

# **EEE6009 Advanced Instrumentation Electron microscopy - Lecture 2**



#### In lecture EM1 we discussed:

- The physical, mechanical and electrical properties of many solid state materials is governed by their structure at the micro (10-6m), nano (10-9m) and atomic (10-10m) scale.
- To control these properties for component manufacture we need a means of understanding and examining these structures.
- Conventional optical microscopy provides limited resolution and scope for analysis required for many of todays unsolved materials problems.
- Electron microscopy offers the increased spatial resolution, due to the significantly shorter wavelength of the electron, compared to conventional optical microscopy.
- The electron beam matter interaction generates several useful signals that can be used for both the formation of images and provide chemical (photon/X-ray spectroscopy and electron energy-loss spectroscopy) and structural information (electron diffraction).

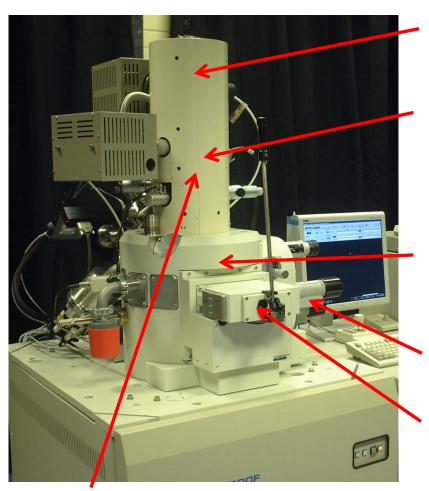


#### In lecture EM1:

- The range of instruments and techniques associated with electron microscopy is vast however from an instrumentation perspective it can be broken down roughly into two sectors:
- Scanning electron microscopy (SEM): focused probe scanned across specimen surface; topographical and near surface analysis; imaging resolution down to 5nm (new advanced instruments can now achieve <1nm); larger specimen format (defined by the specimen chamber size); chemical and structural analysis possible; moderate specimen vacuum compatibility required (10-4Pa to 10-6Pa); moderate skill required to operate.
- Transmission electron microscopy (TEM and STEM): electrons *pass* through the specimen to reveal the internal structure; imaging resolution down to 0.2nm (0.05nm possible with aberration correction (see lecture EM3); electron transparent specimen <100nm; specimen size limited to 3mm diameter disc; high precision chemical analysis possible; different types of imaging modes and methods to determine atomic structure (diffraction): high specimen vacuum compatibility required (10-6Pa); highly skilled operator needed.



# Revision from lecture 1 – Scanning electron microscope



Electron gun: (accelerating voltages from as low as 1kV up to 30-50kV)

Beam passes through a condenser lens system/apertures and scan coils to deflect the focused spot in a raster over the specimen scanning very similar to cathoderay tube display

Electron beam formed into a small spot (down to ~1nm) and scanned across the specimen surface

Specimen chamber and stage with x, y, z, rotation and tilt axis adjustment

Specimen exchange airlock

Electron-optical column, with electromagnetic lenses, under high vacuum



### secondary electron (SE) contrast

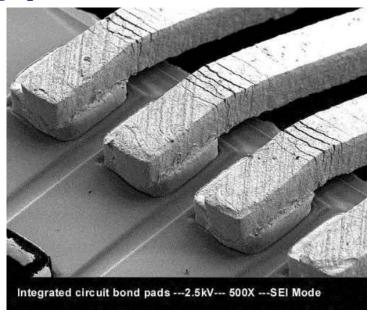
process: an incident electron passes close to an atom in the specimen and ionises it. This causes energy loss and a slight path change in the incident electron. The ionised electron can leave the atom with a very small kinetic energy (1eV...50eV) and is hence termed a "secondary electron". Each incident primary electron can produce several secondaries. Due to their low energy only secondaries near the surface (a few nm) can exit the sample and be examined. This yields strong topographical contrast of the surface.

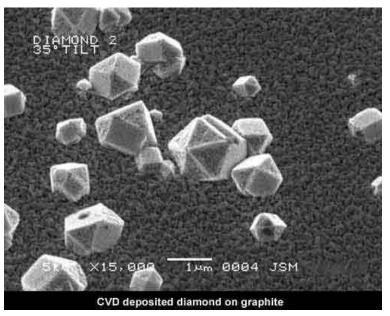
detection: collection of these electrons is aided by using a "collector" in conjunction with a scintillator-photomultiplier as detector. The collector is a grid or mesh at positive potential and is placed in front of the detector, attracting the negatively charged secondary electrons to it which then pass through the grid-holes and into the detector to be counted. The number of secondary electrons reaching the detector is converted into brightness for each image point during scanning and depends on illumination and collection angle. Steep surfaces and edges appear brighter than flat surfaces, which gives the images a three-dimensional impression. A spatial resolution of about 1 nm is possible in this mode.



#### secondary electron (SE) contrast

process: an incident electron passes close to an atom in the specimen and ionises it. This causes energy loss and a slight path change in the incident electron. The ionised electron can leave the atom with a very small kinetic energy (1eV...50eV) and is hence termed a "secondary electron". Each incident primary electron can produce several secondaries. Due to their low energy only secondaries near the surface (a few nm) can exit the sample and be examined. This yields strong topographical contrast of the surface.







#### back-scattered electron (BSE) contrast

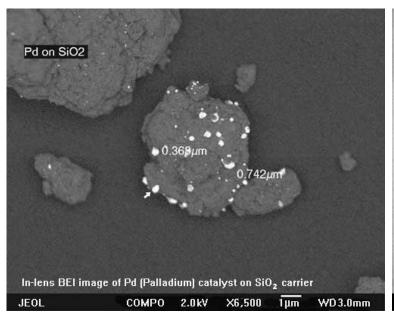
process: backscattered electrons are caused by an incident electron colliding head-to-head with an atom in the specimen. The incident electron is then scattered "backward" by ~180 degrees. Different scattering cross-sections causes higher atomic number elements to appear brighter than lower atomic number elements. This atomic number contrast adds to the weaker topographical contrast also present. Backscattered electrons can also be used to form an electron backscatter diffraction pattern (EBSD) for determining the crystallographic structure of the specimen surface region.

detection: there are fewer backscattered electrons emitted from a sample than secondary electrons. The number of backscattered electrons leaving the sample surface upward might be significantly lower than those that follow sideward trajectories. Additionally, in contrast with the case with secondary electrons, the collection efficiency of backscattered electrons cannot be significantly improved by a positive bias common on an Everhart-Thornley detector. This detector positioned on one side of the sample has low collection efficiency for backscattered electrons due to small acceptance angles. The use of a dedicated backscattered electron detector above the sample in a "doughnut" type arrangement, with the electron beam passing through the hole of the doughnut, greatly increases the solid angle of collection. Both spatial resolution and signal-to-noise ratio are inferior to secondary electron images.



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process: backscattered electrons are caused by an incident electron colliding head-to-head with an atom in the specimen. The incident electron is then scattered "backward" by ~180 degrees. Different scattering cross-sections causes higher atomic number elements to appear brighter than lower atomic number elements. This atomic number contrast adds to the weaker topographical contrast also present. Backscattered electrons can also be used to form an electron backscatter diffraction pattern for determining the crystallographic structure of the specimen.





