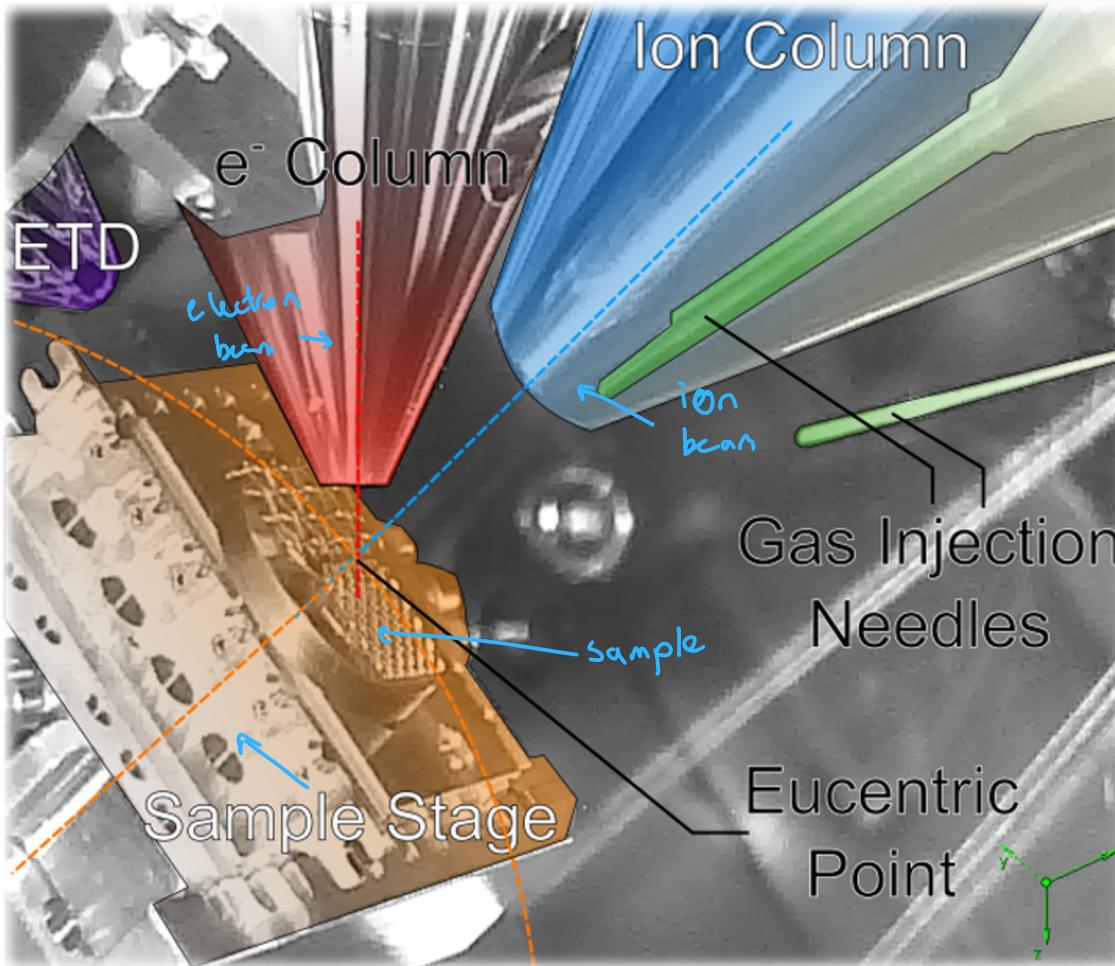




68320 Nanofabrication and Nanocharacterization Techniques



Lecture ³ ~~4~~ ???

Focused Ion Beam (FIB)
microscopy and
nanofabrication

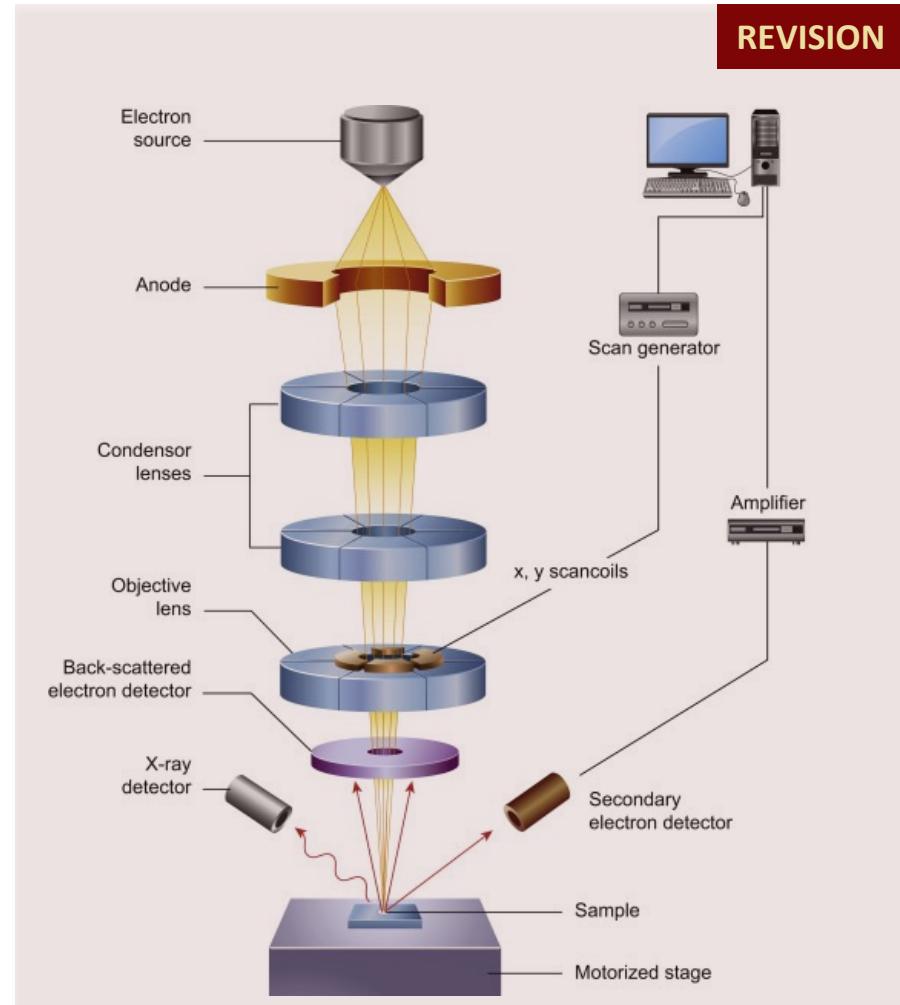
Focused ion beam (FIB) column

inside of ion beam column → conceptually the same



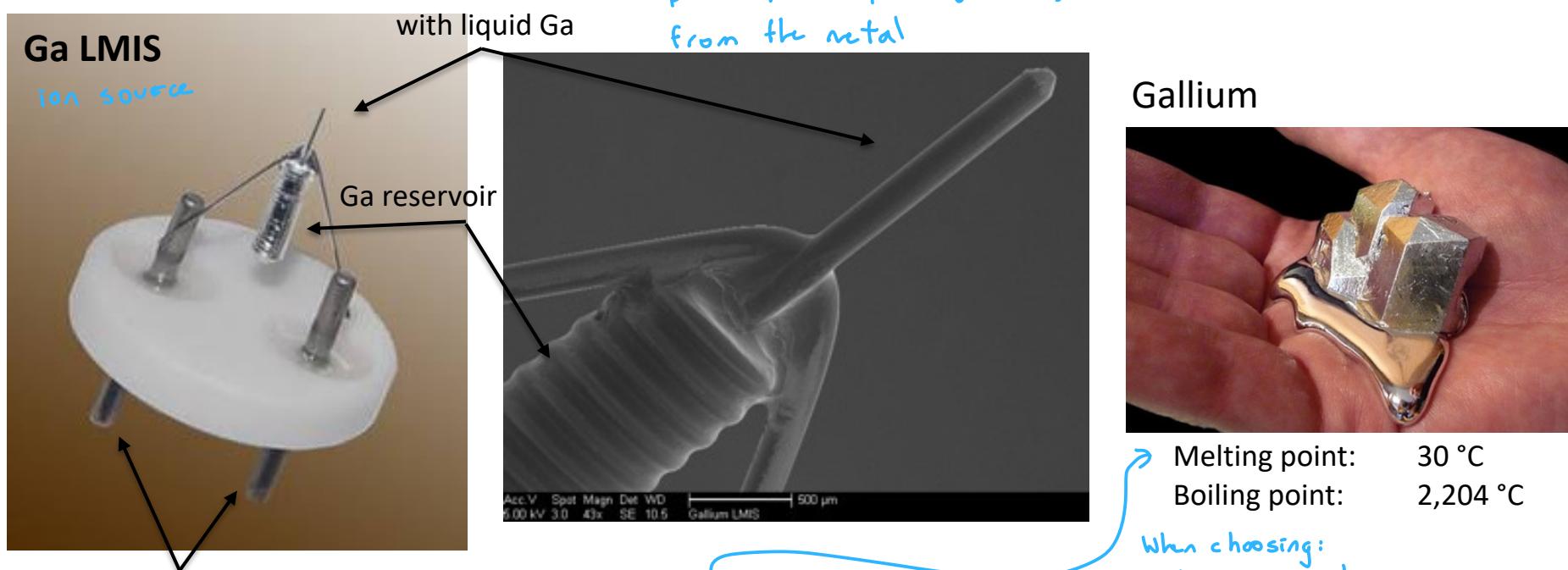
Ion source (LMIS) ↳ except positive

Similar to SEM column



REVISION

Liquid metal ion source (LMIS)



H	Li	Be	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	N	O	F	Ne
	Li	Be																He
	Na	Mg																
K	Ca		Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr		Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Lu	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac	Lr	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn						

