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EEE6009: Advanced Instrumentation

- This sub-module of EEE6009 Advanced Instrumentation consists of three lectures on Electron Microscopy (EM1-3) and six demonstration classes. It is designed to provide an overview and appreciation of a range of micro and nano-scale imaging, and analysis techniques. The module will highlight the impact of **electron and focused ion beam microscopy** based research to the development and characterisation of modern engineering materials and electronic devices.

The module will provide:

- the general principles behind the operation of scanning and transmission electron microscopes and an introduction to focused ion beam microscopy,
- an appreciation of each type of instrument in terms of sample formats, specimen preparation methods, length scales and signals recorded,
- an understanding for the kind of structural and chemical information that can be obtained,
- a basis for solving basic problems involving the application of electron or focused ion beam microscopy in an informed manner.

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Lecture 1: Thursday 6th March 2014 10.00 LT 11

introduction to electron optics and resolution; construction of electron microscopes: scanning (SEM) and transmission electron microscopes (TEM); scanning transmission electron microscopy (STEM), Basic concepts of electron diffraction.

Lecture 2: Monday, 10th March 2014 10.00 LT10

Interaction of electrons with matter; introduction to analytical techniques; SEM versus TEM/STEM; diffraction techniques in the SEM, X-ray energy dispersive spectroscopy (EDS); electron energy-loss spectroscopy (EELS); energy filtered imaging in the TEM; advances in aberration corrected TEM and STEM.

Lecture 3: Thursday, 13th March 2014 10.00 LT11

Introduction to focused ion microscopy; focused ion miller (FIB); beam induced chemical vapour deposition; specimen preparation for TEM; semiconductor device fabrication and failure analysis.

Assignment: written assessment details tbc.

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Practical operation and image optimization in the SEM.

Demonstration group 6:–Monday 17th March 2014 – 10-11am

Demonstration group 5:–Thursday 20th March 2014 – 10-11am

Demonstration group 4:–Thursday 24th March 2014 – 10-11am

Demonstration group 3:–Thursday 27th March 2014 – 10-11am

Demonstration group 2 - Thursday 31st March 2014 – 10-11am

Demonstration group 1 - Monday 3rd April 2014 – 10-11am

All above demonstrations will be in Room C148/C147c of the Mappin Building (NOTE: attendance forms part of your course assessment).

1.1 Motivation for electron microscopy

Some revision:

Microscopy: *the means of viewing the detail of an object using a microscope that cannot be resolved using the unaided eye.*

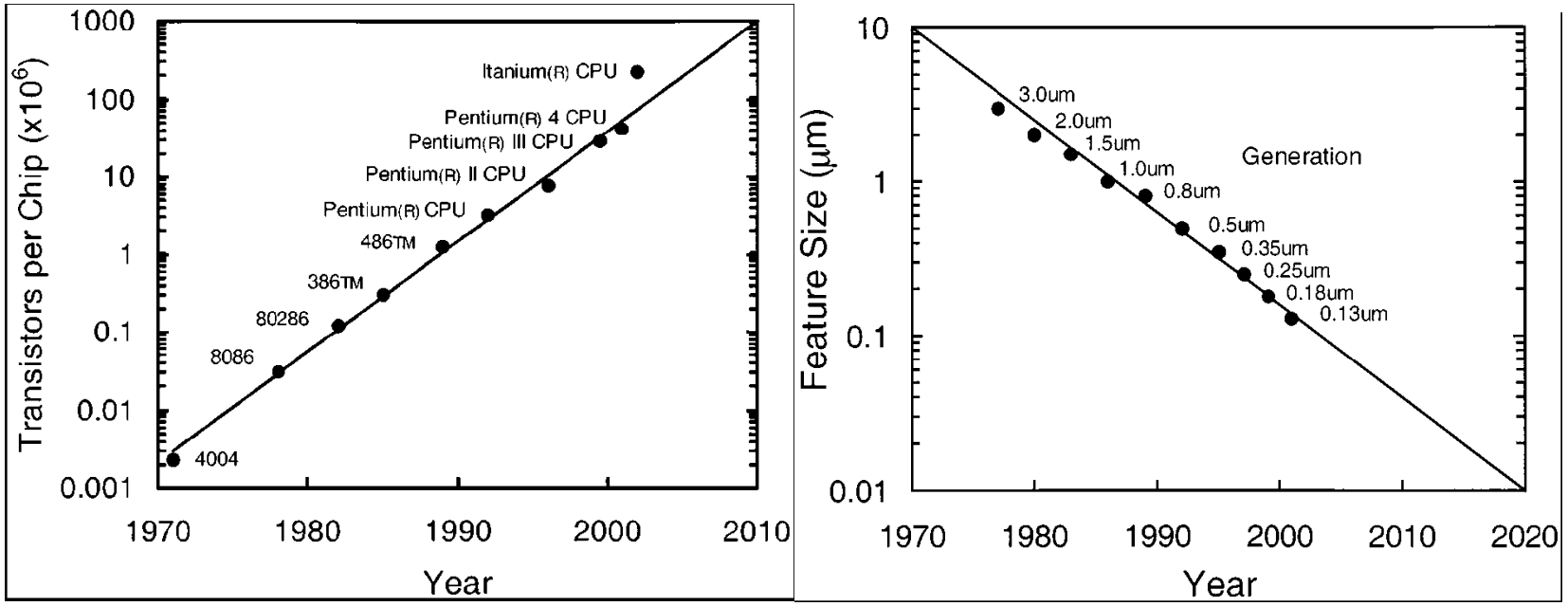
Micro-structure: *the structure of a material revealed when observed using a microscope, typically in the range ~100μm-10nm*

Nano-structure: *typically features of the order 100nm to <1nm – (high resolution microscopy)*

Atomic-structure: *<1nm, typical inter-atomic spacing in many technologically important materials lie between 0.5 and 0.05nm.*

1.1 Motivation for electron microscopy

- steady decrease in the size of many semiconductor devices, with a corresponding improvement in device performance and reduction in production costs. *Moore's Law* from 1965 stated that the number of transistor on a computer chip would double every 18-24 months.



MT Bohr, IEEE Trans. Nanotechnol. 1 (2002) 56-62

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1.1 Motivation for electron microscopy

- Most physical properties of a material such as strength, hardness, toughness, thermal stability, electrical conductivity and so on are normally measured by methods that provide useful information on the bulk properties. Many of these properties are however governed by the materials micro and nano structure.

- Understanding the micro and nano-structure allows us to explain many of these properties and provides a means to predict and modify the materials properties through careful control of the micro/nano-structure during processing and manufacturing.

- Electron microscope provides the most readily available method to study sub-micron and sub nanometer (atomic scale) structures.

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1.1 Motivation for electron microscopy

- continuing miniaturization only possible through detailed correlation of device properties with microstructure and chemistry on the nano-scale
- **electron microscopy** provides important research tools in the development and characterisation of new semiconductor device structures
- compliments bulk analysis techniques such as X-ray diffraction or secondary ion mass spectroscopy and surface analytical techniques such as atomic force microscopy or scanning tunnelling microscopy.
- **transmission electron microscopy** and its associated analytical techniques provide the only real direct method of examining the internal structure of many of these devices at an **atomistic level**.

1.1 Motivation for electron microscopy

Electron microscopy has applications in:

- Research
- Process development
- Manufacturing
- Quality control

of a huge range of electronic and engineering components and processes in addition to:

- Biological and Medical
- Forensics
- Failure analysis

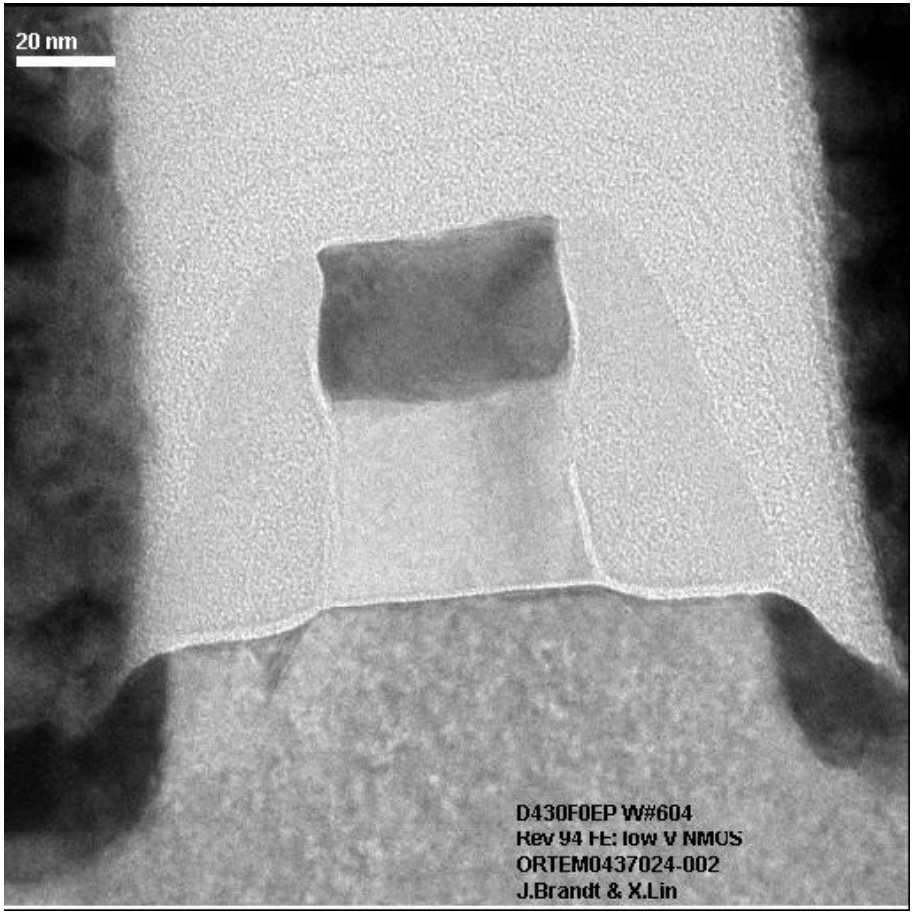


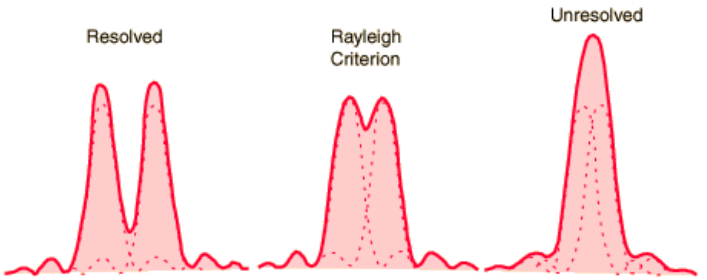
Image of currently smallest CMOS transistor with 35nm gate length