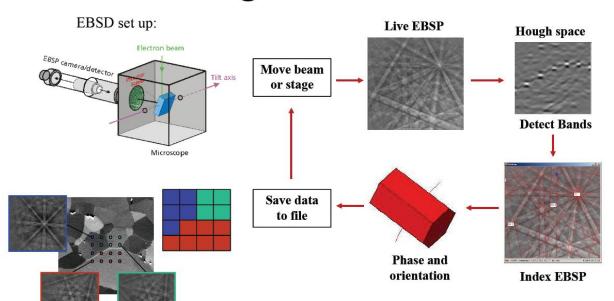


#### **Electron diffraction in the SEM**

#### electron back-scatter diffraction (EBSD)

A single automated EBSD run can provide rather complete characterisation of the microstructure: it maps the local sample crystallographic orientation (down to ~20nm resolution if the voltage is reduced to ~5kV)

## Indexing & Automation



from which one can construct maps of

- phase distribution
- grain size distribution

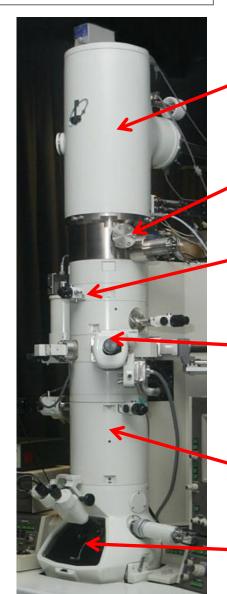
and thus measure also

- grain misorientations
- intra-granular deformation

Prerequisite: a very clean surface (produced e.g. by electro-polishing, etching, ion beam milling)

images courtesy of HKL Inc.





## Revision from lecture 1 – Transmission electron microscope

Electron gun: accelerating voltages from 80kV to 1200kV (typically 80-120kV for biological, 200-400kV for materials research).

Field emission gun requires a gun isolation valve to maintain high vacuum (~10<sup>-7</sup>Pa).

Condenser lens assembly and aperture defines the illumination which can be broad (TEM) or a focused probe scanned across the specimen (STEM)

Specimen air-lock, specimen held in beam path mounted at the end of a special holder (various designs, x, y tilt, rotation, heating/cooling LN<sub>2</sub>/LHe, electrical connections) Column vacuum ~ $10^{-6}$ Pa.

Post specimen projector lens system, magnifies the image or diffraction pattern.

 Viewing chamber for direct image observation or projection onto CCD camera or film

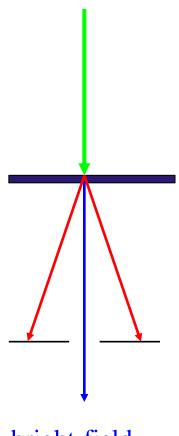


## **Basics of TEM image formation**

#### mass thickness (amplitude) contrast

- scattered electrons have an altered energy spread and angular range relative to the primary beam
- inserting a small moveable aperture into the backfocal (diffraction) plane of the objective lens (objective aperture or high contrast aperture just below the specimen) selects the electrons allowed to form the final image:

aperture centred on the optical axis — regions of the specimen which are thicker or of higher density will scatter more strongly and appear darker in the final image — *bright-field image* 



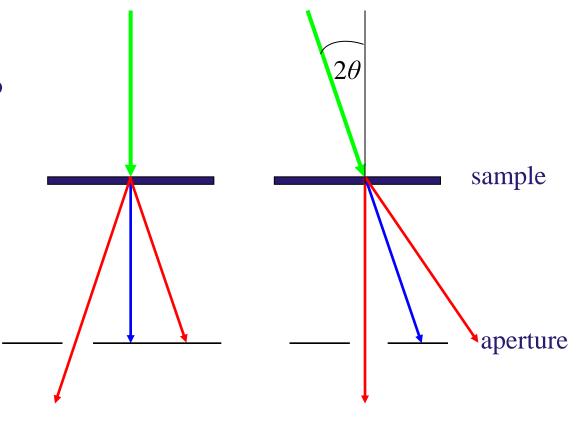
bright-field



## **Basics of TEM image formation**

#### mass thickness (amplitude) contrast

For crystalline samples, if particular diffracted beams are selected by an aperture to contribute to the final image it is possible to obtain important information about crystal defects such as dislocations, stacking faults and precipitates – *dark-field image* 

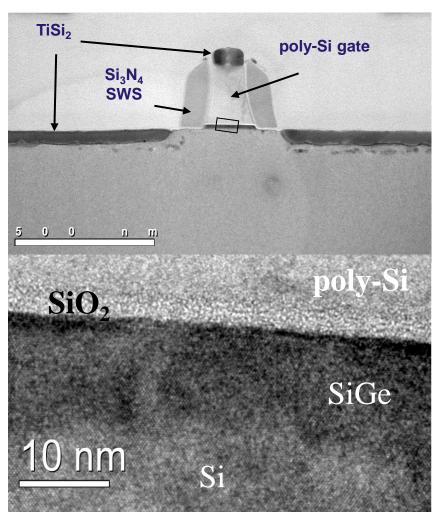


dark-field

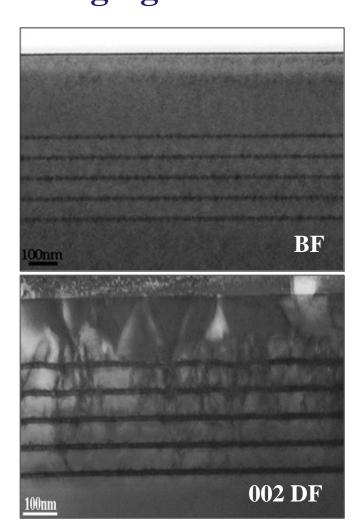


# bright-field (BF) and dark-field (DF) imaging in the TEM

#### mass thickness (amplitude) contrast



A.C.K. Chang et al., Thin Solid Films 496 (2006) 306-310



InGaAs(N)/GaAsN multiple quantum wells Gutierrez et al 2003

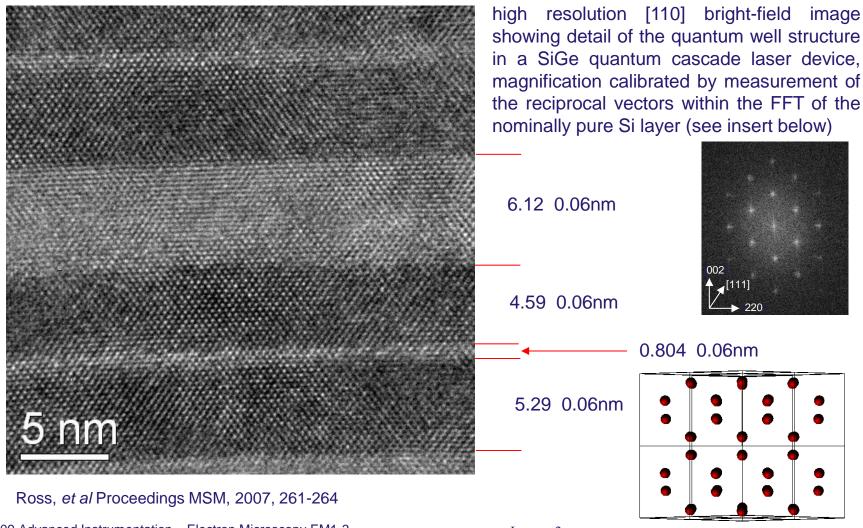


#### phase contrast:

- Relative *phase shifts* are introduced into the scattered electron waves by different regions of the sample.
- When many beams are accepted by the objective aperture and interfere
  with the main (un-scattered) beam constructive and destructive
  interference occurs.
- With correct defocus and at very high magnification, the image shows phase contrast which can reveal detail of the corresponding crystal lattice
   high resolution transmission electron microscopy (HREM)
- Can show atomic columns running through the crystal as dark or bright features – care required for interpretation; necessary to perform image simulation to confirm experimental data!