Gallium is similar to Hg since it melts at

30 °C
Is non toxic tho

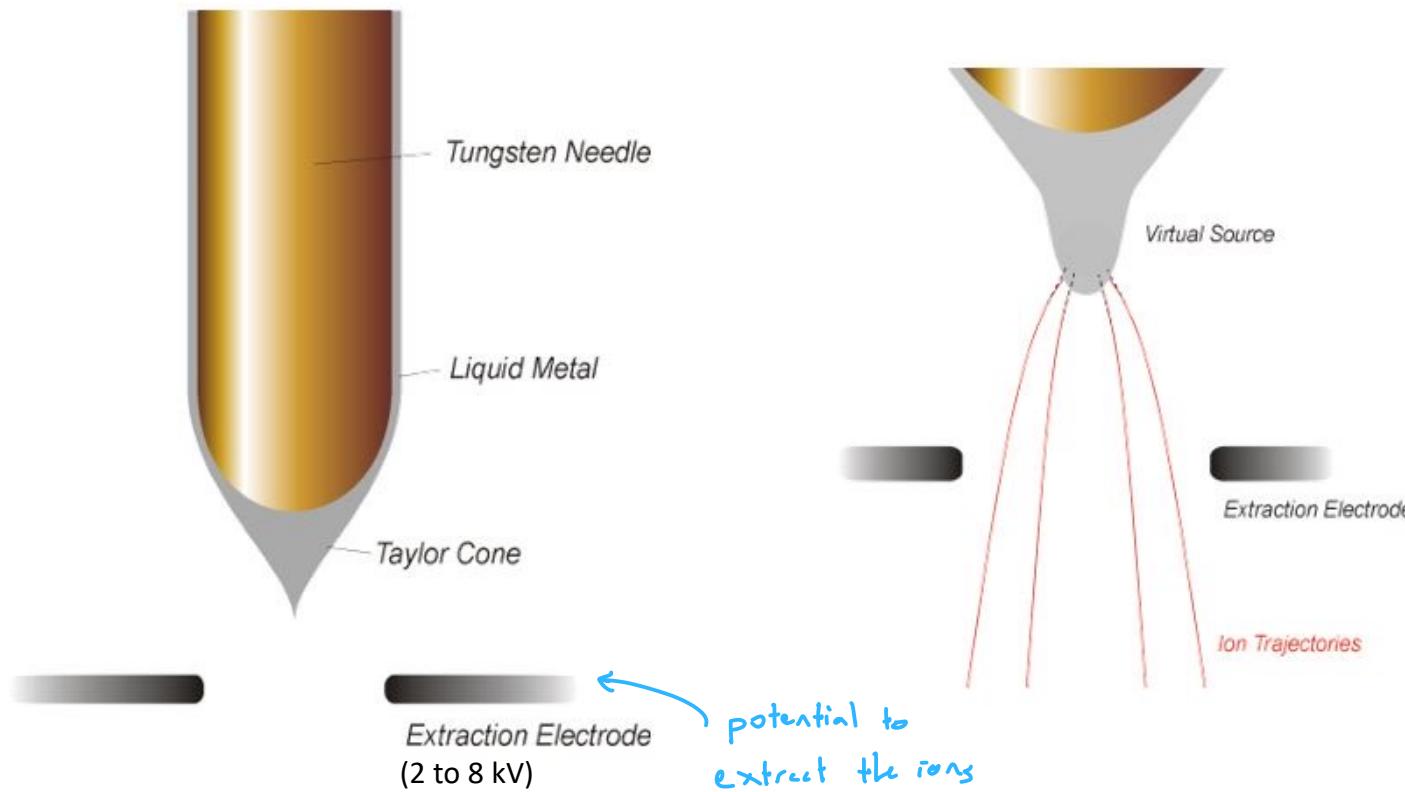
Melting point: 30 °C
Boiling point: 2,204 °C

When choosing:
• Higher mass creates more damage
• Can contaminate the sample

Ga is the most common LMIS, but other liquid metal ion sources are available (shown in red), including alloys: AuGe, AuBeSi, GeMn, PtB...

La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No

Liquid metal ion source (LMIS)



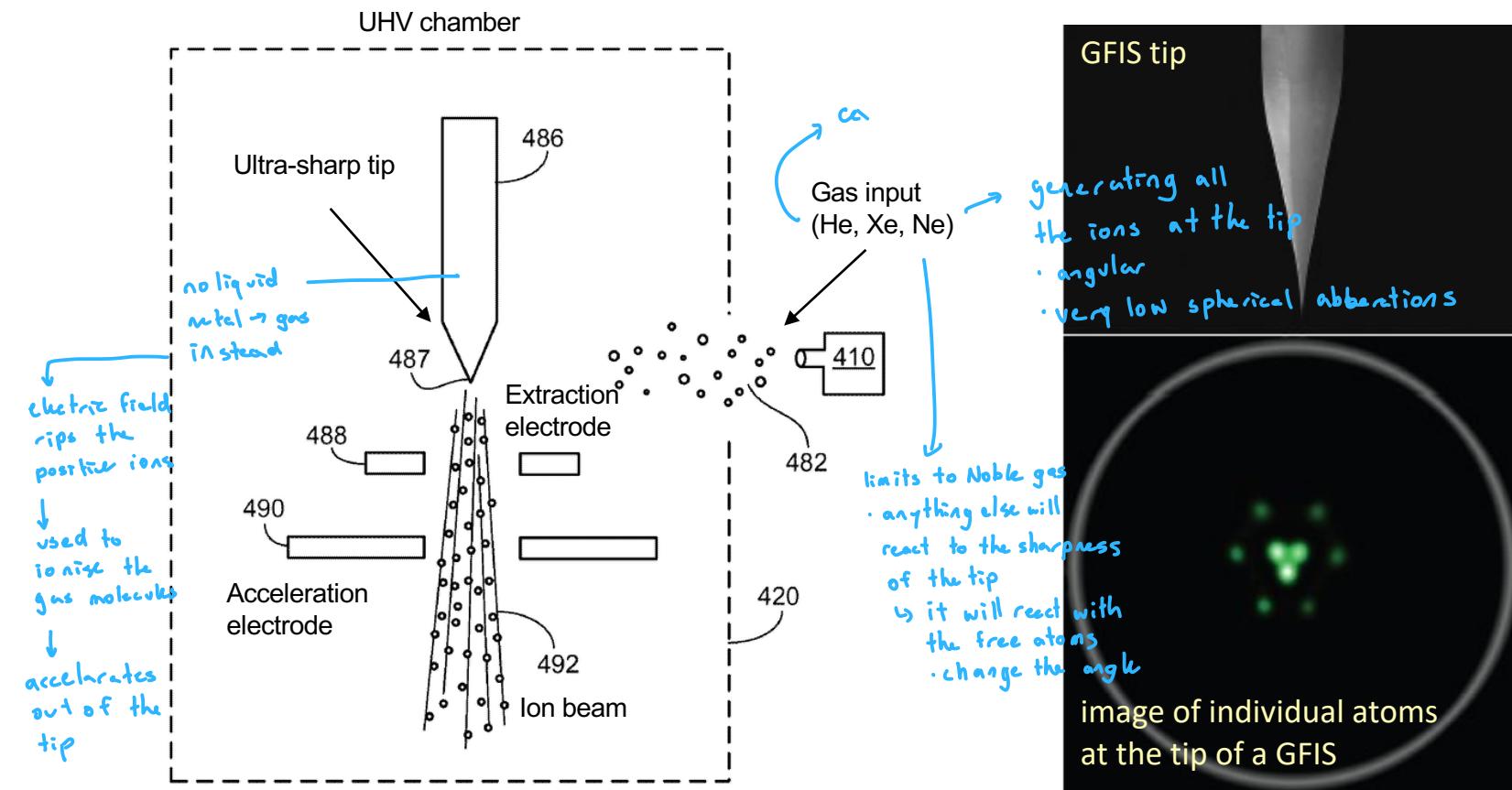
LMIS characteristics

- Angular distribution is relatively uniform (compared to electron sources) – ie: **LMIS brightness is low.**
- Ion energy spread is relatively large (~ 15 eV), so chromatic aberrations are significant.

↳ energy spread is fairly high

Ga: fairly cheap but aberrations are significant due to spread

Gas field ionization source (GFIS)

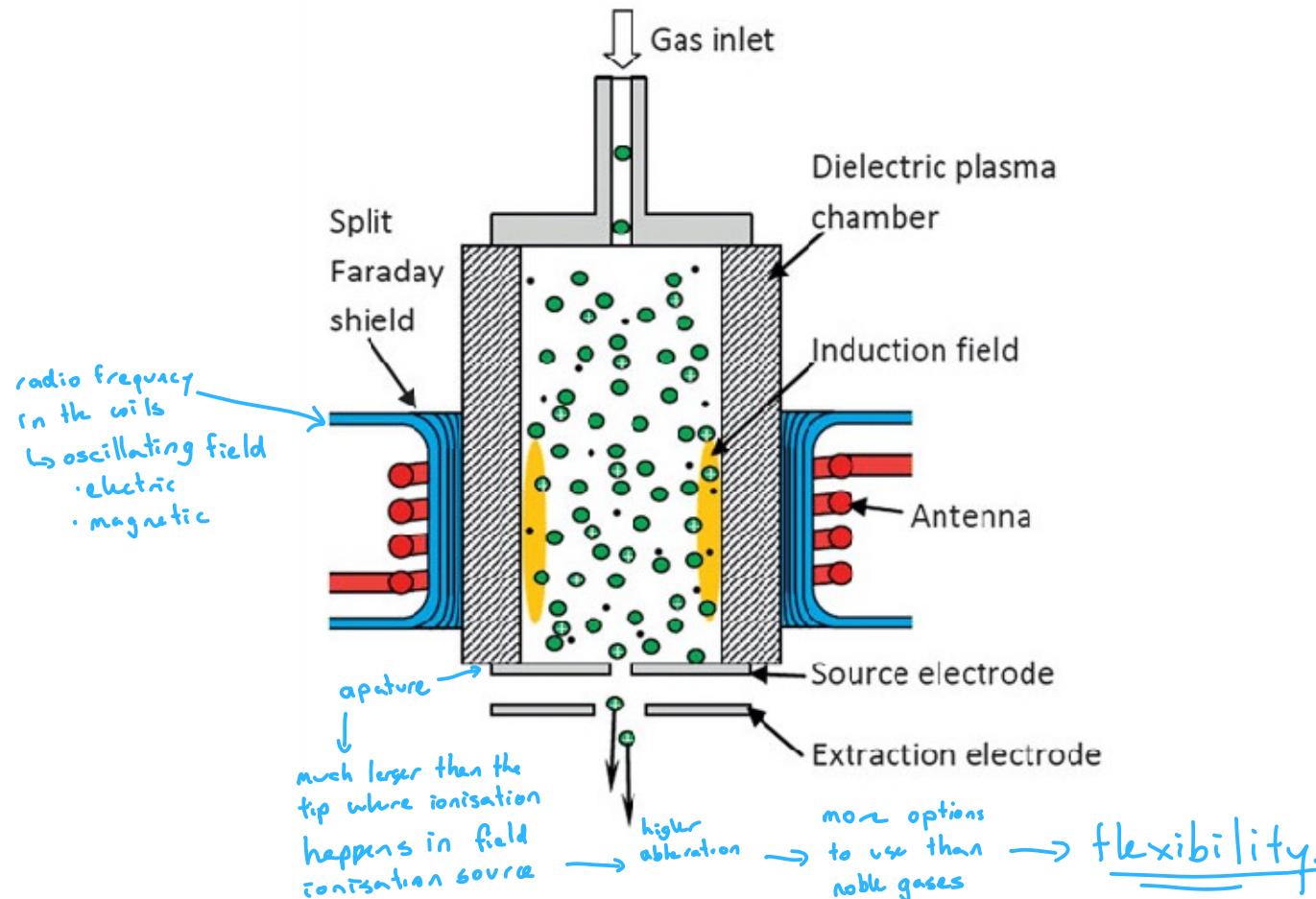


- Very intense electric field ionizes gas molecules.
- Ultra-sharp tip.
- Ion emission comes from 1 – 3 atoms at the tip only → **very small source size**.