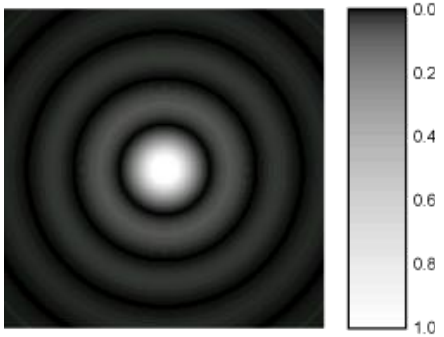
P. Bai et al., Proc. 50th IEEE Intern. Electron Devices Meeting, San Francisco (2004) 657-660

1.1 Motivation for electron microscopy

resolution improvement compared to light microscopy

- resolution – defined as the smallest spacing of two points that can be observed as two distinct entities
- For a perfect lens system, resolution is limited by **diffraction**. The Rayleigh criterion states that the smallest spacing *r* between two resolved points is given by the distance at which the intensity maximum at the central Airy disc falls into 1st minimum of the other [J. W. Strutt, (III. Lord Rayleigh), Philos. Mag. VIII (1879) 261, 403, 477]:





$$r = 0.61\lambda / (n \sin\alpha)$$

where λ = wavelength of illumination
n = refractive index
α = half angle subtended by an aperture

violet light λ =400nm, angle a=65°, oil with n=1.56: r=194nm≈ **0.2μm (200nm)**
on-line simulation: (http://www.olympusfluoview.com/java/resolution3d/index.html)

1.1 Motivation for electron microscopy

wavelength of fast electrons is much shorter than that of light

- electrons have wavelengths (that are useful for microscopy) of 1-10 **millionth** of that of visible light
- wavelength of electrons is related to their energy, i.e. accelerating voltage:

  allowing for relativistic effects

$$\lambda^2 = h^2 / (2m_0eV + e^2V^2/c^2)$$

where:	$c = 2.998 \times 10^9 ms^{-1}$	velocity of light
	$e = 1.602 \times 10^{-19} C$	charge on electron
	$h = 6.62 \times 10^{-34} Js$	Planks Constant
	$m_0 = 9.109 \times 10\text{-}31 kg$	rest mass of electron

This can be rearranged to give:

$$\lambda = [1.5/(V + 10^{-6}V^2]^{1/2} \text{ nm}$$

for example: at 20kV: λ=0.0086nm, 200kV: λ=0.0025nm, 1000kV: λ=0.0009nm

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1.1 Motivation for electron microscopy

diffraction limit in electron microscopy

- for electrons in vacuum (n=1) and for small angles $\sin\alpha \approx \alpha$:

$$r = 0.61\lambda/\alpha$$

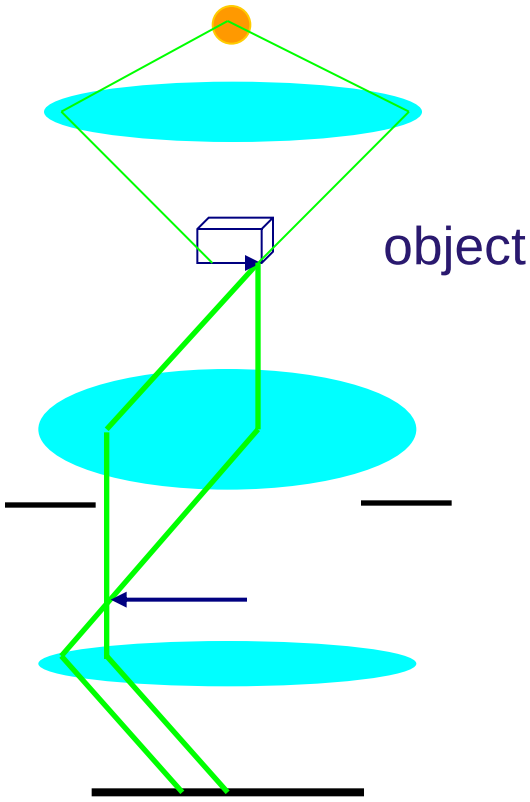
  For a 200kV electron microscope where λ=0.00251nm with α typically 0.001-0.02 radians the diffraction limited resolution becomes 0.015-0.08nm for large and small apertures, respectively. This allows **atomic resolution at 200kV in a transmission electron microscope (TEM)**!

- actual resolution of a TEM instrument is not limited by wavelength but lens aberrations, which restrict the angular range that can be used (cf. Lecture 2).

1.2 Basics of emitters and electron optics

general optical elements necessary for a microscope

element	light
illumination source	light bulb
condenser	glass lens
objective	strong glass lens
aperture	pin-hole
projector	glass eye-piece
detector	eye, film, CCD



1.2 Basics of emitters and electron optics

general optical elements necessary for a microscope

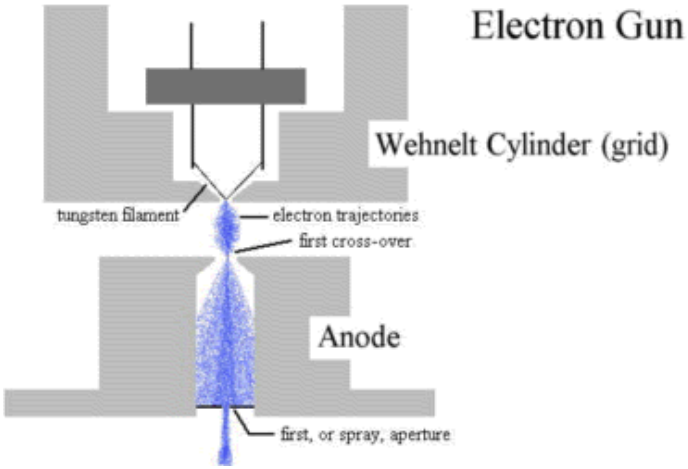
element	light	electrons
illumination source	light bulb	cathode
condenser	glass lens	electromagnetic lens
objective	strong glass lens	electromagnetic lens
aperture	pin-hole	fine aperture
projector	glass eye-piece	electromagnetic lens
detector	eye, film, CCD	phosphor screen, film

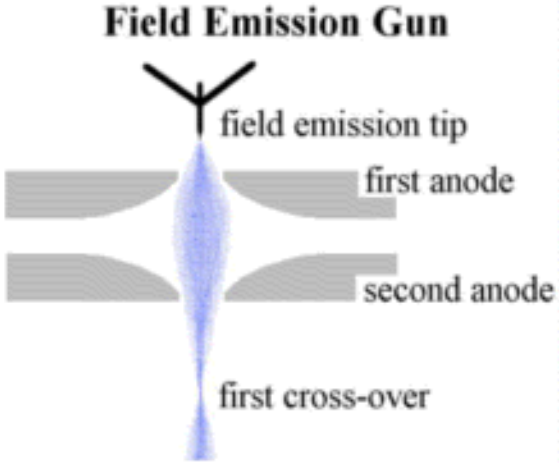
1.2 Basics of emitters and electron optics (How do we generate a beam of electrons in the electron microscope?)

1.2.1 cathodes

two principles:

heating to electron emission (thermal) or field-emission by strong electric field





cathodes	W filament	LaB_6	Schottky FEG	cold FEG
principle	thermal	thermal	thermal + field	field-emission
virtual source size $2r$	~0.1mm	~μm	few nm	few Å
brightness $\beta=I(\pi r\alpha)^{-2}$ [10^6A/cm^{2}sr]	~1	10-50	100-500	1000
vacuum [mbar]	10^{-5}	10^{-6}	10^{-9}	10^{-10}

1.2 Basics of emitters and electron optics

1.2.2 electromagnetic lenses

principle: use of Lorentz Force*

$\boldsymbol{F} = -e\,(\boldsymbol{E} + \boldsymbol{v} \times \boldsymbol{B})$ to deflect electrons in an inhomogeneous field; result: no velocity change but **rotation and focusing** towards optical axis like a convex lens; problem: affects off-axis electrons stronger, hence **spherical aberration**