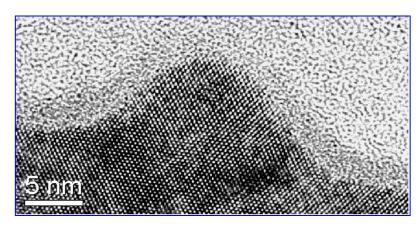


## Examples of phase contrast lattice images



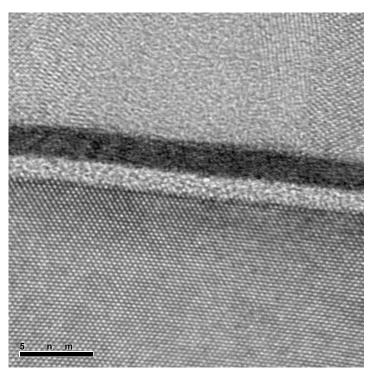


### lattice fringe spacings: more examples



high resolution TEM cross-section of an InAs/GaAs (001) quantum dot showing atomic columns

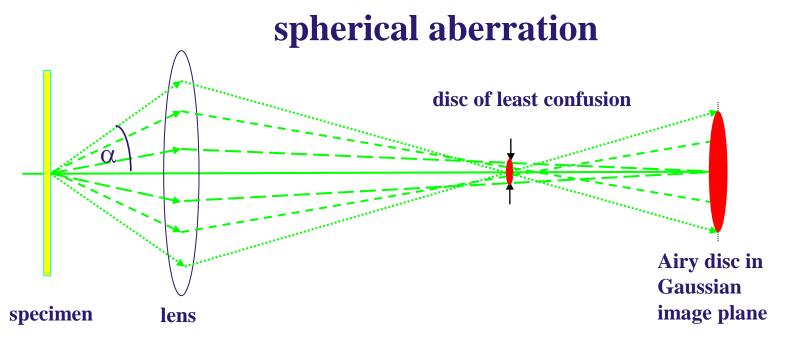
*Lattice fringe imaging* in the TEM can also be used to provide information about *localised strain* (P.H. Jouneau *et al.*, J. Appl. Phys. <u>75</u> (1994) 7310-6) and *chemical composition* (T. Walther *et al.*, Philos. Mag. A <u>72</u> (1995) 1015-1030)



high resolution TEM cross-section of the gate oxide layer in a state-of-the-art transistor device



lens aberrations I



The portion of the lens furthest from the optical axis brings rays to focus nearer the lens than the central portion of the lens. This results in a disc of least confusion of diameter d that is related to  $\alpha$  (the semi-angle at which rays leave points on the specimen) by:  $d_{\min} = 0.5 C_s \alpha^3$ 

 $C_{\rm s}$  is called spherical aberration constant; for most instruments it is ~1 mm.



The objective lens induces a **phase shift** of the electron beam which gives rise to the **contrast transfer function** which is in first order given by

$$\chi(g) = \pi/2(Cs\lambda^3g^4 + 2f\lambda g^2)$$

where Cs is the spherical aberration coefficient,  $\lambda$  is the wavelength of the incident electrons, g the reciprocal lattice vector and f the defocus.

Importantly, this means both focus and spherical aberration determine the contrast of the atomic columns in the lattice resolved HREM image.

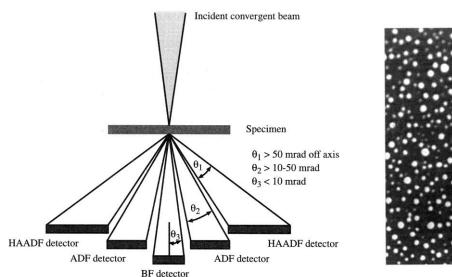
If we take the effects of spherical aberration into consideration and combine this with defocus at a particular value of  $\Delta f$  known as the Scherzer focus, the contrast transfer function shows a broad transfer and atomic columns appear dark. From this we get the so called **point resolution**:

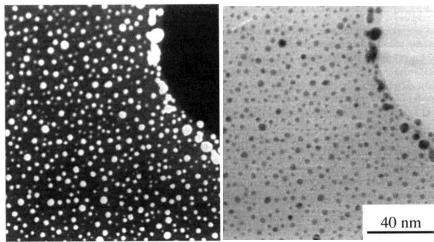
$$\approx 0.66 (C \mathrm{s} \lambda^3)^{1/4}$$

Hence, for a 200kV TEM with a Cs of 0.5mm the point resolution is  $r \approx 0.19$ nm. Information can be transferred down beyond the point resolution to the so called *information limit* but the details are not directly interpretable any more.



# scanning transmission electron microscopy (STEM)





STEM image formation: the diagram ,left, shows the arrangement of detectors and collection angles for bright field and Z-contrast (annular dark field) imaging in the STEM. the bright-field (BF) detector placed in the plane conjugate to the back focal plane to intercept the direct beam and beams scattered through low angles. The concentric annular dark field (ADF) detector(s) intercepts the diffracted beam (B). The signals from either detector are amplified and modulate the STEM CRT. Images on the right above show gold islands on carbon film, ADF and BF image respectively. (D.B. Williams and C.B. Carter: TEM – Vol.1: Basics, Springer, New York, 1996, p. 146)

Other STEM references:

Jesson & Pennycook, Proc. Roy. Soc. Lond. A <u>441</u> (1993) 261 Browning & Pennycook, J. Microsc. <u>180</u> (1995) 230 Nellist & Pennycook, Ultramicroscopy <u>78</u> (1999) 111



## scanning transmission electron microscopy (STEM)

### high-angle annular dark-field imaging or 'Z-contrast'

- HAADF image is formed by using a large annular detector. Regions of the specimen which scatter electrons through high angles (>50mrad) are recorded and appear with bright contrast in the final image.
- In the HAADF image the image intensity increases with specimen thickness and average atomic number (Z) of the specimen. Regions of high Z appear with brighter contrast compared to regions of lower Z. Quantification is possible, because for large angles Rutherford scattering dominates the elastic scattering (E. Rutherford, Philos. Mag. 21 (1911) 669).
- For pure Rutherford scattering the contrast intensity scales with a Z<sup>2</sup> relationship however in practice this may vary and careful extrapolation methods are required (T. Walther, J. Microsc. <u>221</u> (2006) 137-144).
- The contrast in HAADF is generally unaffected by small changes in objective defocus and specimen thickness.

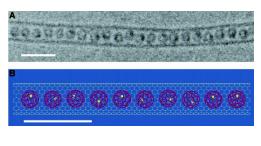


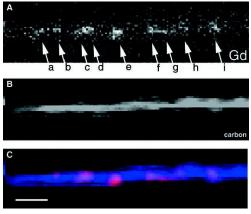
# scanning transmission electron microscopy (STEM)

#### annular dark-field (ADF) imaging

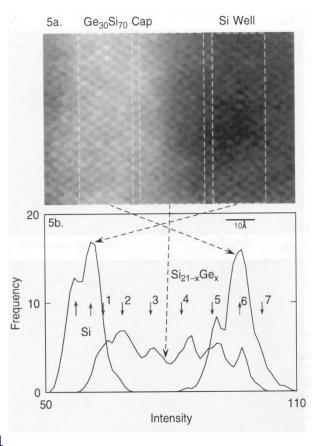
single atom detection of heavy atoms (noble metals etc.) next to light atoms

- U, Au, Pt within large organic molecules on amorphous substrate:
  - Crewe et al., Science <u>168</u> (1970) 1338
- Gd in C82 nano-tubes (mainly STEM-EELS): Suenaga et al., Science 290 (2000) 2280





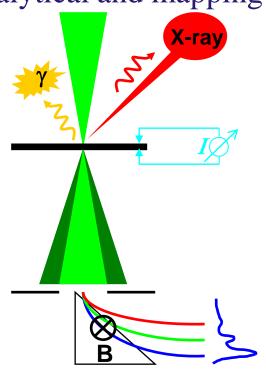
- Au crystallites on a-C support: Batson et al., Nature 418 (2002) 617
- Ge in bulk SiGe: Batson, J. Microsc. <u>180</u> (1995) 204
- Sb dopants in Si: Voyles et al., Ultramic. <u>96</u> (2003) 251





## scanning transmission electron microscopy (STEM)

analytical and mapping capabilities



analytical signals can be collected simultaneously with the **ADF** signal while a focused electron beam scans; [also possible are: secondary, back-scattered or Auger electrons]