Plasma source

↳ will be using it the next two weeks

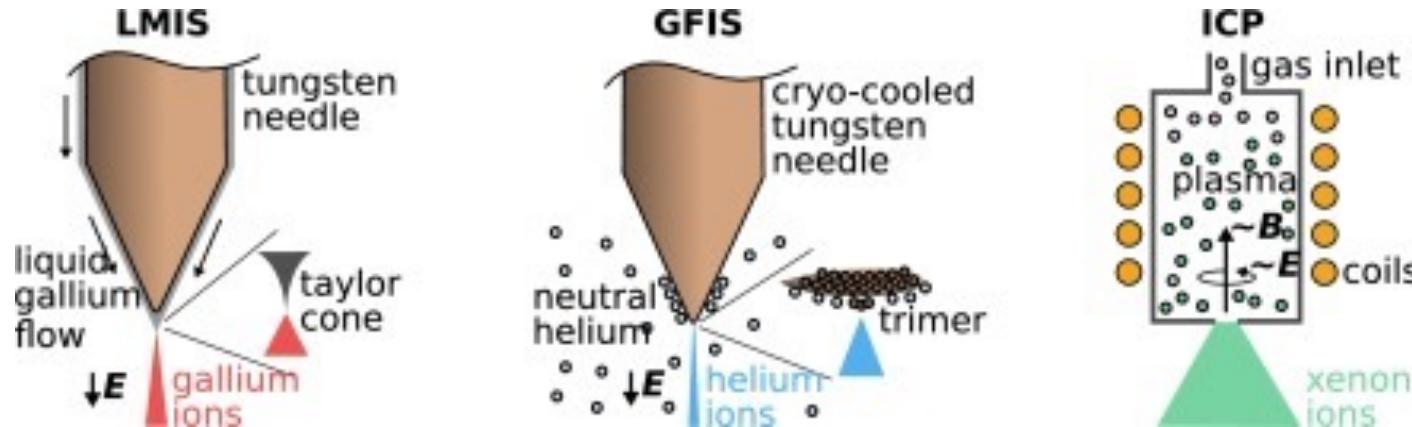
a range of gases is available: Xe, Ar, He, O₂, N₂...



ions generated in the radio frequency (RF) plasma are extracted and focused using an ion beam column

Common ion source types: Summary

Revision slide to the 3 sources!



Liquid metal ion source (LMIS)

- Most widely used.
- Cost is low & the technology is very mature.

Gas field ionization source (GFIS)

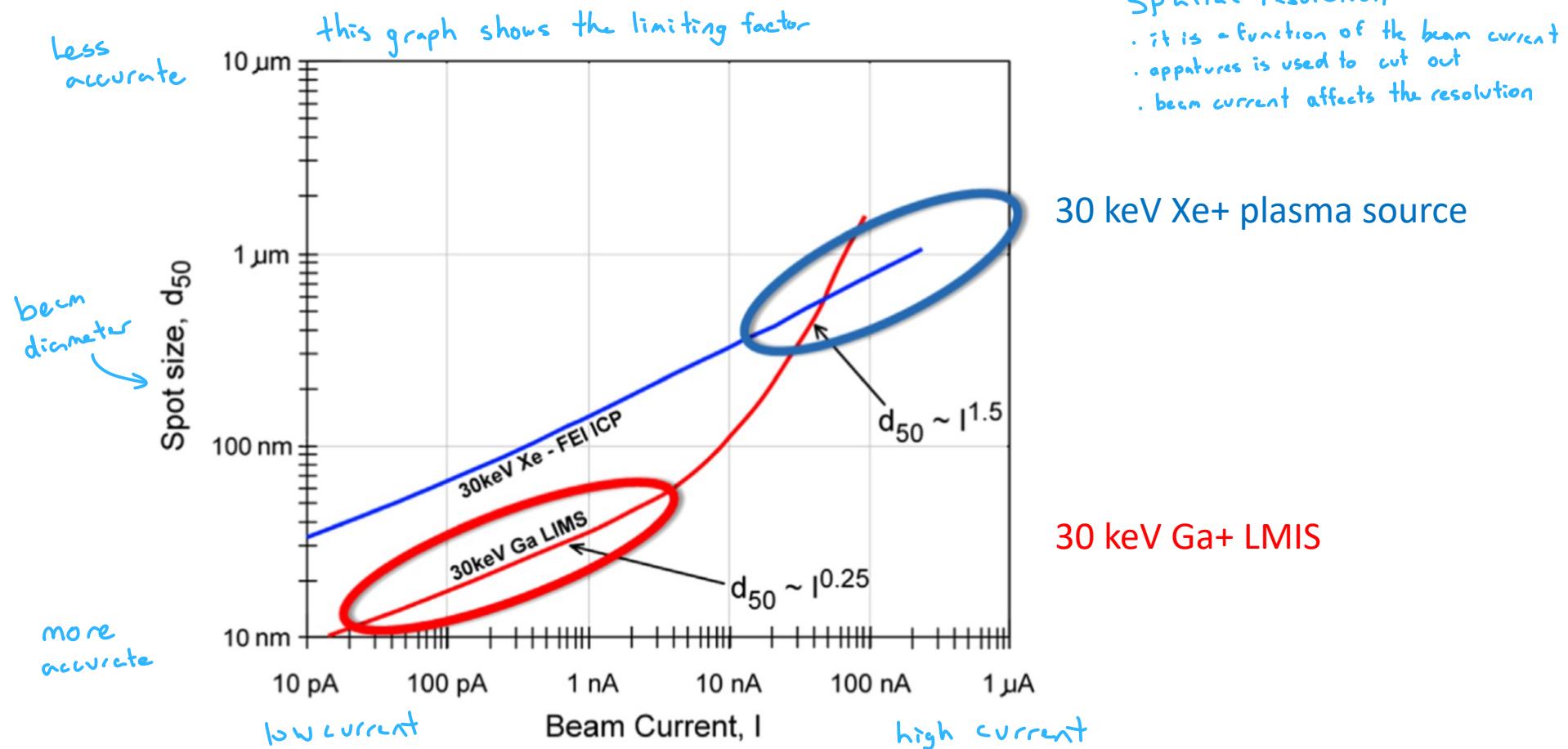
- Smallest virtual source size (3 atom wide emission).
- Best for high resolution imaging and high resolution (but very slow) milling (using He ions).
- More efficient milling possible using Xe ions.

Plasma source

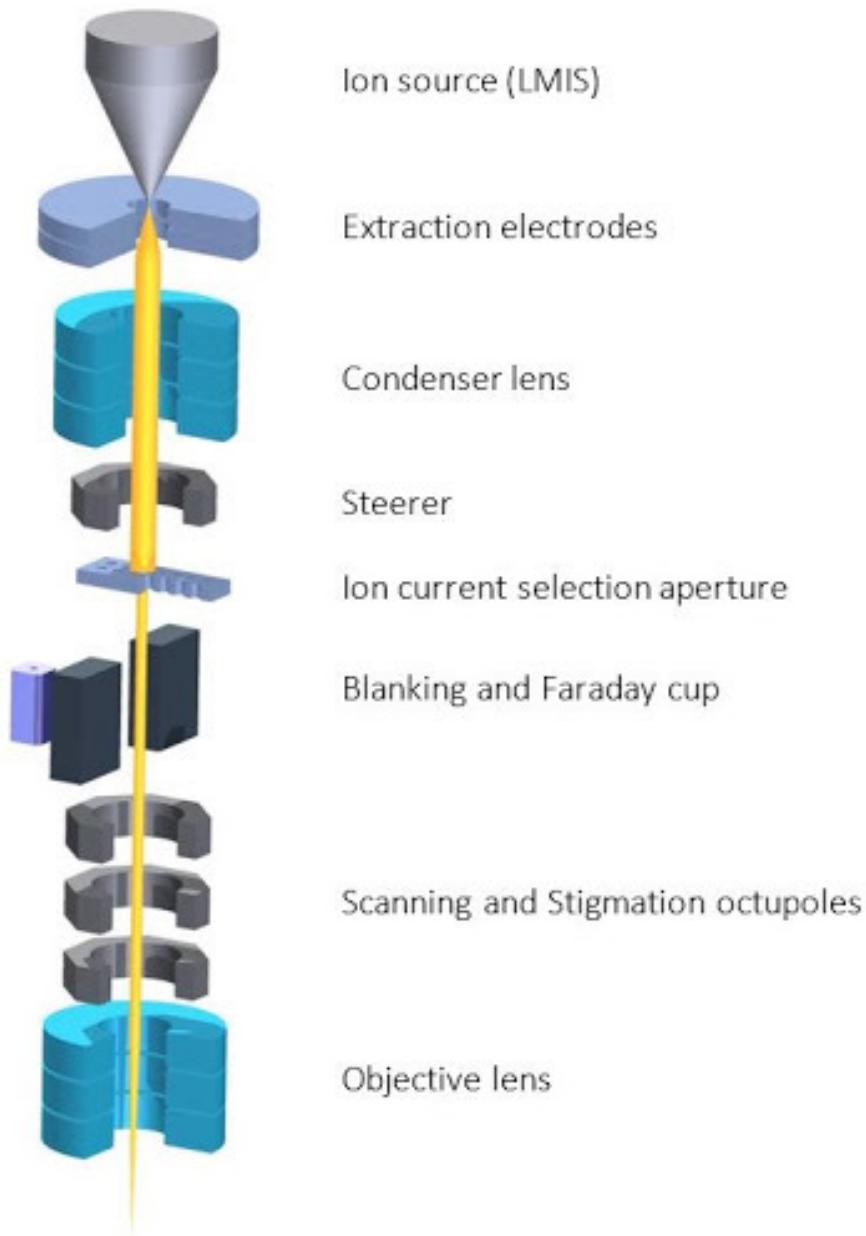
- Very versatile – easy to switch gas species and numerous gases are available.
- Ultimate resolution is inferior to LMIS & GFIS-based FIBs (virtual source size is large)
- Superior resolution at very high beam currents (ie: best for fast milling).

Beam diameter vs beam current

- Resolution decreases with increasing beam current (the same principle applies to SEM).
- Scaling depends on the FIB source – eg: Ga+ LMIS is better at low currents, but Xe+ plasma source is superior at very high beam currents.



Focused ion beam (FIB) column



Magnetic lenses are used in SEM.

Electrostatic lenses are used in FIB instruments.

... lets briefly review some basics of electromagnetism ...

Lorentz force

~Recap

- Understanding why the difference in the -ve vs. the +ve
- Can't just flip everything!

$$\begin{aligned} F &= q(E + v \times B) \\ &= F_E + F_B \end{aligned}$$

force (N) charge (C) electric field (NC^{-1}) magnetic field (T)
velocity (m s^{-1}) cross product

• mass is larger in the ions compared to electrons

↳ 'B' changes the direction only

- E = electric field [units: $\text{NC}^{-1} = \text{Vm}^{-1}$]
- B = magnetic field [$T = \text{NC}^{-1}\text{m}^{-1}\text{s}$]

↳ this affects the velocity → hence ions use 'E'

↳ electric field uses electrodes to change the direction for ions

- $qE = F_E = \text{electric force}$ [N]
- $qv \times B = F_B = \text{magnetic force}$ [N]
- $F = \text{"Lorentz force"}$ [N]

↳ Trade off: 'B' is convenient, doesn't change the speed (chromatic aberrations)
 ↳ 'E' changes the energy so it compromises the resolution

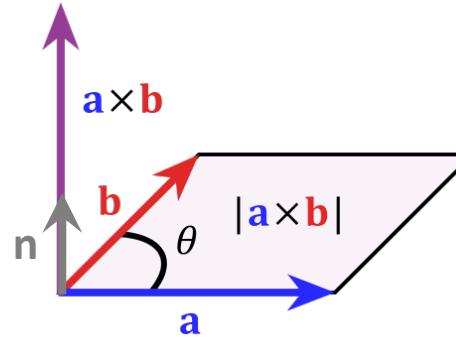
- if $q = 0$, then $F_E = F_B = 0$ (no force unless particle is charged)
- if $v = 0$, then $F_B = 0$ (no magnetic force unless particle is moving)

• CAN USE WIKI FOR FURTHER REVISION

- Electric fields are created by charged particles & exert forces on charged particles.
- Magnetic fields are created by & exert forces on moving charged particles.
- Unlike an electric field, a constant magnetic field changes the direction of motion of a charged particle, but does not change the speed of the particle.