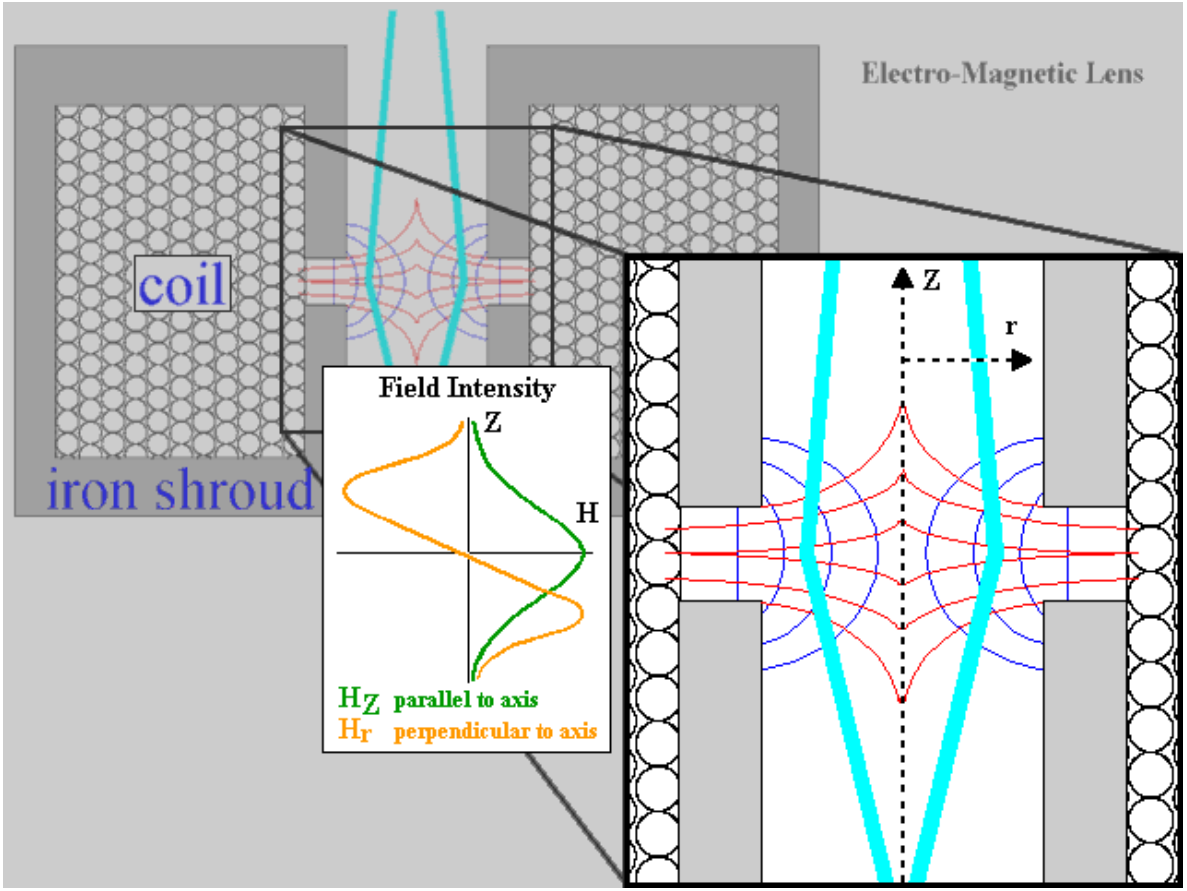


image courtesy by AR Sampson, Advanced Research Systems

*http://en.wikipedia.org/wiki/Lorentz_force

1.3 Scanning electron microscopy

Scanning Electron Microscope (SEM)

- **used to study the structure, composition and electronic properties of surface/near surface regions of bulk materials**
- **can examine relatively large samples**:
  - limitation is size of specimen chamber & sample stage, typically a few cm
- **specimens usually require little special preparation**
  - non-conducting samples may require coating with carbon or gold to reduce charging under electron beam
  - specimens must be vacuum compatible generally (although low pressure environmental instruments have recently been developed)

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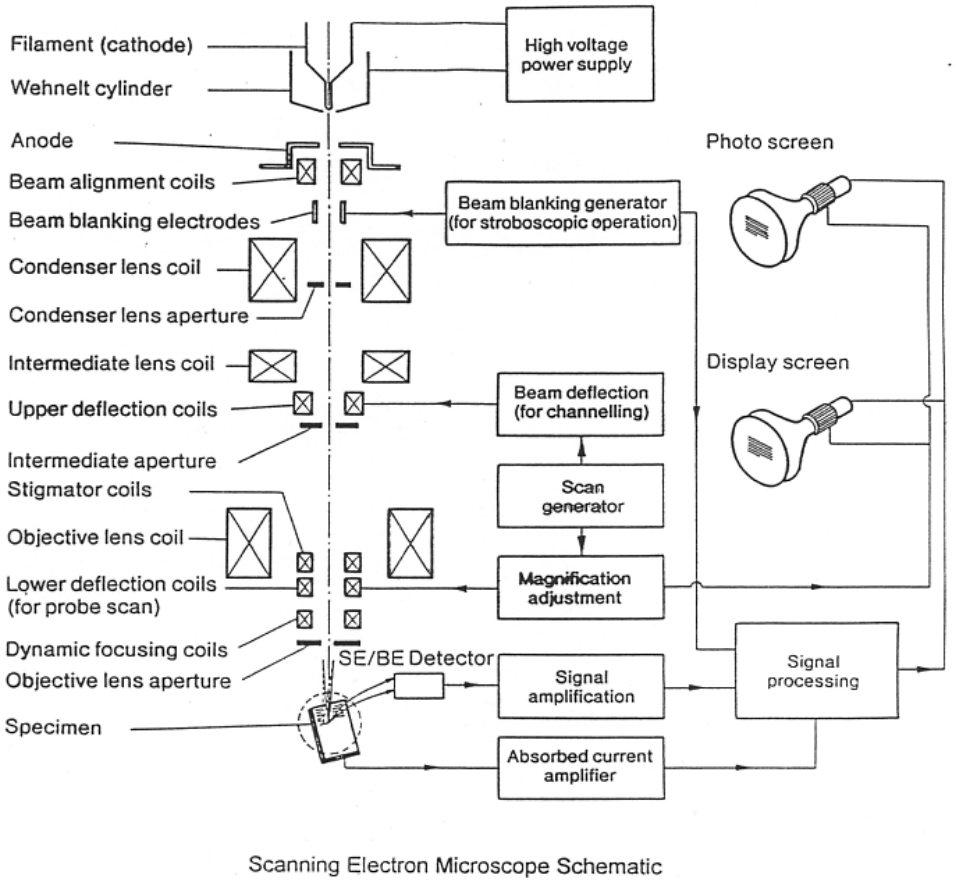
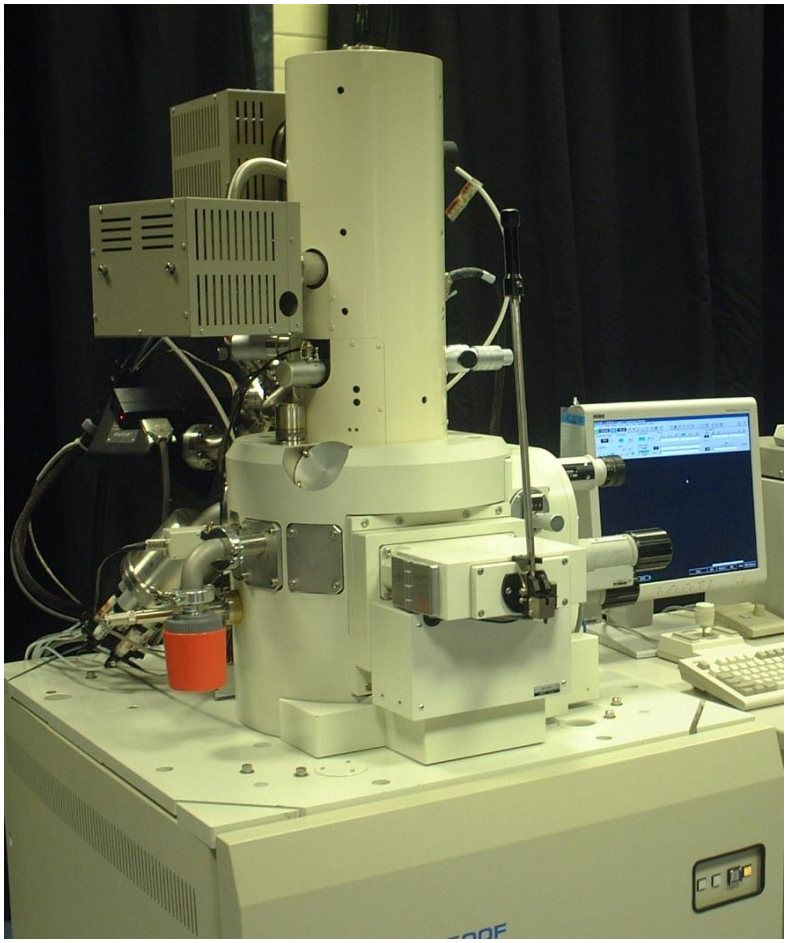
1.3 Scanning electron microscopy



Scanning Electron Microscope Schematic

sketch of typical SEM layout



One of several Scanning Electron Microscopes at the University of Sheffield used for advanced materials research.