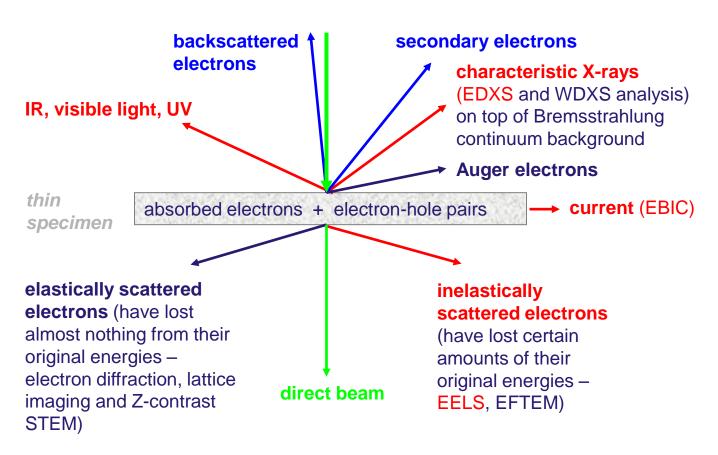
#### examples:

- DLTS, cf: Petroff & Lang, Appl. Phys. Lett. <u>31</u> (1977) 60
- CL, cf: Pennycook & Brown, J. Luminescence 18 (1979) 905
- EBIC, cf: Perreault & Ast, J. Phys. E 21 (1988) 1175
- EDXS, cf: Wood et al., J. Microsc. <u>133</u> (1984)255
- EELS, cf: Browning et al., Nature 336 (1993) 143



## electron-specimen interactions

#### incident high-energy electrons





## electron-specimen interactions

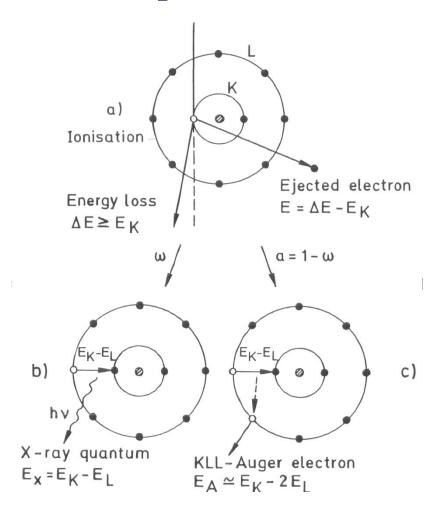
- first: ionisation of atom (excitation as primary effect)
  - The primary electron interacts with bound electrons. By exciting an electron from e.g. the innermost orbital (K-shell) it looses at least the ionisation energy, i.e.  $\Delta E = E_K + \delta E \ge E_K$  and is (slightly) deflected -> **EELS**
- then: de-excitation (secondary effects)
  - **radiative** by emission of an X-ray of discrete energy, e.g.  $E_{\gamma} = E_{K} E_{L}$  with a probability given by X-ray fluorescence yield for transition from K to L-shell -> **EDXS**

#### or

- **non-radiative** by transfer of energy to another (*third*) electron which is then ejected as an Auger electron of energy  $E_A = E_K E_L E_I \approx E_K 2E_L$ , where  $E_I$  is the ionisation energy of this electron in the presence of a vacancy in a subshell, taking relaxation into account
- -> Auger electron emission



## electron-specimen interactions



sketch of EELS (a), EDXS (b) and Auger electron emission (c)

L. Reimer: Transmission Electron Microscopy, 2<sup>nd</sup> ed.(1989), Springer, Berlin, pp. 188-190



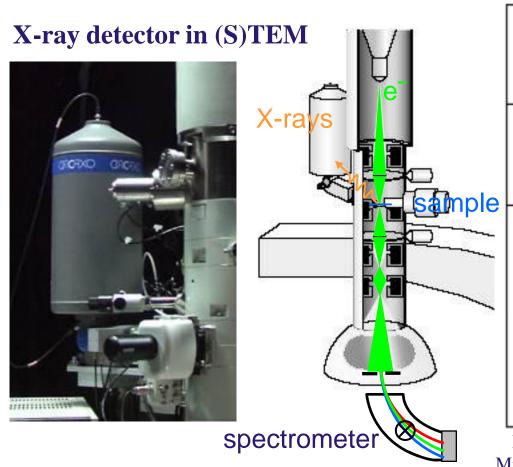
(EDXS)

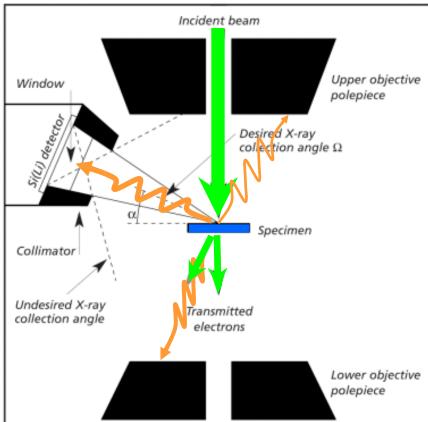
### principles of X-ray generation and detection

- small fraction of X-Rays emitted isotropically by irradiated specimen are collected and analysed
  - X-ray detector provides a spectrum of photon intensity as a function of photon energy (hence the name energy-dispersive)
- Each element produces a series of characteristic X-rays on top of a weak background of Bremsstrahlung
  - EDXS can be used to detect the presence of specific elements within the specimen interaction volume (generally for elements above sodium).
  - qualitative analysis: provides information about which elements are present.
  - As the number of X-ray counts of a specific energy is in first approximation proportional to the amount of the element present it is possible to obtain *quantitative* analysis information about the amount or relative amounts of each element present (after appropriate processing).
  - single spot analysis or generate elemental distribution maps / profiles



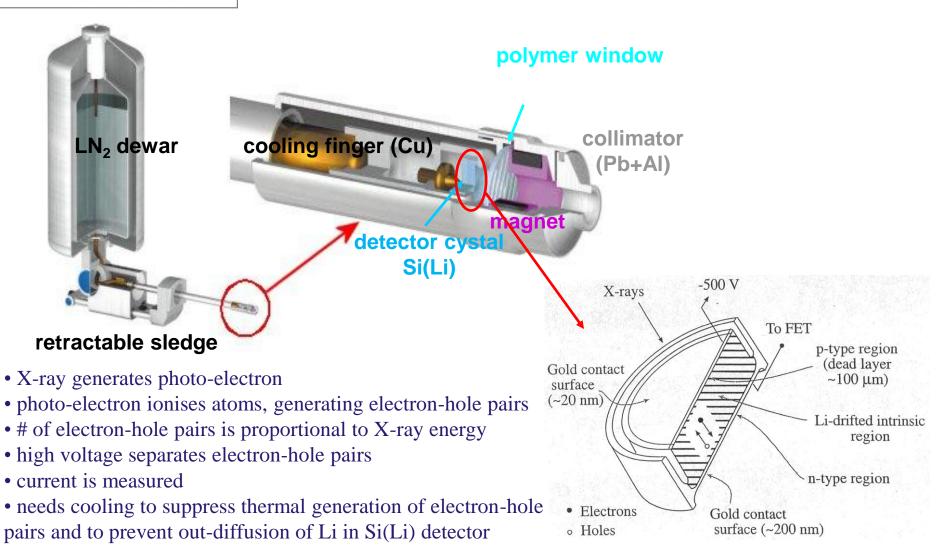
#### principles of X-ray generation and detection





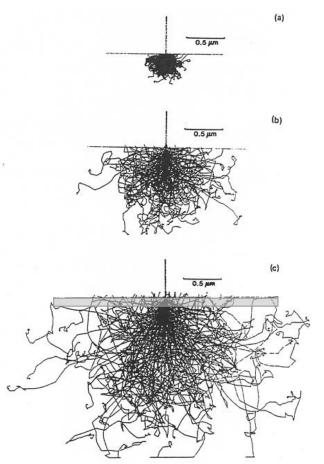
D.B. Williams and C.B. Carter: Transmission Electron Microscopy – IV: Spectrometry, Springer, New York, 1996







#### Simulation of electron beam broadening



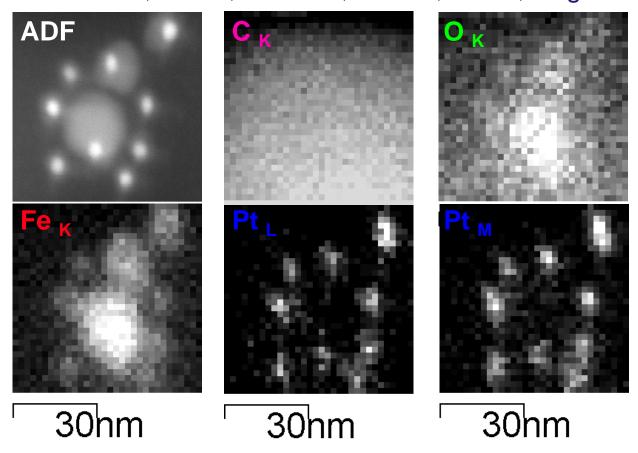
TEM vs. SEM: The spatial resolution of EDX analysis in the SEM is limited to ~1μm due to beam broadening within the interaction volume. The size of the interaction volume can be minimised by using low kV. In the TEM it can reach ~2nm because in a thin foil specimen the pear-shaped interaction volume is cut off.