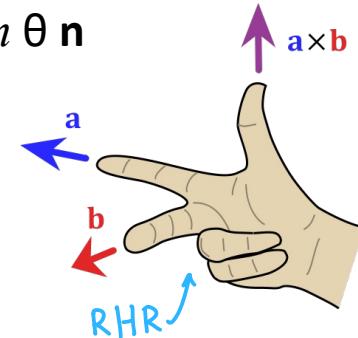
Typically, magnetic fields are used to focus electron beams and electric fields to focus ion beams

vector cross product ×



$$a \times b = |a| |b| \sin \theta n$$

the direction of the unit vector n is given by the right-hand rule



Focused ion beam (FIB) column

Main Parts of the column

Ion source: LMIS or GFIS or plasma

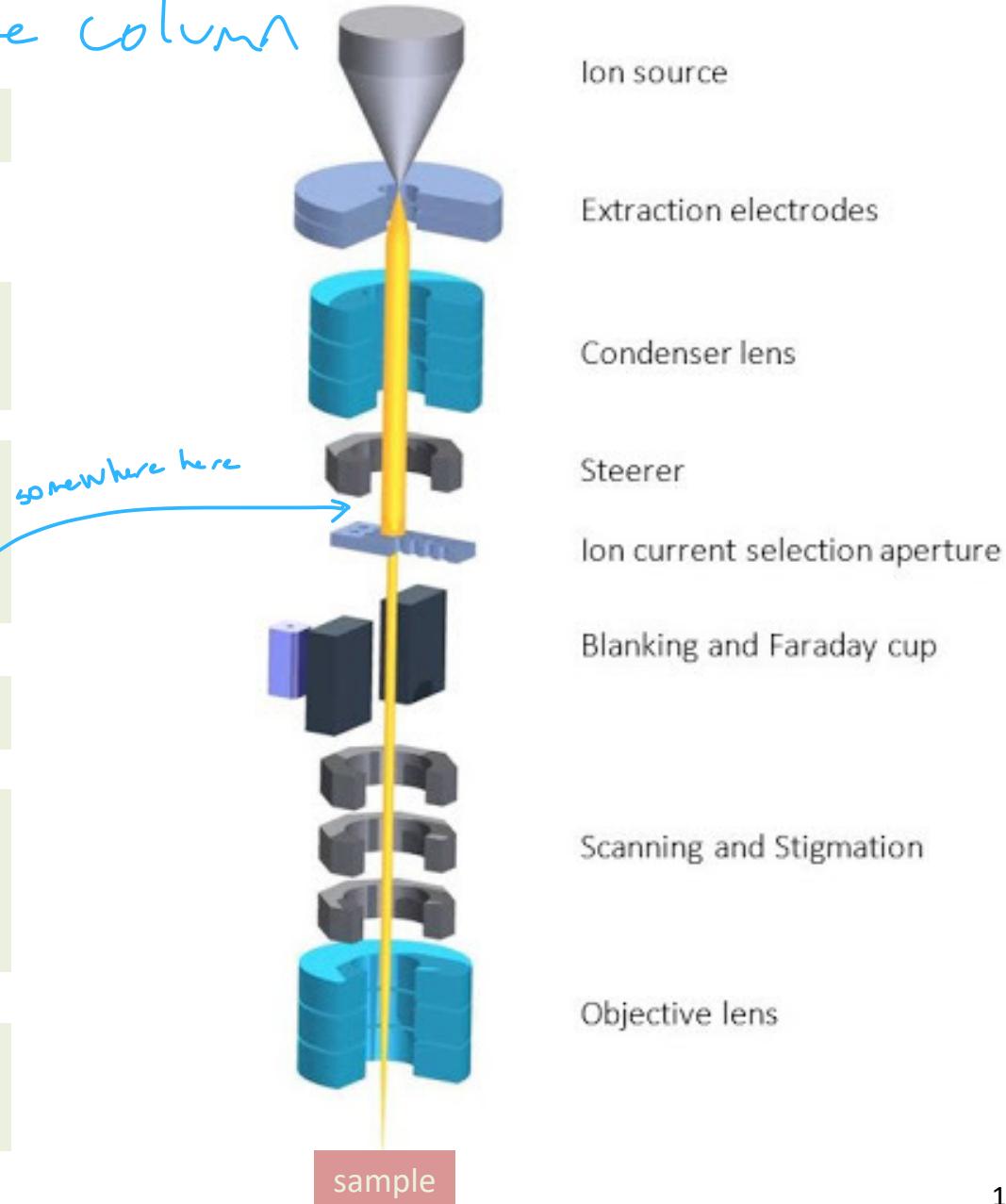
Condenser lens (and aperture) used to shape beam & reduce current

Wein filter (and slit or aperture) sometimes used to select the ion species [not shown here]

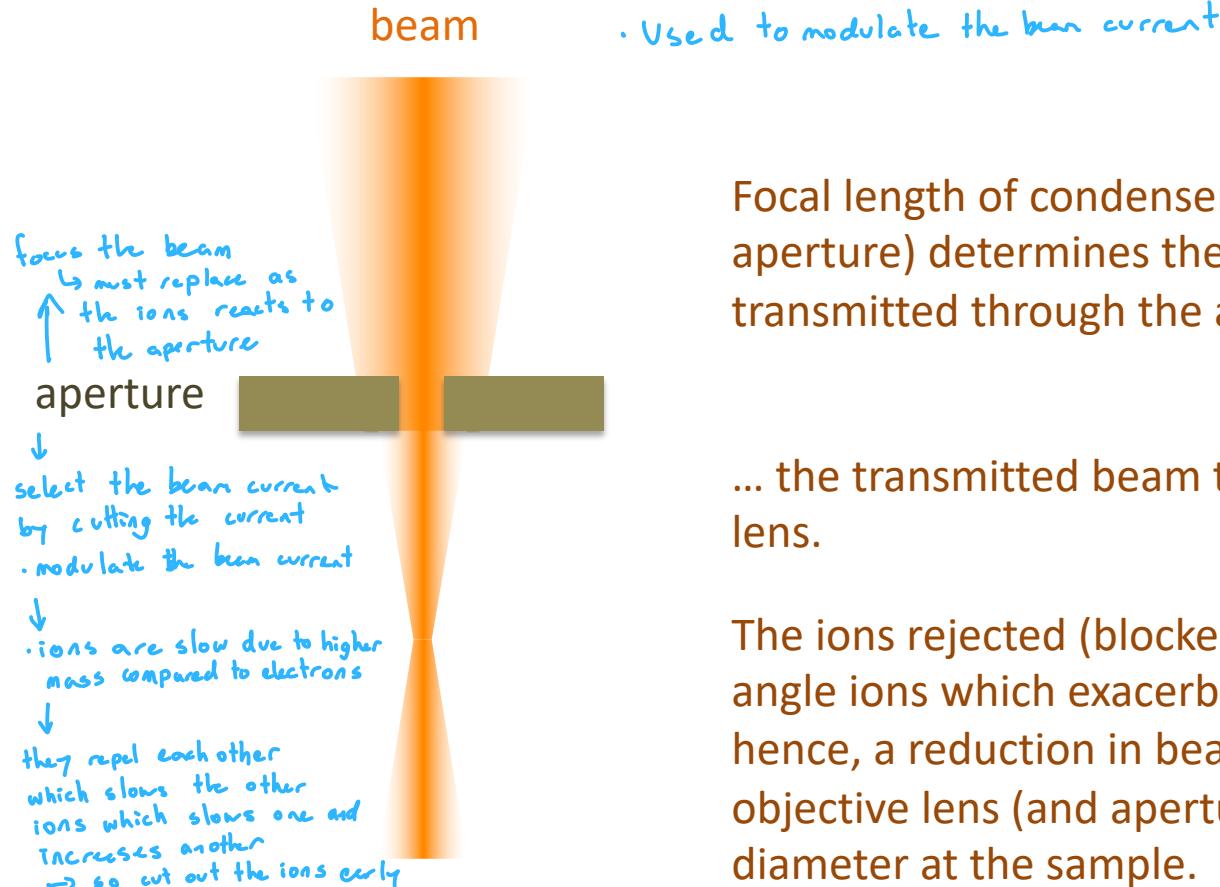
Beam blunker & Faraday cup

Electrodes used to correct astigmatism (beam shape) & to scan the ion beam

Objective lens used to focus the beam onto the sample



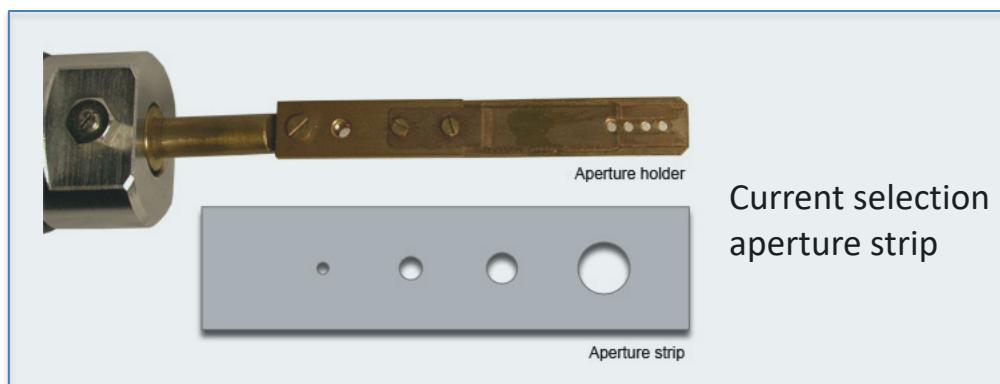
Condenser lens & aperture



Focal length of condenser lens (located above the aperture) determines the fraction of the beam that is transmitted through the aperture...