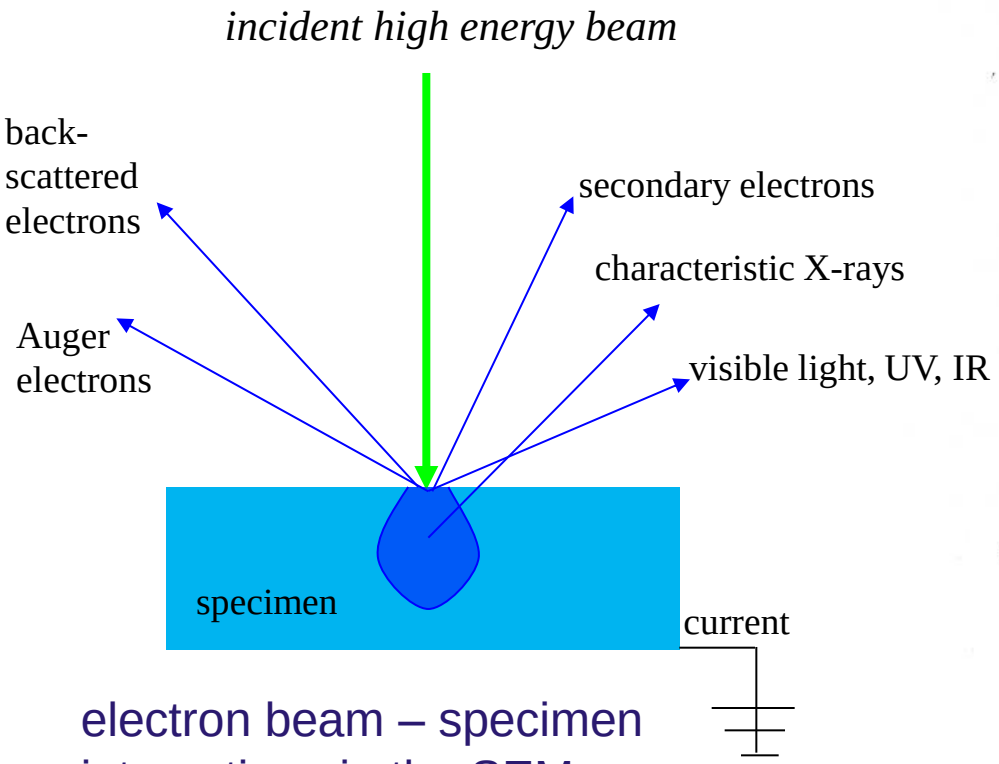
1.3 Scanning electron microscopy

1.3.1 structure of the SEM

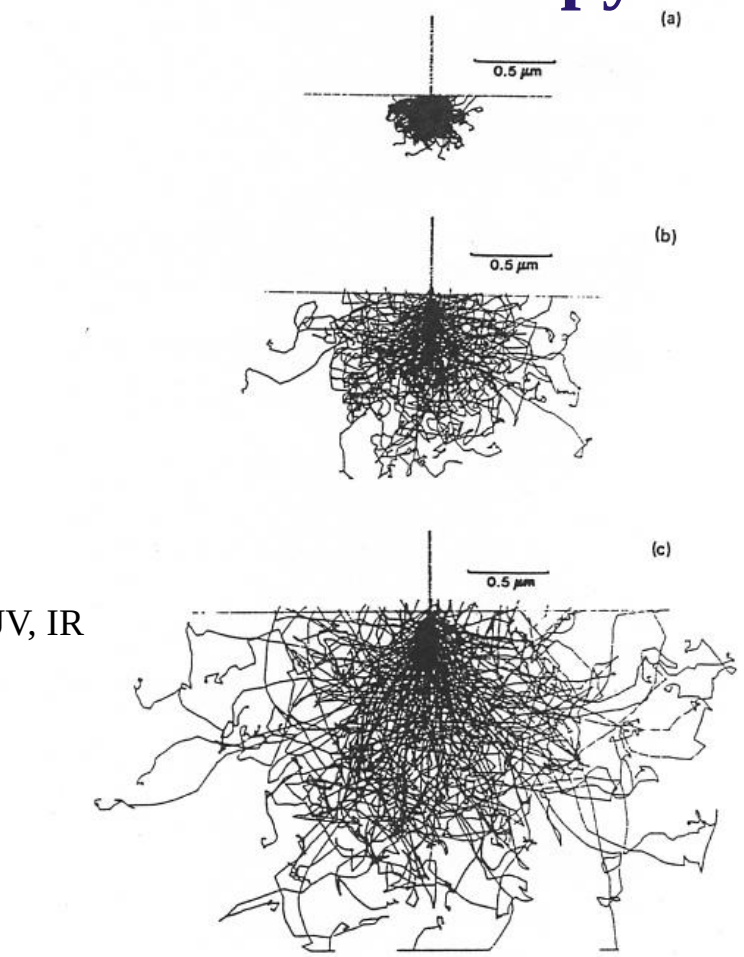
- electron-optical column, with electromagnetic lenses, under high vacuum
- electron gun: tungsten, LaB_6 or W/ZrO field-emitter
  - accelerating voltages from as low as 1kV up to 30-50kV
- electron beam formed into a small spot (down to ~1nm)
  - beam passes through scan coils to deflect the spot in a raster over the specimen: scanning very similar to cathode-ray tube monitor
- incident electrons interact with the specimen to generate a range of signals
  - for each signal, a separate detector is used
  - detector signals can be read out and stored point-by-point to generate images and distribution maps

1.3 Scanning electron microscopy

1.3.2 signals and detectors



electron beam – specimen interactions in the SEM



Monte Carlo calculations of the interaction volume in iron as a function of accelerating voltage: (a) 10keV, (b) 20keV (c) 30keV

DC Joy: Monte Carlo Modeling for Electron Microscopy and Microanalysis, OUP, New York, 1995

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1.3 Scanning electron microscopy

1.3.2 signals and detectors

- **secondary electron (SE) imaging**
  - relatively low energies (<50eV)
  - shows surface structure and strong topography contrast
  - detected using scintillator / photomultiplier
  - lateral resolution down to a few nm with ultra small spots (field-emission gun) and thin samples (to minimise scattering volume)
- **backscattered electron (BSE) imaging**
  - high energies (up to primary electron beam energy)
  - detected using solid state diodes
  - carries information on topography, atomic number and crystallography
- **analytical modes – these include**:
  - energy-dispersive X-ray spectroscopy (EDXS) or microanalysis
  - Auger electron spectroscopy (in special UHV instruments)
  - cathodoluminescence (CL) imaging
  - electron beam induced current (EBIC)

1.4 Transmission electron microscopy

Transmission Electron Microscope (TEM)

- **used to study the internal structure and composition of materials**
  - requires thin electron transparent specimens: <100nm thick
  - samples typically mounted on 3mm diameter disks or mesh grids (mesh grids coated in a very thin amorphous film are often used for the examination of nano-particles)
- **specimens preparation a specialised procedure**
  - typically material mechanically polished to 10-15μm thickness and reduced to electron transparency using Ar^+ ion milling
  - non-conducting samples may require a thin coating with carbon to reduce charging
  - recent developments include the application of focused ion beam instruments to prepare site-specific cross-sections (see lecture 3)

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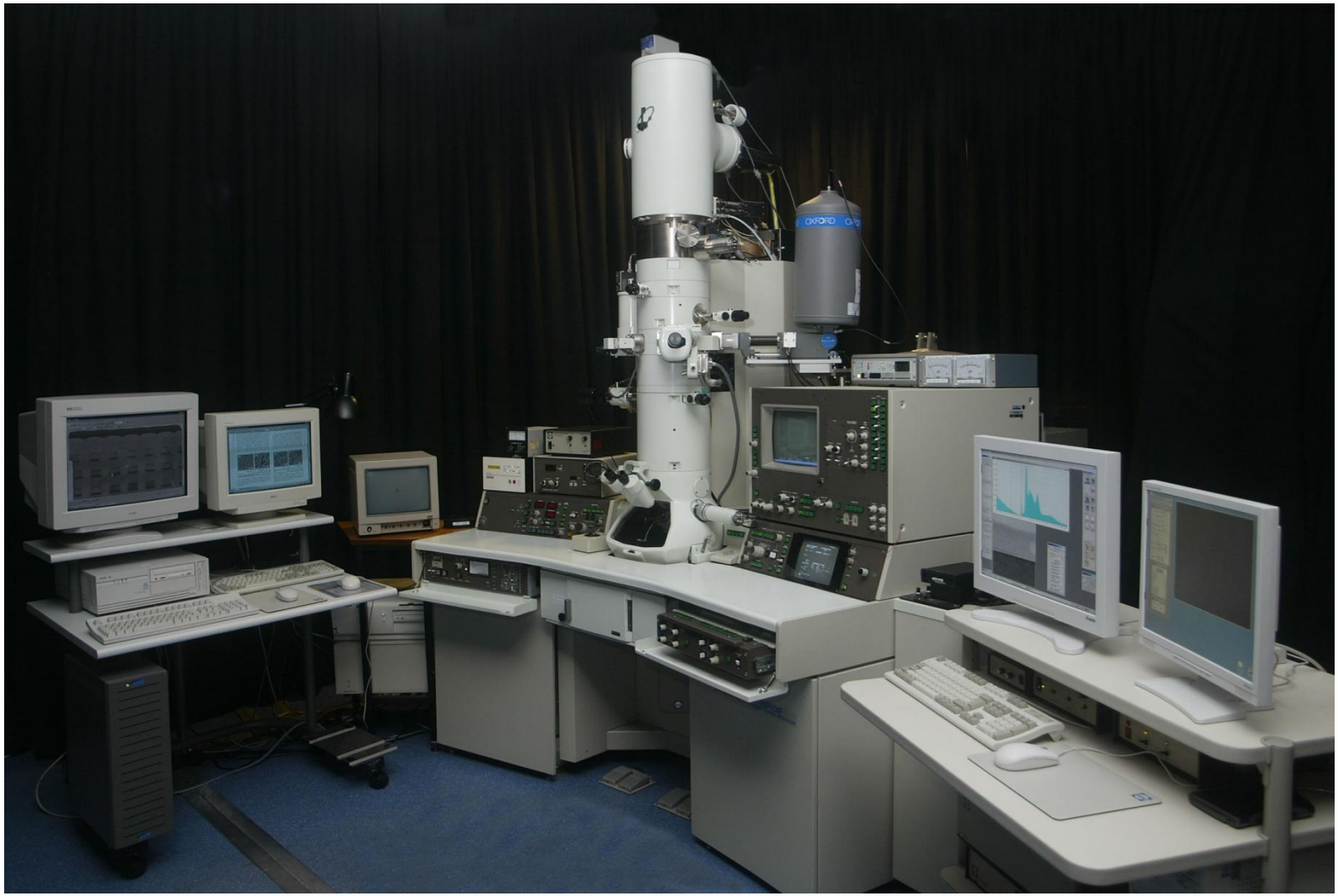
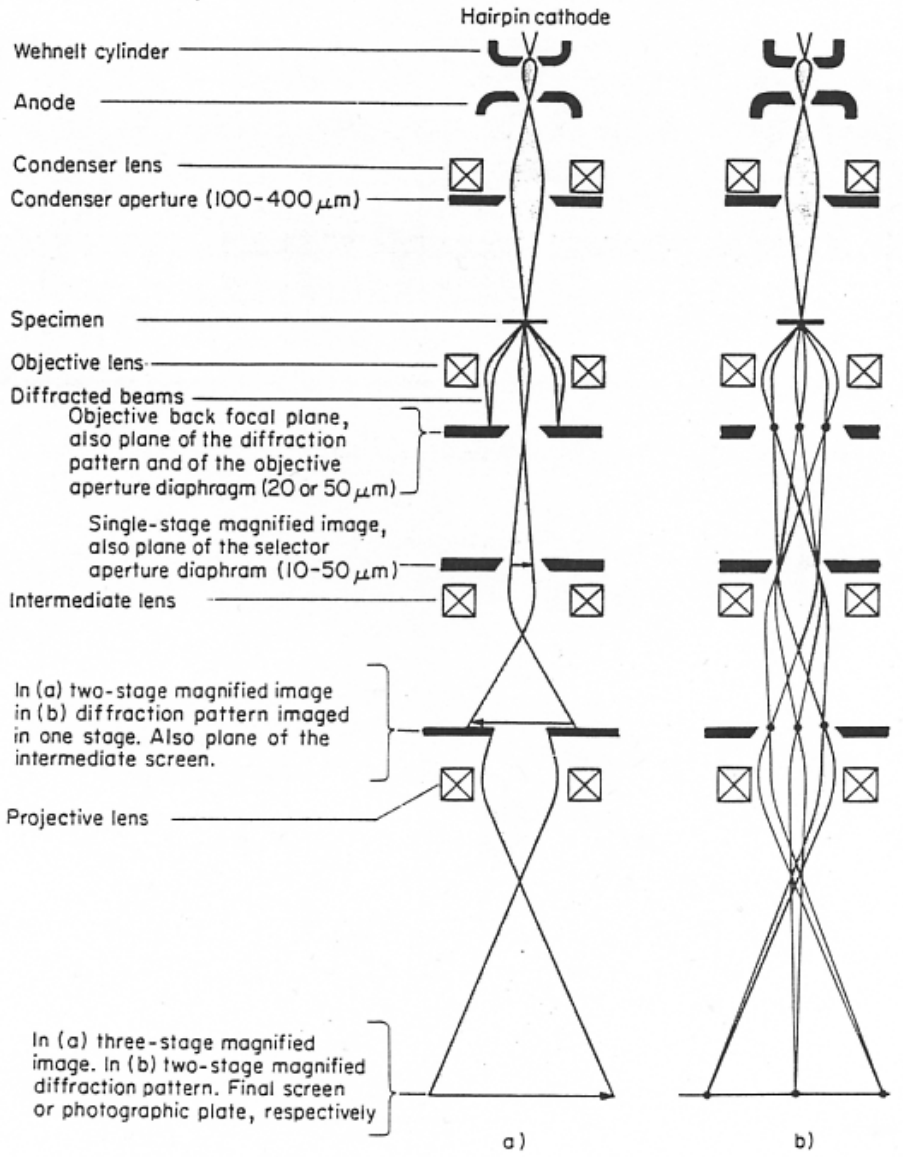