Random walk ('Monte Carlo') simulations of electron paths taking into account side-wards and inelastic scattering yield estimates of the beam broadening and the backscattered electron yield.

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



**example**: X-ray mapping of 7nm Pt @ Fe<sub>3</sub>O<sub>4</sub> core/shell particles with: 1.1nm FWHM beam, 1.6nA, 26 mrad, 40min., 0.3eV, single scan (!)



T. Walther, unpublished

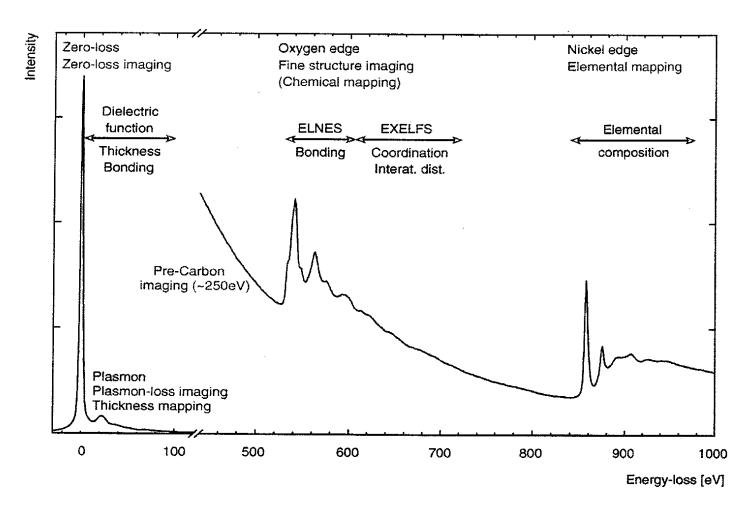


### spectral information

- EELS analyses can be performed in a transmission electron microscope (TEM) with broad beam illumination or a scanning transmission electron microscope (STEM) with a focused electron beam.
- EELS measures the energy loss of electrons within the sample.
- An EELS spectrometer collects energy-loss spectra that can provide highly sensitive information about specimen thickness, elemental composition, valence, bonding and co-ordination.
- An imaging energy filter can in addition produce images or diffraction patterns of a narrow range of energy-losses.

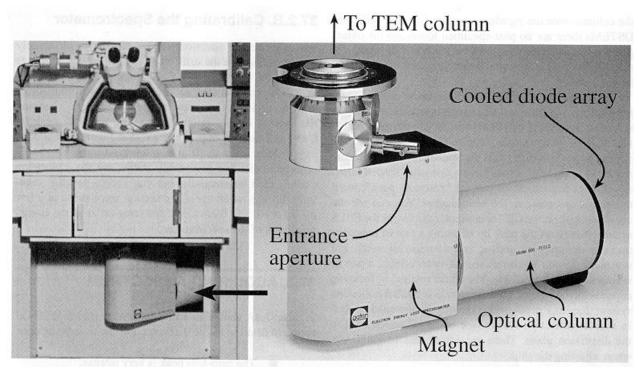


## spectral information





## spectral information spectrometers as energy analysers

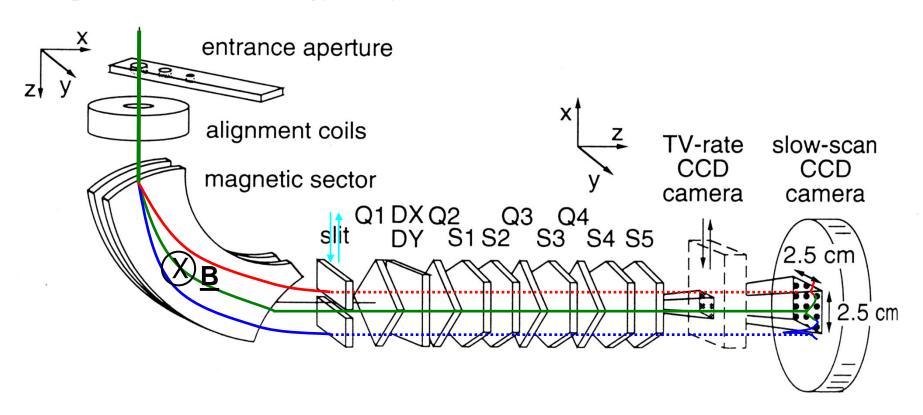


Gatan parallel-detection magnetic prism spectrometer (PEELS)

D.B. Williams and C.B. Carter: Transmission Electron Microscopy – IV: Spectrometry, Springer, New York, 1996



## spectral information spectrometers as energy analysers

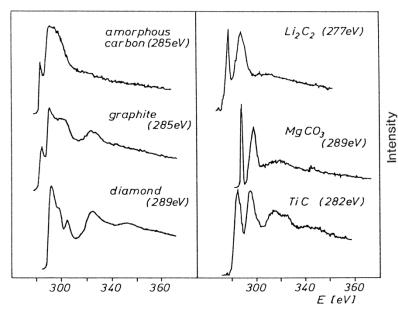


Gatan imaging filter (GIF) with 2D-detector (CCD or TV) to record EEL spectra (slit retracted) or energy-filtered images or diffraction patterns (slit inserted)

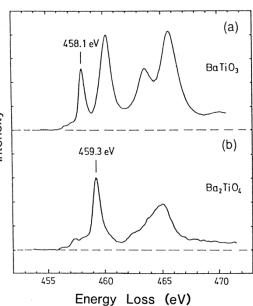


### energy-loss near-edge structure (ELNES)

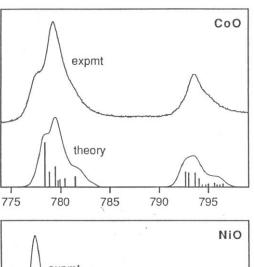
#### carbon compounds

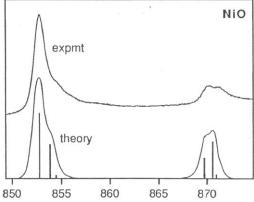


#### titanate









Energy Loss (eV)

bond type, bond symmetry and valence determine both form and position of the ELNES

textbook by Egerton



energy-loss near-edge structure (ELNES)