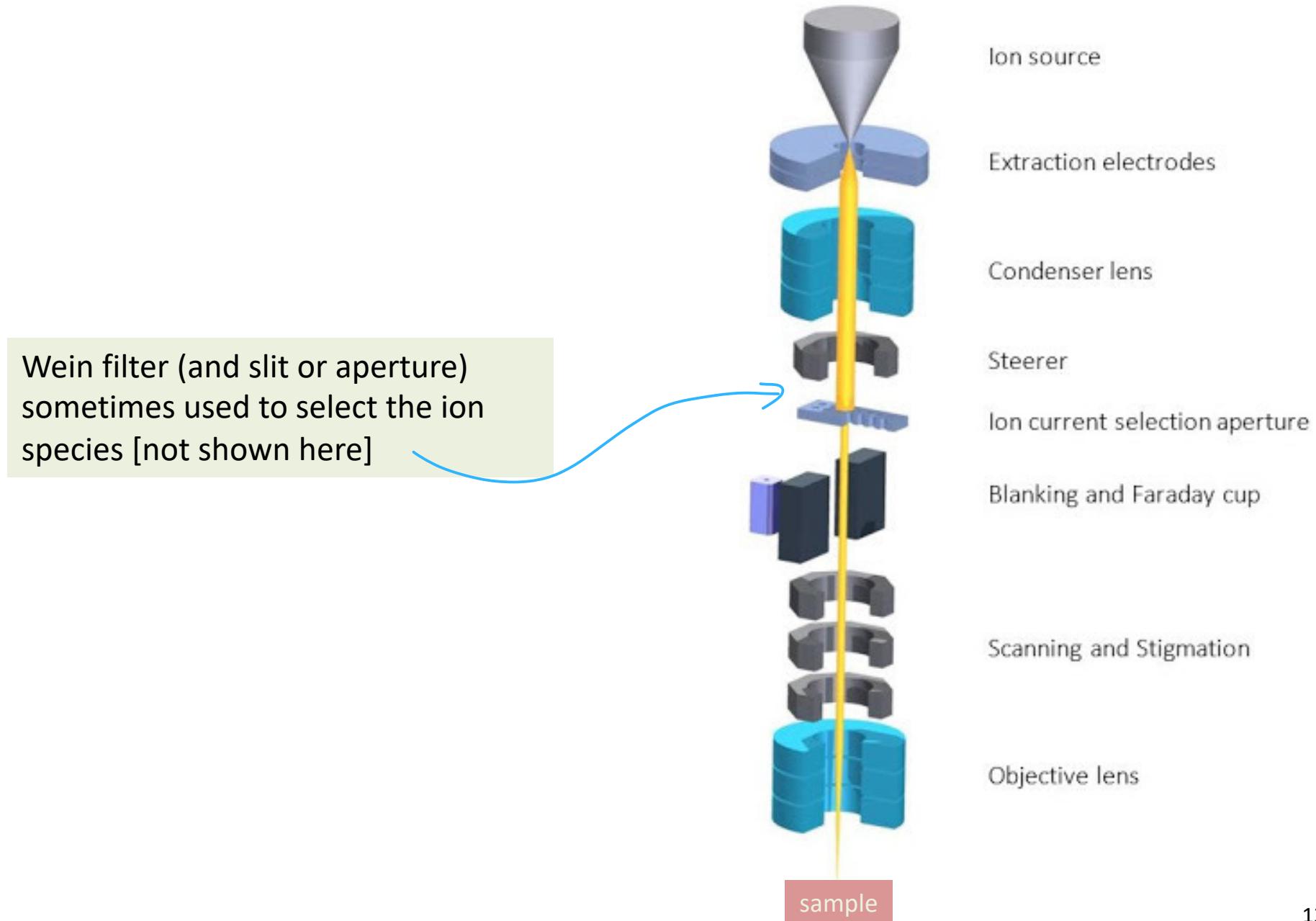
... the transmitted beam then travels to the objective lens.

The ions rejected (blocked) by the aperture are high angle ions which exacerbate spherical aberrations – hence, a reduction in beam current made by the objective lens (and aperture) reduces the beam diameter at the sample.



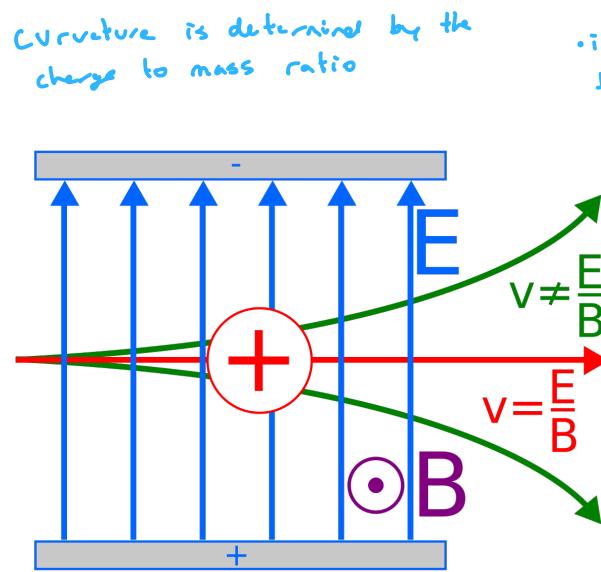
These principles also apply to SEM columns: A condenser lens and an aperture is used to control the electron beam current, which affects resolution.

Focused ion beam (FIB) column

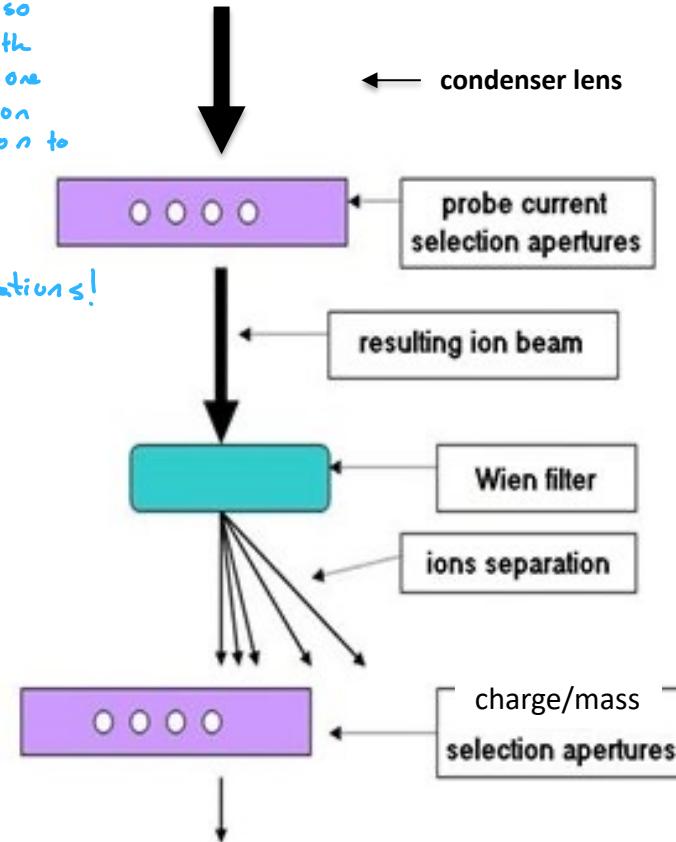


Wien filter

- Ion sources produce a number of ion species corresponding to different:
 - Charge states (eg: Ga^+ , Ga^{2+} ...)
 - Elements in an alloy LMIS (eg: AuGe, AuBeSi, GeMn, PtB)
 - Impurities (eg: residual gases in a plasma source)
 - Isotopes (eg: ^{124}Xe , ^{125}Xe ... ^{136}Xe in a plasma FIB)
- A Wien filter (and aperture/slits) is used to reject unwanted ion species from the beam.