Sheffield University JEOL 2010F TEM/STEM with add-on detectors

General ray diagrams for a Transmission Electron Microscope (TEM)

a) imaging mode

b) diffraction mode



1.4.1 structure of the TEM

- electron-optical column, with electromagnetic lenses, under high vacuum
- electrons generated in a ***gun*** and accelerated by application of a high voltage (typically 100kV – 1.25MV)
- electron spot formed on thin specimen using a 2- or 3-stage ***condenser lens system*** and aperture arrangement;
- 2 basic operation modes:
  - stationary convergent spot-mode illumination by condenser (de-magnifies electron source) or
  - transmission imaging operation with parallel beam illumination
- electrons transmitted by specimen pass through ***objective lens***
  - magnified first **image formed in image plane**
  - **diffraction pattern simultaneously formed in back-focal plane**
- ***intermediate and projector lenses*** further magnify and project beam on to final fluorescent screen or photographic film/CCD camera for live recording
  - image magnifications from 50 up to 1.5×10^6 directly displayed
  - TV camera/intensifier provides images up to > $\times 10^7$ magnification
  - diffraction pattern can also be projected onto film/camera, depending on lens settings
- add-on detectors for chemical analysis and special signal recording

Scanning transmission electron microscopy

1.4.2 structure of the STEM

- basic construction similar to that of TEM, but:
  - electron beam is focused and scanned in a raster across the specimen (as in an SEM but different from the static beam illumination used in a conventional TEM)
  - ultimate spatial resolution of a STEM depends on the probe forming capabilities of the illumination system: ~0.1nm now possible, i.e. atomic column chemical analysis by electron energy-loss spectroscopy (EELS)
  - needs ultra-high vacuum (UHV) to prevent sample contamination during long (serial) data acquisition
  - electrons transmitted through the sample are detected on a range of electron detectors
  - STEM imaging can be performed in either a dedicated instrument or a convention TEM equipped with a means to: form a small probe (need FEG source), scan the beam and record the image

left: dedicated VG HB501 STEM (photo courtesy of Dept. Materials, Oxford University); right: JEOL 2010F FEG-TEM equipped with a scanning unit and bright-field and annular dark-field electron detectors

1.4.3 signals and detectors

- for TEM imaging and diffraction:
  - phosphor screen, film (negative plates), TV camera, charge-coupled device (CCD) camera
- for BF and ADF-STEM imaging:
  - pneumatically retractable solid-state detectors
- for spectroscopy:
  - imaging energy filter for electron energy-loss spectrometer (EELS)
  - thin-window semiconductor detector for energy-dispersive X-ray spectroscopy (EDXS)
  - possibly other special detectors for secondary electrons, cathodoluminescence etc. (like in SEM);

    problem: much weaker signals than in SEM (smaller volumes),

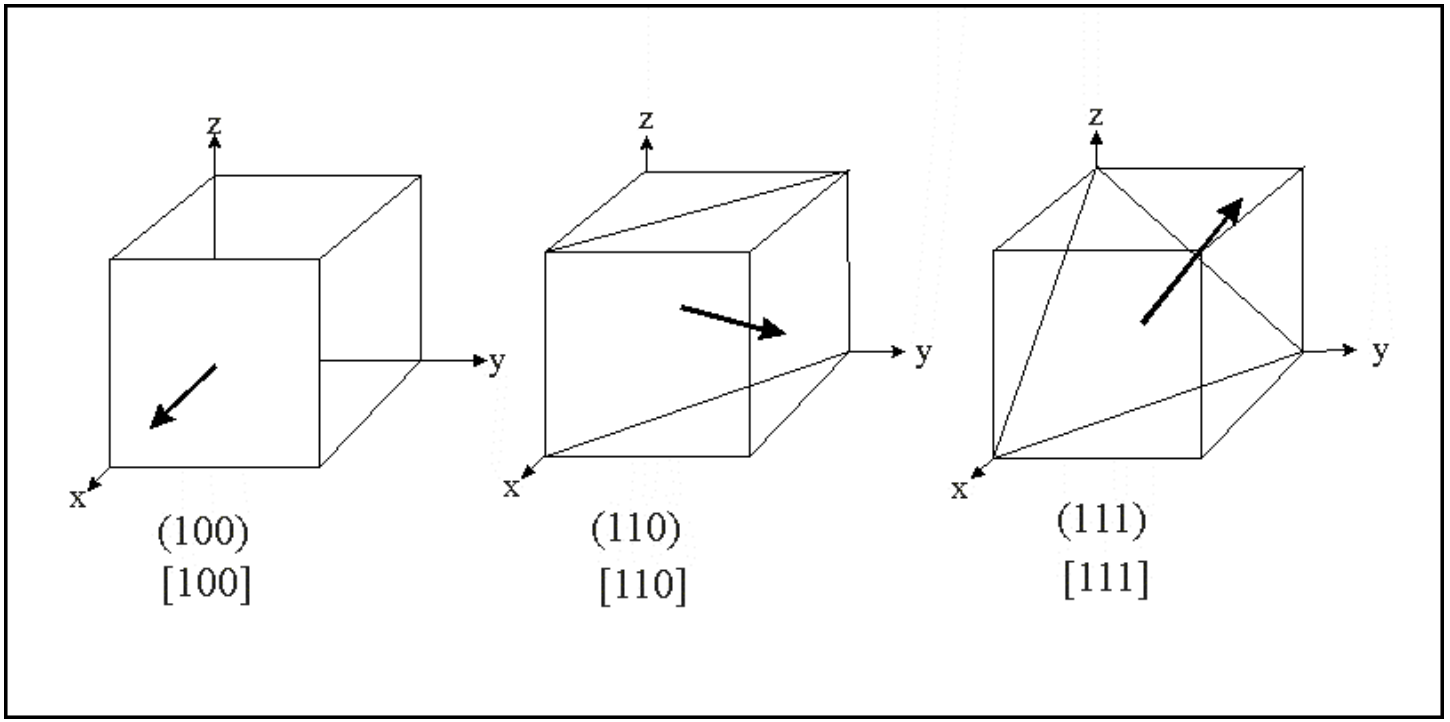
    advantage: higher spatial resolution obtainable

Why Is Diffraction of Interest?

- useful information about general structure
  - crystalline or amorphous
  - symmetry and atomic arrangement (crystallographic space groups)
  - planar spacings and lattice parameters
  - identification of phases, crystallite sizes
  - microstructure
  - defects (dislocations, twins, stacking faults, inversion domain boundaries, grain boundaries)
  - local lattice strains
  - inclusions (precipitates, grain boundary phases)

1.5 Basic concept of electron diffraction

Miller Indices of Crystallographic Planes

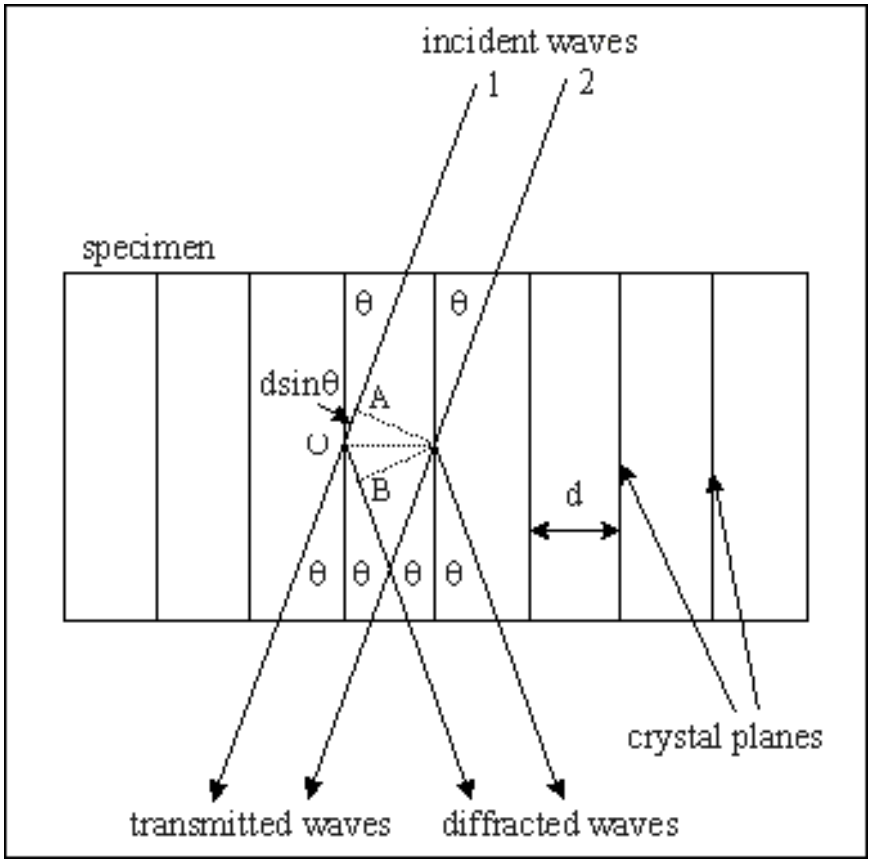


unique plane (hkl), set of planes {hkl)