## chemical shifts due to crystal symmetry and valence

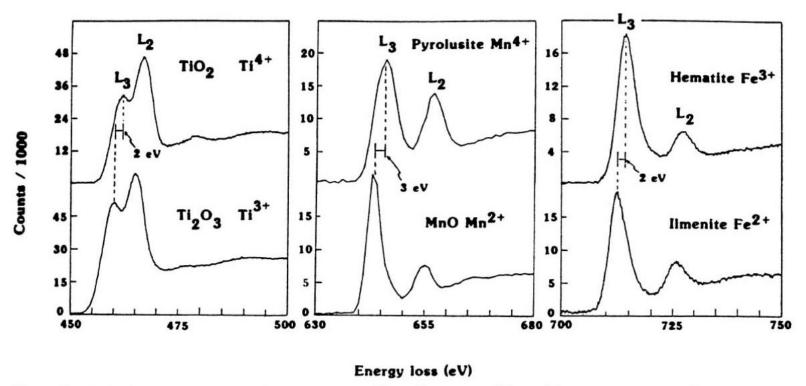


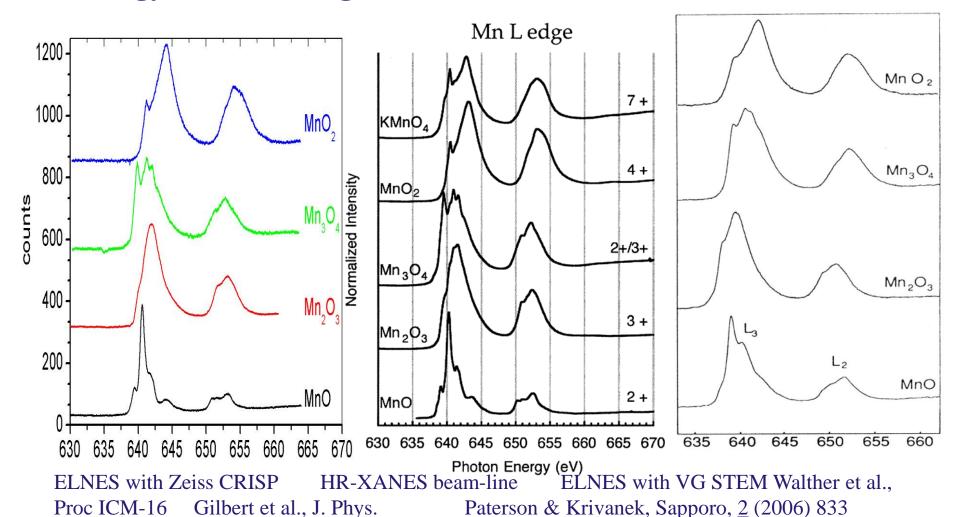
Fig. 3. Chemical shifts as a function of oxidation state for Ti<sup>3+</sup>-Ti<sup>4+</sup> (2 eV), Mn<sup>2+</sup>-Mn<sup>4+</sup> (3 eV) and Fe<sup>2+</sup>-Fe<sup>3+</sup> (2 eV).

M.T. Otten, B. Miner, J.H. Rask and P.R Buseck, Ultramicroscopy <u>18</u> (1985) 285-289



# electron energy-loss spectroscopy (EELS)

#### energy-loss near-edge structure (ELNES)



Ultramic. 32 (1990) 319

EEE 6009 Advanced Instrumentation - Electron Microscopy EM1-3

Chem. A 107 (2003) 2839



#### comparison of EDXS with EELS

#### **EDXS**

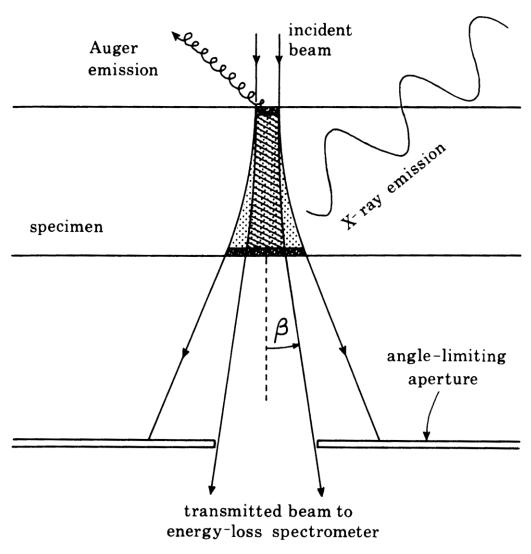
- X-rays provide elemental information only
- inefficient signal generation, collection and detection
- slow technique (minutes to hours for X-ray mapping)
- X-ray spectra can contain artefacts
- good detection efficiency for high
   Z elements
- energy resolution >100eV causes
   frequent overlaps
- spectral processing standardised
- spatial resolution depends on specimen thickness (>1nm)

#### **EELS**

- elemental, chemical, and dielectric information
- very efficient in every respect higher sensitivity for most elements
- fast technique (seconds to minutes for EFTEM or spectrum imaging)
- EELS spectra usually have no such artefacts
- high detection efficiency for low Z elements
  - energy resolution ~1eV (0.15eV with monochromator) gives fewer overlaps
- more complex processing required
- spatial resolution depends on energy loss and collection aperture



#### comparison of EDXS with EELS

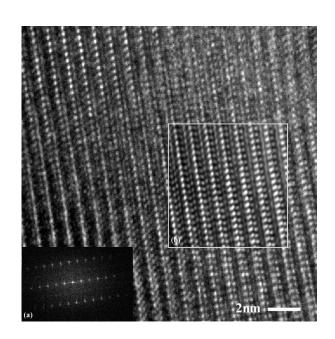


after: S.A. Collett, L.M. Brown and M.H. Jacobs, Proc. Conf. Quant. Microanalysis with High Spatial Resolution, Manchester, The Metals Society, London (1981) pp. 159-164

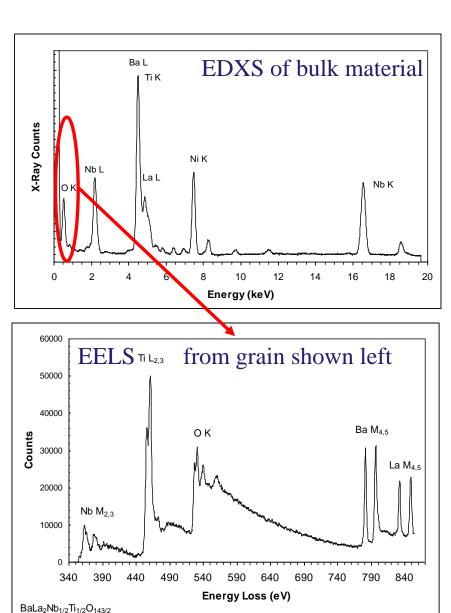


#### comparison of EDXS with EELS

BaLa<sub>2</sub>Nb<sub>1/2</sub>Ti<sub>1/2</sub>O<sub>143/2</sub> electroceramic as an example.



(courtesy of I.M. Ross, University of Sheffield)





#### principle:

use imaging energy filter and slit aperture to select scattered electrons of a certain energy window and use electron energyloss spectrum to interpret the image contrast