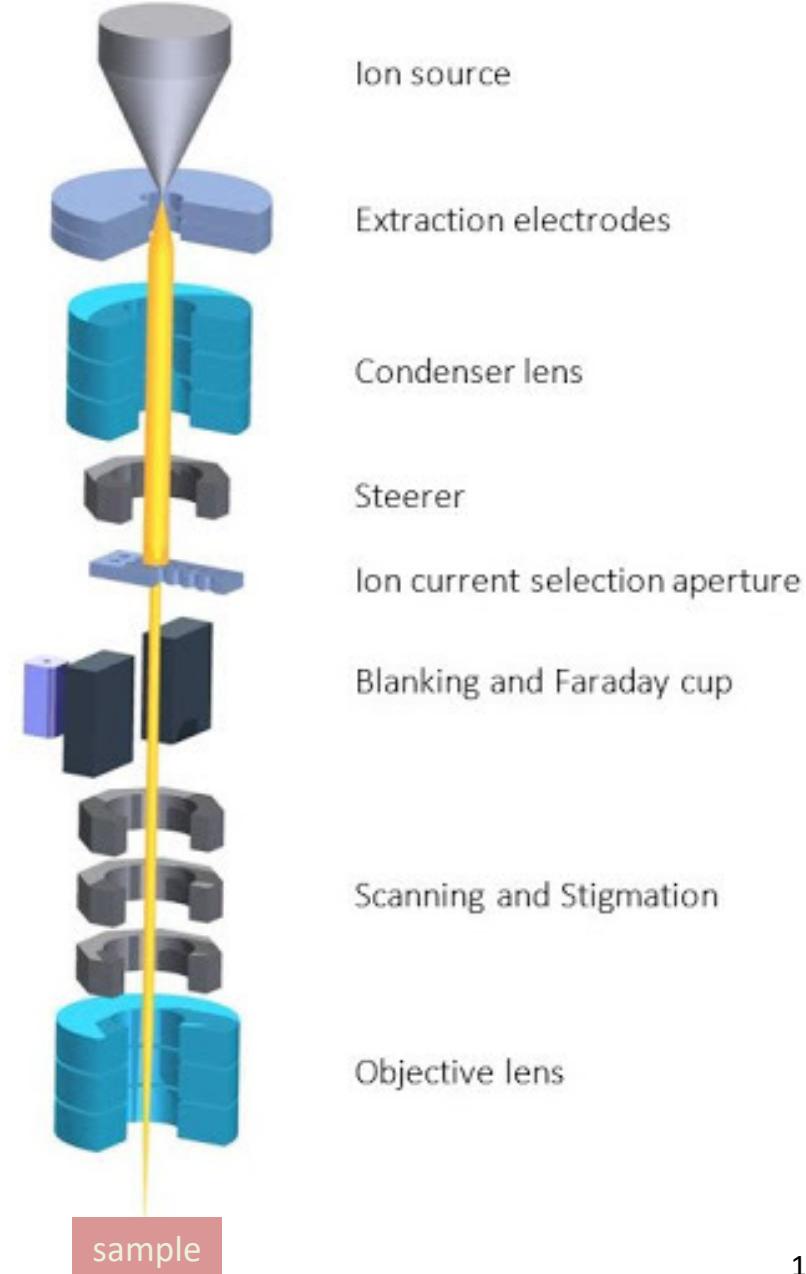
$$F = q(E + v \times B)$$



Focused ion beam (FIB) column

Angelina
Acosta

Beam blunker & Faraday cup

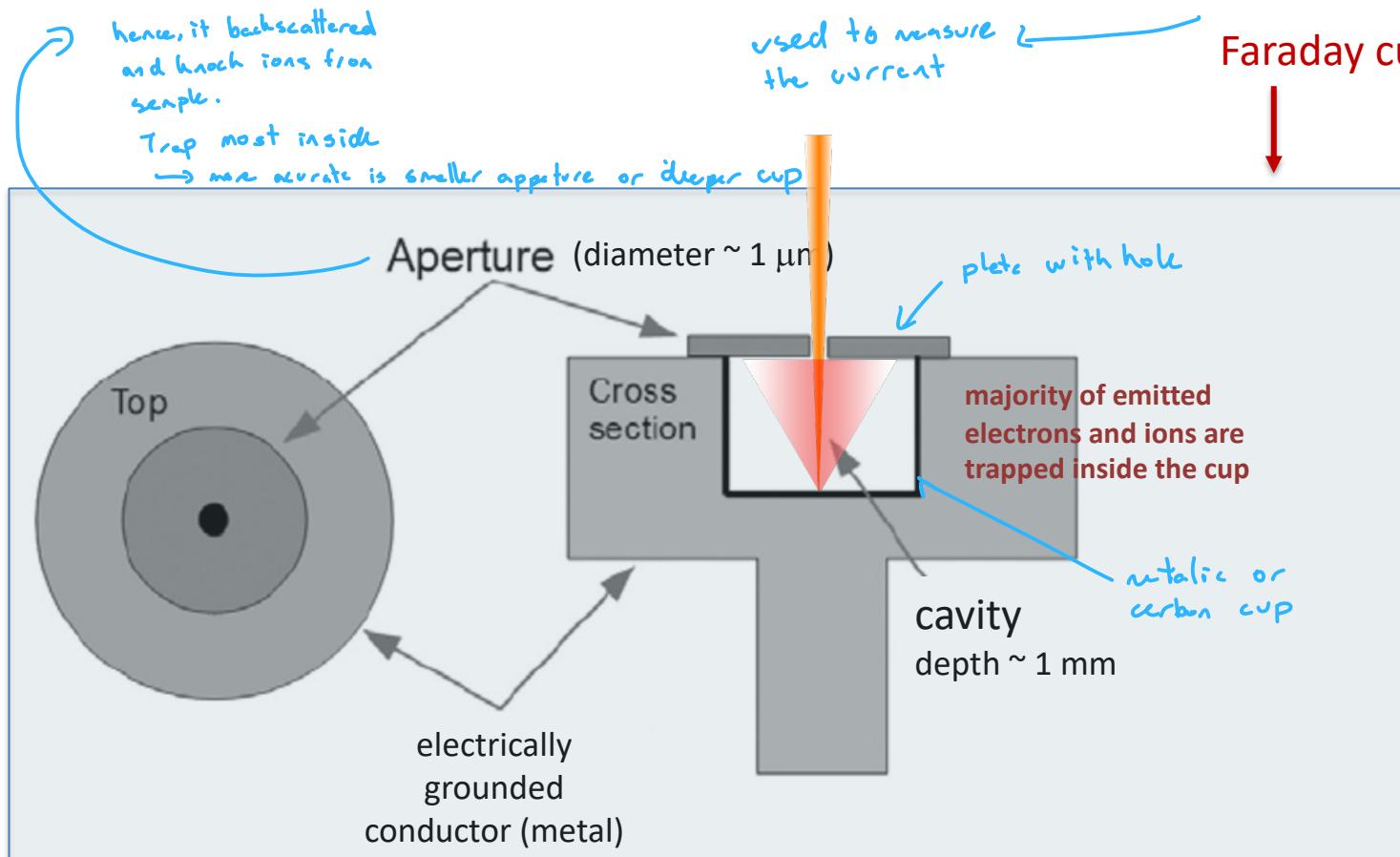


Beam blanker & Faraday cup

Faraday cup can be located:

- in the column (as shown here)
- next to the sample

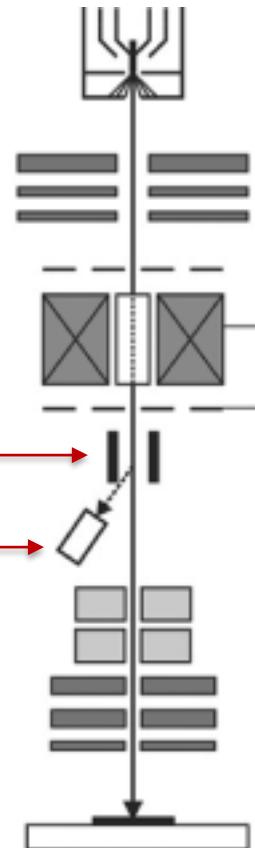
Used in both FIB & SEM instruments.



Measure the current!

↳ Important! beam deflection electrodes

Faraday cup



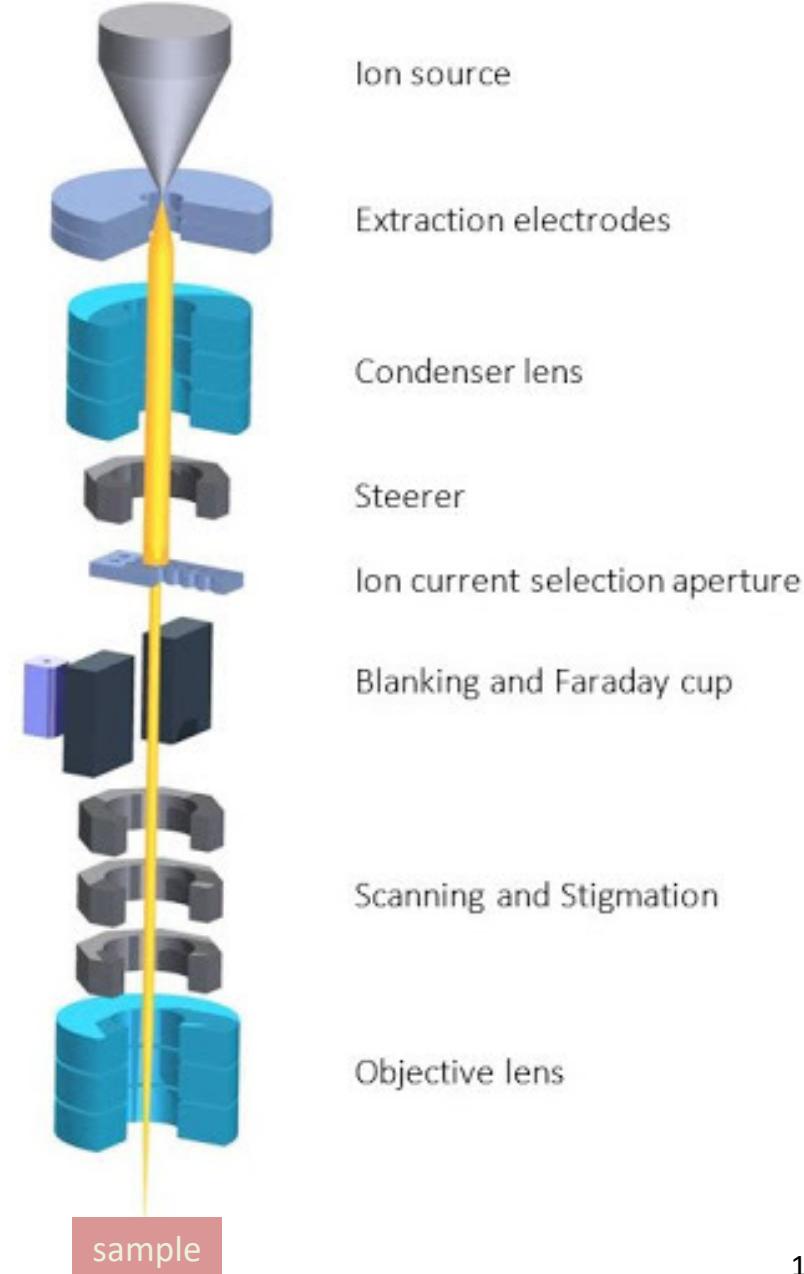
Ask if it reflects back

Electrostatic lenses for beam steering and focusing

Condenser lens (and aperture) used to shape beam & reduce current

Electrodes used to correct astigmatism (beam shape) & to scan the ion beam

Objective lens used to focus the beam onto the sample

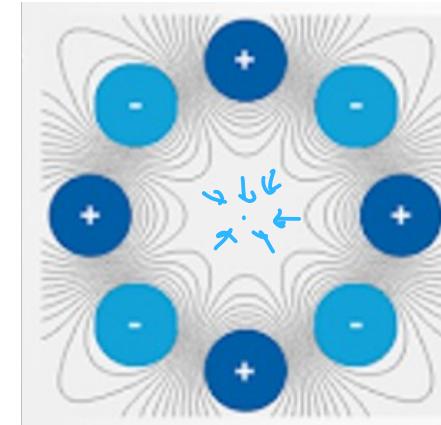


Scanning and astigmatism correction: Multipole ion lenses

quadrupoles used
to guide ions



equipotentials of an
octupole ion lens



diff voltages change the
field which can distort it
which can fix ellipsis shape to
circular

Potential field distributions for
common multipoles:

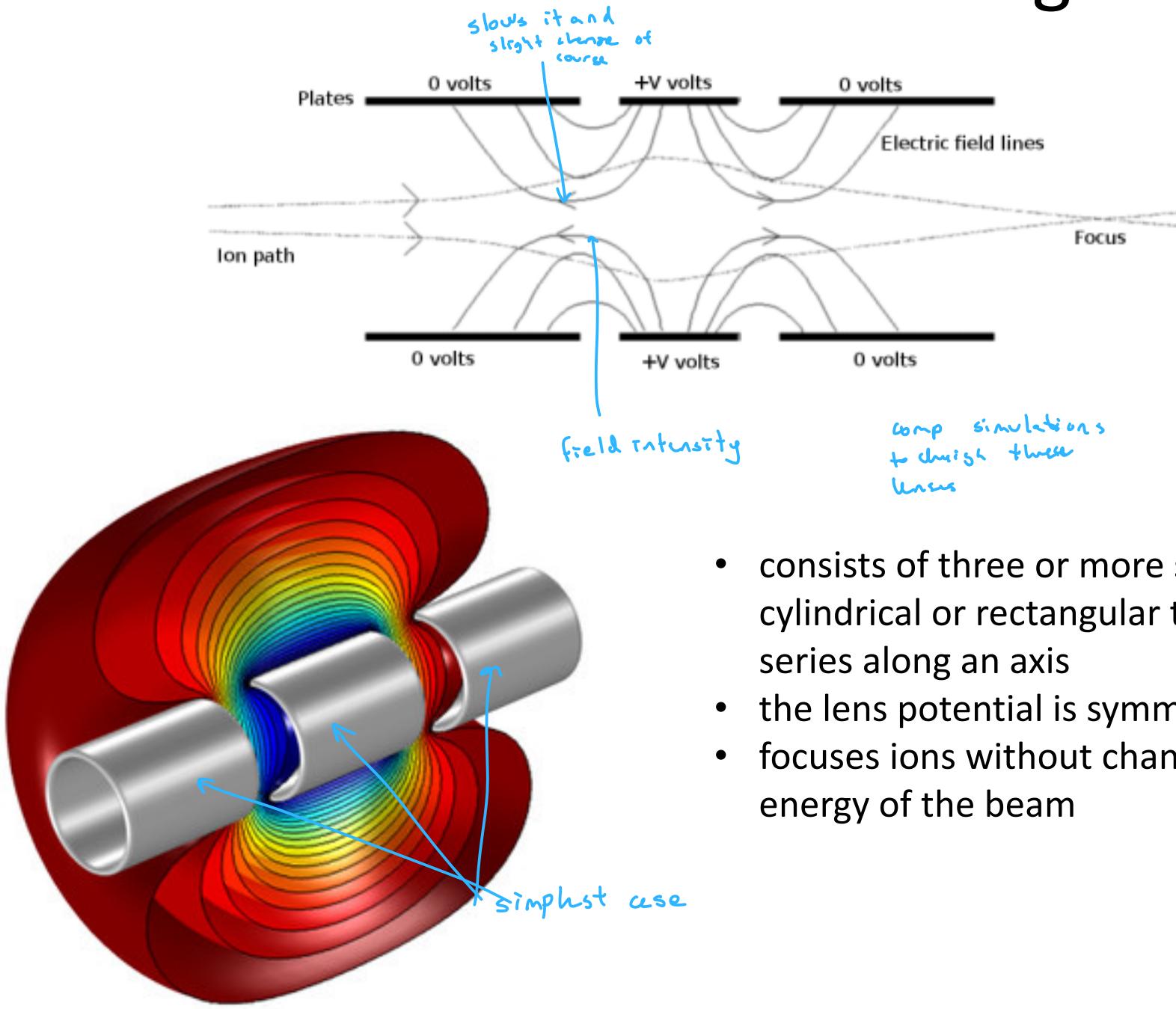
Quadrupole (N=2): $\Phi_2(x,y) = (x^2 + y^2)/(r_0^2)$