unique direction [hkl], set of directions <hkl>

1.5 Basic concept of electron diffraction

1.5.1 Bragg's Law



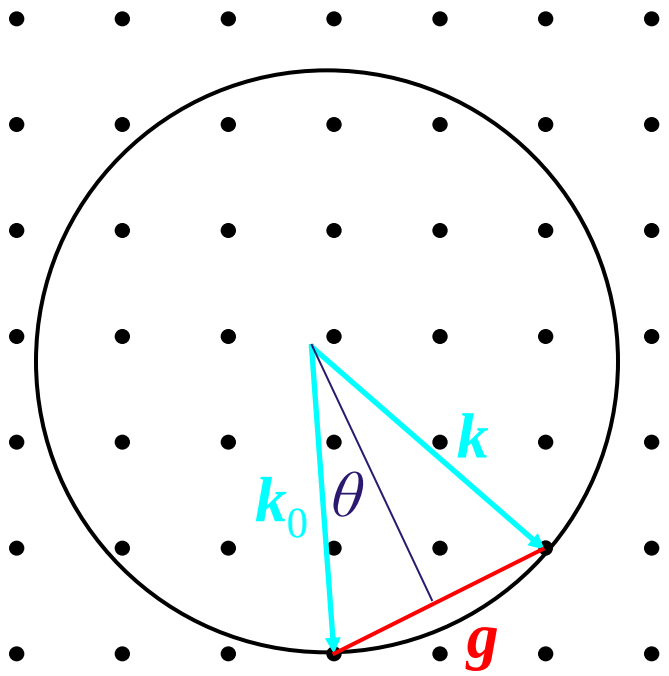
- optical path difference between diffracted waves 1 and 2 is $2d \sin\theta$
- If $2d \sin\theta = n\lambda$ then the waves are in-phase and interference with each other constructively
- **therefore the conditions for diffraction :**

$$2d \sin\theta = n\lambda$$

BRAGG'S LAW

1.5 Basic concept of electron diffraction

1.5.2 Ewald's sphere construction



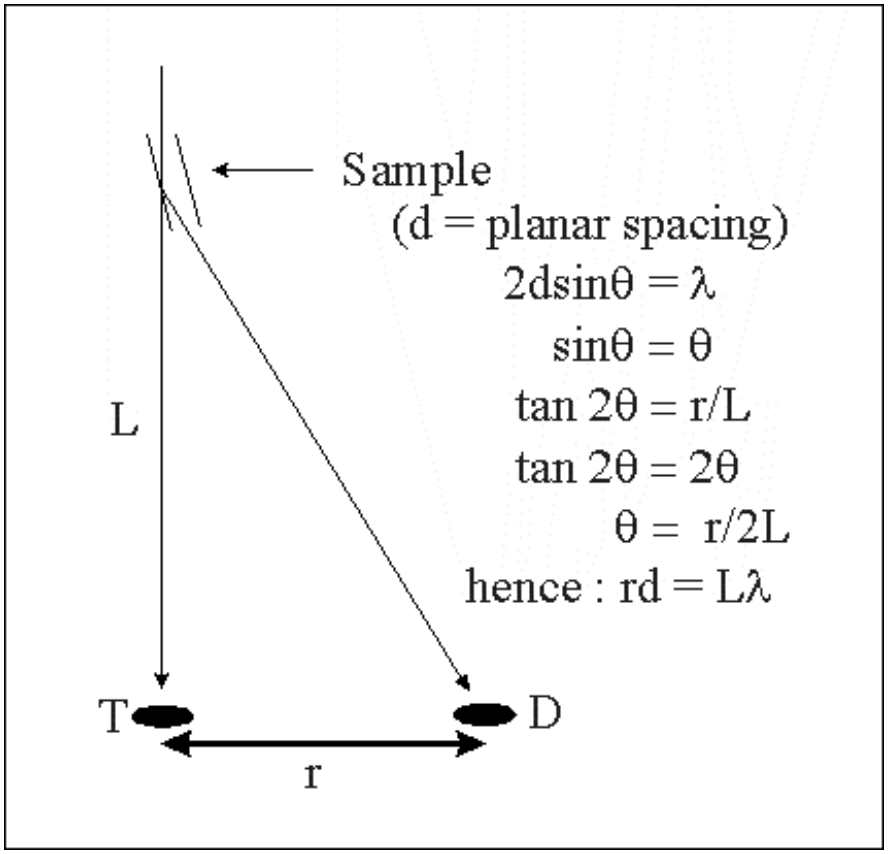
construct so-called 'reciprocal lattice' with points of all crystal reflections, then draw circle with radius $k_0=1/\lambda$ and determine the directions for the incoming beam $\boldsymbol{k}_0$ and the scattered beam $\boldsymbol{k}$. Diffraction then occurs only if difference is a reciprocal lattice point, i.e.:

$\sin\theta=n(g/2)/k_0=n\lambda/2d$

same result as Bragg's Law!

1.5 Basic concept of electron diffraction

1.5.3 The spot diffraction pattern



Each set of lattice planes near the Bragg condition gives a **point** in the diffraction pattern.

The spacing "*r*" in the diffraction pattern of back focal plane is *inversely* proportional to the real crystallographic interplanar spacing "d" because their product is a constant, namely the product of camera length *L* and wavelength λ.

1.5 Basic concept of electron diffraction

1.5.4 The structure factor (kinematical diffraction theory)

The structure factor describes the way in which an incident beam of electrons is scattered by the atoms of a crystal unit cell

The wavefunction of a diffracted wave in the Born (i.e. high energy) approximation by a crystal potential $V(\boldsymbol{r})$ is given by the following formula wherein $\Delta\boldsymbol{k}=\boldsymbol{k}_0-\boldsymbol{k}$ with $\Delta k=2\sin\theta/\lambda$:

$\Psi_s(\boldsymbol{k},\boldsymbol{r})= A_0$ amplitude of ingoing wave

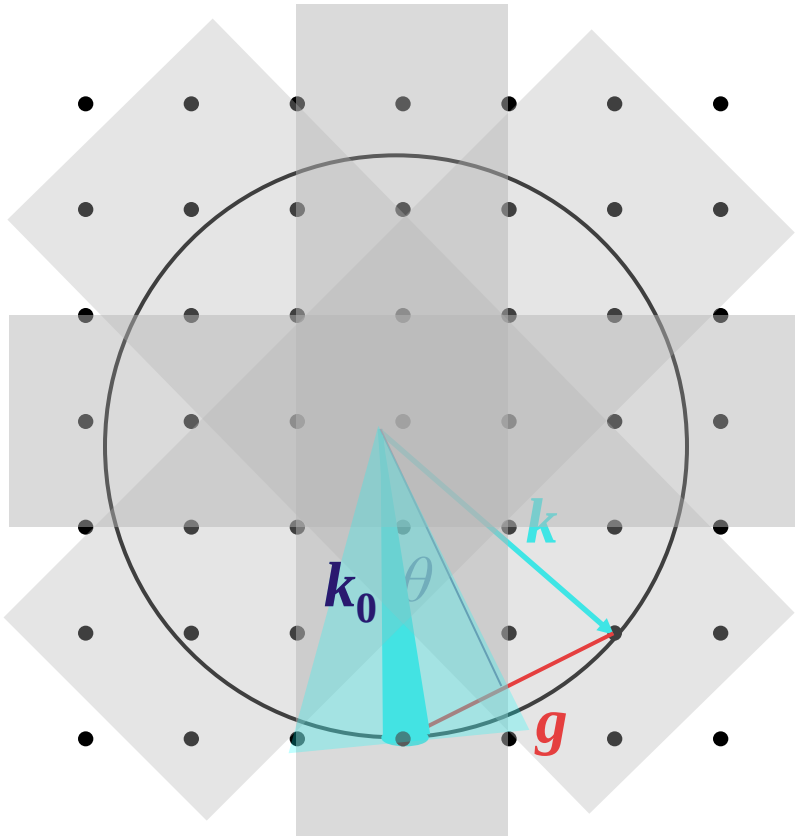
- $\exp(2\pi i\boldsymbol{kr})/r$ spherical wave
- $\Sigma_n \exp(-2\pi i\Delta\boldsymbol{kr}_n)$ sum over all unit cells
- $\Sigma_m \exp(-2\pi i\Delta\boldsymbol{kr}_m)\ 2\pi m_0 e h^{-2} \Sigma_{atom} V(\boldsymbol{r}_j) \exp(-2\pi i\Delta\boldsymbol{kr}_i)\ d\tau_j$

$f_m(\theta)$: atomic scattering factor (tabulated)

$\boldsymbol{F_n(\theta)}$: **structure factor** (can be easily calculated)