- main applications of EFTEM:
  - contrast enhancement

to improve visibility – particularly useful in biological specimens

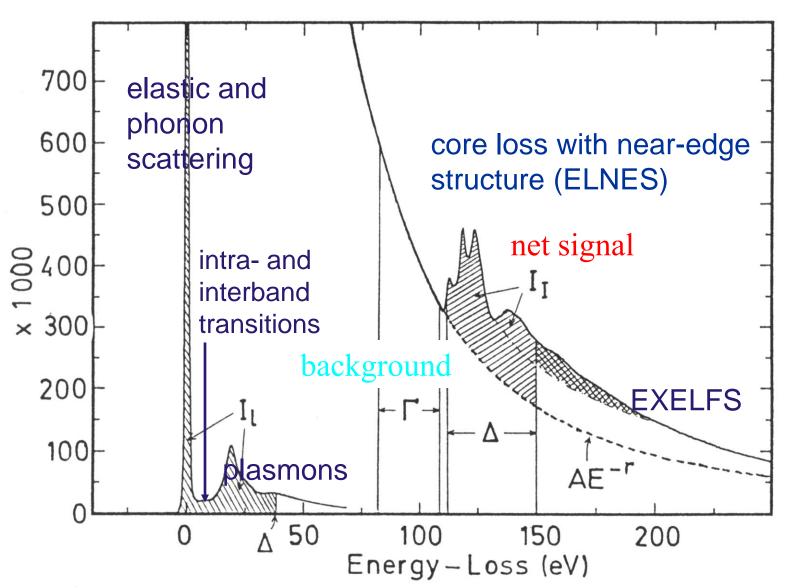
elemental mapping

to investigate the elemental distribution within a specimen

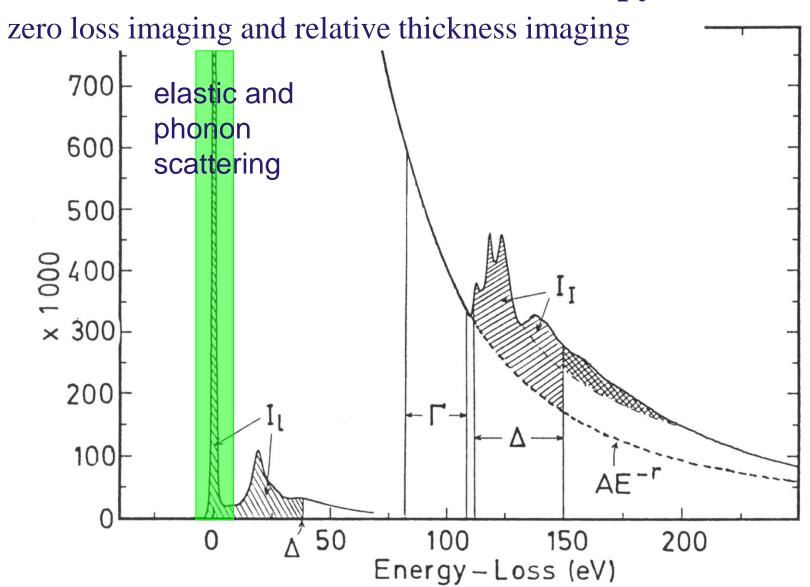
chemical state mapping

to reveal the chemical states within a sample by enhancing the contrast in the image as a function of chemical state







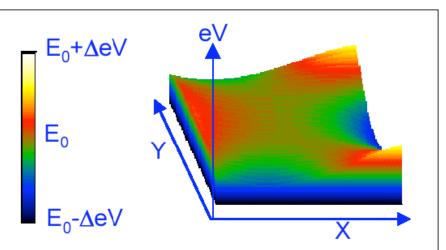




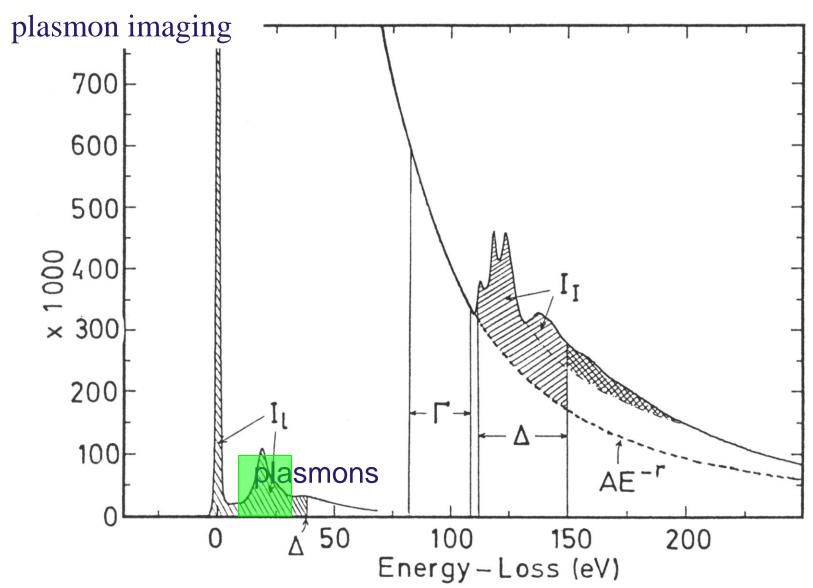
#### zero loss imaging and relative thickness imaging

principle: use small slit aperture to select only elastically scattered electrons around the zero loss peak;
 gives improved contrast (chromatic aberrations reduced);
 can be used to calculate relative thickness map from log of ratio of unfiltered to elastic intensity in terms of inelastic mean free path L:
 t/L= ln (I<sub>total</sub>/I<sub>0</sub>)

limit: non-isochromaticity of filter



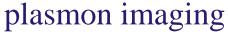


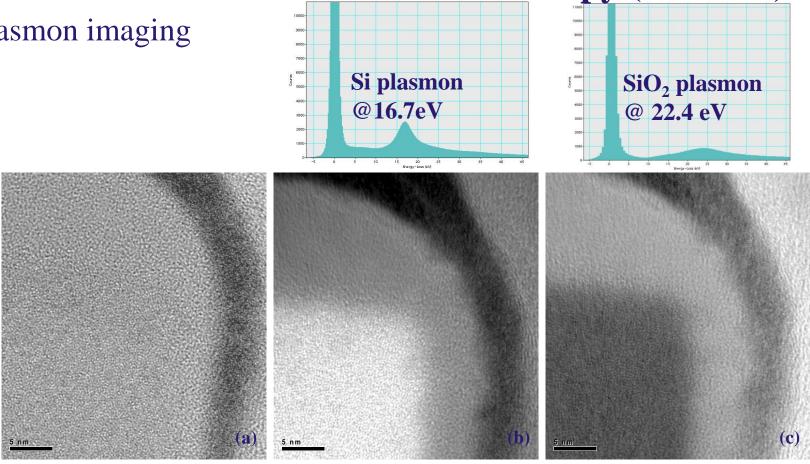




energy-filtered transmission

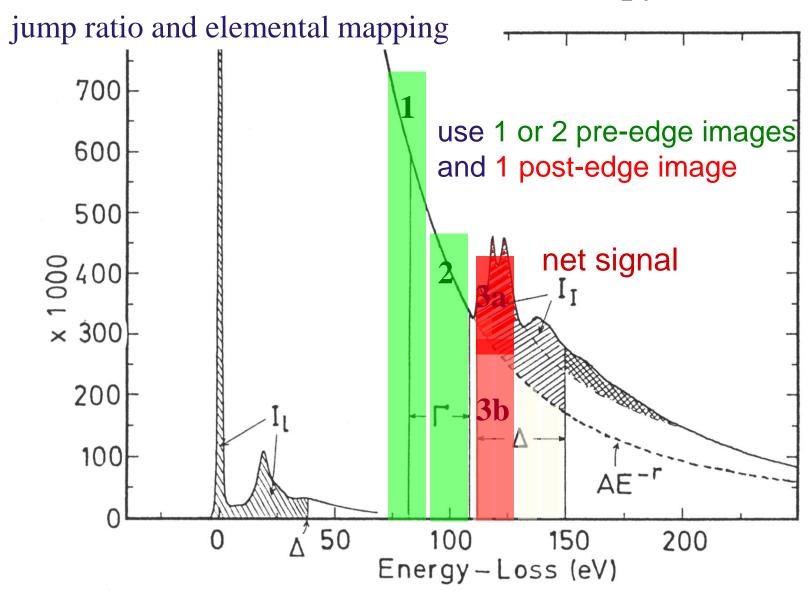
electron microscopy (EFTEM)



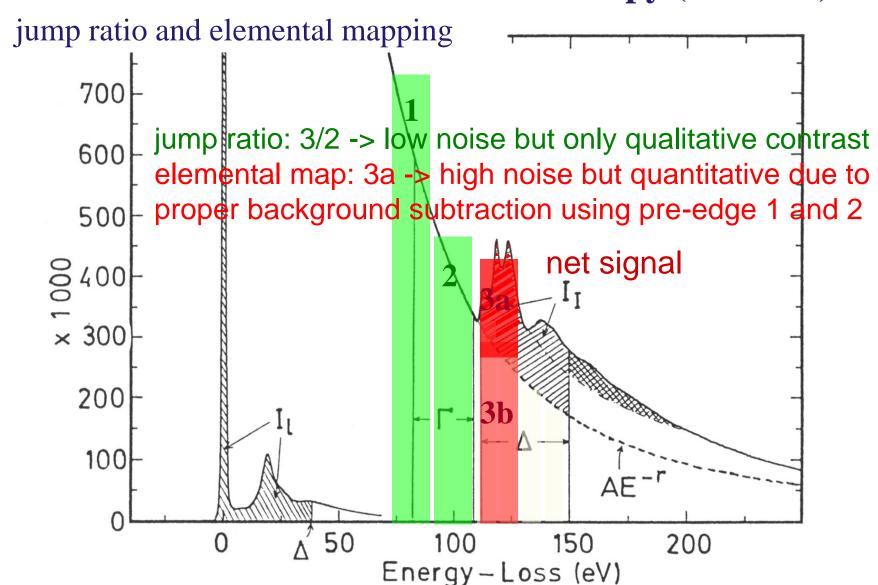


Higher magnification TEM images of the top right-hand corner of the device, (a) bright-field zeroloss image, (b) Si plasmon image and (c) SiO<sub>2</sub> plasmon image highlighting the distinction between the amorphous upper gate electrode region and the amorphous poly re-oxide layer. data courtesy of I.M. Ross, University of Sheffield



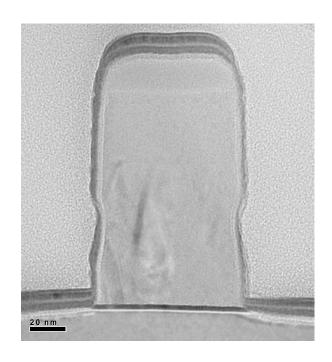




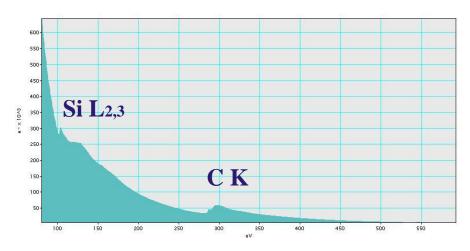


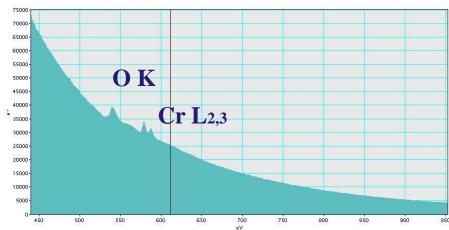


#### jump ratio and elemental mapping



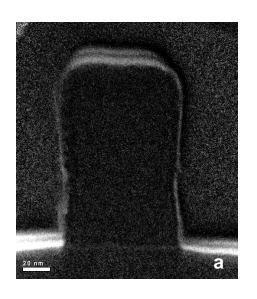
TEM bright-field and energy-filtered TEM (EFTEM) image of a transistor device. Right: corresponding EELS spectra derived from the whole device.

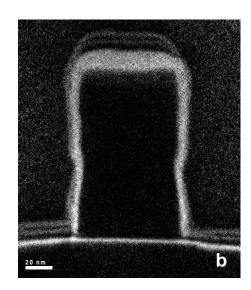


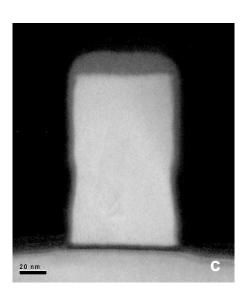




#### jump ratio and elemental mapping







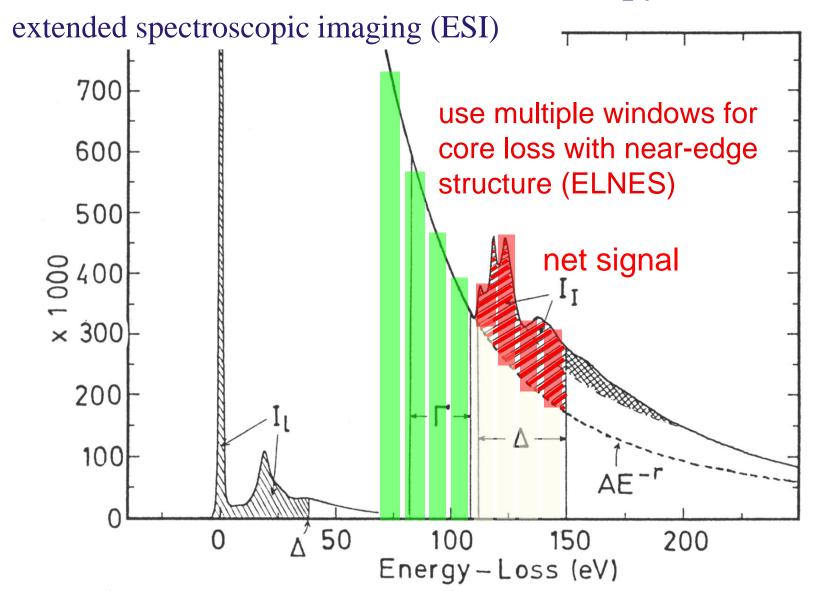
Single stack transistor device: Energy-filtered TEM (EFTEM) images showing **(a)** chromium, **(b)** oxygen and **(c)** silicon distributions respectively.



#### jump ratio and elemental mapping

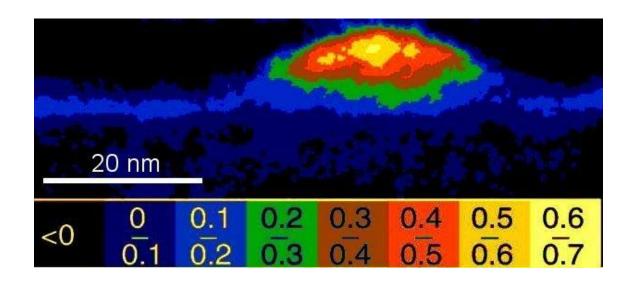
- spatial resolution of inner-shell loss images depends on several factors; ~1nm spatial resolution attainable in the best case:
  - delocalisation of inelastic scattering ionisation of an atom by a fast electron which is some distance away
  - diffraction limit due to objective aperture
  - spherical aberration of the objective lens (Cs)
  - chromatic aberration of the objective lens (Cc)
  - statistical noise due to low inelastic cross sections
  - radiation damage of the specimen
  - instrumental instabilities high voltage drift, sample drift, contamination etc







extended spectroscopic imaging (ESI)



In<sub>x</sub>Ga<sub>1-x</sub>As/GaAs quantum dot nano-analytical image showing indium concentration profile

T. Walther et al., Phys. Rev. Lett. 86 (2001) 2381



#### **Textbooks for further reading!**

- R.F. Egerton: Electron Energy-Loss Spectroscopy in the Electron Microscope, 2<sup>nd</sup> ed., Plenum, New York, (1996): the definitive EELS reference handbook for physicists, materials scientists and chemists
- J.I. Goldstein, H. Yakowitz, D.E. Newbury, E. Lifshin, J.W. Colby, J.R. Coleman, R.B. Bolon and M.F. Ciccarelli: Practical Scanning Electron Microscopy: Electron and Ion Microprobe Analysis, Plenum, New York (1975): good overview over SEM
- P.B. Hirsch, A. Howie, R.B. Nicholson, D.W. Pashley and M.J Whelan: Electron Microscopy of Thin Crystals, Butterworths, London (1965): comprehensive treatment of diffraction contrast in TEM
- D.C. Joy: Monte Carlo Modeling for Electron Microscopy and Microanalysis, Oxford University Press, New York and Oxford (1995): detailed models and program descriptions for BSE and EDXS analysis in SEM and STEM
- R.J. Keyse, A.J. Garratt-Reed, P.J. Goodhew and G.W. Lorimer: Introduction to Scanning Transmission Electron Microscopy, Bios Scientific, Oxford (1998): brief introduction to STEM for engineers
- L. Reimer: Transmission Electron Microscopy; Physics of Image Formation and Microanalysis, 2<sup>nd</sup> ed., Springer, Berlin (1989): a rather complicated but precise and reliable textbook for physicists
- J.C. H. Spence: Experimental High-Resolution Electron Microscopy, 2<sup>nd</sup> ed., Oxford University Press, New York (1988): good handbook for HREM
- D.B. Williams and C.B. Carter: Transmission Electron Microscopy, Springer, New York (1996): a very good general textbook (paperback in four volumes!)

Goodhew PJ and Humphreys FJ, Electron Microscopy and Analysis, 1988, Taylor and Francis, London.