Hexapole (N=3): $\Phi_3(x,y) = (x^3 - 3xy^2)/(r_0^3)$

Octopole (N=4): $\Phi_4(x,y) = (x^4 - 6x^2y^2 + y^4)/(r_0^4)$

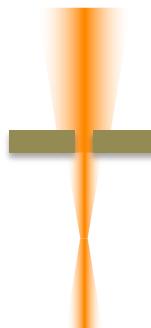
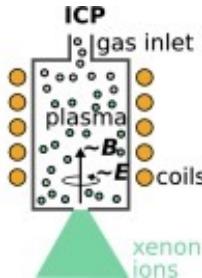
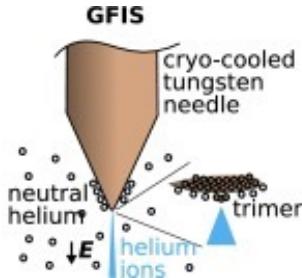
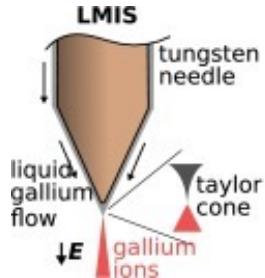
Decapole (N=5): $\Phi_5(x,y) = (x^5 - 10x^3y^2 + 5xy^4)/(r_0^5)$

Einzel electrostatic ion focusing lens



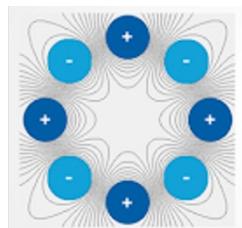
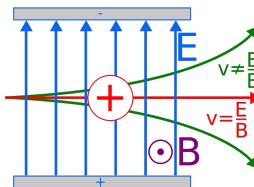
- consists of three or more sets of cylindrical or rectangular tubes in series along an axis
- the lens potential is symmetric
- focuses ions without changing the energy of the beam

FIB column: Summary

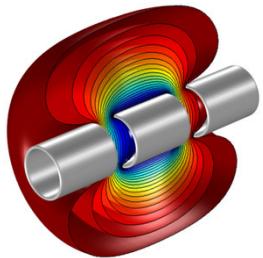


Einzel condenser lens (and an aperture) used to reduce the beam current

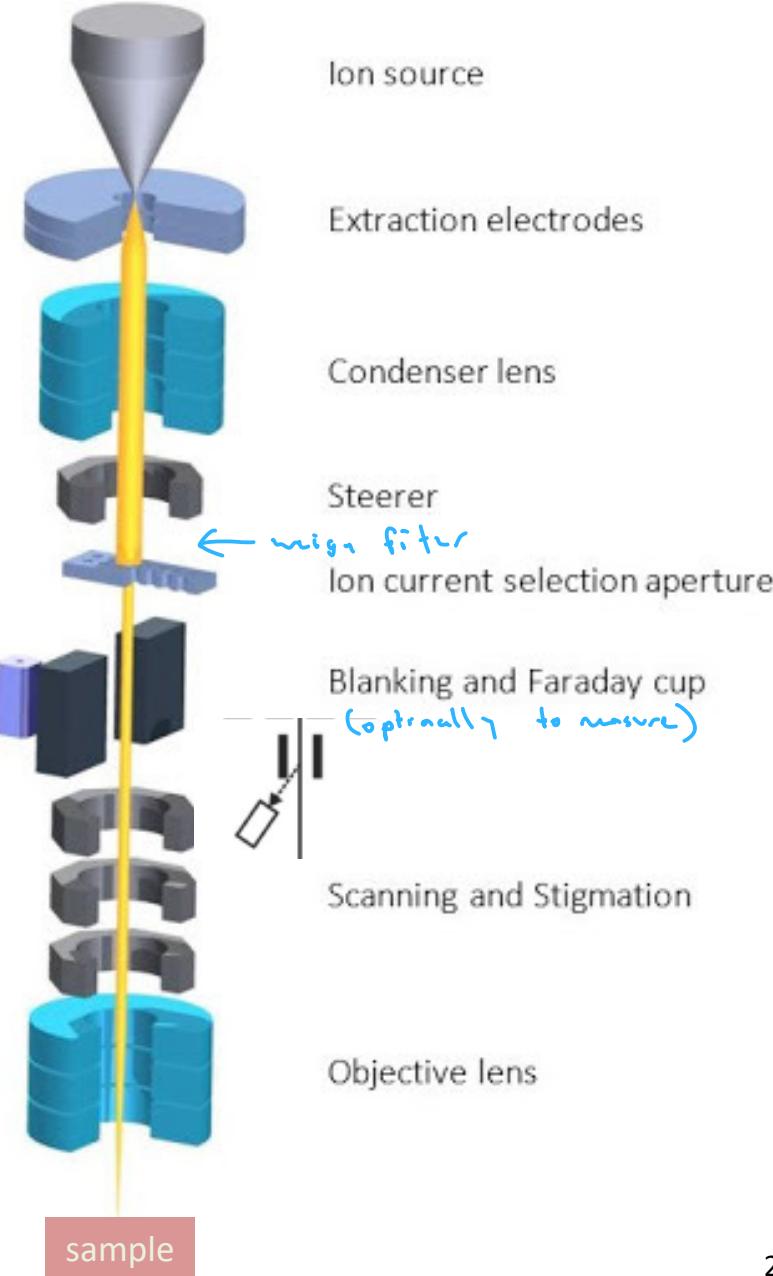
Wien filter (and a slit or aperture) is sometimes used to select the ion species [not shown in this column schematic]



Octupoles used to correct astigmatism (beam shape) & to scan the ion beam



Objective lens used to focus the beam onto the sample (there is a number of different lens types)



Ions vs electrons

electrons
↳ light

light ions

disperse energy
due to the HUGE
diff of mass
heavy ions

mass; momentum: $p = mv$ (at a given energy)

light
↳ lower momentum

$$K = \frac{1}{2} mv^2$$

heavy
higher momentum

velocity: $E = 0.5 mv^2$ (at a given energy)

faster
↳ less time in the column

ions don't penetrate
as deep as electrons
↳ electron is more likely to ionisation
· conserves both energy & momentum

there is math for this
will be explored in a
later lecture
OMG!

slower
↳ more aberrations
↳ more interaction
↳ less likely to ionise

penetration range (in a sample)

deeper
penetration

lower penetration
· more likely interact which
slows due to loss of energy

sample interactions & DAMAGE

defects caused
is less

density of
damage is
higher

nanofabrication SPEED (throughput)

· milling as fast
as possible

ions can remain in the sample – ion “implantation” / “doping”
↳ good if on purpose but can be bad bc impurities
↳ heavier is better

Papers This is on CANVAS!

Focused Ion Beam Microscopy and Micromachining

C.A. Volkert and A.M. Minor, Guest Editors

Abstract

The fairly recent availability of commercial focused ion beam (FIB) microscopes has led to rapid development of their applications for materials science. FIB instruments have both imaging and micromachining capabilities at the nanometer–micrometer scale; thus, a broad range of fundamental studies and technological applications have been enhanced or made possible with FIB technology. This introductory article covers the basic FIB instrument and the fundamentals of ion–solid interactions that lead to the many unique FIB capabilities as well as some of the unwanted artifacts associated with FIB instruments. The four topical articles following this introduction give overviews of specific applications of the FIB in materials science, focusing on its particular strengths as a tool for characterization and transmission electron microscopy sample preparation, as well as its potential for ion beam fabrication and prototyping.

Introduction

The focused ion beam (FIB) microscope has gained widespread use in fundamental materials studies and technological applications over the last several years because it offers both high-resolution imaging and flexible micromachining in a single platform.

The FIB instrument is similar to a scanning electron microscope (SEM), except that the beam that is rastered over the sample is an ion beam rather than an electron beam. Secondary electrons are generated by the interaction of the ion beam with the sample surface and can be used to obtain high-spatial-resolution images. In most commercially available systems, Ga ions are used, and their sputtering action enables precise machining of samples. In conjunction with the gas-injection capabilities on these systems, which enable ion-beam-activated deposition and enhanced etching, a range of sample fabrication schemes are possible.

During the last 25 years, FIB instrumentation has become an important technology for a wide array of materials science applications, from circuit editing and transmission electron microscopy (TEM) sample preparation to microstructural analysis and prototype nanomachining. Most modern FIB instruments supple-

ment the FIB column with an additional SEM column so that the instrument becomes a versatile “dual-beam” platform (FIB-SEM, see Figure 1) for imaging, material removal, and deposition at length scales of a few nanometers to hundreds of microns. The FIB instrument becomes a powerful tool for nanomanipulation and fabrication through the augmentation of an FIB instrument with micromanipulators and gas injection for local chemical vapor deposition (CVD).

The first FIB instruments evolved from advances in field ion microscopes¹ and through the development of high-resolution liquid metal ion sources (LMISs).^{2–4} In the 1980s, FIB instruments were embraced by the semiconductor industry as offline equipment for mask or circuit repair. It was not until the 1990s that FIB instruments began to be used in research laboratories, and today there are commercial instruments available from multiple manufacturers.⁵ With the popularity of FIB instruments for TEM sample preparation, microstructural analysis, and nanomachining, dual-beam FIB instruments are becoming a versatile and powerful tool for materials researchers.

This introductory article focuses on the FIB instrument itself and the basic

ion–solid interactions that lead to the various functionalities of FIBs. In the topical articles that follow, the major subspecialties of FIB research are discussed.

The FIB Instrument

The basic functions of the FIB, namely, imaging and sputtering with an ion beam, require a highly focused beam. A consistent tenet of any focused beam is that the smaller the effective source size, the more current that can be focused to a point. Unlike the broad ion beams generated from plasma sources, high-resolution ion beams are defined by the use of a field ionization source with a small effective source size on the order of 5 nm, therefore enabling the beam to be tightly focused.

The ion source type used in all commercial systems and in the majority of research systems designed with micromachining applications in mind is the liquid-metal ion source (LMIS).^{6,7} Of the existing ion source types, the LMIS provides the brightest and most highly focused beam (when connected to the appropriate optics). There are a number of different types of LMIS sources, the most widely used being a Ga-based blunt needle source. Ga has decided advantages over other LMIS metals such as In, Bi, Sn, and Au because of its combination of low melting temperature (30°C), low volatility, and low vapor pressure. The low melting temperature makes the source easy to design and operate, and because Ga does not react with the material defining the needle (typically W) and evaporation is negligible, Ga-based LMISs are typically more stable than other LMIS metals. During operation, Ga flows from a reservoir to the needle tip (with an end radius of about 10 µm), where it is extracted by field emission. A large negative potential between the needle and an extraction electrode generates an electric field of magnitude 10⁹ V/m at the needle tip. The balance between the electrostatic forces and the Ga surface tension wetting the tapered W needle geometry results in the formation of a single Taylor cone at the needle tip. For typical emission currents used in FIB microscopes (~2 µA), a cusp forms at the tip of the Taylor cone with a tip radius of approximately 5 nm.