1.5 Basic concept of electron diffraction

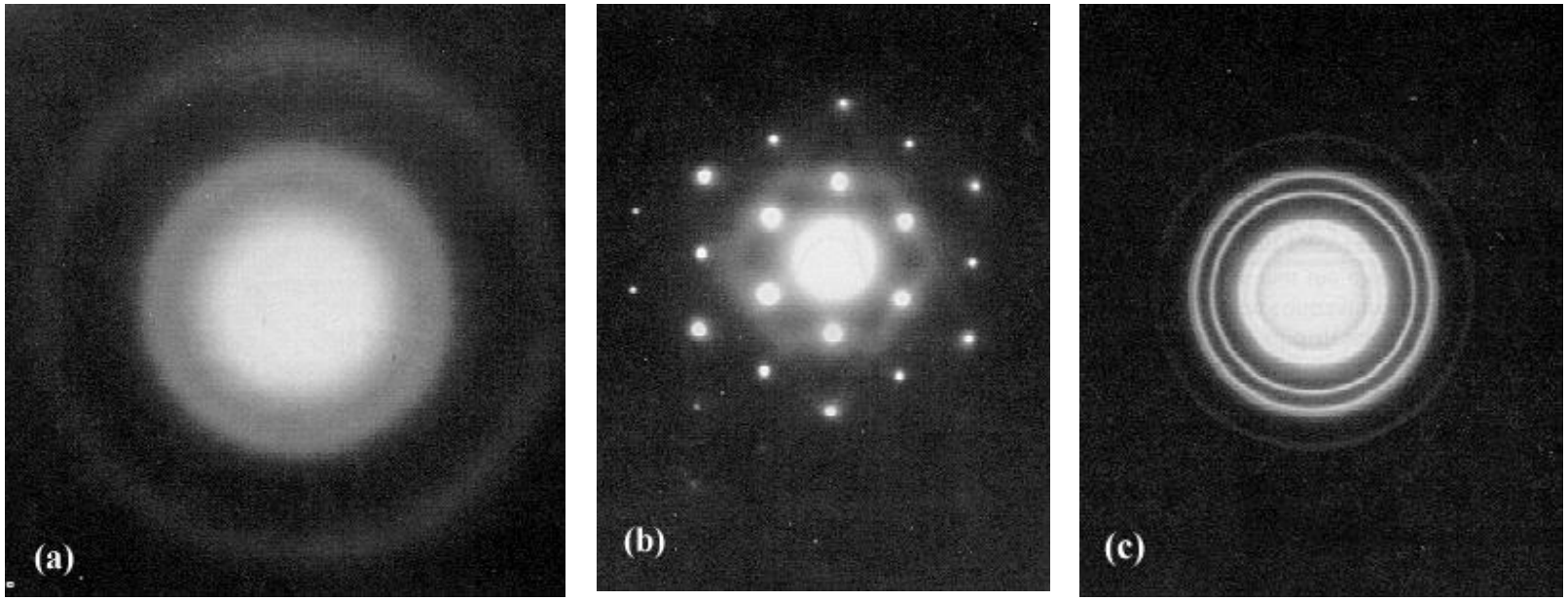
1.5.5 Kikuchi bands



For convergent beam illumination or multiple inelastic scattering **in a thick specimen**: replace arrow for direction of incidence by a **cone** and obtain a **Kikuchi pattern with bright ('excess') and dark ('deficiency') lines** instead of a spot pattern. The pattern's symmetry contains detailed crystallographic information.

2. Electron diffraction techniques

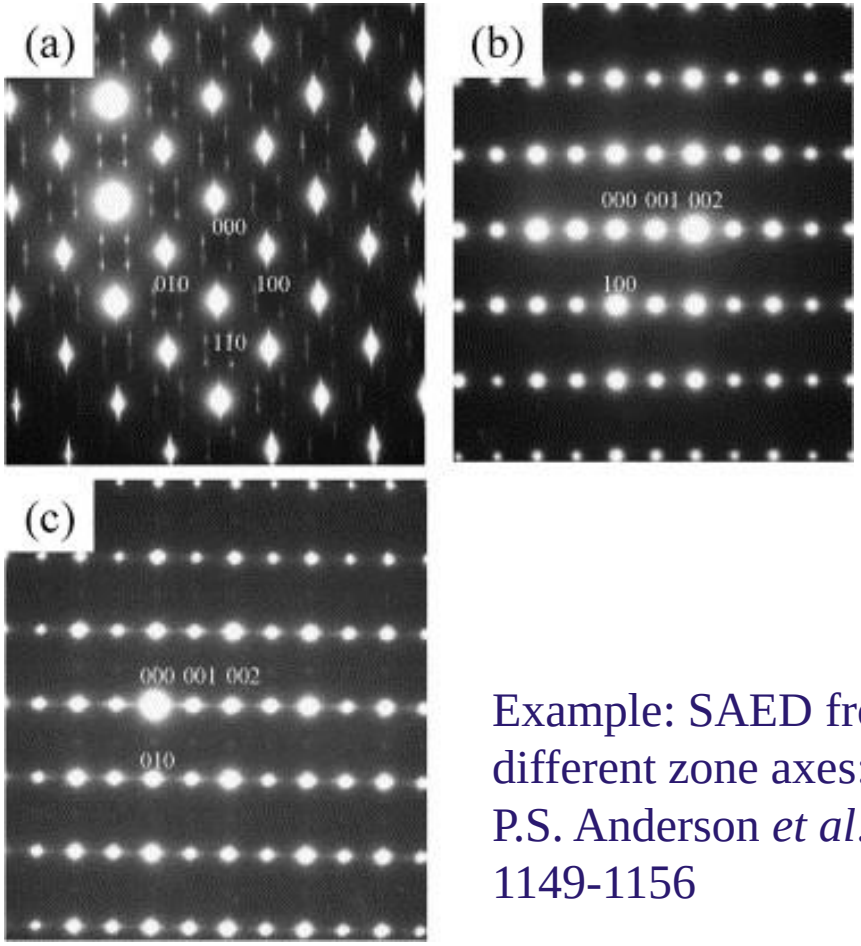
2.2 Selected-area electron diffraction (SAED) in the TEM



selected-area electron diffraction patterns obtained from three different materials: (a) amorphous carbon film, (b) Aluminium single crystal and (c) poly-crystalline gold. images courtesy of IM Ross, Univ. Sheffield

2. Electron diffraction techniques

2.2.1 Space group determination from tilting experiments

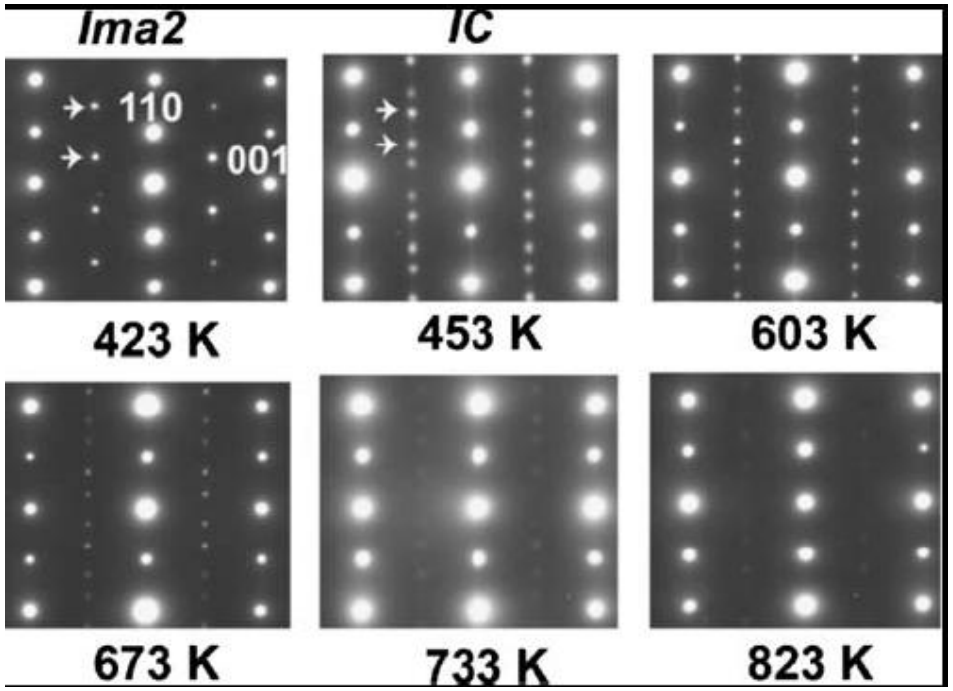


Principle: take diffraction patterns from various directions, then try to index the spot patterns and find forbidden reflections (F=0)

Example: SAED from $NdBaCo_2O_{5.5}$ along three different zone axes: <100>, <010>, <001>
P.S. Anderson *et al.*, Sol. State Sciences 7 (2005) 1149-1156

2. Electron diffraction techniques

2.2.2 Study of phase transitions as a function of temperature

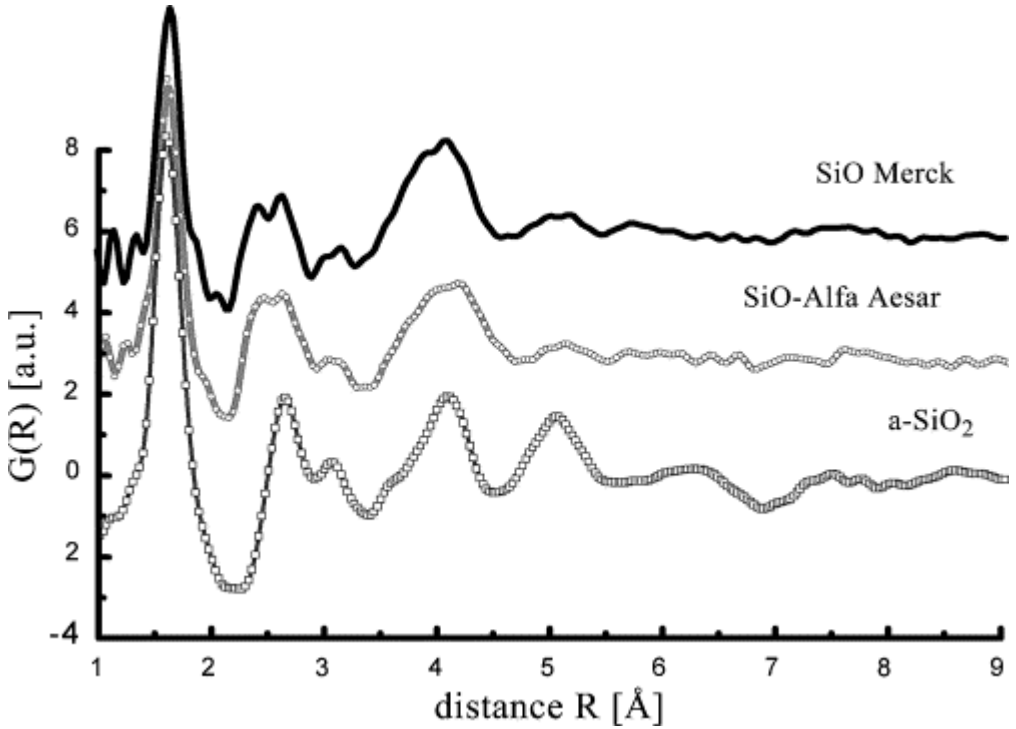


Example: Ima2 – IC – P4/mbm phase transition in <110> $Ba_2NdNb_3Ti_2O_{15}$ bronze
I. Levin *et al.*, Appl. Phys. Lett. 89 (2006) 122908