The simplest and most widely used ion beam columns consist of two lenses (a condenser and objective lens) to define the beam and then focus it on the sample, beam-defining apertures to select the beam diameter and current, deflection plates to raster the beam over the sample surface, stigmation poles to ensure a spherical beam profile, and a high-speed beam blanker to quickly deflect the beam

Nanoscale

MINIREVIEW



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Recent advances in focused ion beam nanofabrication for nanostructures and devices: fundamentals and applications

Ping Li,^a Siyu Chen,^b Houfu Dai,^a Zhengmei Yang,^a Zhiqian Chen,^a Yasi Wang,^a Yiqin Chen,^a Wenqiang Peng,^{a,c} Wubin Shan^a and Huigao Duan^{a,b}

The past few decades have witnessed growing research interest in developing powerful nanofabrication technologies for three-dimensional (3D) structures and devices to achieve nano-scale and nano-precision manufacturing. Among the various fabrication techniques, focused ion beam (FIB) nanofabrication has been established as a well-suited and promising technique in nearly all fields of nanotechnology for the fabrication of 3D nanostructures and devices because of increasing demands from industry and research. In this article, a series of FIB nanofabrication factors related to the fabrication of 3D nanostructures and devices, including mechanisms, instruments, processes, and typical applications of FIB nanofabrication, are systematically summarized and analyzed in detail. Additionally, current challenges and future development trends of FIB nanofabrication in this field are also given. This work intends to provide guidance for practitioners, researchers, or engineers who wish to learn more about the FIB nanofabrication technology that is driving the revolution in 3D nanostructures and devices.

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rsc.li/nanoscale

1. Introduction

Recent years have witnessed rapidly increasing research interest in functional nanostructures and devices with configurations from 0D to 3D. Owing to their unique structural features and excellent physical properties, functional nanostructures and devices are of great significance for a broad range of applications, such as the information industry, optoelectronics, micro/nano electromechanical systems (MEMS/NEMS), biomedical, and micro-energy.^{1–4} The white paper “Nanoscience and Technology: Status and Prospects 2019” predicts that the expected annual global economic impact of nanotechnology closely related to nanostructures and devices, by 2024, will contribute more than \$125 billion US dollars.

For decades, continuous research efforts have been devoted to the development of novel nano-manufacturing technologies that enable the precise fabrication of nanostructures and devices consistent with rational design. To be precise, nanostructures and devices are dependent on nanofabrication tech-

niques to structure matter with nanoscale (refers to the range from 1 to 100 nm) features and nanoscale or even sub-nanometer accuracy. Up to now, a multitude of nano-manufacturing technologies, including optical/electron-beam lithography, nanoimprint lithography (NIL), self-assembly, atomic layer deposition (ALD), chemical mechanical polishing (CMP), laser nanopatterning, 3D printing, and nanotransfer printing (nTP), have demonstrated distinct advantages and revealed enormous potential for the flexible fabrication of nanostructures and devices.^{3,5–7} However, all these nanofabrication technologies have encountered insurmountable problems in the fabrication of arbitrary and high-resolution 3D nanostructures and devices. For instance, lithography techniques have been widely employed for fabricating 2D patterns that can accurately and reproducibly be generated in a specialized layer of material called the resist; nonetheless, those patterns then need to be transferred to another functional layer to achieve 3D fabrication, which is an arduous process.¹ For bottom-up nanofabrication, self-assembly provides a controllable avenue to construct 2D/3D nanostructures with fabrication accuracy at the atomic or molecular level, but it is only applicable to a small number of organic materials and the assembly process cannot be predesigned freely.⁶ To realize atomic or close-to-atomic scale manufacturing, ALD has been widely adopted as a kind of advanced deposition technology with controllable film thickness and excellent shape retention. However, nanostructures and devices with complex configurations cannot be

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^cCollege of Aerospace Science and Engineering, National University of Defense Technology, Changsha 410073, P. R. China

Sample test question: Ion beam lens

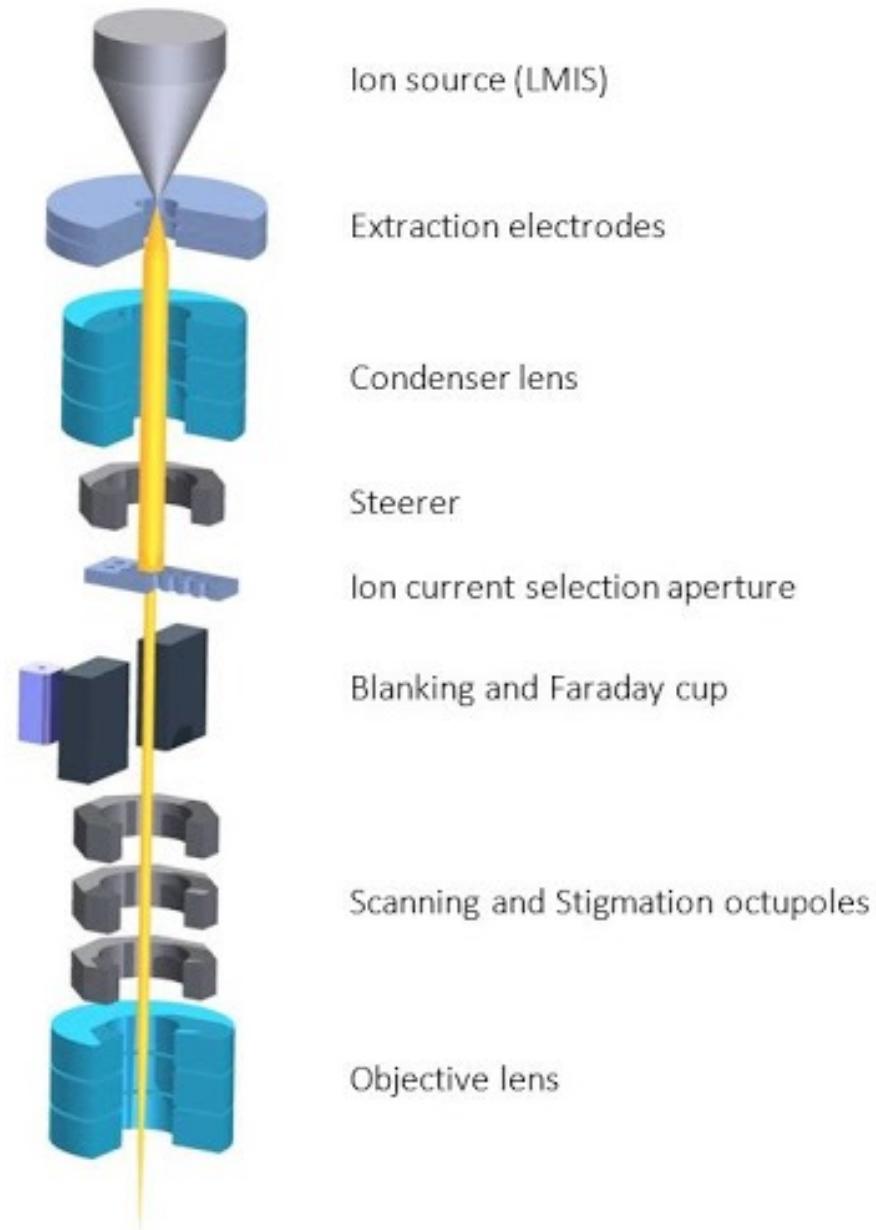
The operator of a focused ion beam (FIB) microscope increases the magnitude of the voltage applied to a 3 electrode electrostatic Einzel lens. This causes the FIB image to become blurred. Which one of these actions can be used to re-focus the image?

- a) Decrease the working distance.
- b) Increase the ion beam scan speed.
- c) Decrease the ion beam scan speed.
- d) Increase the working distance.
- e) Decrease the ion beam energy.

Sample question: Ion beam column

Consider this schematic of an ion beam column. Why does the focal length (f) of a perfect, symmetric condenser lens affect the spatial resolution of the image?

- a) Because a perfect condenser lens is designed to control the location of the focal plane below the pole piece.
- b) Because f alters the fraction of ions transmitted through the current-limiting aperture.
- c) Because a perfect condenser lens is designed to increase the energy distribution of the ions.
- d) Because a perfect condenser lens is designed to decrease the energy distribution of the ions.



Portfolio 1

- Research question
- Data is given
- Writing a report

- Must write up as if done it for real
 - schematics (cartoon)
 - photo from lab
 - ...

- Assessed on
 - how accurately
 - clearly written
 - references
 - doesn't need to be long
 - quality over quantity

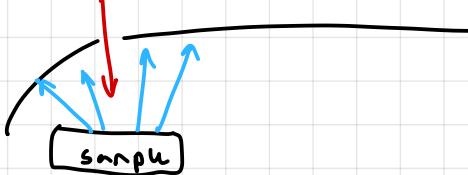
Electron-Solid Interactions

- SEM Beam
- CASINO simulated
- energy is conserved through photons
- also produce catholuminescence

from next weeks tute!
will be released soon!!!

NANOFA
&
NANOCHAR

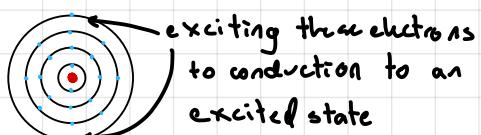
- Then detect the light
eg. mirror



- reflected and into a detector
- the shape ensures reflection with minimal loss
 - green glow is the catholuminescence

- Luminescence is generated by exciting the electron to jump bandgap
 - valence electrons (loosely bound electrons), outer shell electrons
 - ionising the outer shell electrons up into the conduction band
 - the jump is conserved by the photon

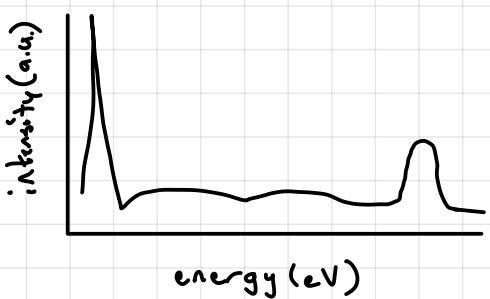
Eg WS_2 ~ hexagonal lattice 3 atoms thick; semiconductor



CL Spectra

→ used in portfolio 1

- the cloud is the conduction band
 - cannot lie between the conduction band & valence band; there isn't always a band gap
 - Spontaneously jumps the E_c
 - Metals don't have band gap

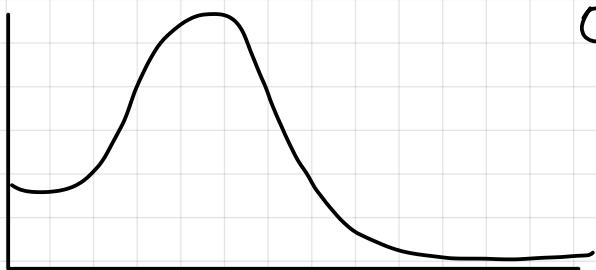


- show the concentration of defects
- impurities
- missing atoms
-

CL spatial resolution

- varying beam area \rightarrow changes the volume
- 30-ish for portfolio 1
- depth distribution
- simulations / equation needed

Normalized CL gun



okie dokie