2. Electron diffraction techniques

2.2.3 Radial distribution functions of amorphous materials

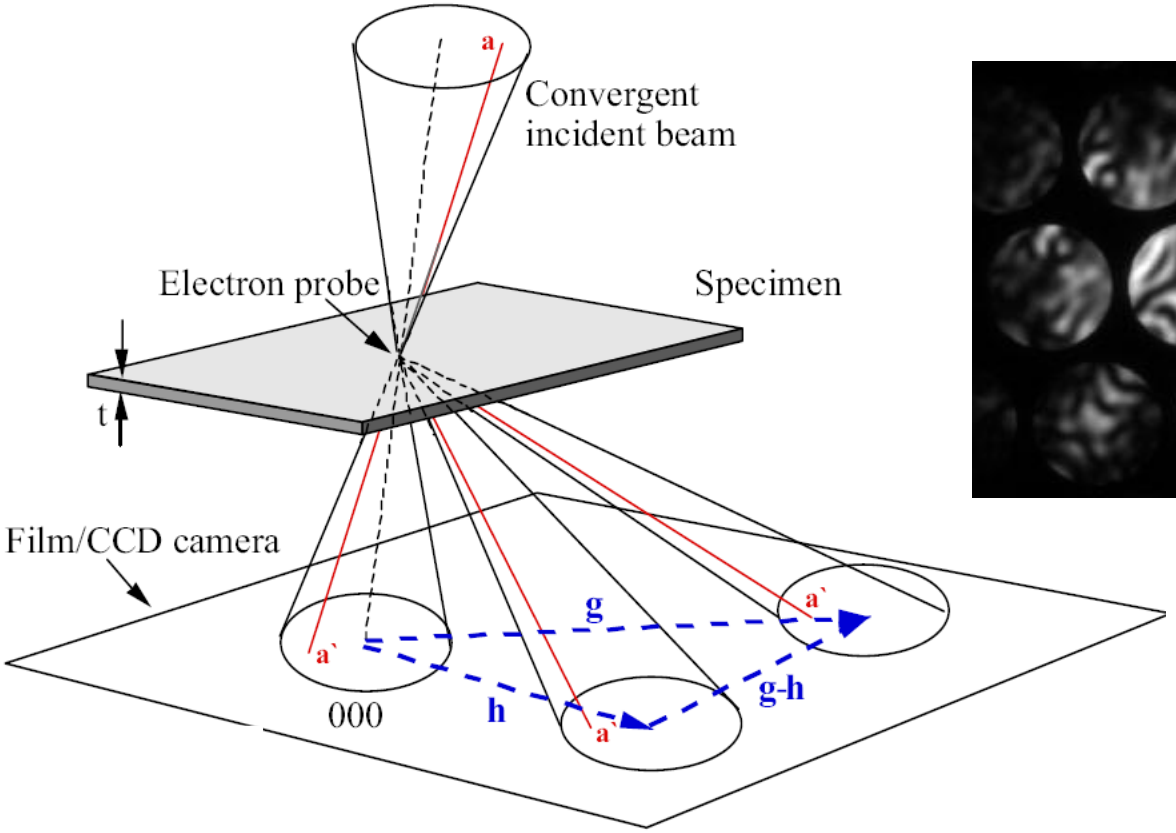
Principle: record diffraction pattern, get radial profile by line averaging along azimuth angle, subtract background, Fourier transform to real space: peaks = bond lengths

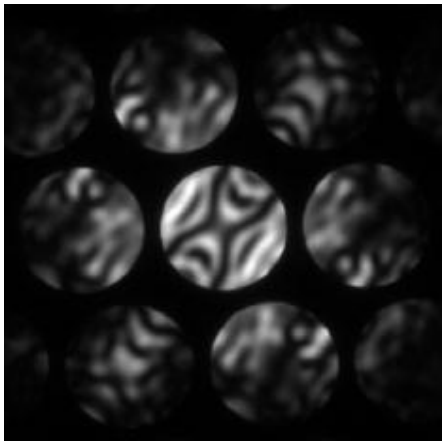


Example: comparison of atomic distances from different oxides
K. Schulmeister and W. Mader, J. non-cryst. Solids 320 (2003) 143-150

2. Electron diffraction techniques

2.3 Convergent-beam electron diffraction (CBED) in the TEM

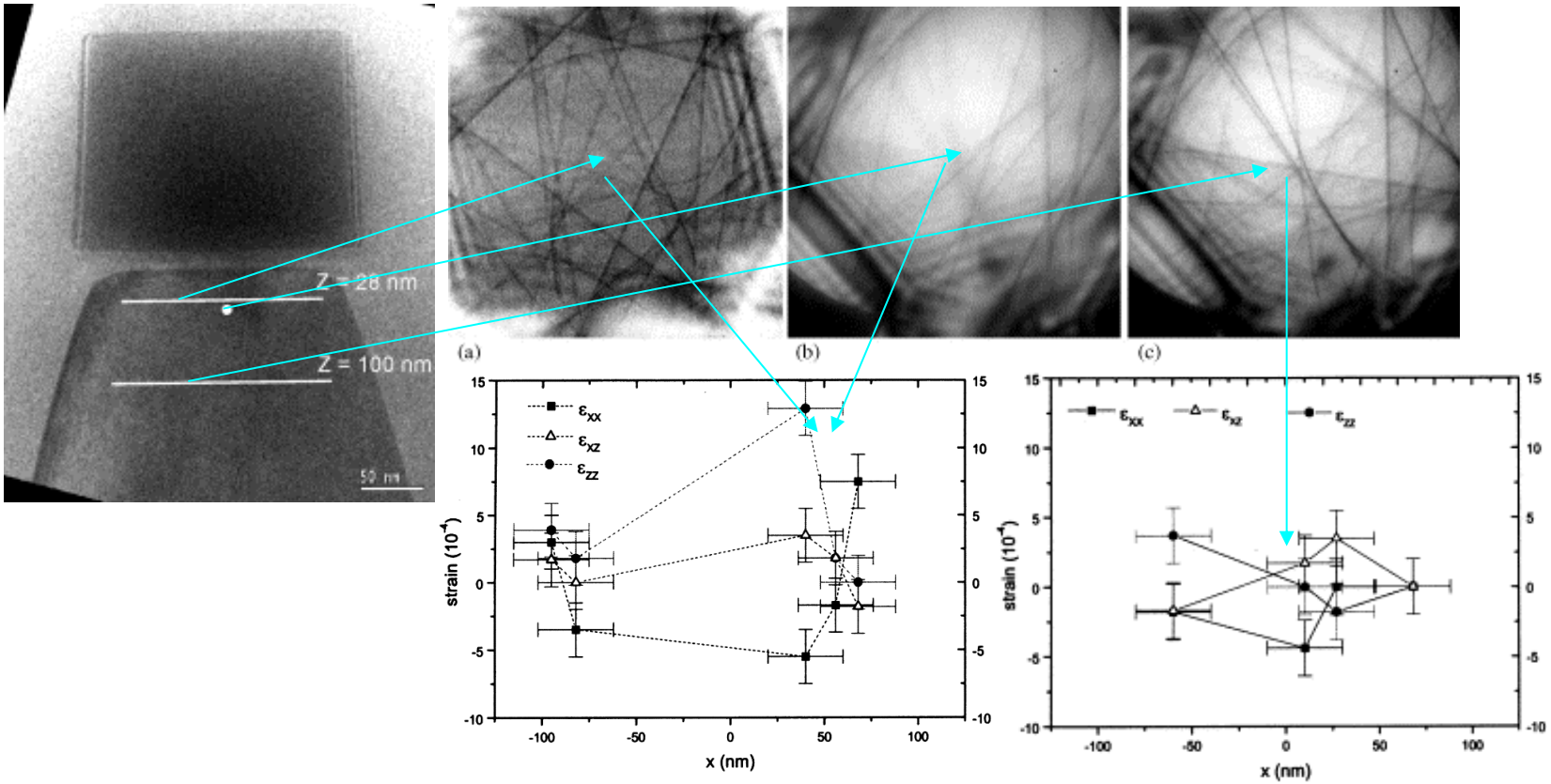




advantages: very small probe possible, each point in the discs refers to one incident beam direction (have multiple directions)
disadvantages: contamination and radiation damage; complicated modelling

2. Electron diffraction techniques

2.3.1 local strain measurements



Example: strain measurement in a shallow trench isolation structure with ~10nm spatial resolution;

A. Armigliato *et al.*, Mater. Sci. Semicond. Process. 4 (2001) 97-99

The University Of Sheffield.

Lecture 1 - Summary

•Electron microscopy offers the increased spatial resolution compared to conventional optical microscopy needed to investigate many modern materials problems.

•Scanning electron microscopy (SEM): focused probe scanned across specimen surface; topographical and near surface analysis; imaging resolution down to 5nm; larger specimen format; chemical and structural analysis possible (next lecture); moderate specimen vacuum compatibility required; moderate skill required to operate.

•Transmission electron microscopy (TEM and STEM): internal structure; imaging resolution down to 0.2nm (0.05nm possible with aberration correction (next lecture); electron transparent specimen <100nm; high precision chemical analysis possible (next lecture); high specimen vacuum compatibility required; highly skilled operator needed.

•Electron diffraction: in TEM or STEM, provides important additional information about the specimens atomic structure and composition: