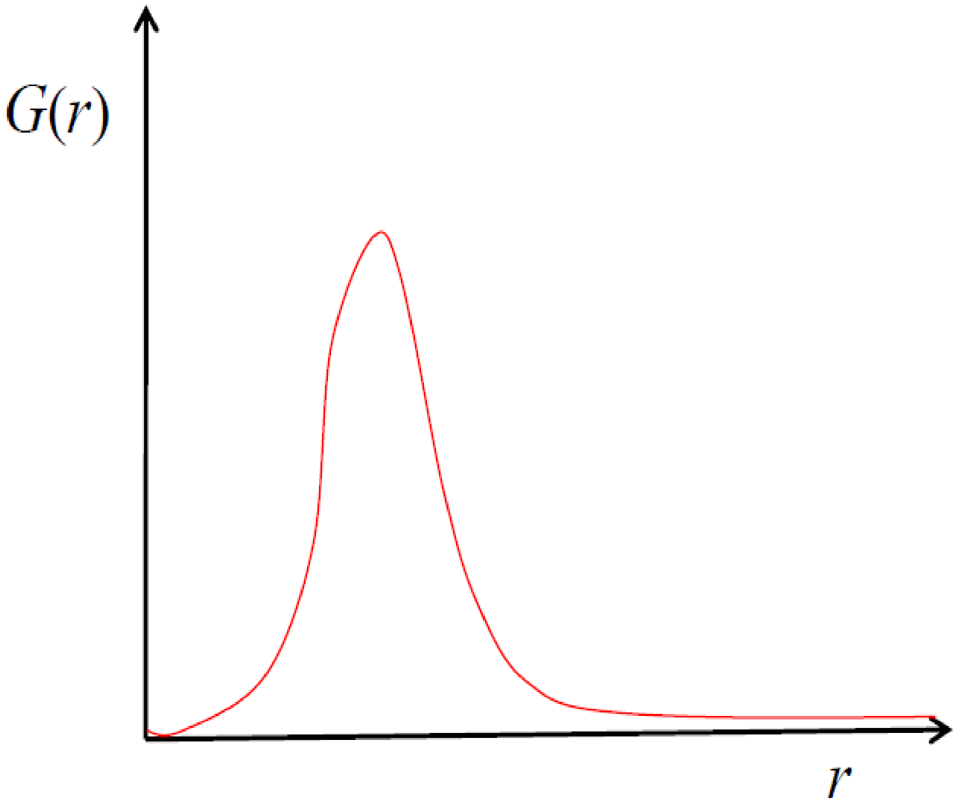
# Neutrons and X-rays

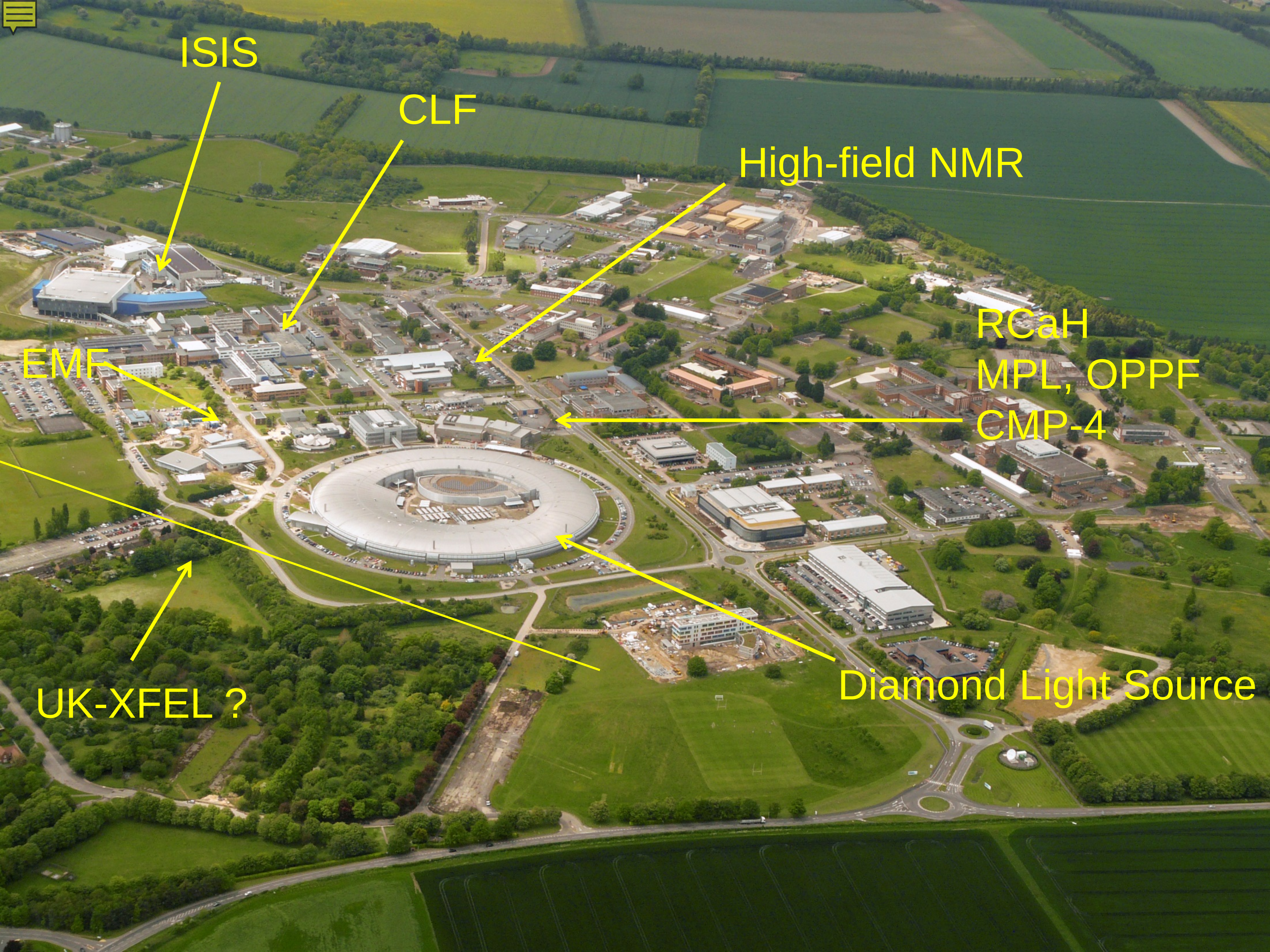
Andrew Harrison, Diamond Light Source

15th Oxford School on Neutron Scattering - 2017

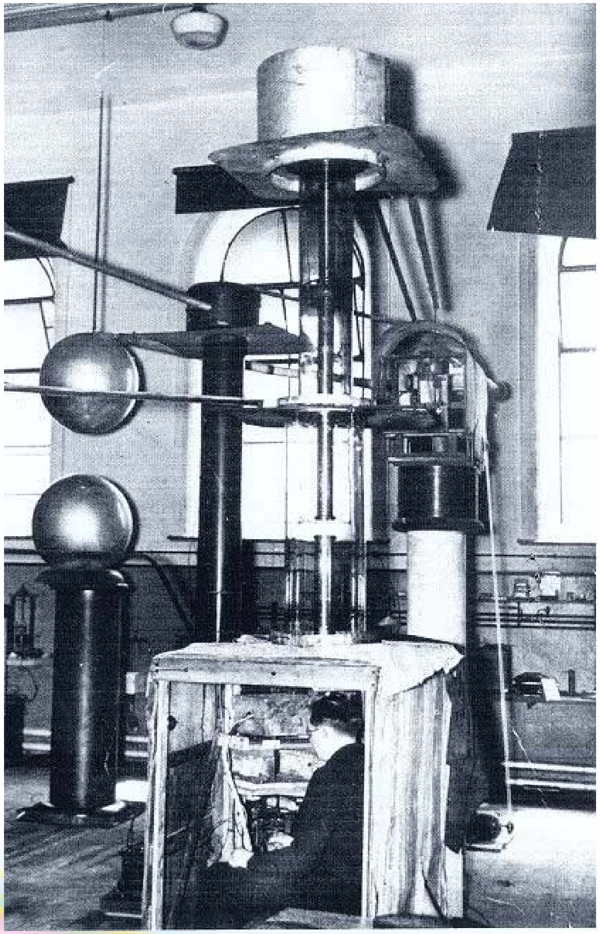
# Neutron source – synchrotron distribution

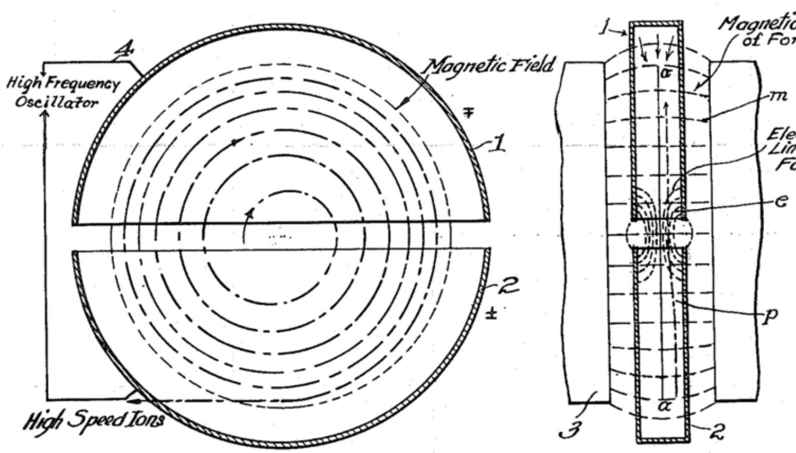


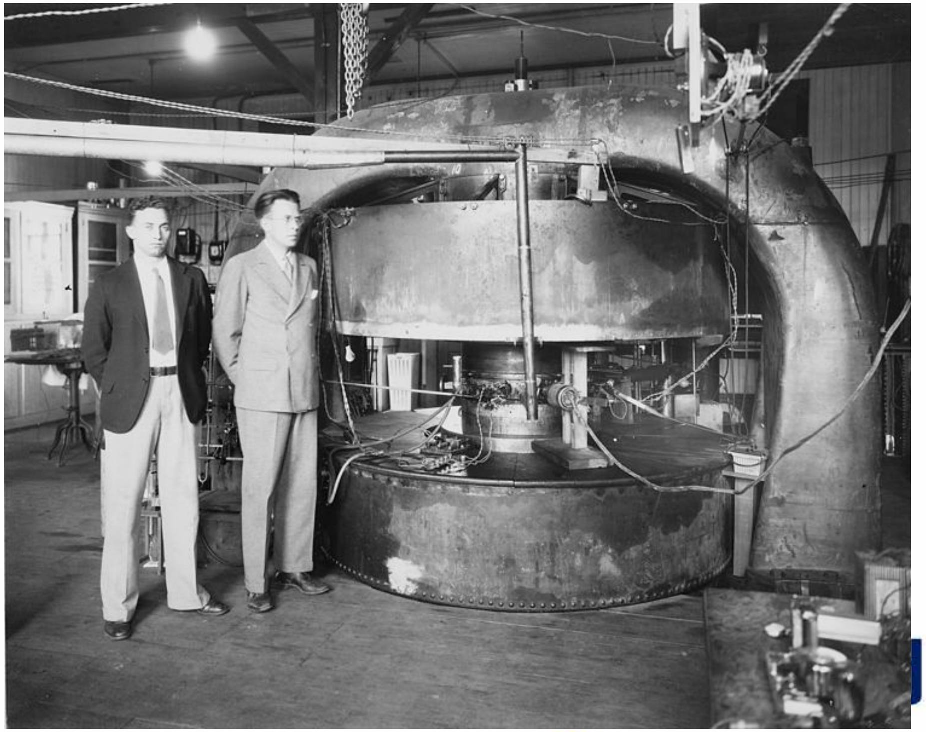

*Acknowledgement: Jon Goff*


ISIS
CLF
High-field NMR
EMF
RCaH
MPL, OPPF
CMP-4
Diamond Light Source
UK-XFEL ?

# Origins





# Ever increasing circles

# Ever increasing circles

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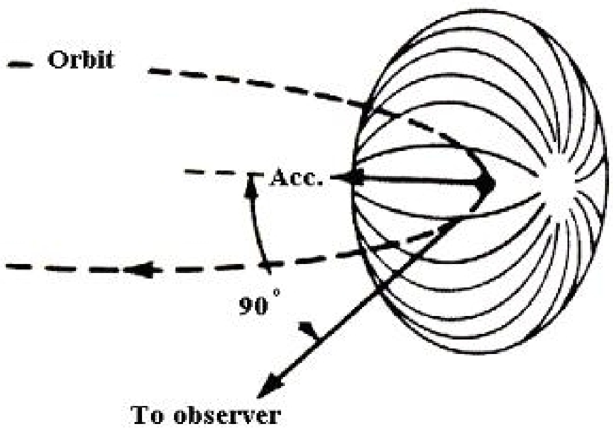
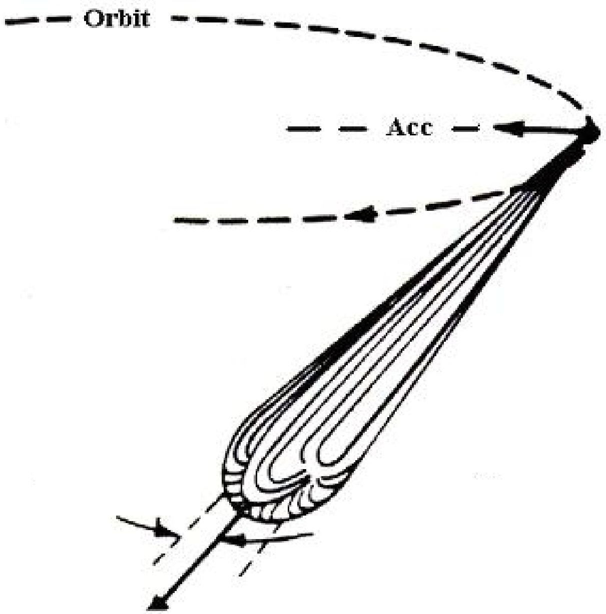
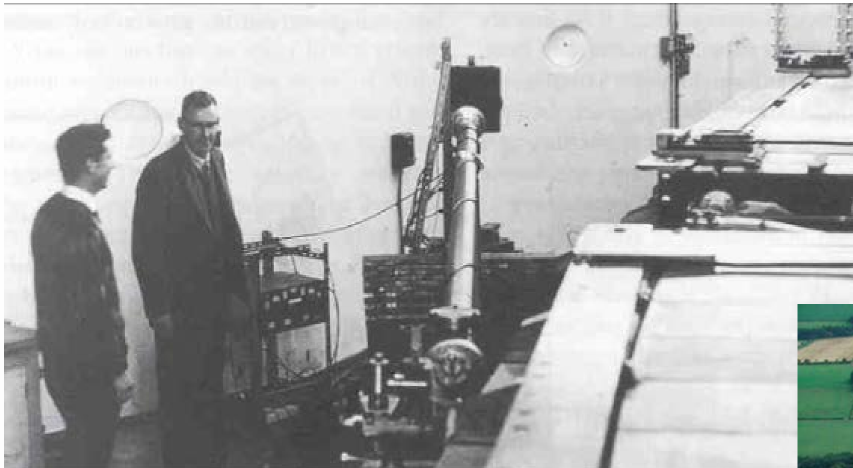
# From Maxwell to synchrotrons

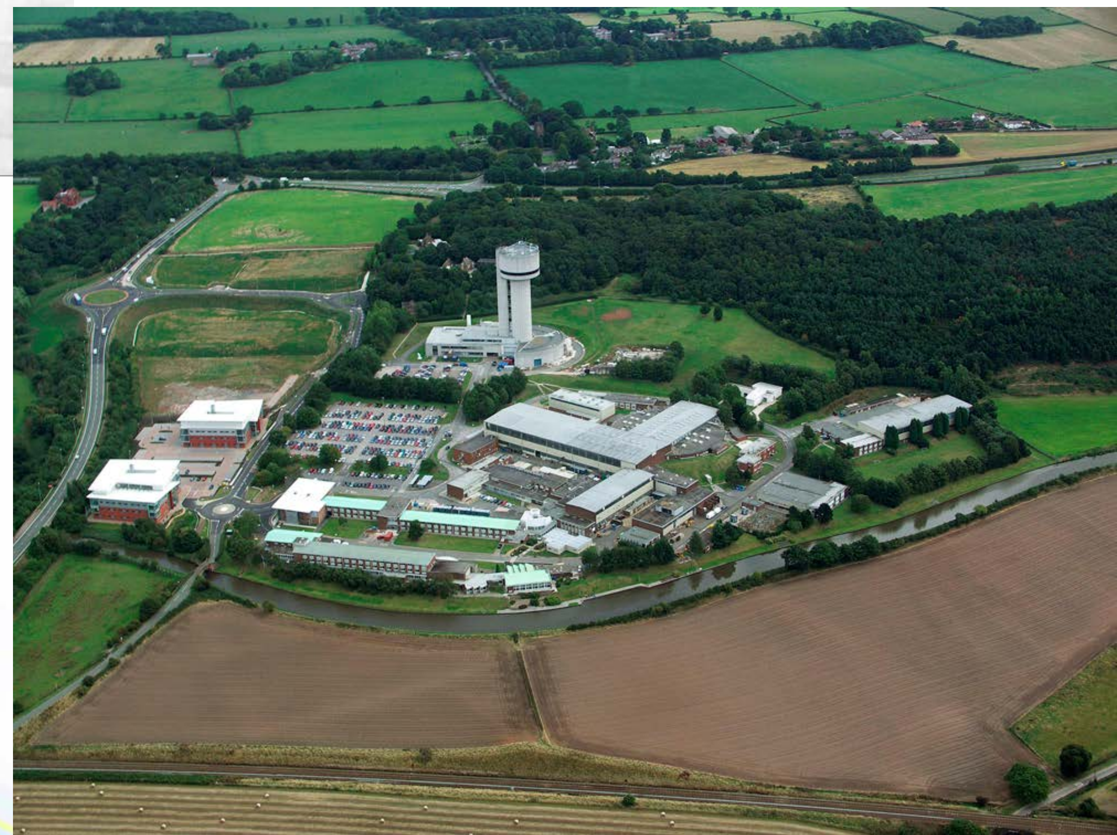


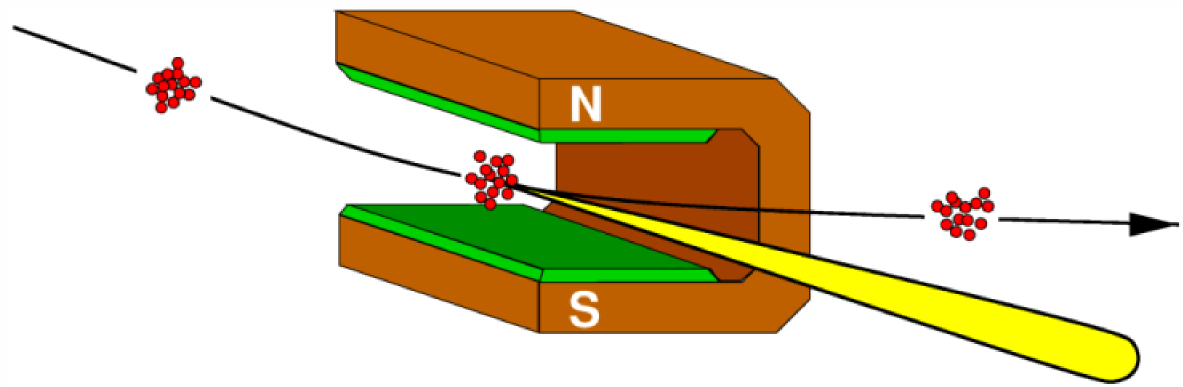

$v \sim c$

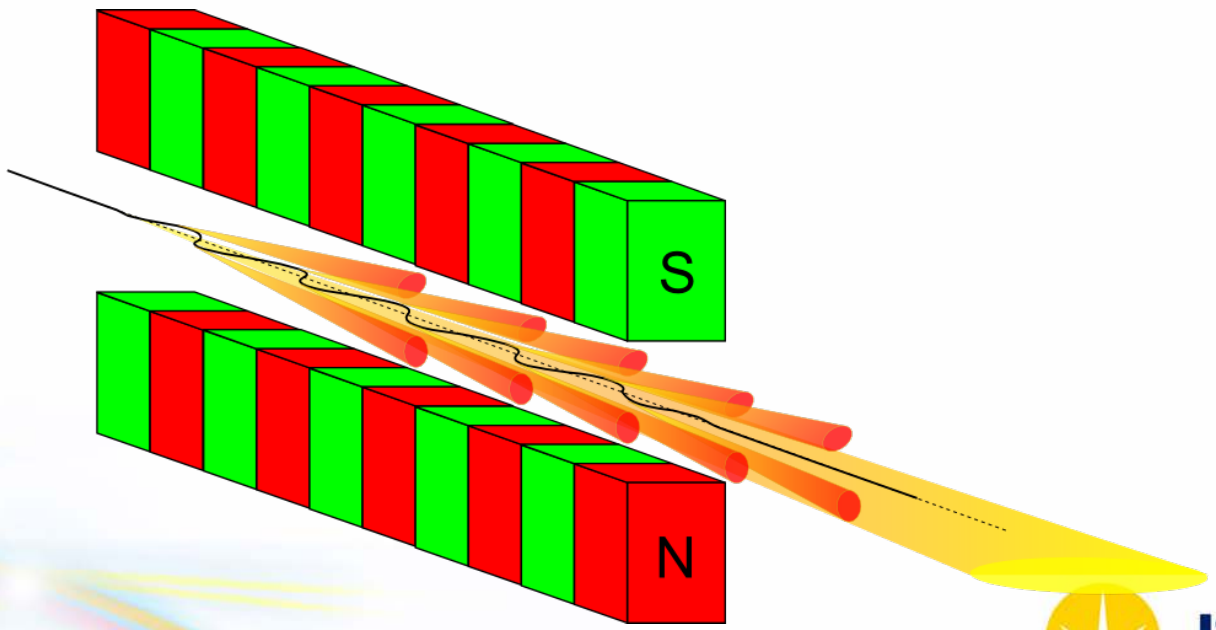
# Jumping a generation (2nd incarnation)

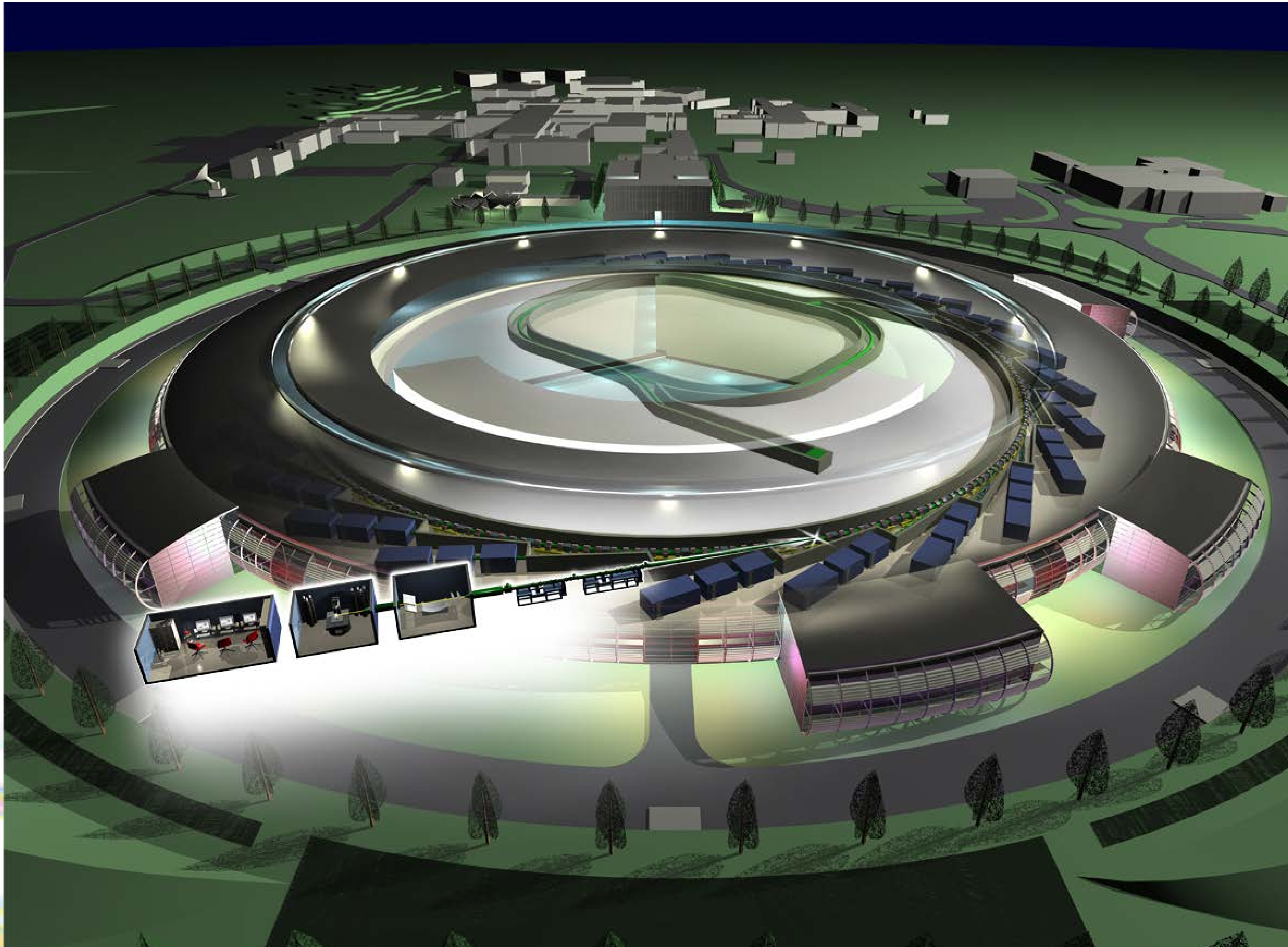




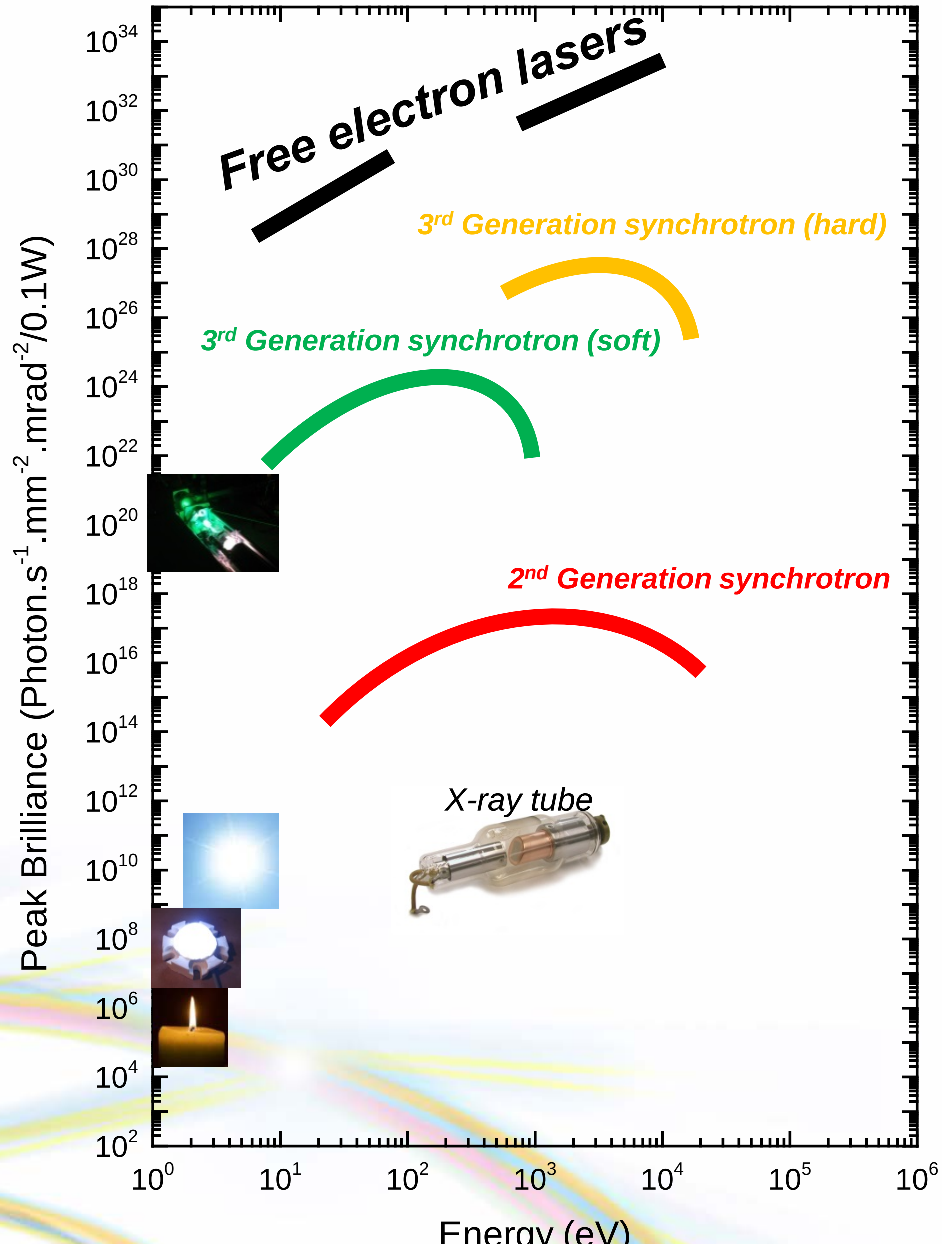
# 2nd and 3rd generation sources





# Not just ordinary light...

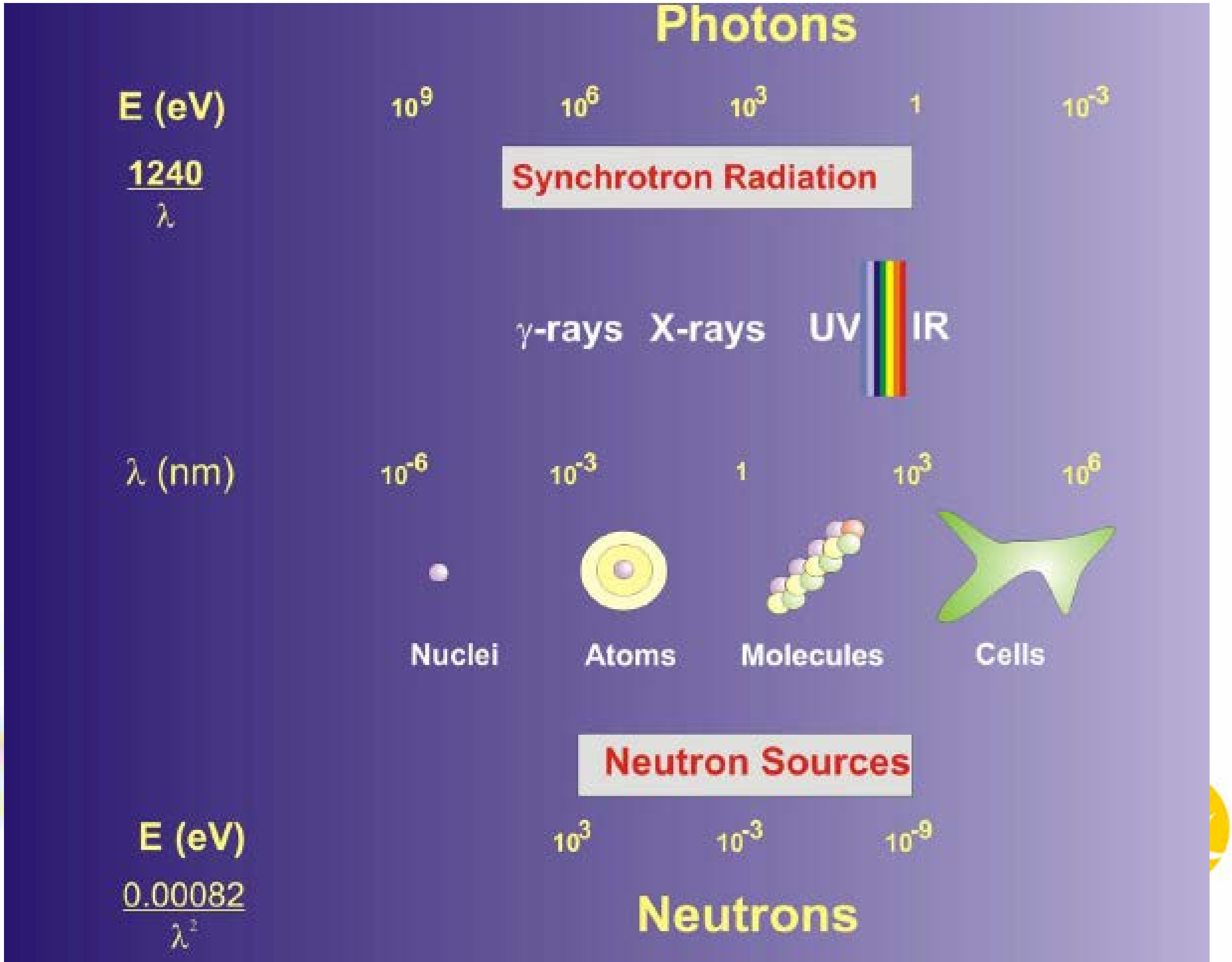



- Brilliant
- Tuneable energy – from IR to X-ray
- Focussing without loss of intensity – 100 mm to 10 nm
- High degree of coherence

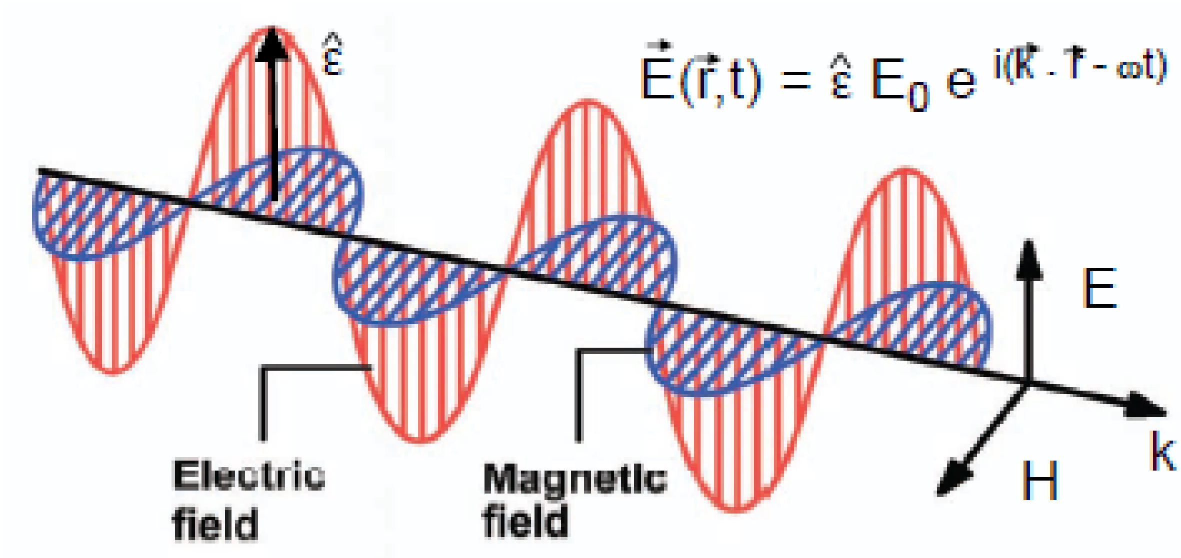
diamond

# Properties of neutrons and X-rays

| | Neutron | X-ray | |
|---|---|---|---|
| **Charge** | 0 | 0 | |
| **Mass/kg** | $1.675 \times 10^{-27}$ | 0 | |
| **Spin** | ½ | 1 | |
| **Magnetic moment** | $-1.913\mu_N$ | 0 | (Nuclear magneton $\mu_N \cong 5.051 \times 10^{-27}$ J/T) |



# EM wave characteristics




| | |
|---|---|
| **Wavelength** | $\lambda$ |
| **Wavenumber** | $k=2\pi/\lambda$ |
| **Frequency** | $\nu=\omega/2\pi$ |
| **Polarisation** | $\hat{\varepsilon}$ |
| **Energy** | $h\nu = \hbar\omega = 12.398$ [keV]/ $\lambda$[Å] |

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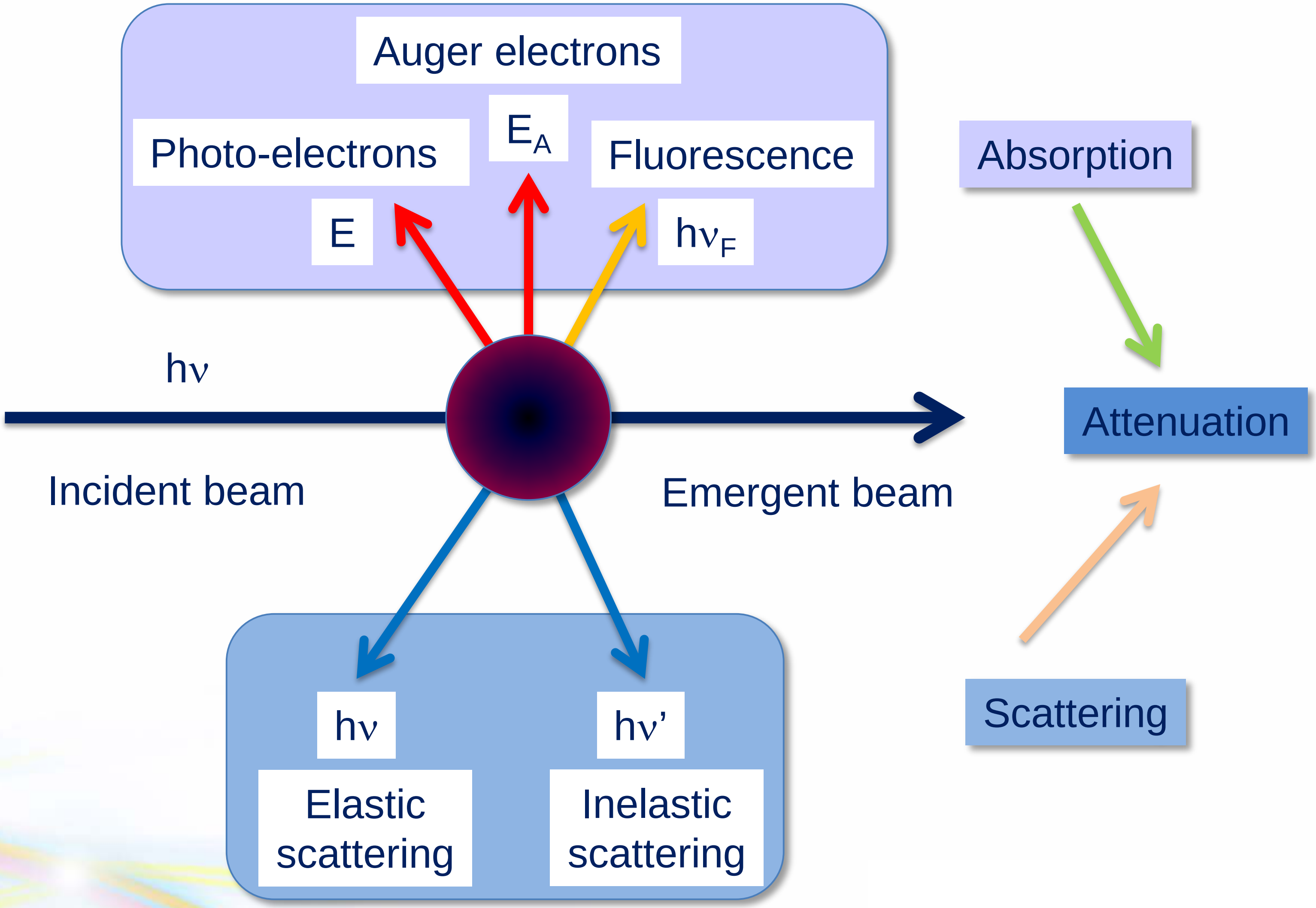
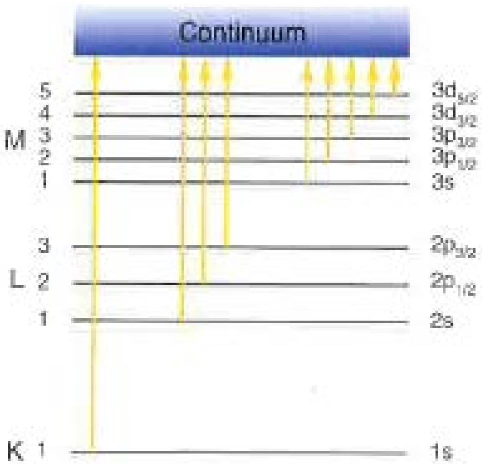
# X-ray – matter interactions



# X-ray absorption/emission processes



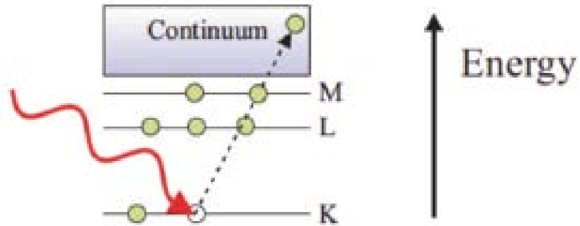
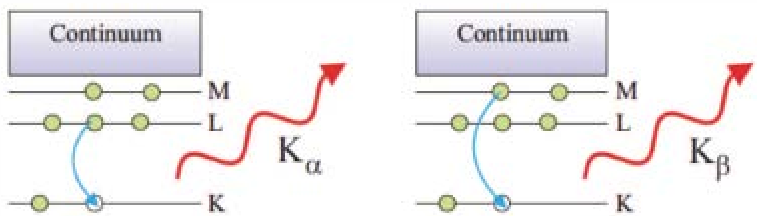
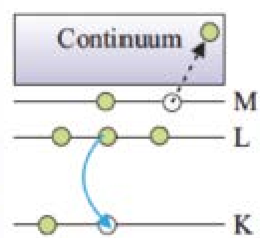



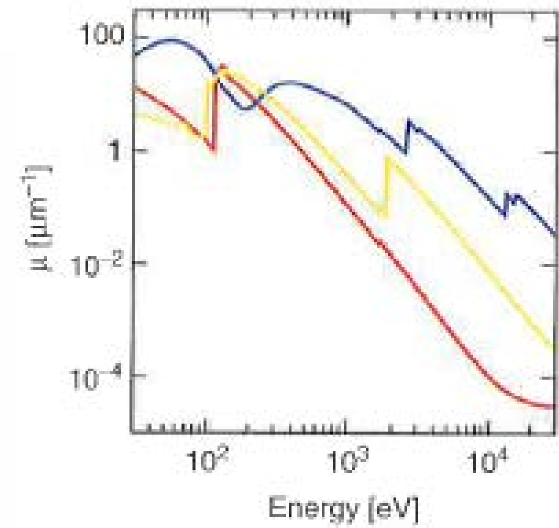

**Photo-electric absorption**

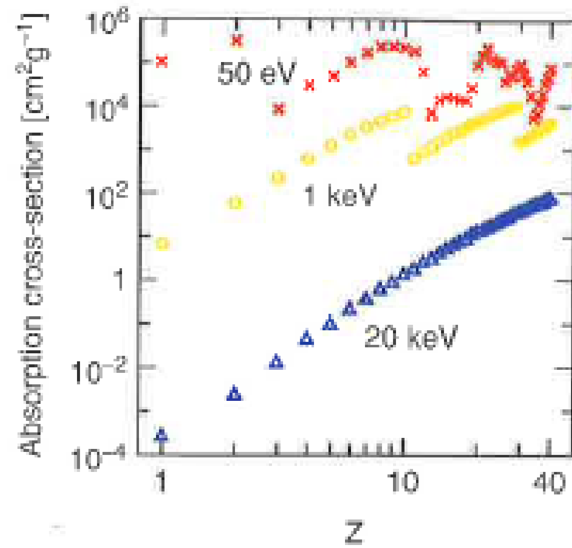



**Fluorescent emission**



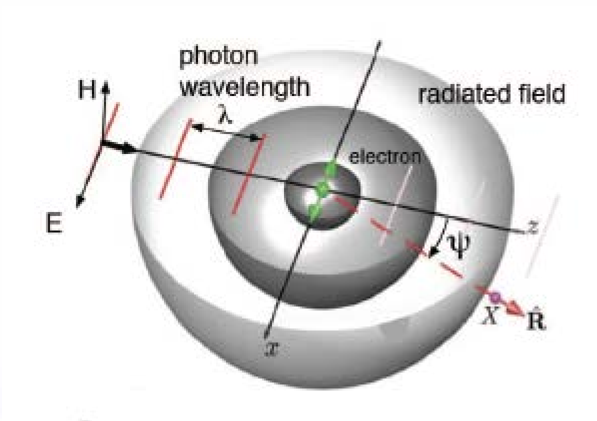

**Auger electron emission**

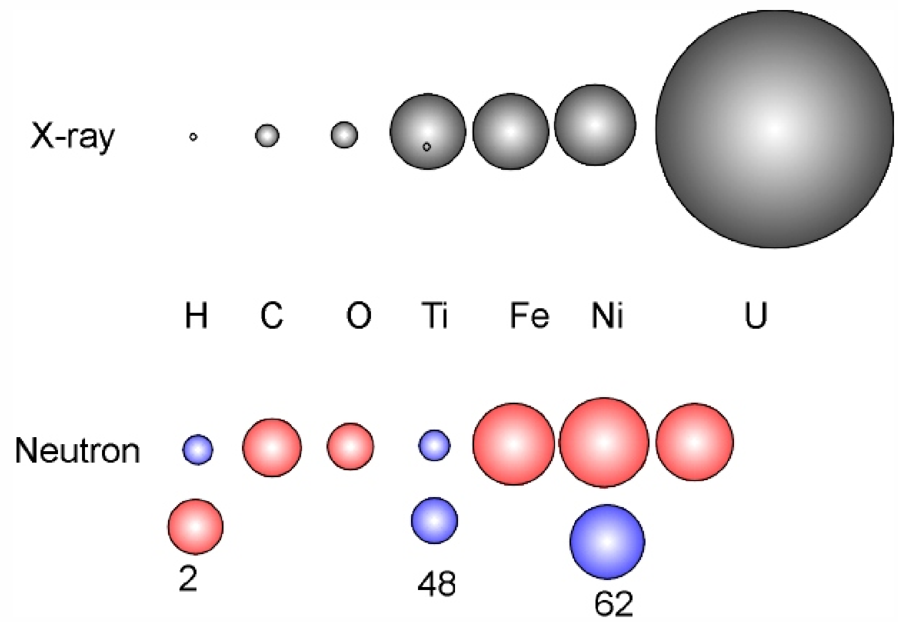


# X-ray scattering - elastic

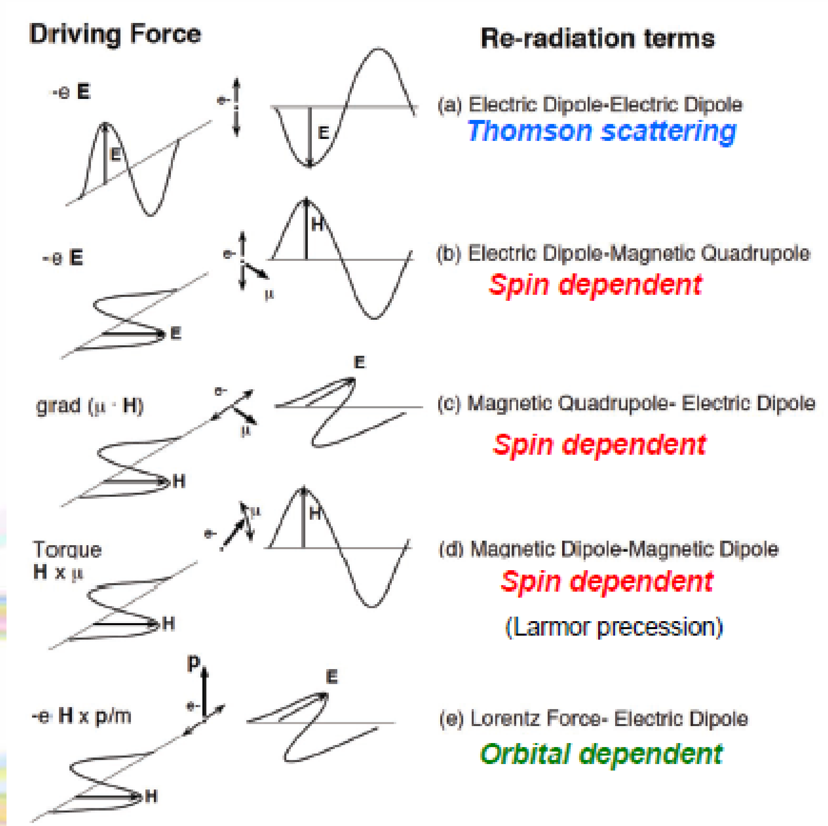
- Leading term is the (Thomson) scattering by electrons
  - The electric field of the incident EM wave drives motion of the electrons and produces spherical waves of the same energy which may interfere to produce a diffraction pattern
  - Elastic scattering intensity is proportional to $Z^2$

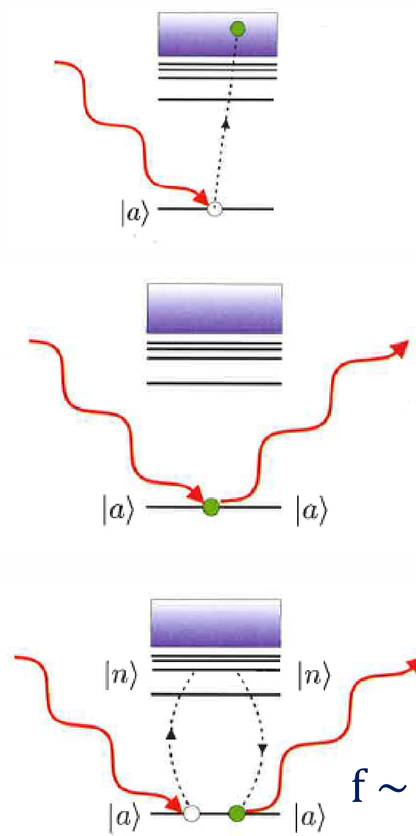


# X-ray scattering - elastic

- Secondary terms are weaker
  - Various (non-resonant) magnetic terms $10^5$ - $10^7$ x weaker
  - If the energy of the incident photons is tuned to resonate with (virtual) transitions to intermediate states very strong amplification of the photon scattering process may occur (see later for RIXS too)




Absorption

Non-resonant scattering

Resonant scattering

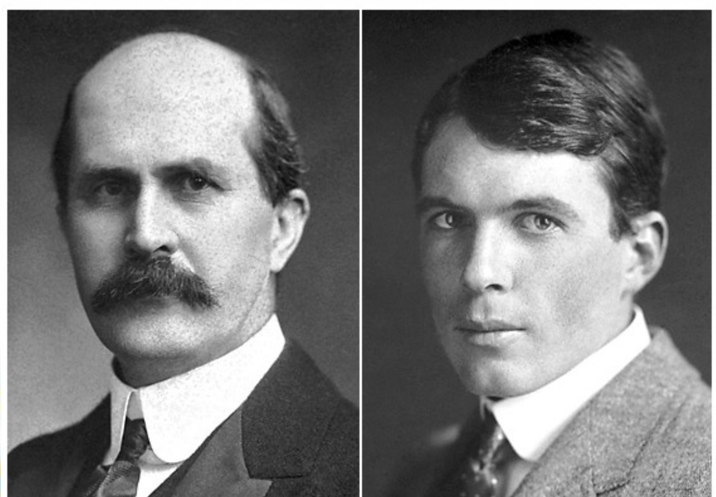
er:

$$f \sim \sum_n \frac{1}{E_n - E_a - \hbar\omega - i\Gamma/}$$

# Diversity - imaging, diffraction, spectroscopy

Wilhelm Röntgen

Max von Laue



William and Lawrence Bragg

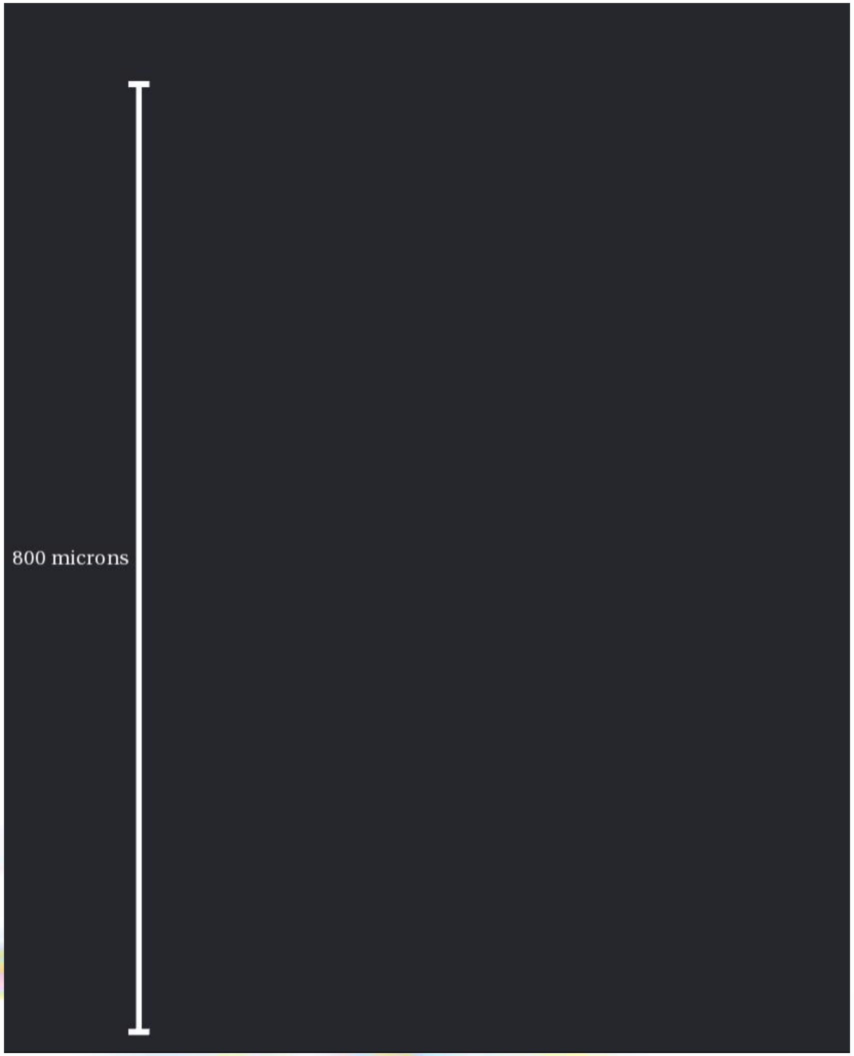
Henry Moseley

diamond

# Origins of imaging

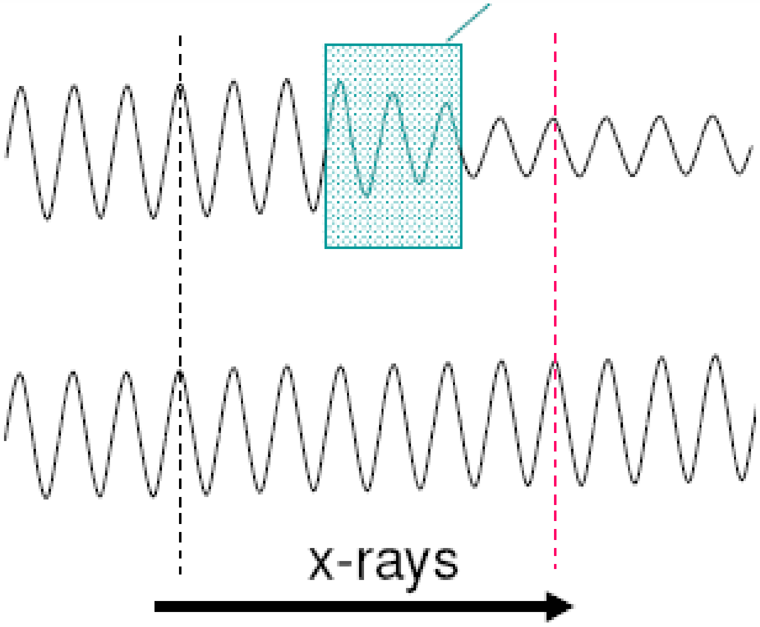
*I have seen my death!*
*(Anna Roentgen, 1900)*

# Finer, faster, three dimensional …




# ...more subtle

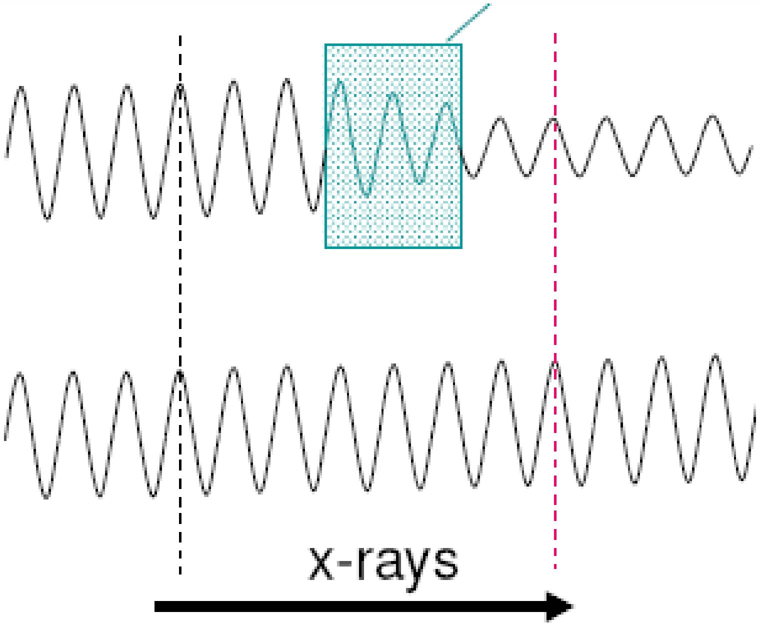
Contrast very poor
if absorption
contrast low

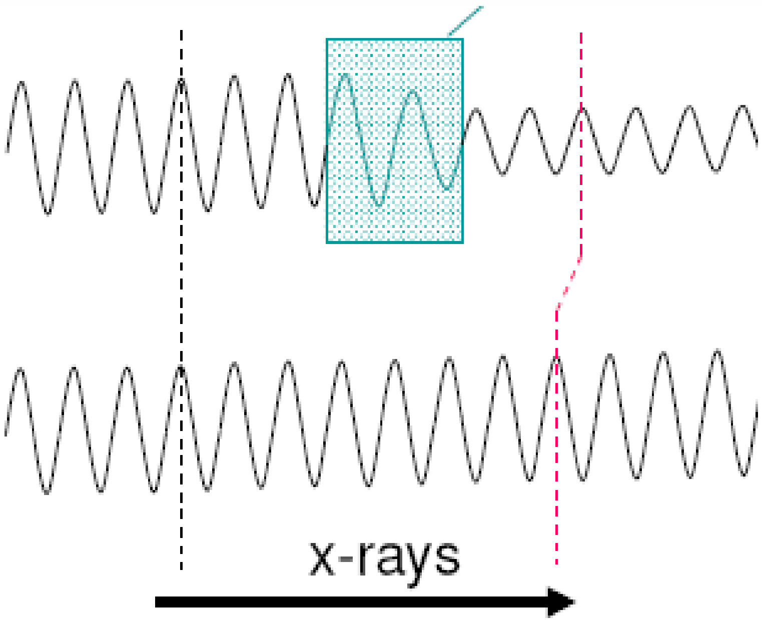



# ...more subtle

Contrast very poor if absorption contrast low

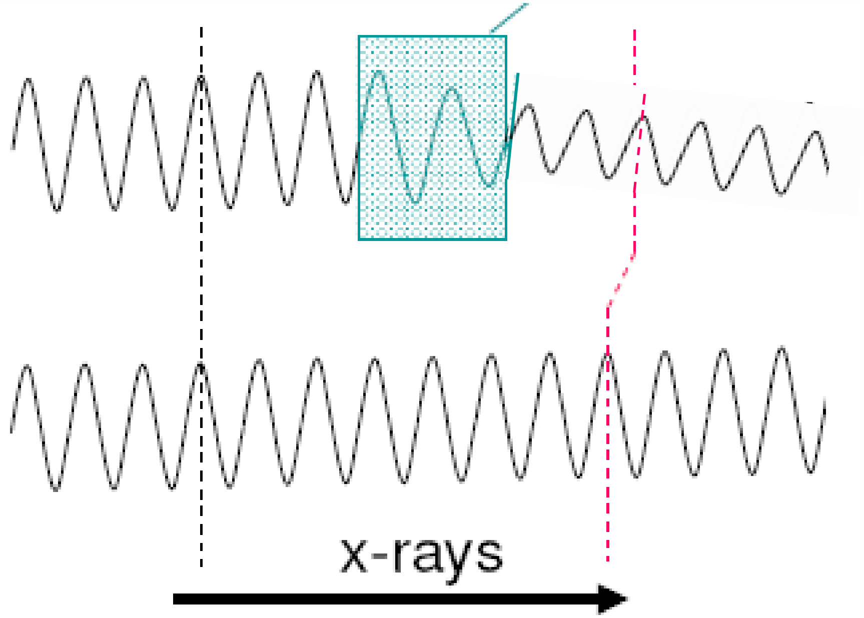
If refractive index different, phase shift occurs



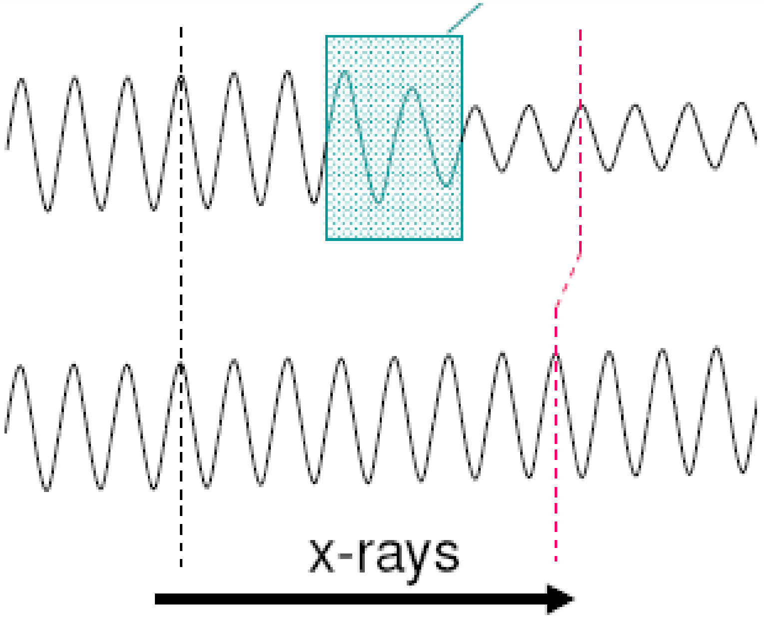



# ...more subtle

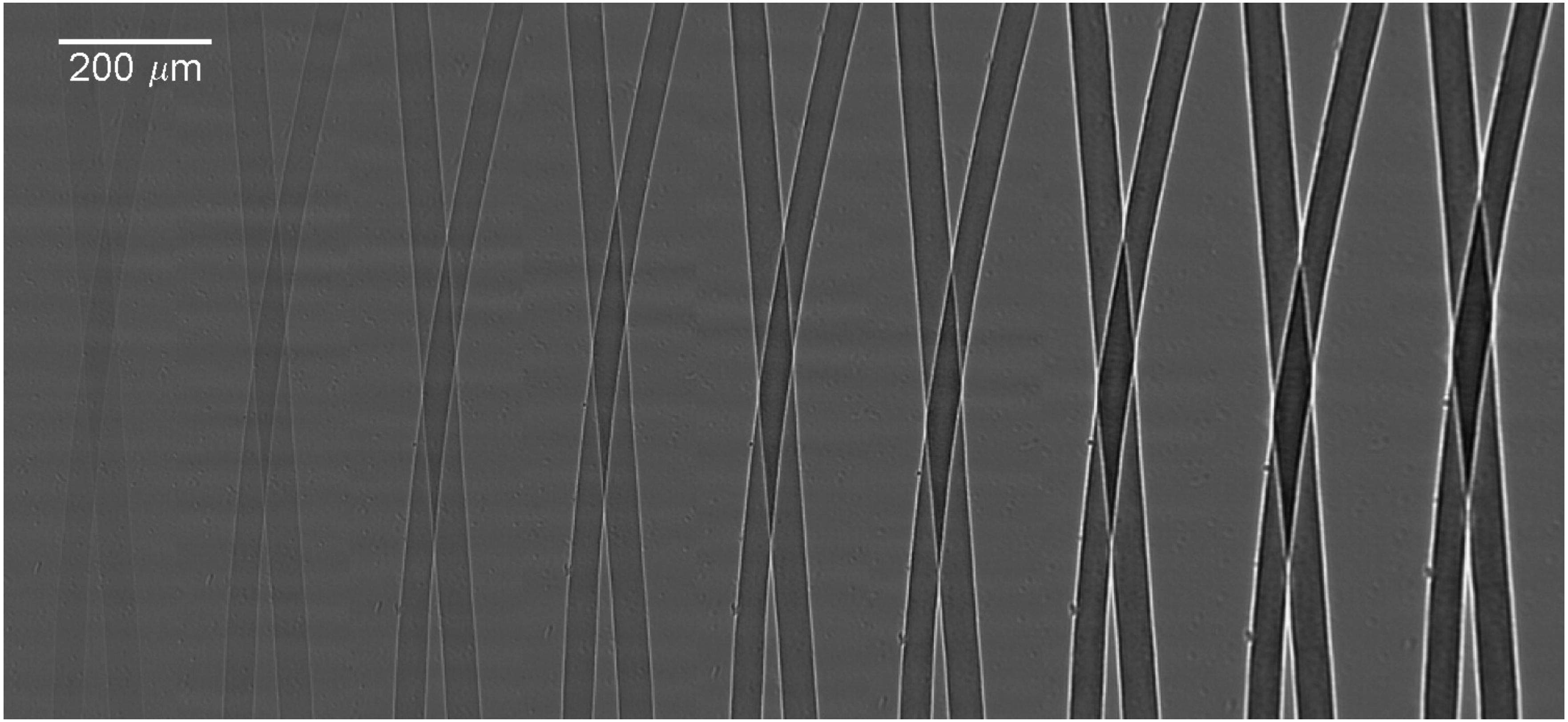
Some diffraction may also occur




If refractive index different, phase shift occurs



# ...more subtle

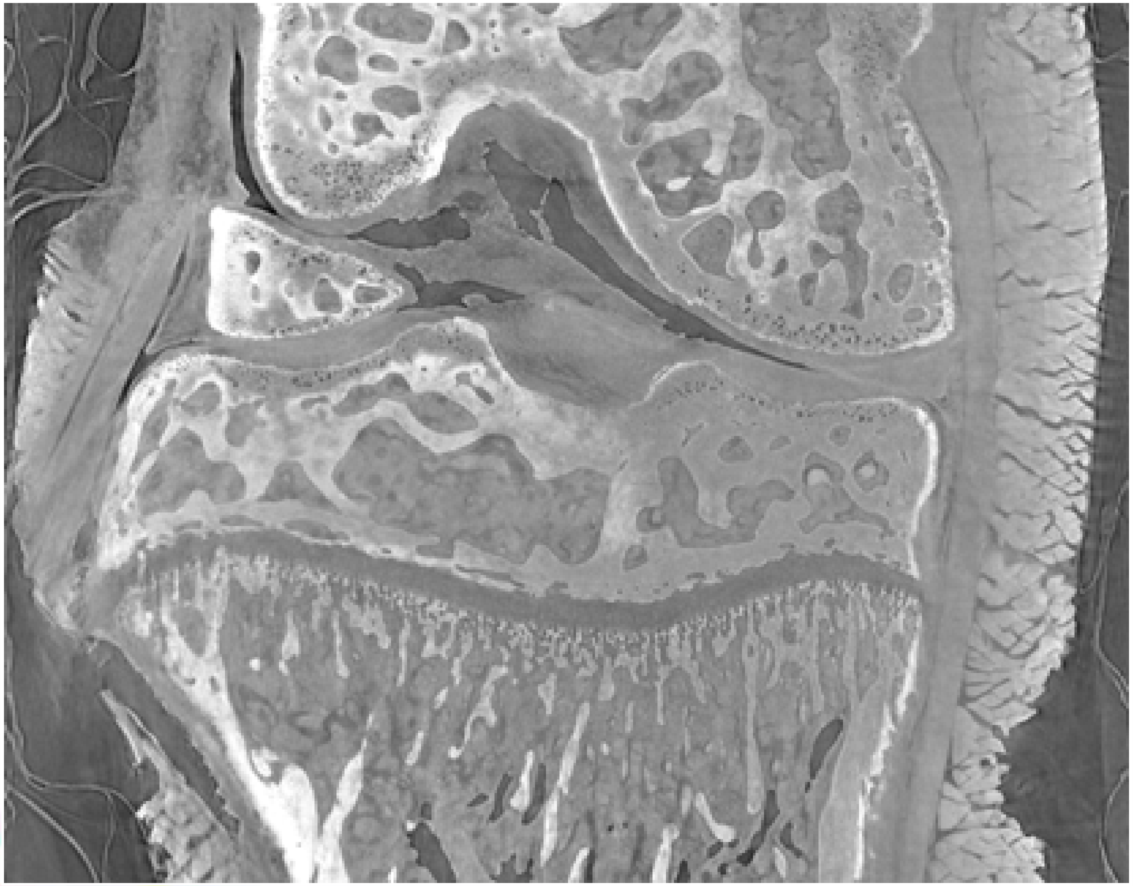



20mm — Distance → 1140mm

E= 20keV

From: A. Rack et al. NIM A, 586 (2), 327-344, (2008).

# Low Z, soft tissue



*Reconstructed slice of mouse knee*

*Results courtesy K. Maadi*

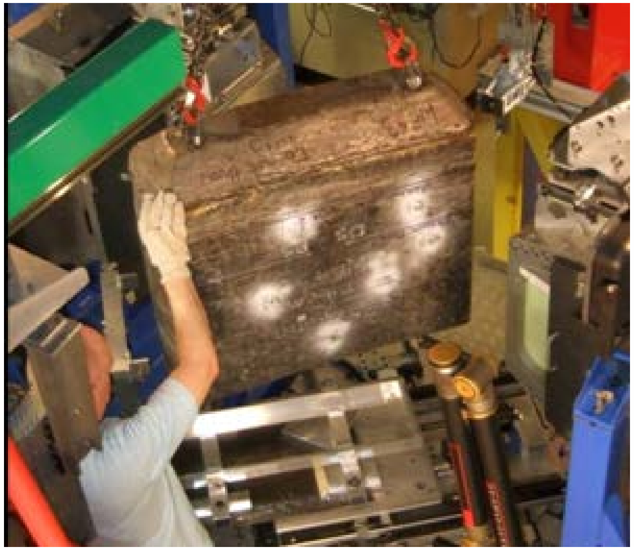
*Image processed with support of A. Bodey*

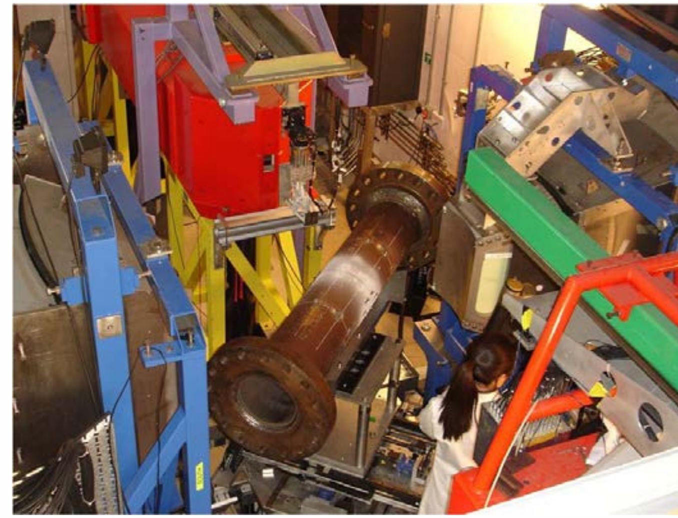
# Complementarity of neutron imaging

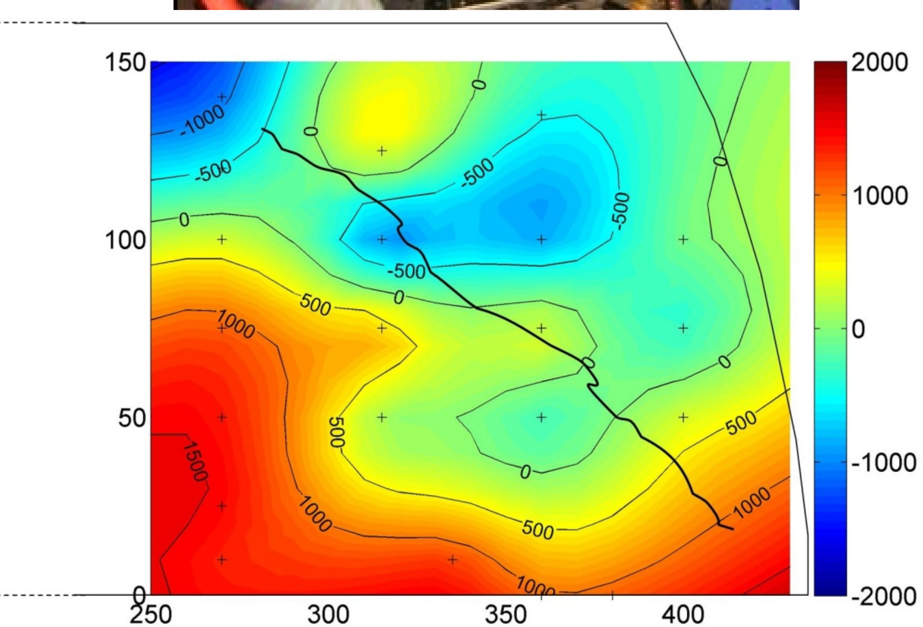
| | Neutrons | Synchrotron X-rays |
|---|---|---|
| Penetration<br>•Steels | 40-60 mm | 20-25 mm |
| Spatial resolution | 0.5 $mm^3$ (cubic gauge volume) | 0.1 mm lateral to incident beam;<br>1 mm parallel to beam (elongated gauge volume) |
| Stress complete tensor | Yes | With problems |
| Coarse grain problem | No | Yes |
| Counting time | 10-30 minutes | Seconds-minutes |

*With thanks to Shu Yan Zhang, ISIS*

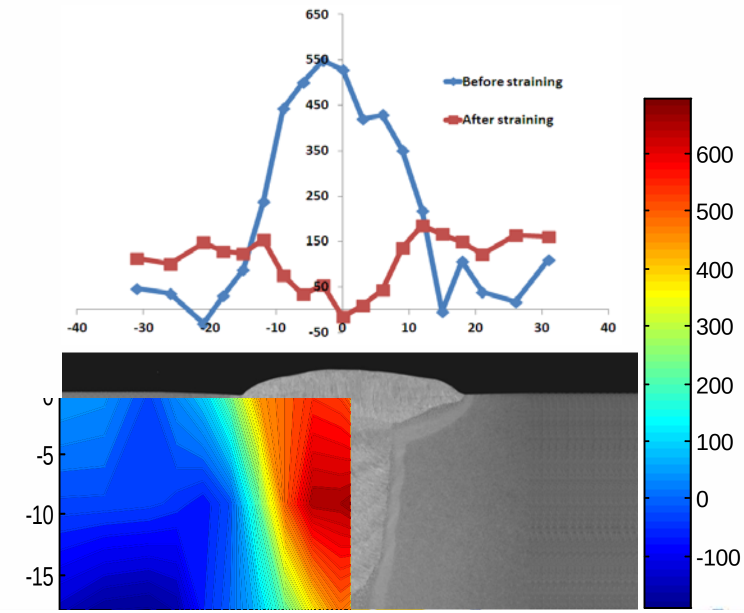
# Complementarity of neutron imaging







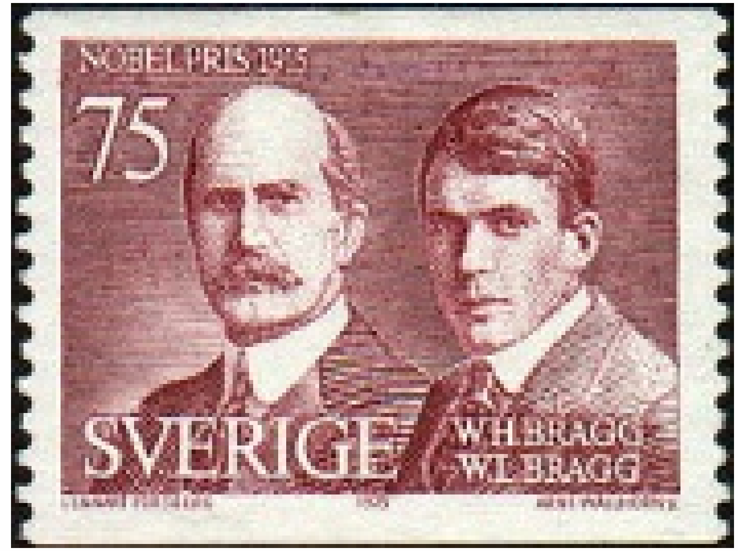

Residual strain profile within the Direct Chill cast Mg alloy slab, with estimated position of the crack in the measurement plane.
*M Turski, et al, Metall Mater Trans A 43A (5) 1547-1557 (2012)*



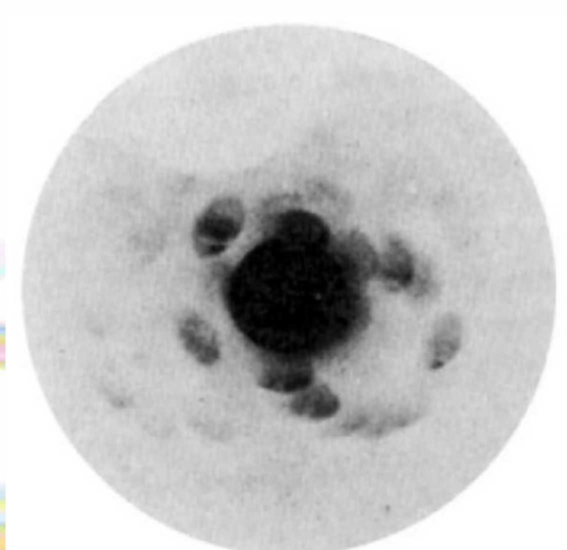

Measure welding residual stresses in pipeline for on-offshore transport of oil and gas

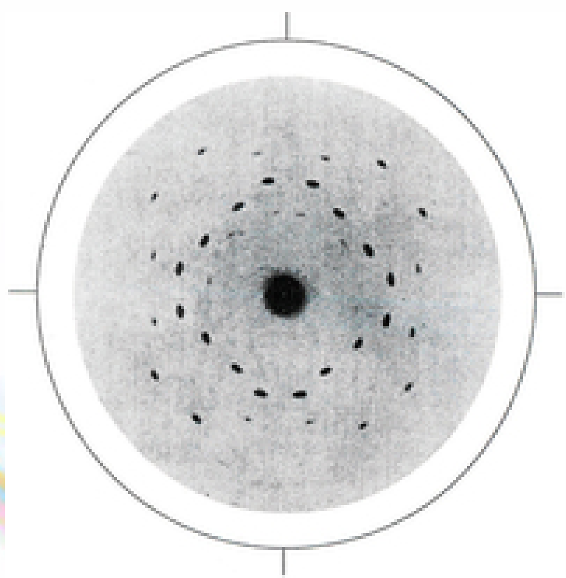
*With thanks to Shu Yan Zhang, ISIS*

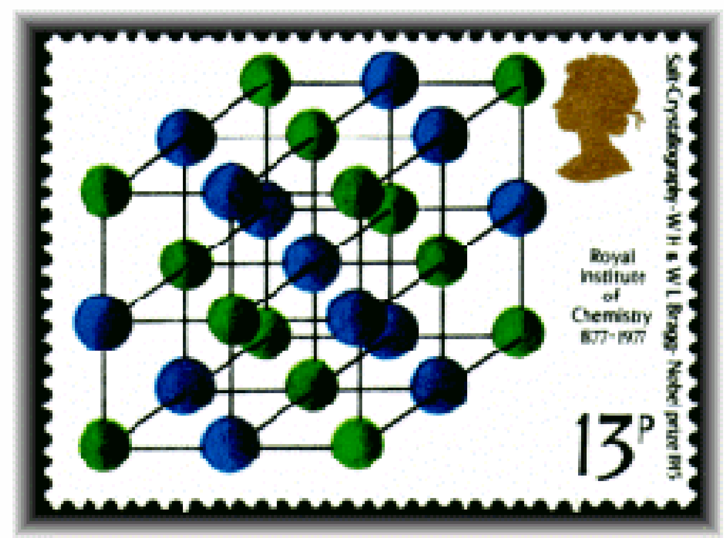
# Origins of diffraction at atomic scales

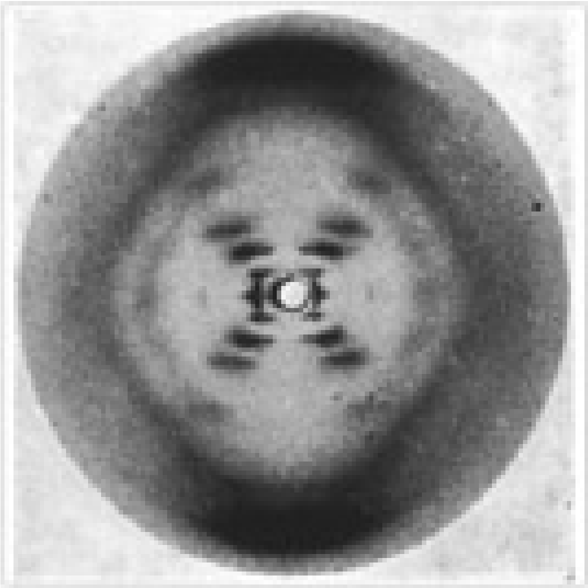

$$n\lambda = 2d.\sin\theta$$

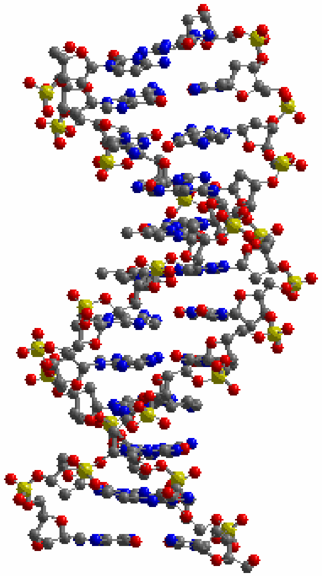


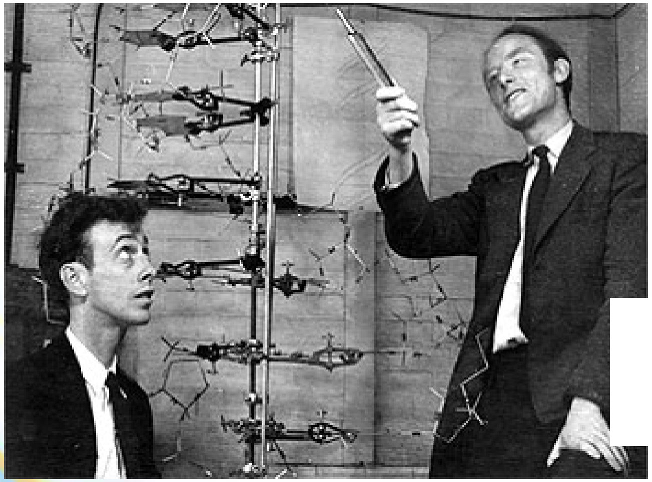




# 40 years on…

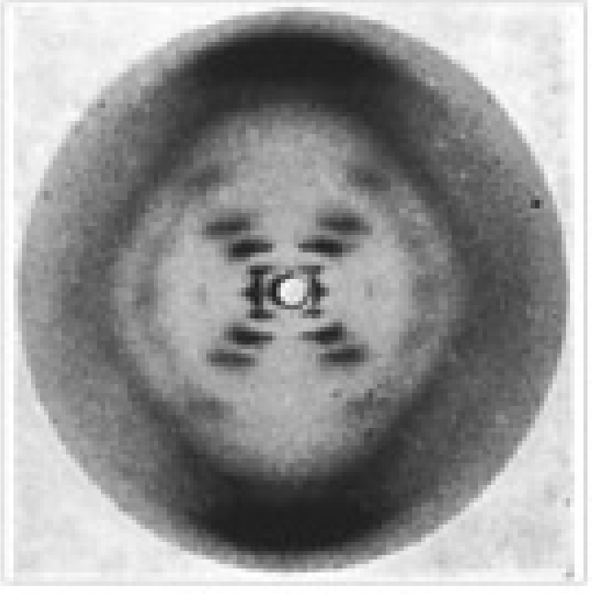


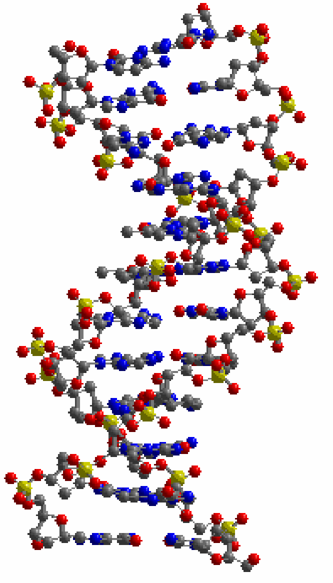


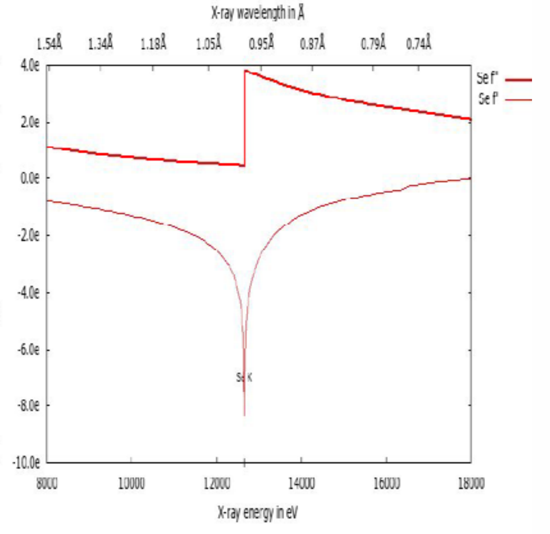


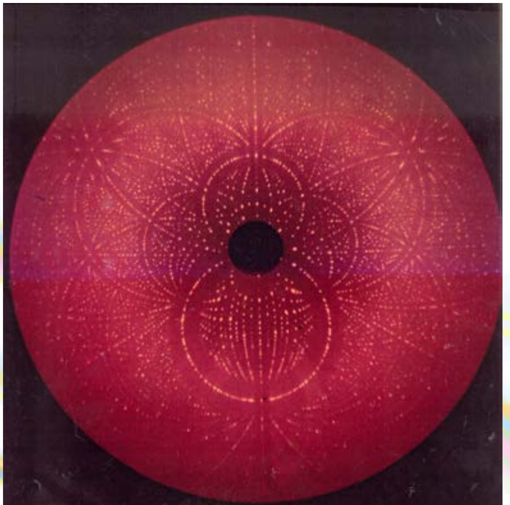
# Macromolecular crystallography today

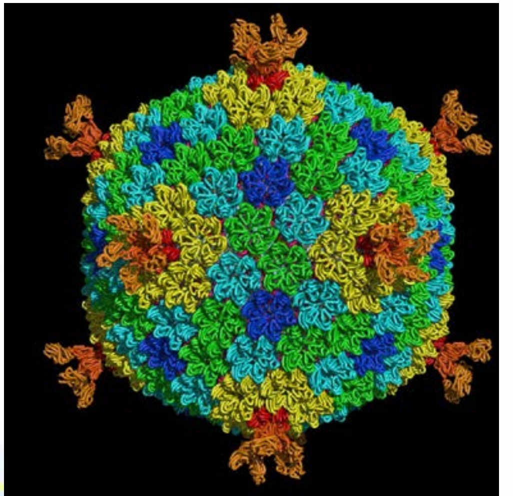
- Brilliance and tunability of photon energy, high-performance computing – > $10^{10}$ times more information/second







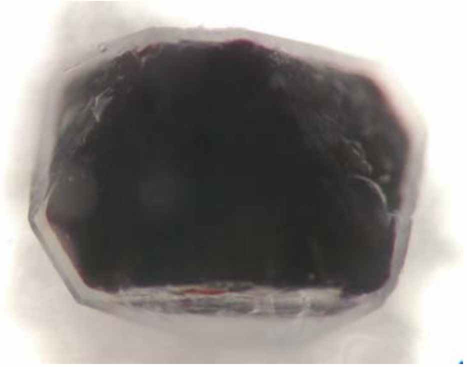





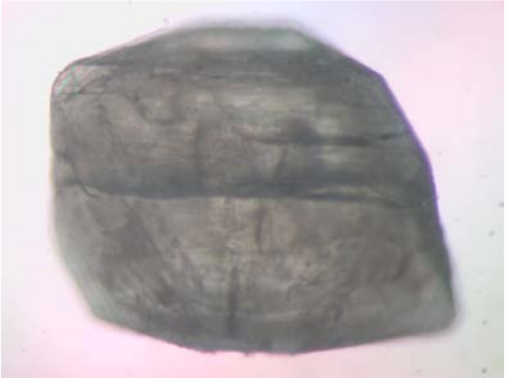
# Complementarity: neutrons

- Location of H atoms when X-rays/chemical ‘rules’ don’t help
- Study of samples susceptible to radiation damage
- Study of Rubredoxin structure illustrates both

  - *Small (~6kD) iron-sulphur containing redox protein – important model system to understand electron transfer processes using redox systems – here $Fe^{3+}$ - $Fe^{2+}$*
  - *$Fe^{3+}$ form very easily reduced in the X-ray beam*



*$Fe^{3+}$*

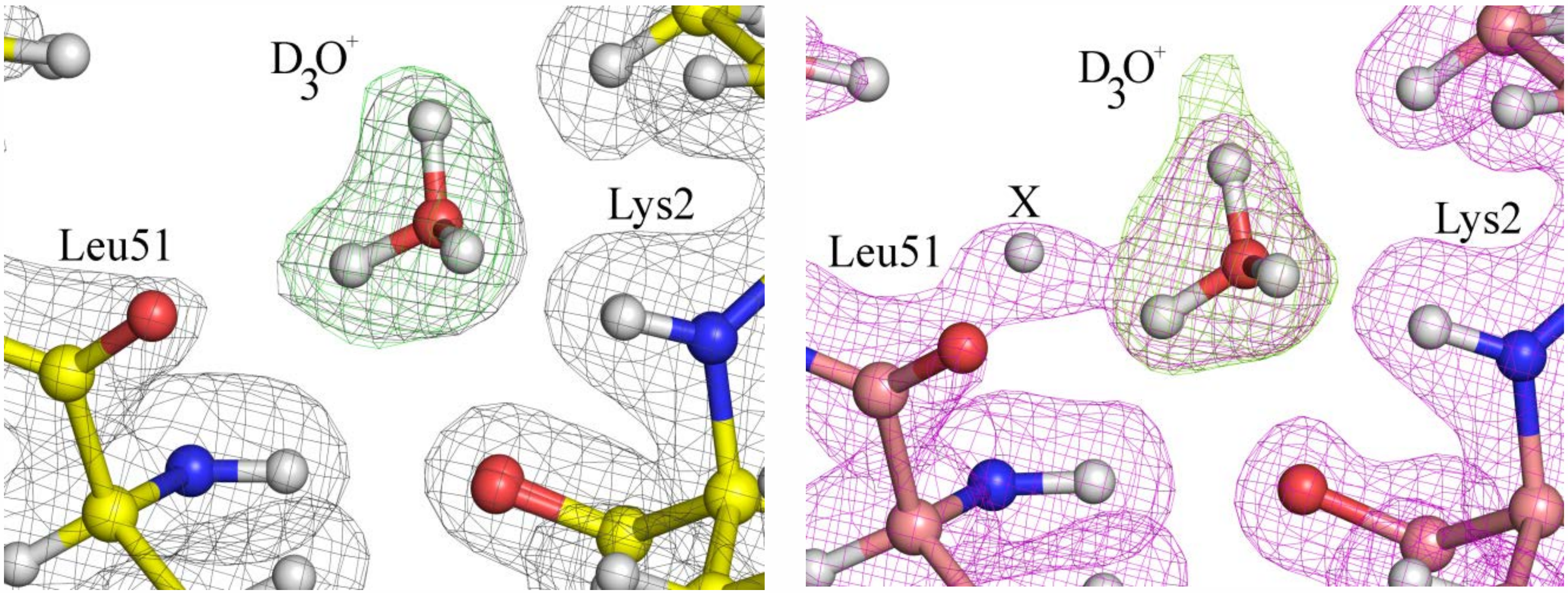


*$Fe^{2+}$*

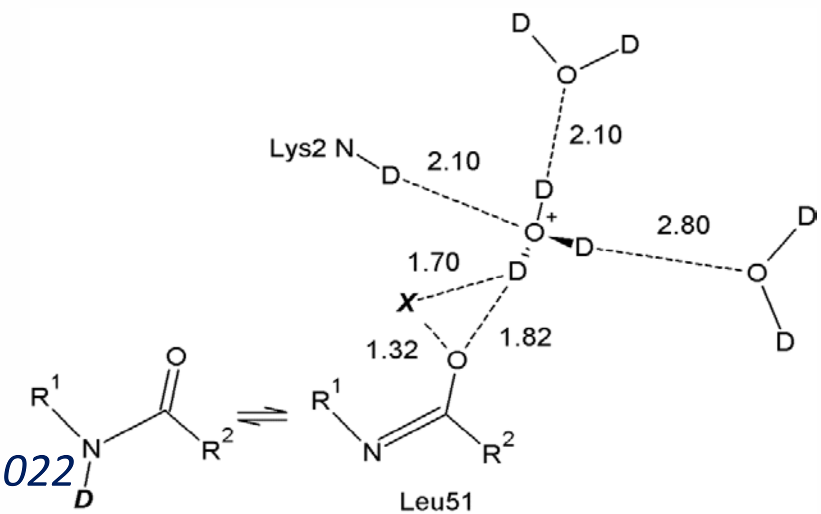
*Max Cuypers et al, Angewandte Chemie 52 (2013) 1022*

# Complementarity: neutrons

- Structure of reduced and oxidised form measured on D19 at ILL



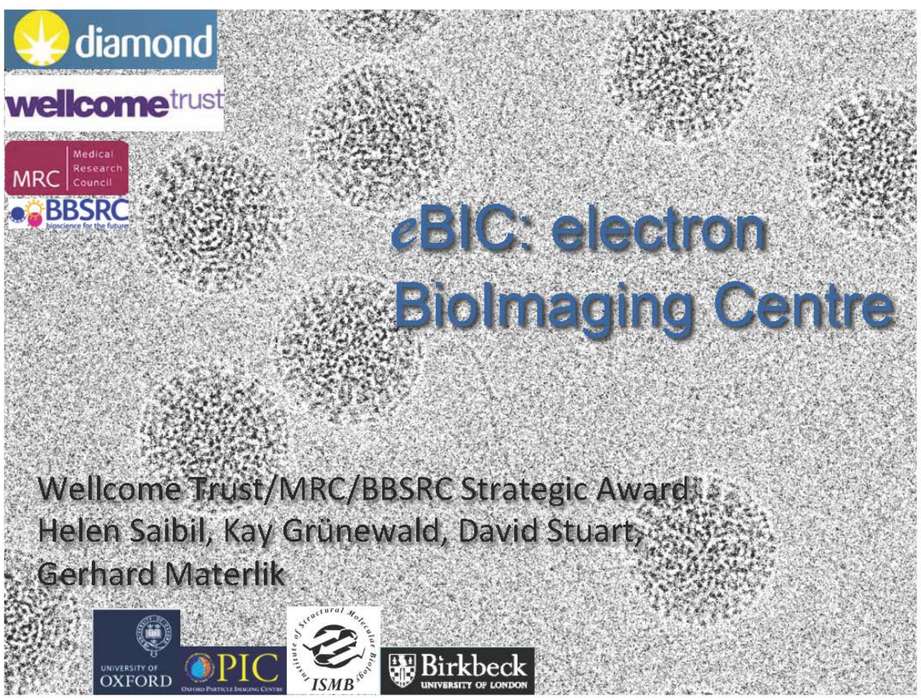

Observation of hydronium (D) ions and of tautomeric shifts following the change from the oxidised form to the reduced form

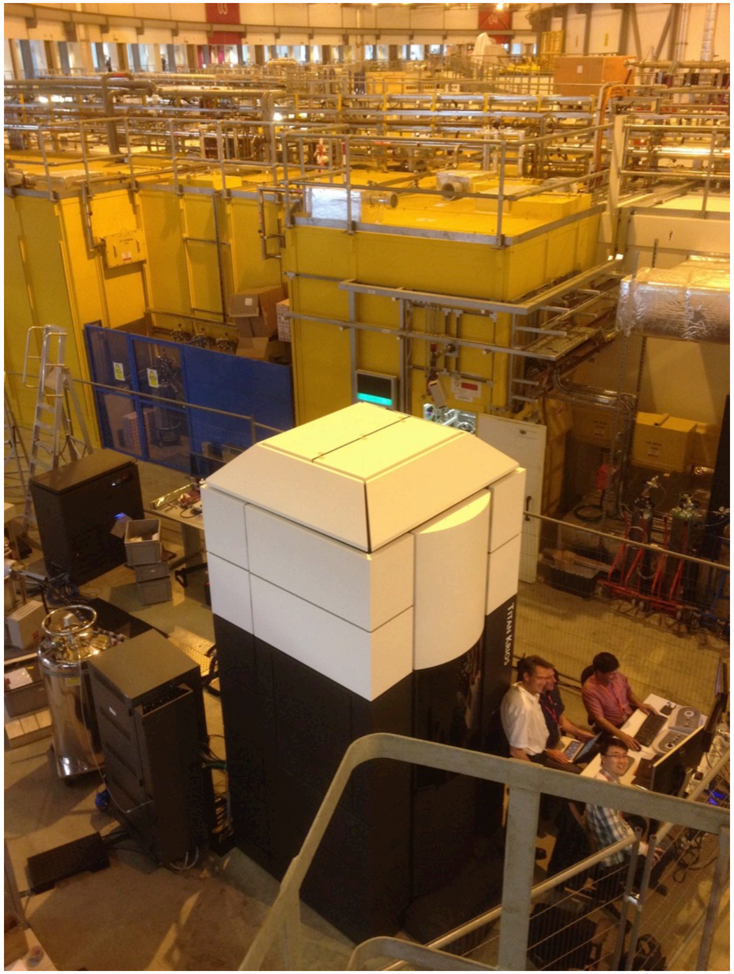



*Max Cuypers et al, Angewandte Chemie 52 (2013) 1022*

# Complementarity: the EM revolution

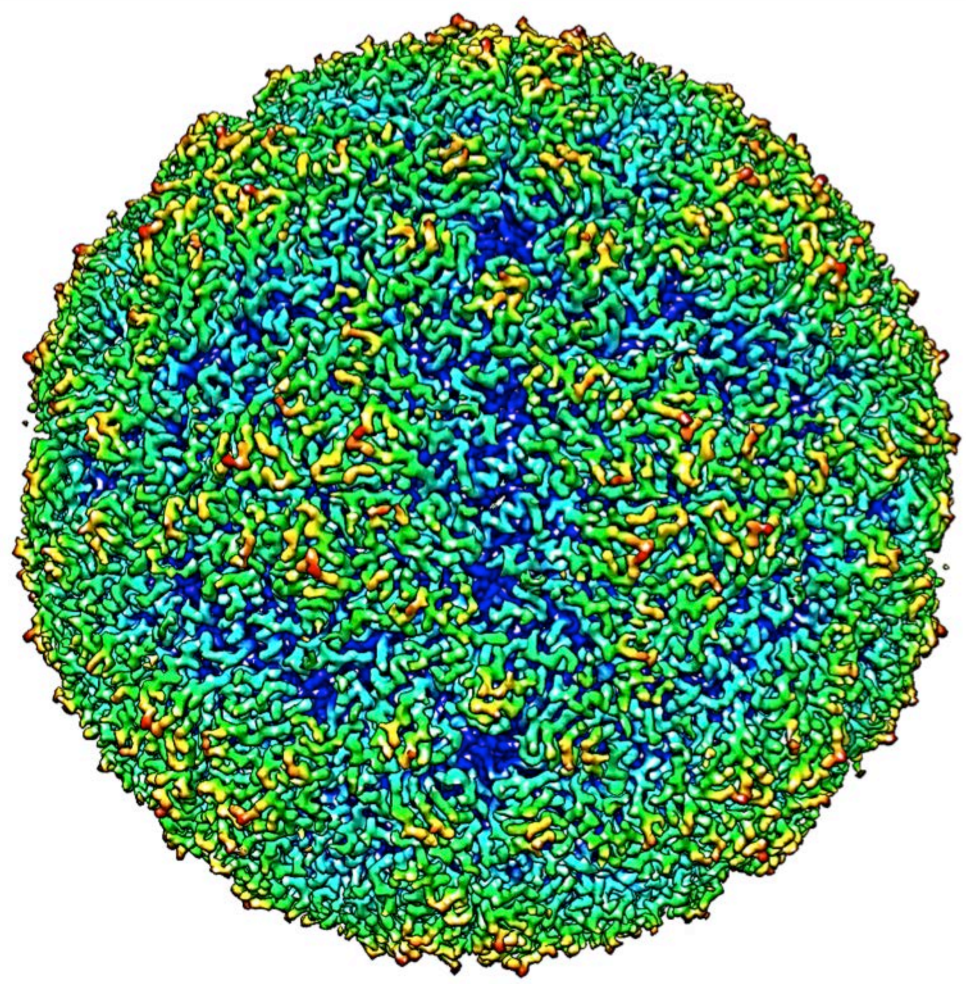
- Wonderful new detectors that collect movies and allow particle movements to be tracked in software to reveal atomic detail
- Native particles frozen in a water ‘glass’

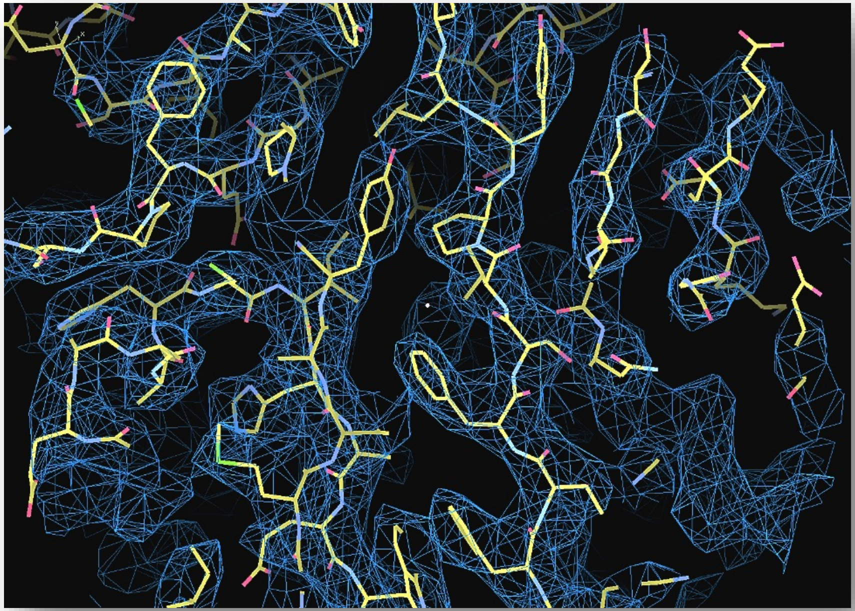


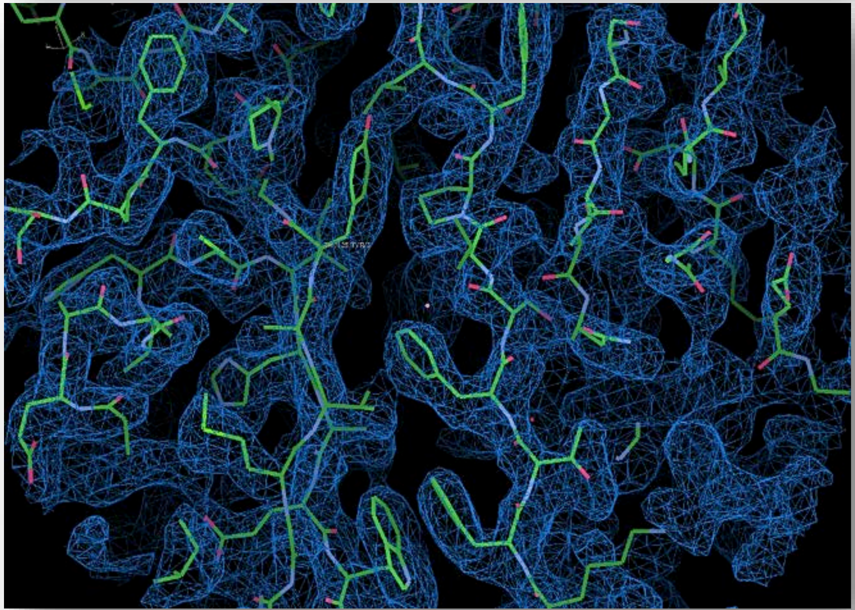



# Complementarity: the EM revolution

- Example from foot-and-mouth disease virus by electron micro-scopy and synchrotron X-rays

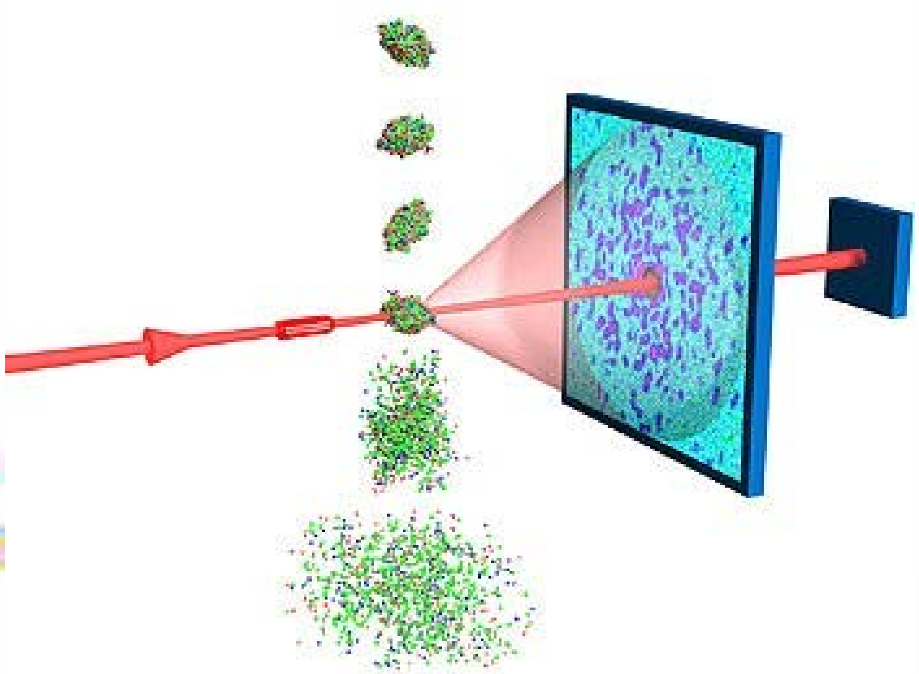






# Complementarity: FELs

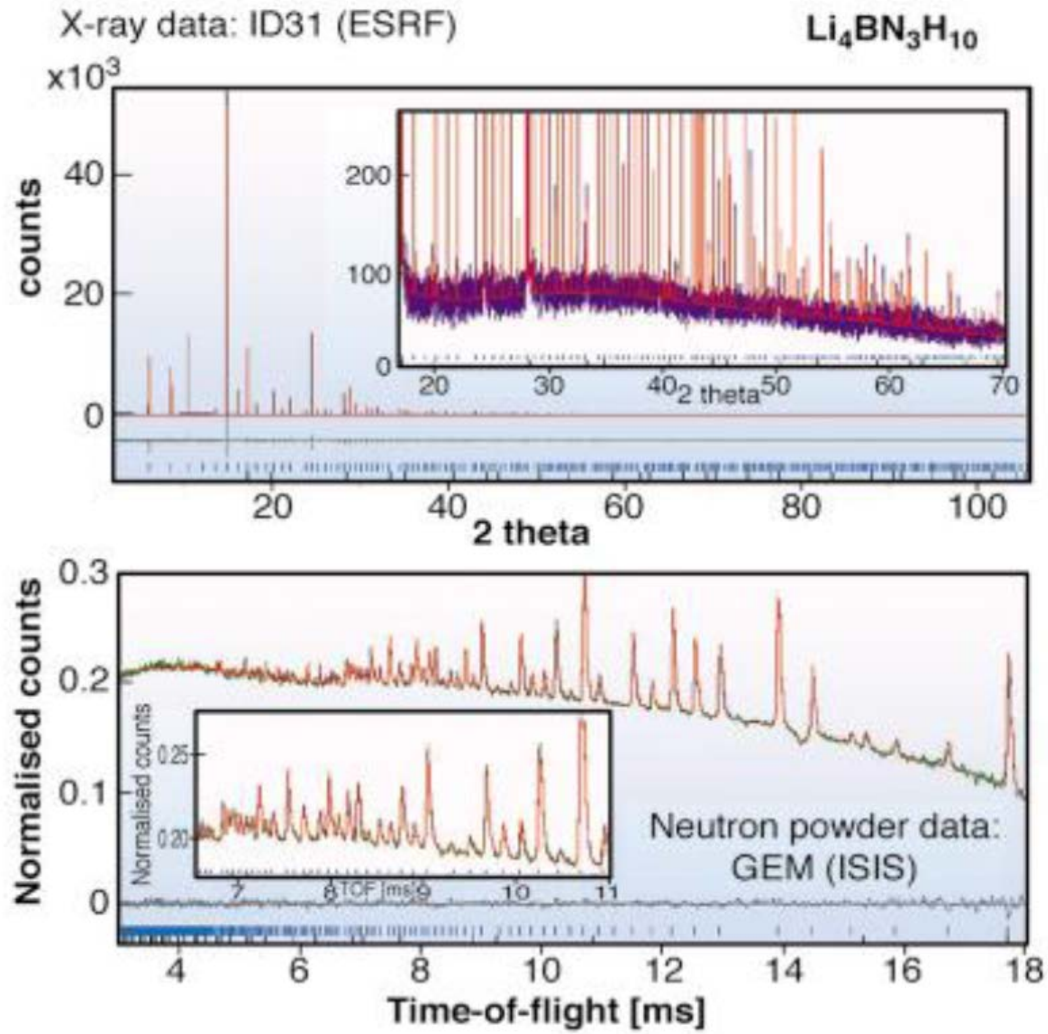
- X-rays pulses in a free electron laser (FEL) are ultrafast (fs) and ultrabright (x $10^{10}$) and capture structure before the sample is perturbed by the beam – ‘diffract and destroy’ !
- Challenge of sample injection, image processing…



# ‘Hard’ materials

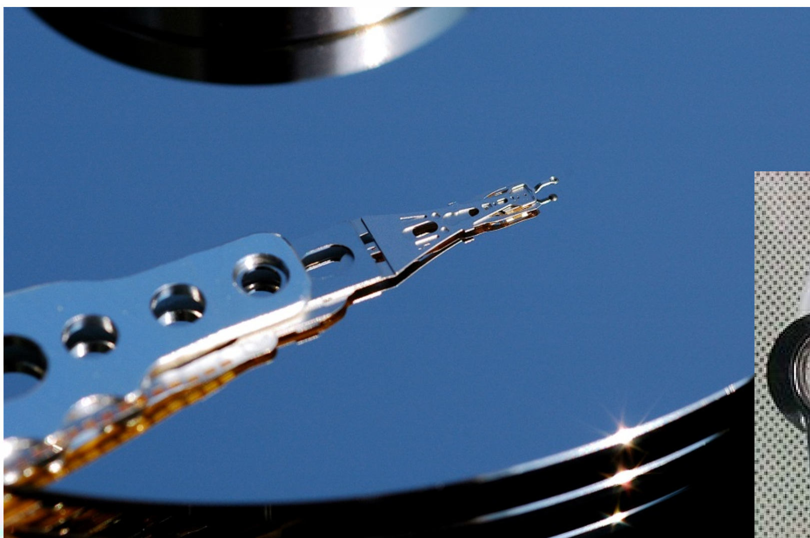
- Many important materials contain hydrogen or other light atoms that are crucial to function – e.g. for hydrogen storage

- Combine X-rays for rapid, high-resolution survey/ study then neutrons to locate H, Li, etc….



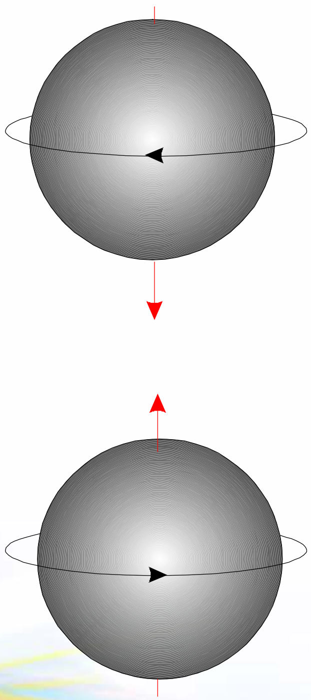
# Magnetic structures

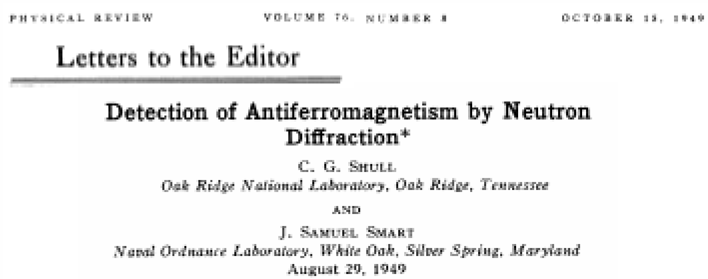
- Need to measure magnetic (spin) structures and excitations at an atomic level to understand and predict magnetic properties
- Important for a wide range of functional materials for sensors, actuators, information storage, nanomedicine, superconductors…




# Magnetic structures: neutrons

- Scattering cross-section relatively simple and comparable in size to the nuclear cross-section





PHYSICAL REVIEW VOLUME 76, NUMBER 8 OCTOBER 15, 1949

## Letters to the Editor

### Detection of Antiferromagnetism by Neutron Diffraction*

C. G. Shull
*Oak Ridge National Laboratory, Oak Ridge, Tennessee*
and
J. Samuel Smart
*Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland*
August 29, 1949

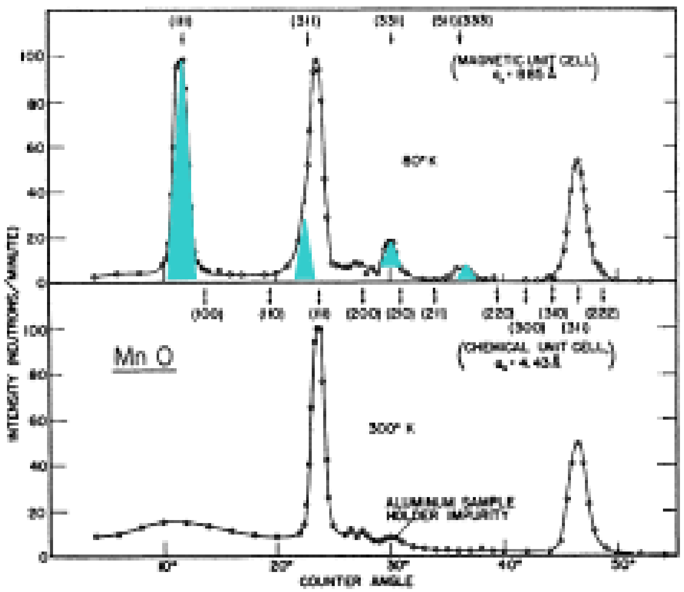
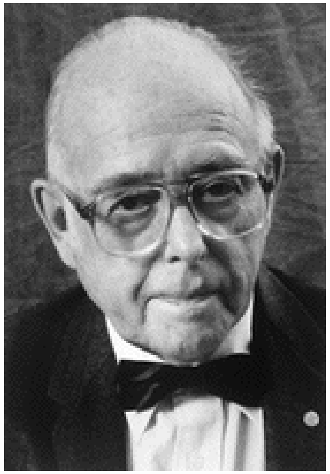
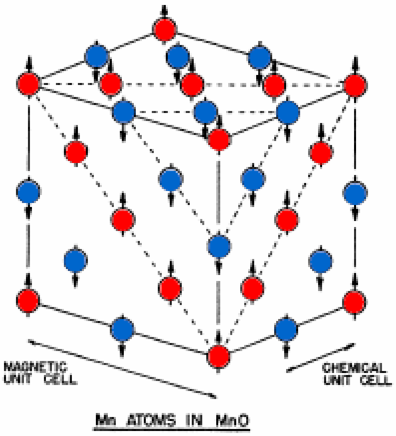


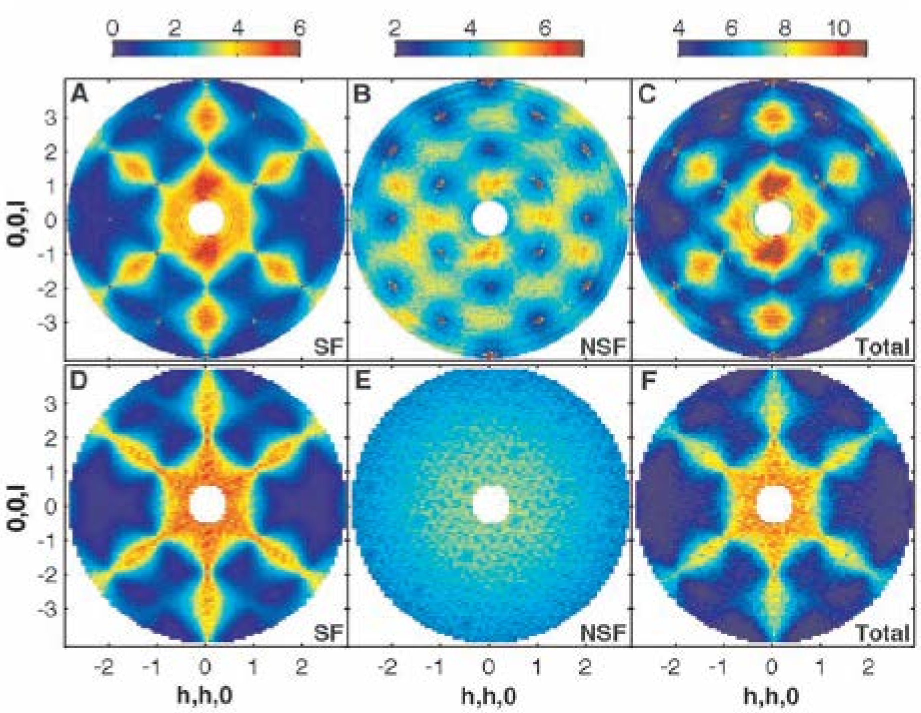

Fig. 1. Neutron diffraction patterns for MnO at room temperature and at 80°K.

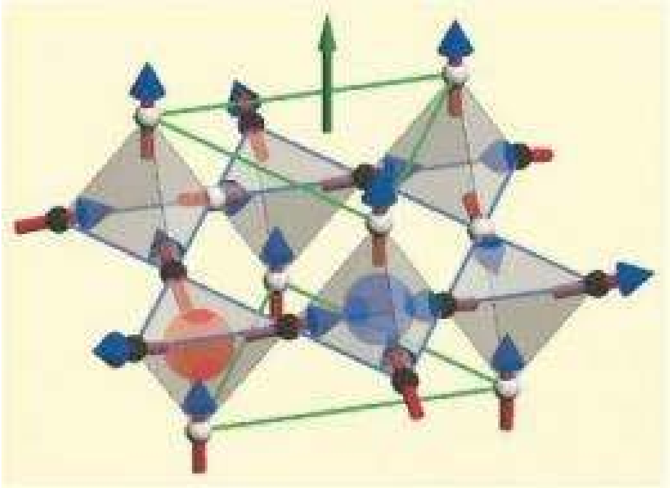




# Magnetic structures: neutrons

- Polarisation techniques can provide all details of spin structure (zero-field polarimetry) and map out diffuse scattering from short-range order
- Penetrating ability of neutrons enables design of sophisticated sample environment and cryogenics to nK, *but* large samples are required



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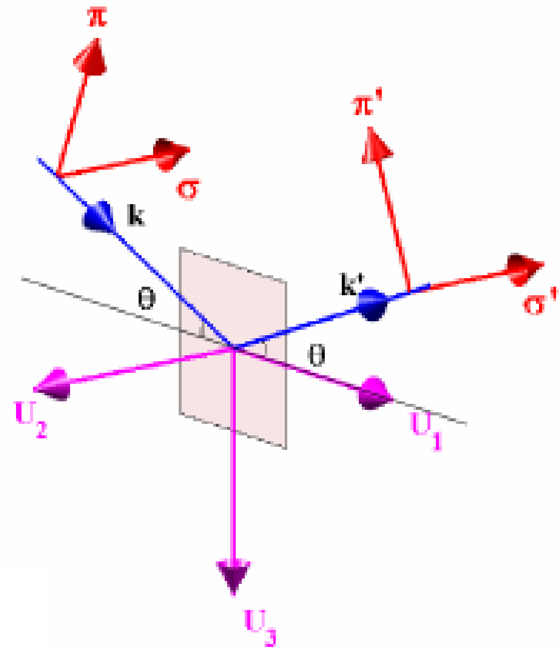
# Magnetic structures: X-rays

- Scattering cross-section generally very weak – typically $10^6$ weaker than the nuclear (Thomson) terms $((\hbar\omega/mc^2) \sim 10^{-2} \rightarrow I^{mag}/I^{at} \sim 10^{-6})$
- Polarisation analysis can be used to separate spin (S) and orbital (L) contributions
- Magnetic scattering amplitude $f^{mag}(Q)$:

$$f^{mag}(Q) = ir_0\left(\frac{\hbar\omega}{mc^2}\right)\left[\frac{1}{2}A''.\,L(Q) + B.\,S(Q)\right]$$

L(Q) and S(Q) are the Fourier transforms of the atomic and spin magnetisation densities; A” and B depend on the relative orientation of k, k’, ε, ε’




$$f^{mag}(\boldsymbol{Q}) = i\,r_0\left(\frac{\hbar\omega}{mc^2}\right) \times$$

| | $\hat{\varepsilon}_\perp = \sigma$ | $\hat{\varepsilon}_\parallel = \pi$ |
|---|---|---|
| $\hat{\varepsilon}'_\perp$ | $\sin 2\theta S_2$ | $-2\sin^2\theta\left[(L_1+S_1)\cos\theta - S_3\sin\theta\right]$ |
| $\hat{\varepsilon}'_\parallel$ | $2\sin^2\theta\left[(L_1+S_1)\cos\theta - S_3\sin\theta\right]$ | $\sin 2\theta\left[2\sin^2\theta L_2 + S_2\right]$ |

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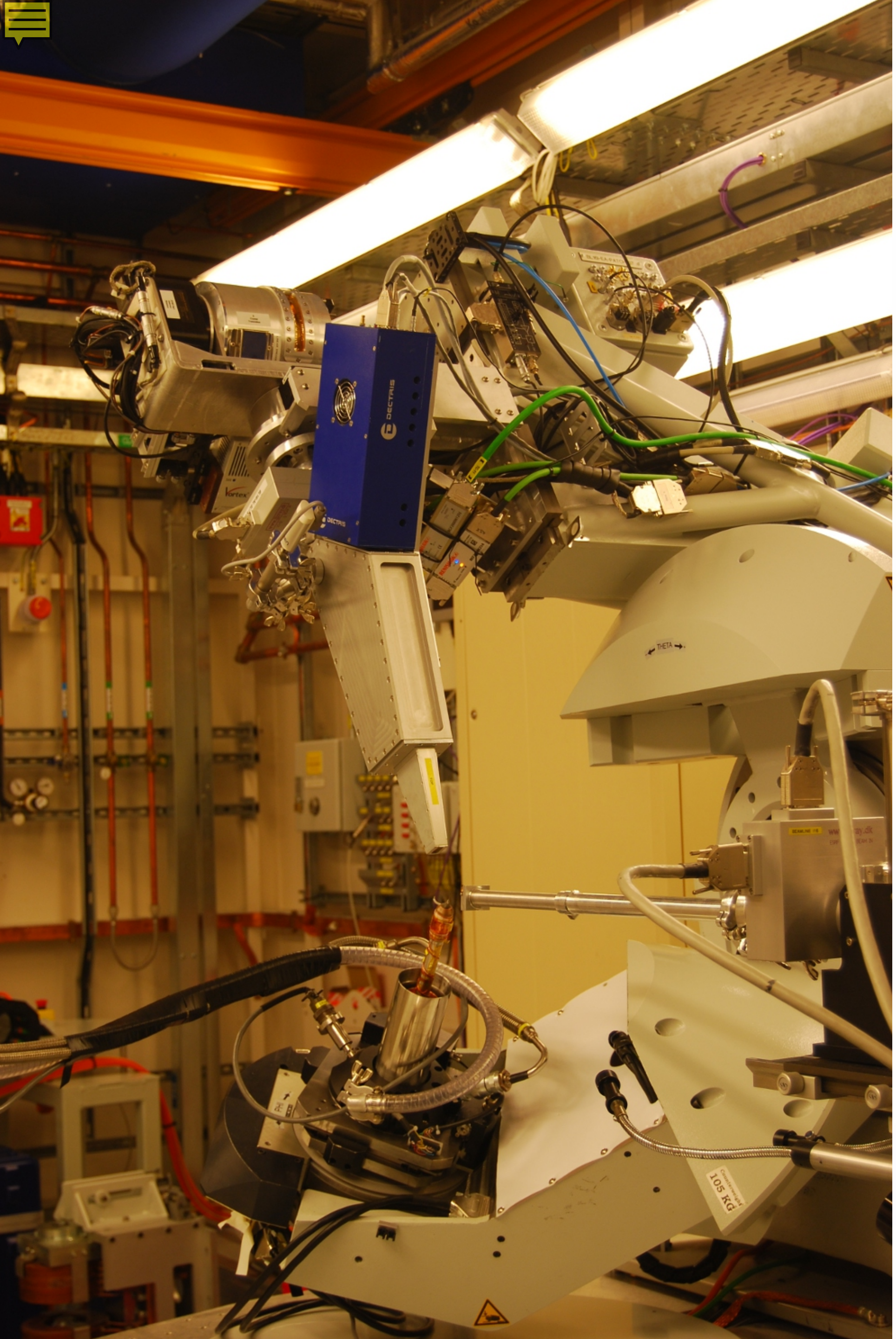
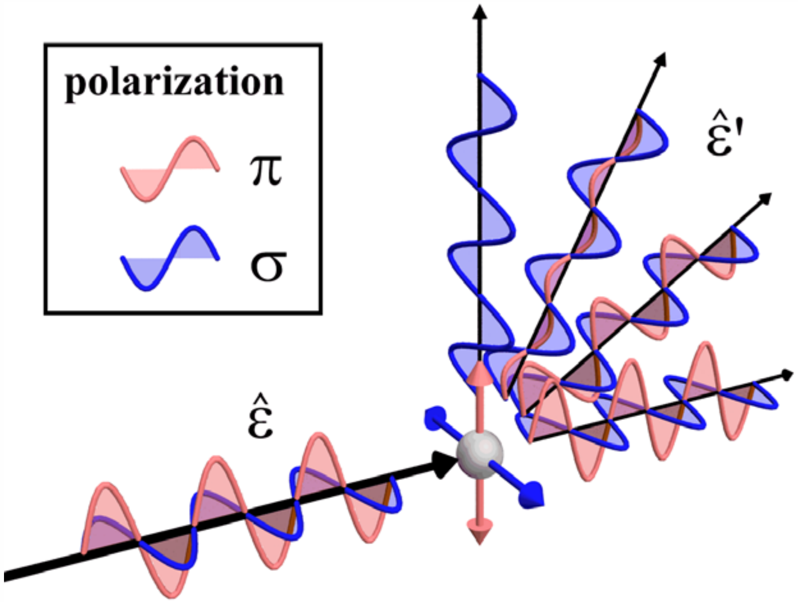



- Versatile diffractometer: polarisation analysis and azimuthal angle
- Energy tunable from 1-15 keV to get good atomic resolution

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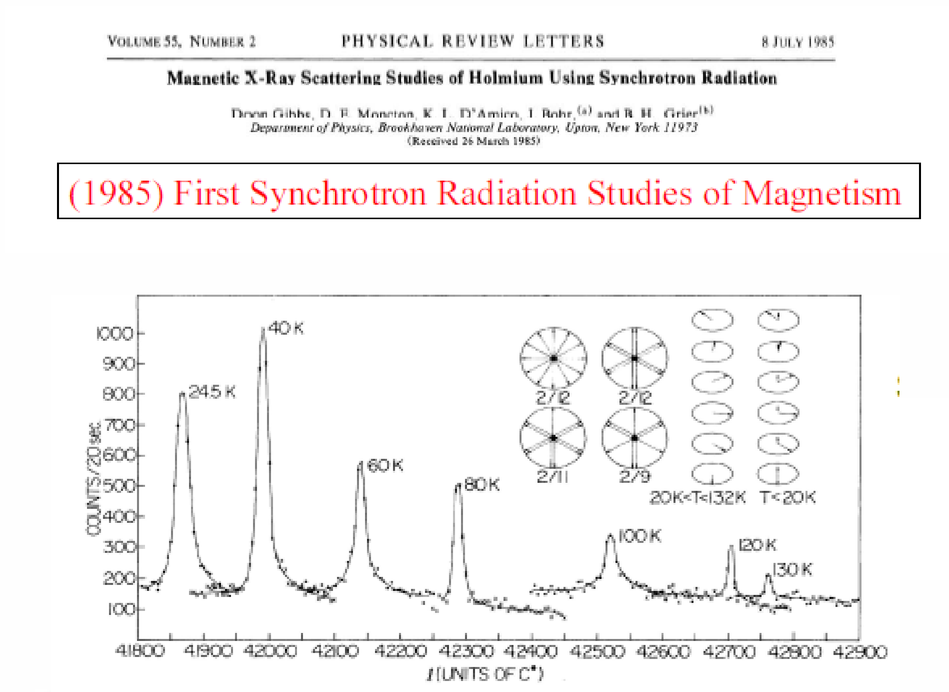
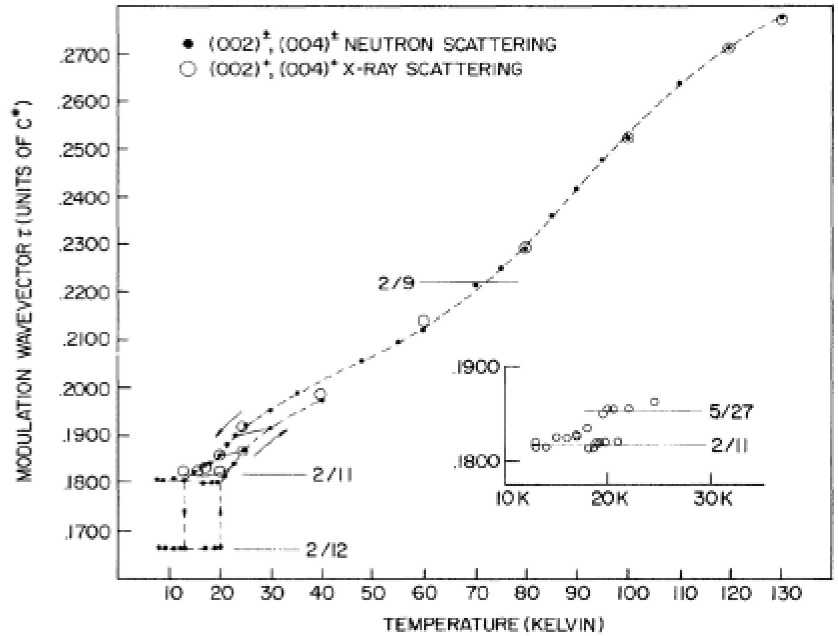
# Magnetic structures: X-rays

- First magnetic structures studied at a synchrotron in 1985 - spiral spin structure of Ho mapped out as a function of temperature




- Advantages of (non-resonant) X-ray magnetic scattering
  - Orbital and spin magnetisation density may be separated
  - Highly-focused beams enable very small samples to be studied
  - High resolution in Q – e.g. for subtle changes in spin structure
  - ***However***, cryogenics are challenging !

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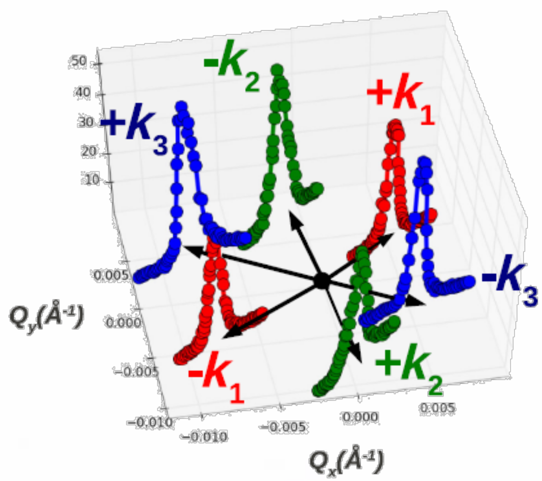
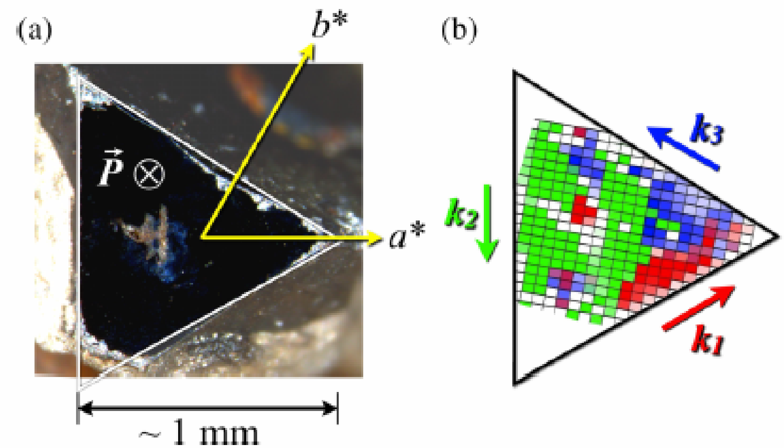
# Magnetic structures: X-rays

- $BiFeO_3$ has room-temperature coupling between magnetic and ferroelectric (FE) order.
- Map domain distribution with 50 μm spot (LD) then probe helicity by CD: same helicity for single FE domain

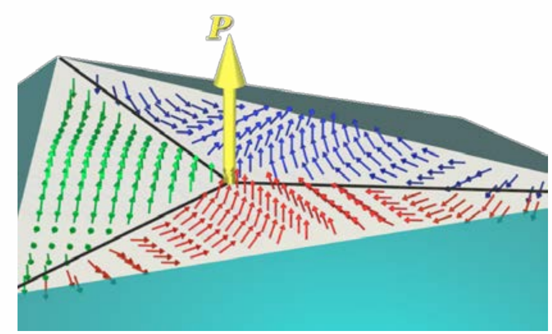



PRL 110, 217206 (2013) PHYSICAL REVIEW LETTERS week ending 24 MAY 2013

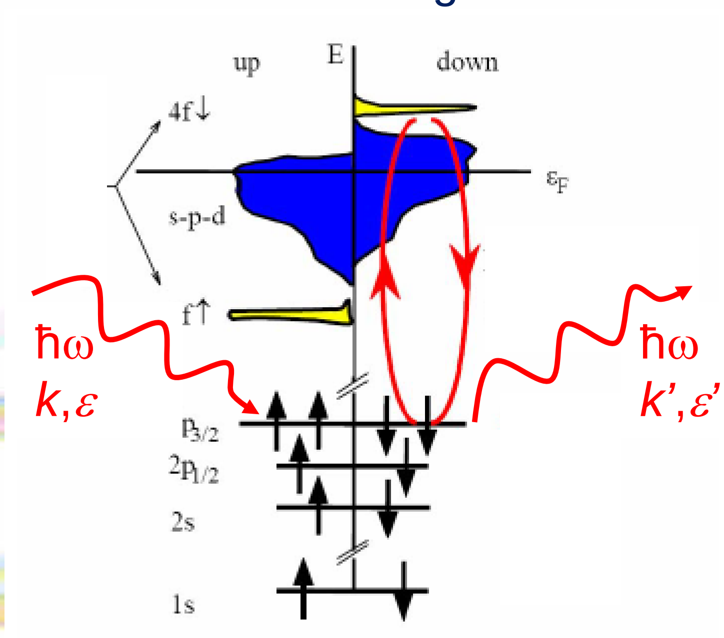
**X-Ray Imaging and Multiferroic Coupling of Cycloidal Magnetic Domains in Ferroelectric Monodomain $BiFeO_3$**

R. D. Johnson,[1,2,*] P. Barone,[3] A. Bombardi,[4] R. J. Bean,[5] S. Picozzi,[3] P. G. Radaelli,[1] Y. S. Oh,[6] S.-W. Cheong,[6] and L. C. Chapon[7]



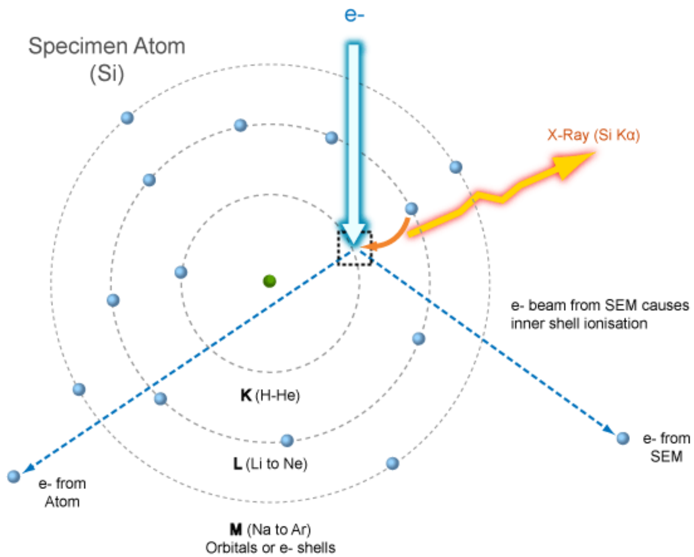
# Magnetic structures: resonant scattering

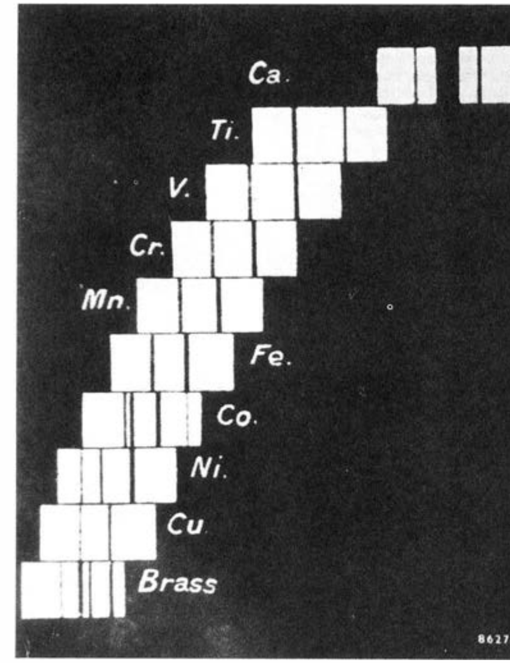
- Tune energy to a core-(empty) valence state transition and exploit resonant scattering to greatly enhance intensity (x$10^3$)
  - Sensitive to specific element and oxidation state
  - Dipole transitions ($\Delta L = 1$) generally stronger than quadrupole transitions ($\Delta L = 2$) and these will also be polarisation dependent; transitions from s to d or p states generally weaker because of lower overlap of wavefunctions
  - Sensitive to spin (S) though spin-orbit coupling
  - However, mostly limited to softer X-rays so will not give atomic resolution – tend to use for larger-scale structures (unit cell, periodic structures)




| elements | edge | transition | energy range [keV] | resonance strength | comment |
|---|---|---|---|---|---|
| 3d | K | 1s → 4p | 5 - 9 | weak | small overlap |
| 3d | $L_I$ | 2s → 3d | 0.5 - 1.2 | weak | small overlap |
| 3d | $L_{II}$, $L_{III}$ | 2p → 3d | 0.4 - 1.0 | strong | dipolar, large overlap, high spin polarisation of 3d |
| 4f | K | 1s → 5p | 40 - 63 | weak | small overlap |
| 4f | $L_I$ | 2s → 5d | 6.5 - 11 | weak | small overlap |
| 4f | $L_{II}$, $L_{III}$ | 2p → 5d<br>2p → 4f | 6 - 10 | medium | dipolar<br>quadrupolar |
| 4f | $M_I$ | 3s → 5p | 1.4 - 2.5 | weak | small overlap |
| 4f | $M_{II}$, $M_{III}$ | 3p → 5d<br>3p → 4f | 1.3 - 2.2 | medium to strong | dipolar<br>quadrupolar |
| 4f | $M_{IV}$, $M_V$ | 3d → 4f | 0.9 - 1.6 | strong | dipolar, large overlap, high spin polarisation of 4f |
| 5f | $M_{IV}$, $M_{II}$ | 3d → 5f | 3.3 - 3.9 | strong | dipolar, large overlap, high spin polarisation of 5f |

# Origins of X-ray spectroscopy




| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | | B | C | N | O | F | Ne |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl | Ar |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr | Y | Zr | Nb | Mo | | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | Xe |
| Cs | Ba | La | | Ta | W | | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | | Rn |
| | Ra | Ac | Th | | U | | | | | | | | | | | | |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ce | Pr | Nd | | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu |

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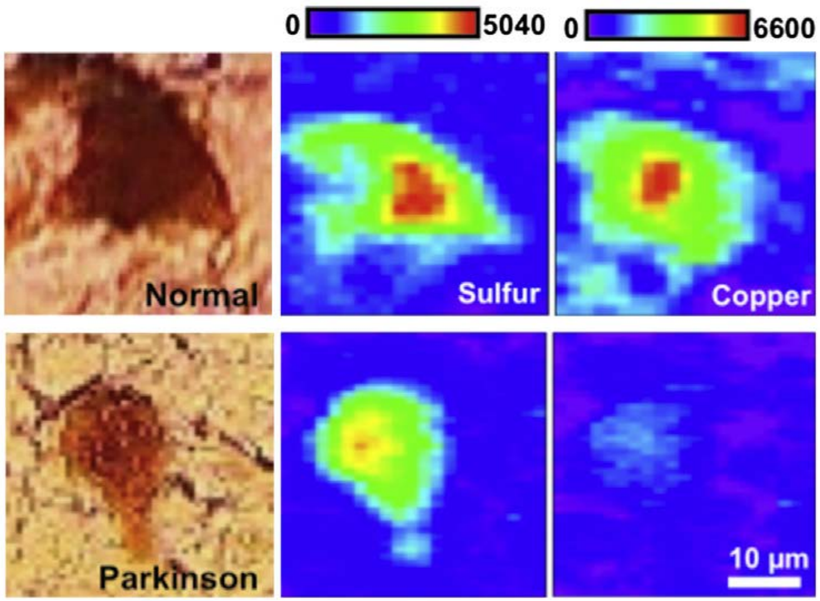
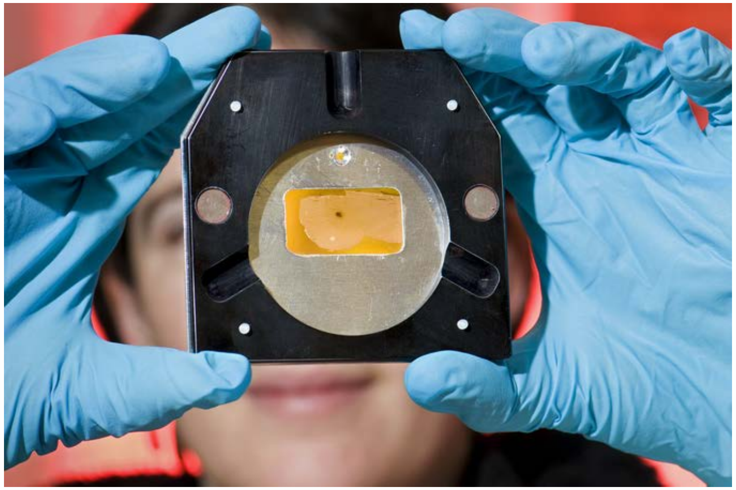
# Mapping elements – but what about chemistry ?



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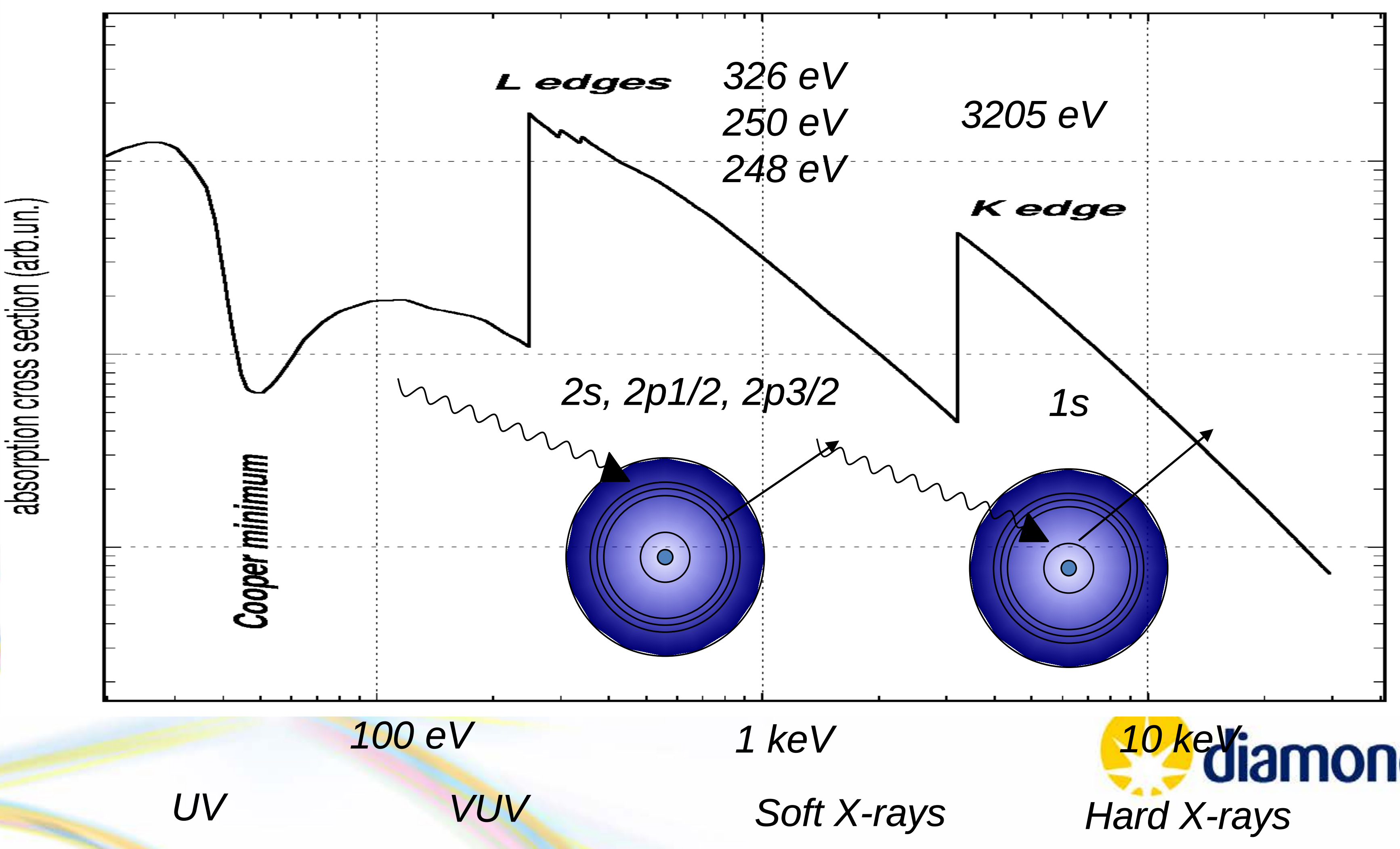
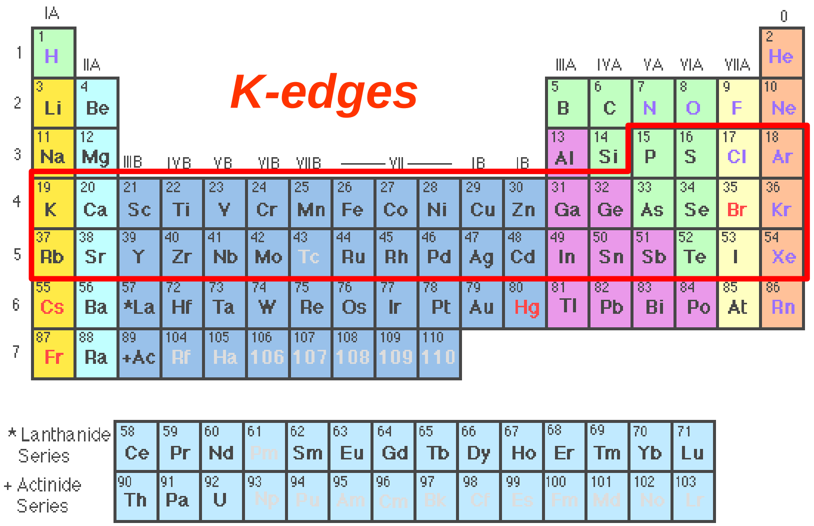
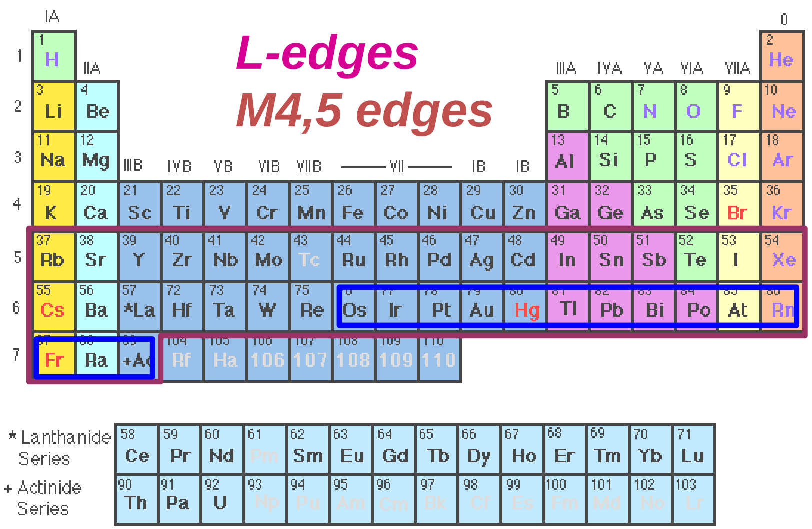
# Exploring electronic structure

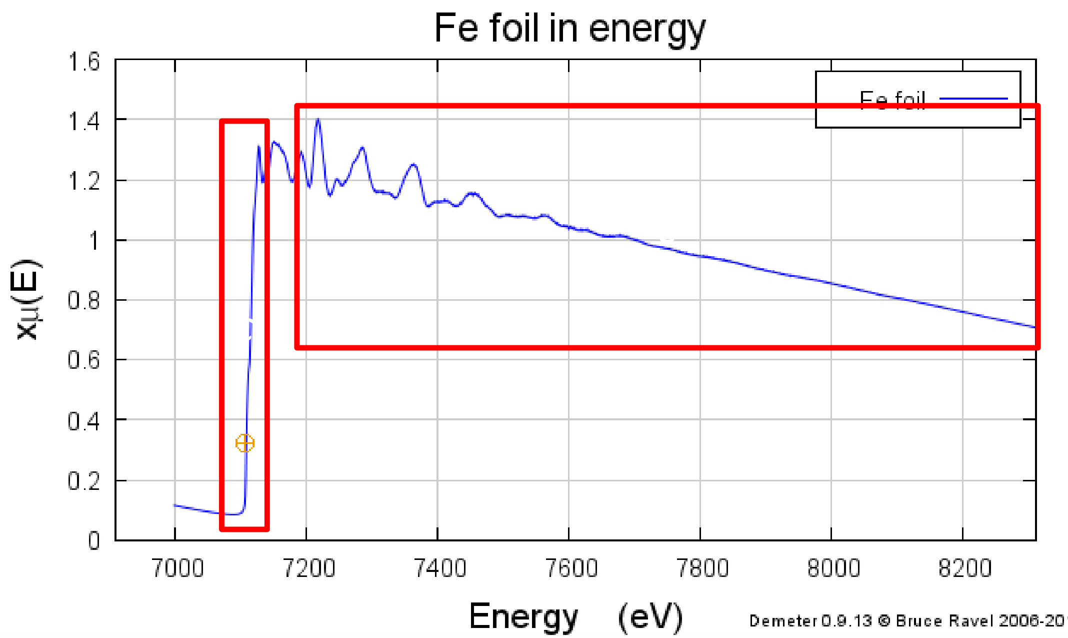
- Atomic levels - e.g. Argon – $1s^2\ 2s^2 2p^6\ 3s^2 3p^6$



# Sensitivity to chemical environment

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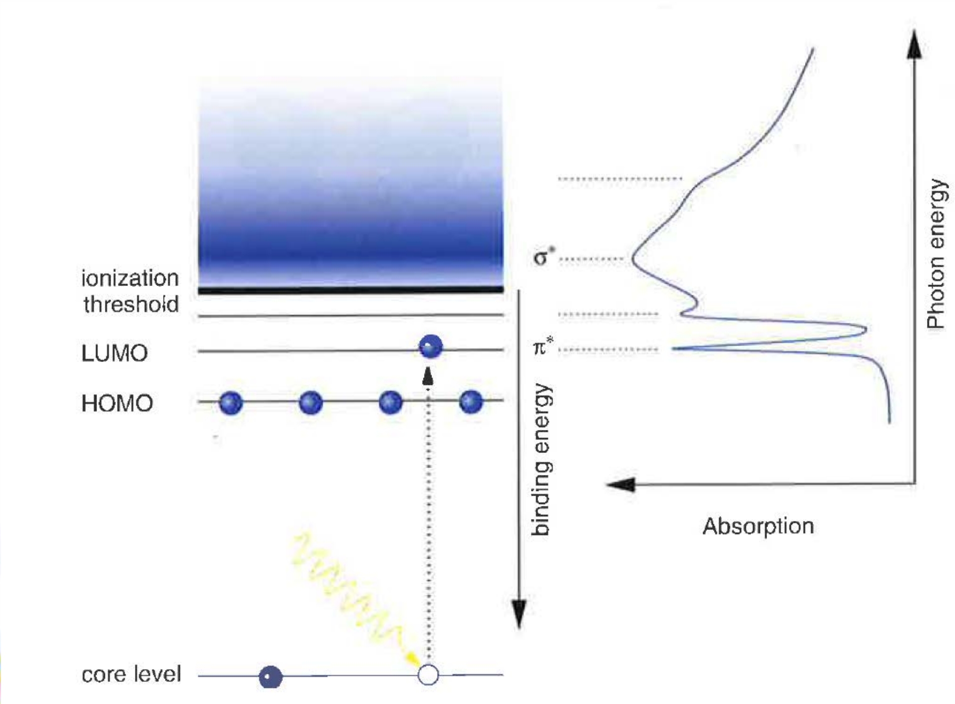
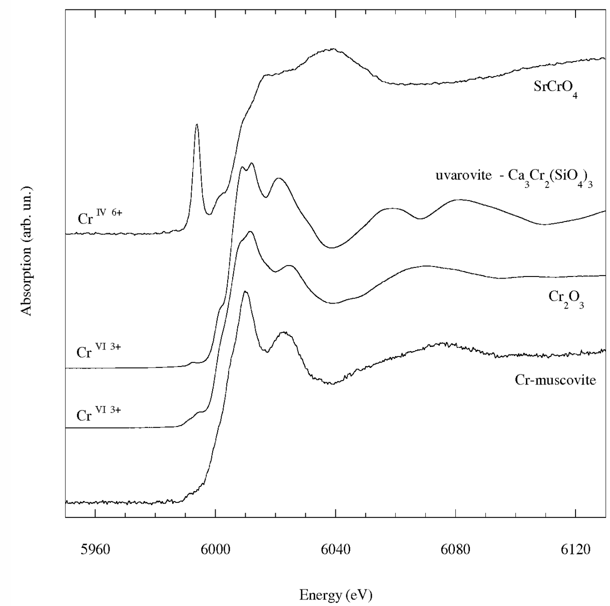
# XANES

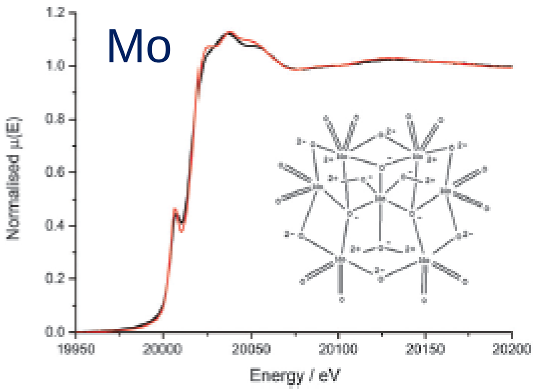
- X-ray Absorption Near Edge Spectroscopy: element-specific oxidation state and bonding in molecules and solids

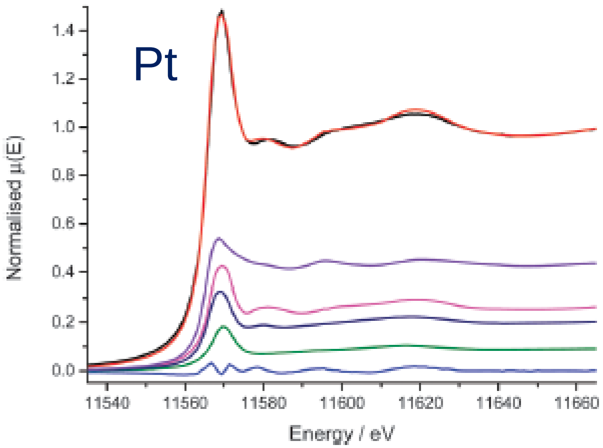



*Cr K absorption edge*

# Unravelling chemical clusters

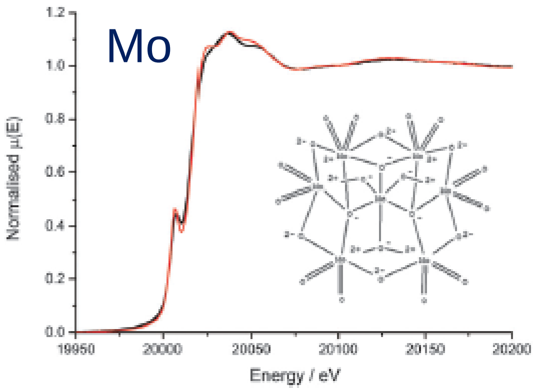


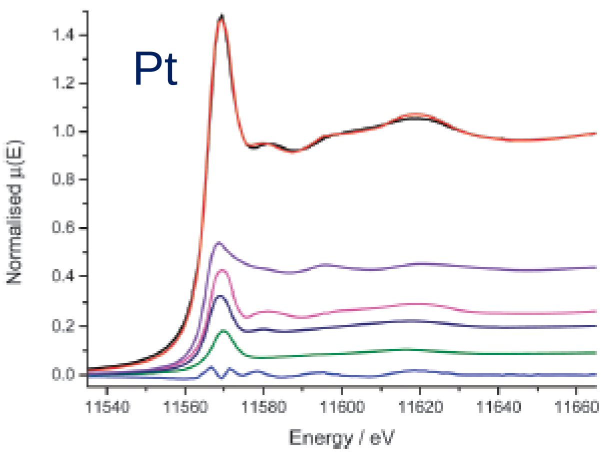




Species identification

Price *et al*, PCCP, Phys. Chem. Chem. Phys., 17 (2015) 521

# …and mapping where they are

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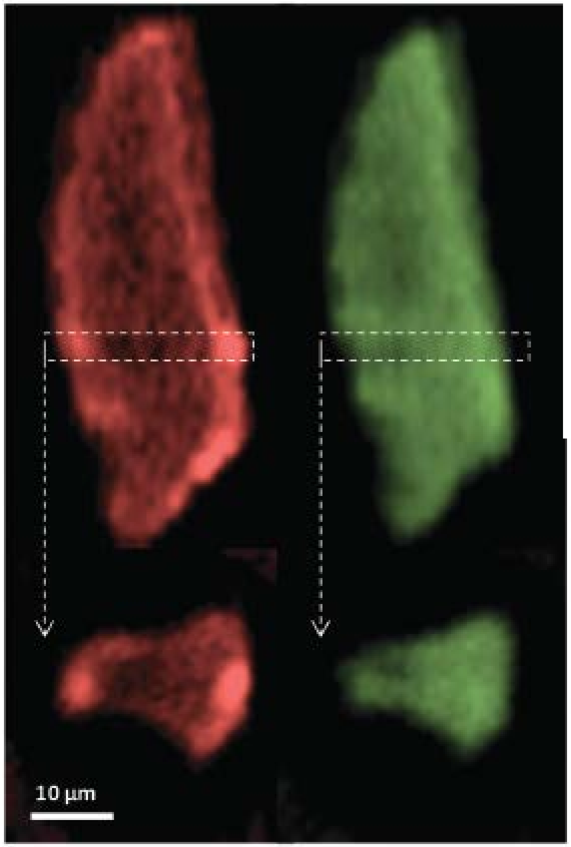
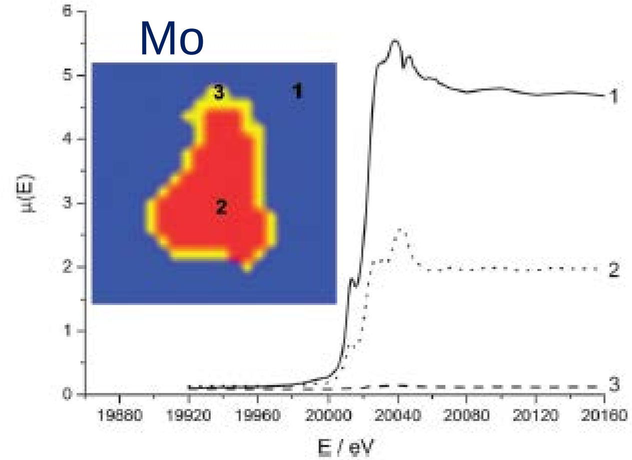
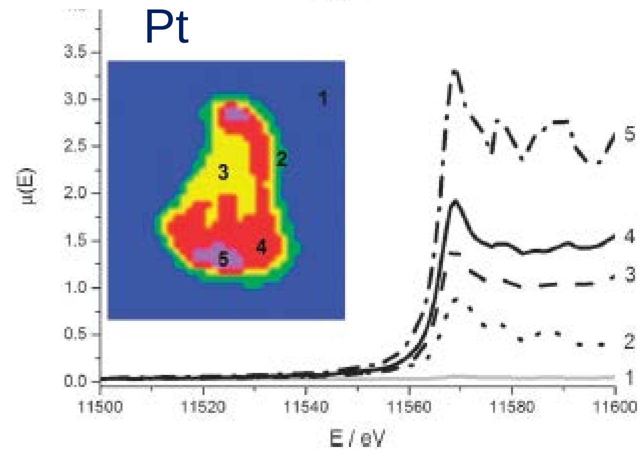



XRF tomography –
Mo (green) and Pt (red)




Species distribution

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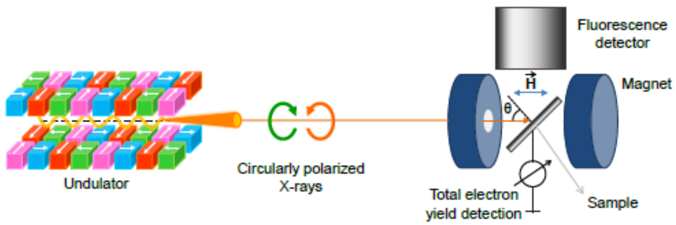
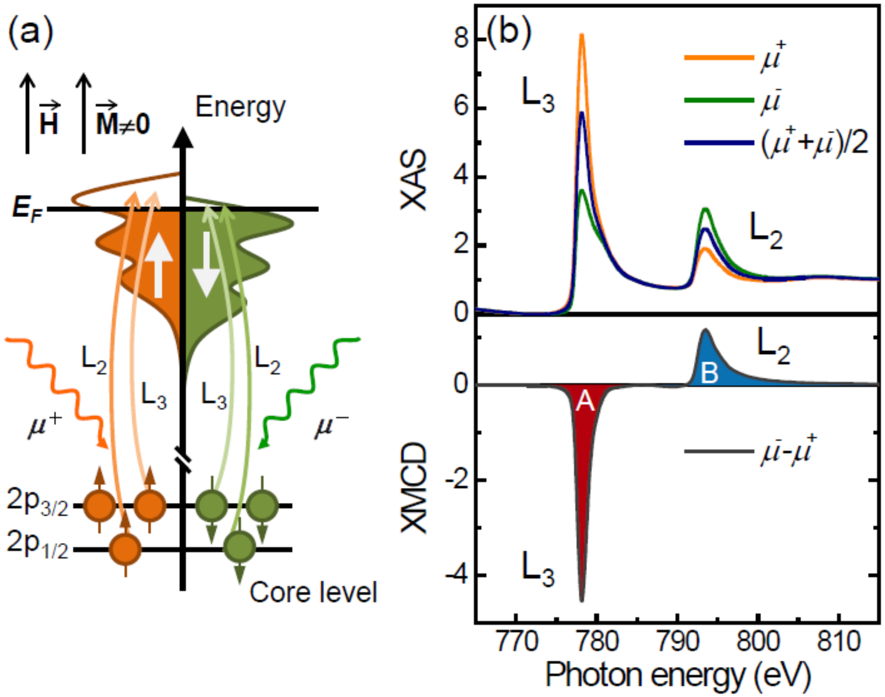
Price *et al*, PCCP, Phys. Chem. Chem. Phys., 17 (2015) 521

# XAS plus magnetic polarisation

- XAS with circularly polarised (CP) light: LCP-RCP probes polarisation o spin states by selectively exciting transitions with $\Delta m = +/- 1 \rightarrow$ XMCD




- XAS with LP radiation: $\Delta (\perp - //) \sim \langle M^2 \rangle \rightarrow$ XMLCD

*G van der Laan and A.I. Figueroa,*
*Coord. Chem. Reviews 277-278 (2014) 95*

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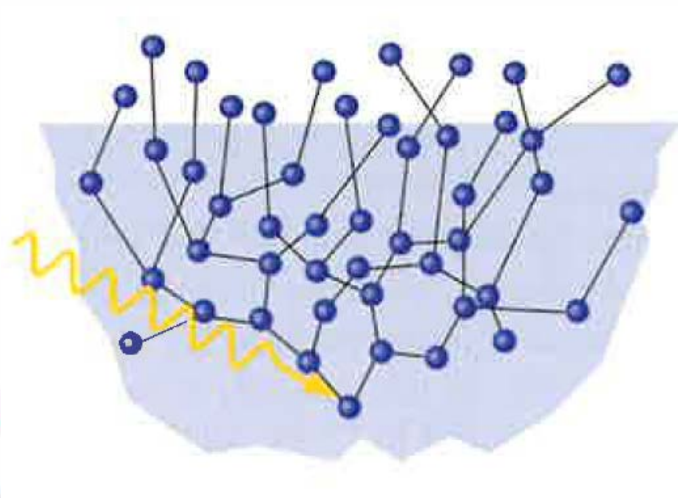
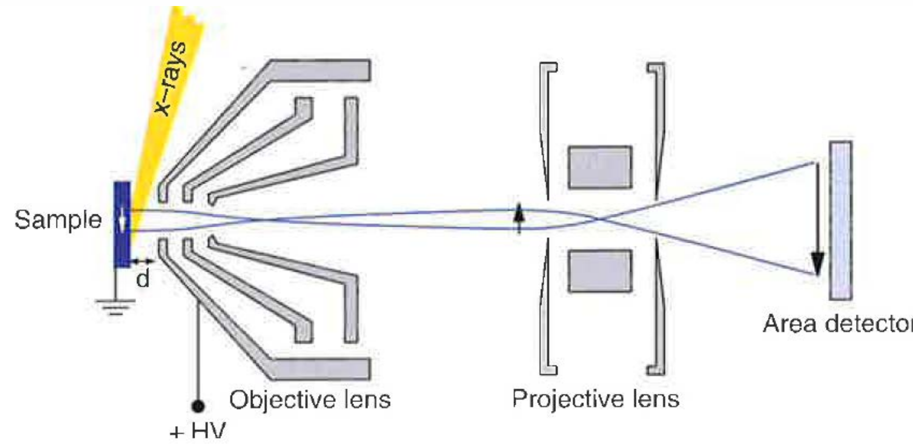
# Mapping nanomagnetism

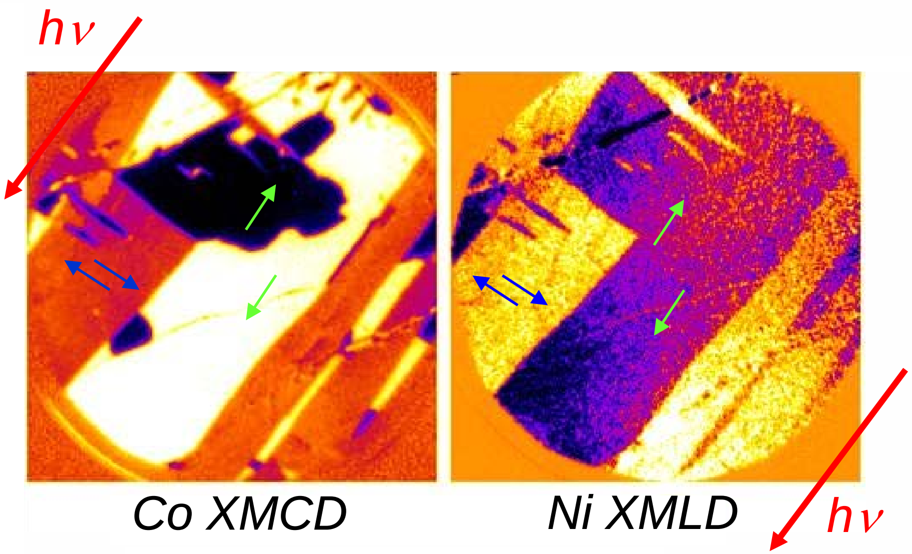
- Resolution to 50 nm with Photoemission Electron Microscopy (PEEM)
- XAS in XANES region produces photoelectrons which create secondary, low-energy (eV) photoelectrons with longer mean-free path. Spatial resolution for imaging these $e^-$ is 10's of nm

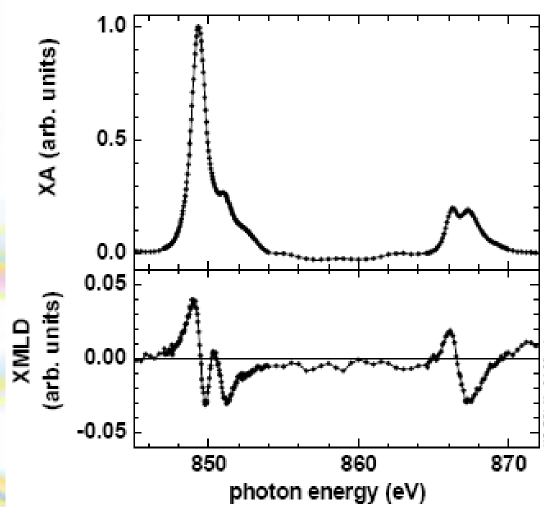




# Mapping nanomagnetism

- PEEM in combination with XMCD, XMLD to map domains to nm lengthscales




- Co/NiO is prototype exchange bias material – need to understand coupling at interface.

- PEEM at I06 with XMCD for (FM) Co L3 and XMLD for (AFM) Ni L2 shows orientation of the two types of moments at the interface is perpendicular – opposite of what was inferred from less precise measurements

*G. van der Laan et al., in prep.*

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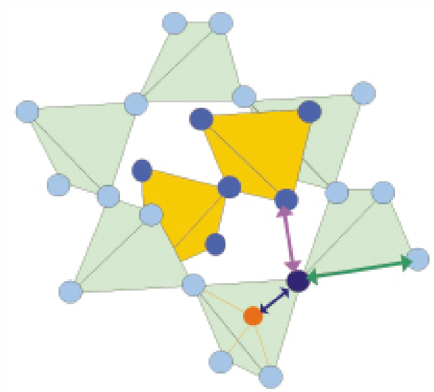
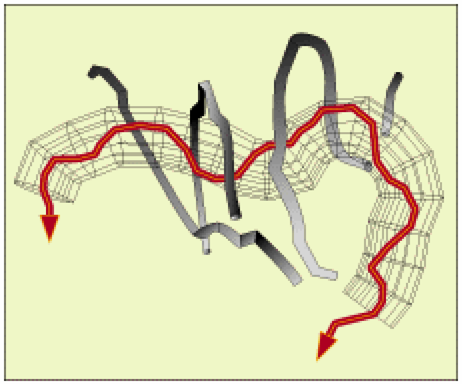
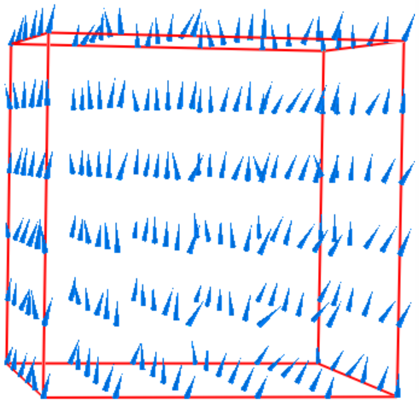
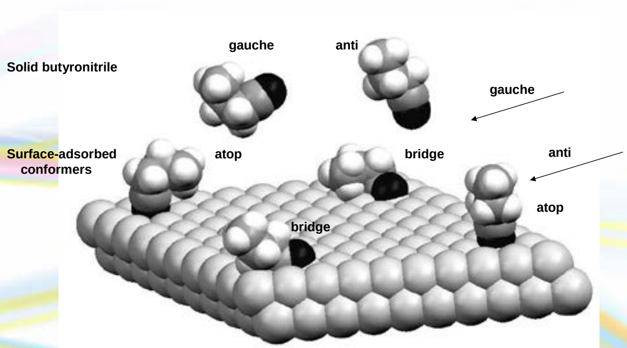
# Higher resolution spectroscopy: atomic and molecular motion in materials

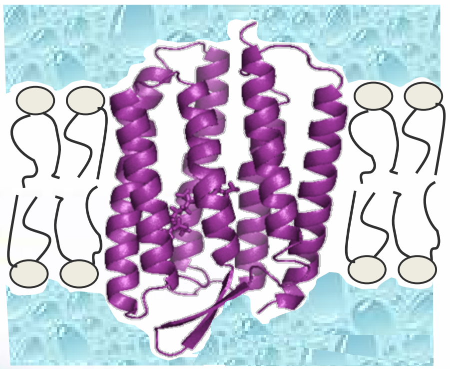
- Vibrations and magnetic excitations in molecules and solids are essential to key processes e.g. thermal conductivity and expansion, polymer processing and properties, biochemical processes, magnetic storage media, superconductors –

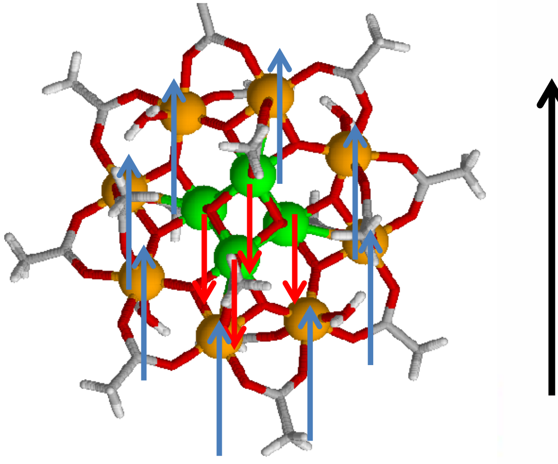








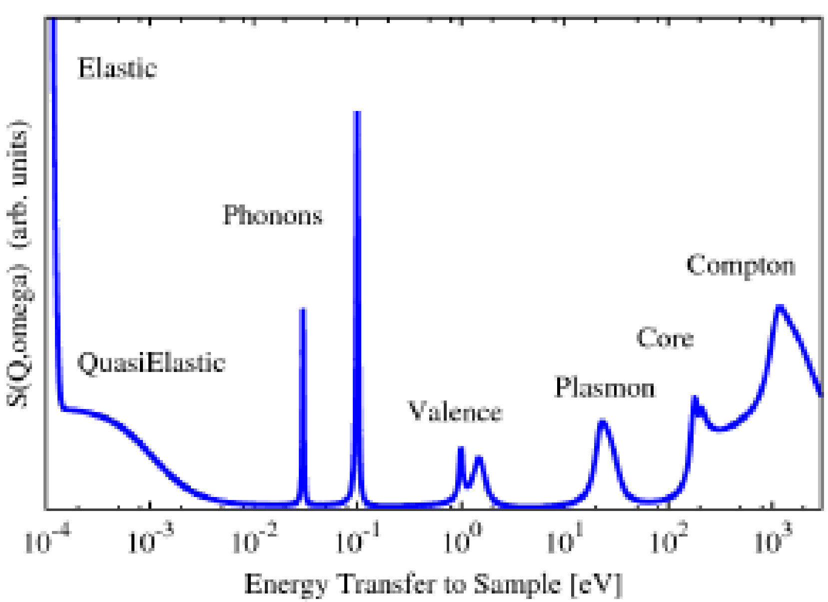



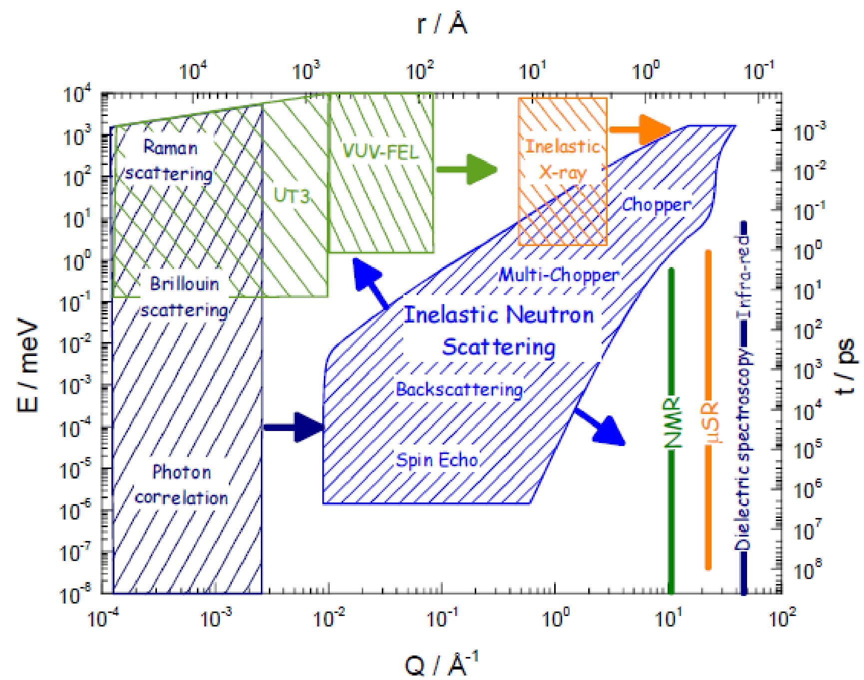


# Atomic and molecular motion in materials

"When a crystal is irradiated with X-rays, the processes of photoelectric absorption and fluorescence are no doubt accompanied by absorption and emission of phonons. The energy changes involved are however so large compared with phonon energies that information about phonon spectrum of the crystal cannot be obtained in this way. The same is true for Compton scattering."[1] W.Cochran, 1966.

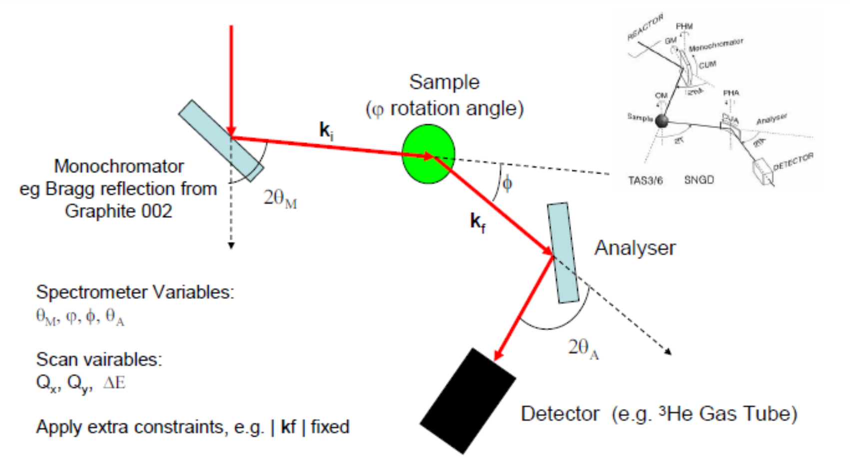
- Typically wish to measure vibrations and magnetic excitations in the range 1-100 meV as a function of q (1/$\lambda$) so the wavelength of the neutrons or X-rays should typically be of the order of 1-2 Å (0.1-0.2 nm) [Q – measured momentum transfer; q – wavevector of the excitation]
- 1 Å corresponds to 12.4 keV for X-rays and 81.8 meV for neutrons so instrumental resolution is ***much*** more challenging for X-rays (10meV: $\Delta E/E \cong 10^{-6}$ vs $10^{-6}$ IXS vs INS)

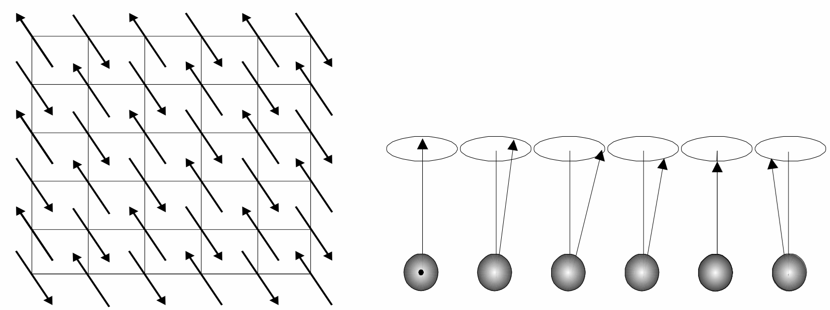


# INS: experimental methods

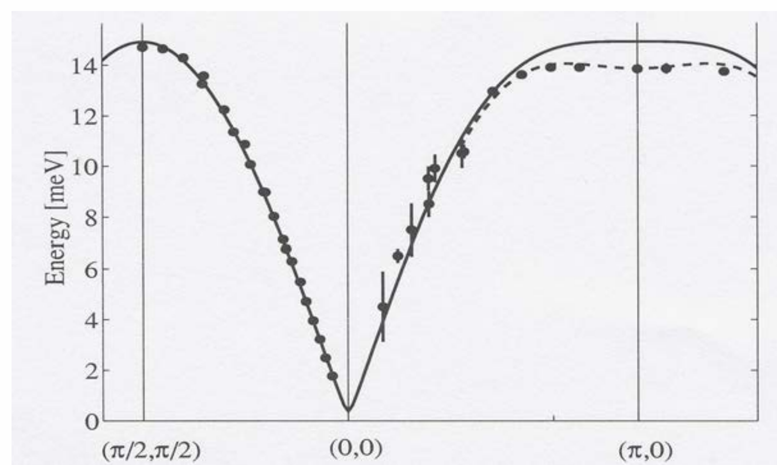
- Triple-axis spectrometer – very versatile means of measuring the change in energy of neutrons scattered by sample as a function of Q (point by point !)



$Q = k_f - k_i$ $\hbar\omega = E_f - E_i$ $E_{f,i} = \hbar^2 k^2/2m_n$

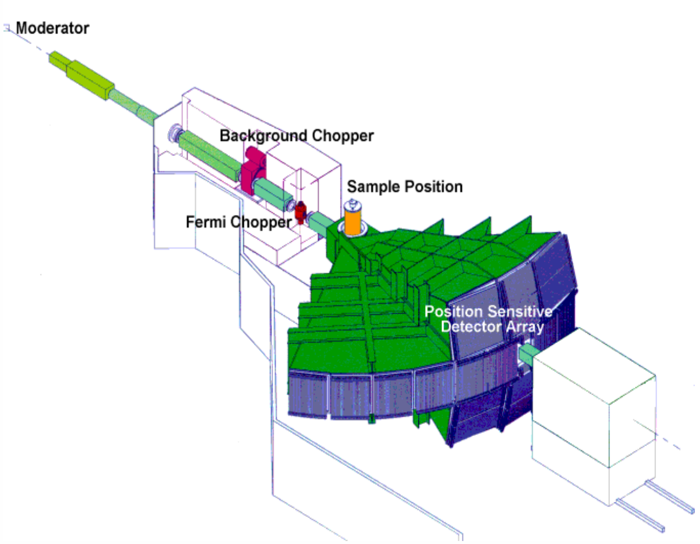
$\lambda=\infty$ $\lambda=2a$

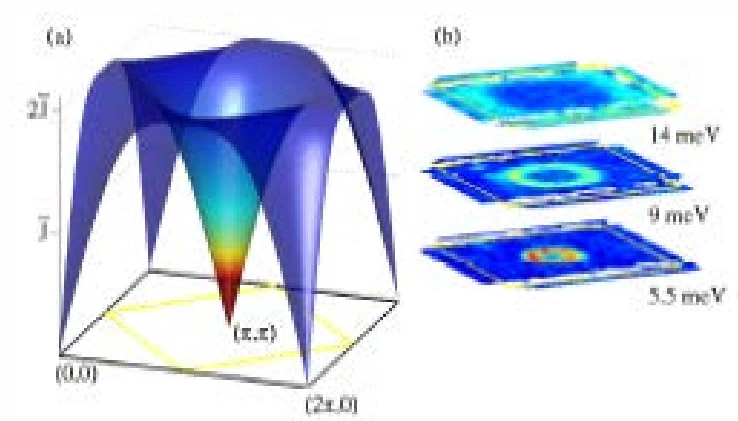


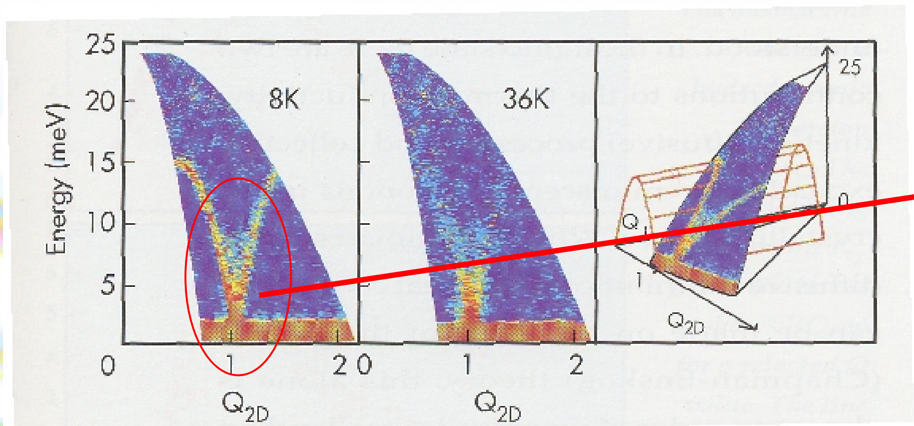

*qa*

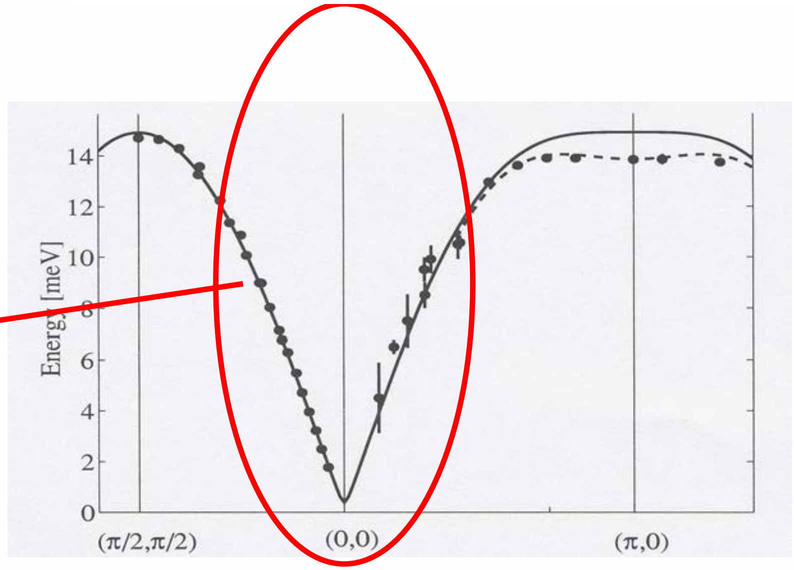
# INS: experimental methods

- With time-of-flight methods and large area detector, can map whole regions of Energy and Q in one fell swoop- often revealing unexpected features



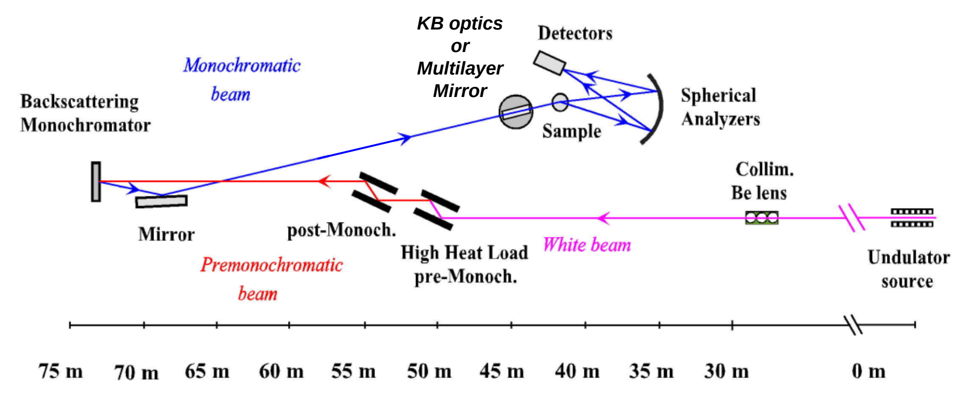



# IXS: experimental methods

- Far more challenging to resolve such excitations by inelastic X-ray scattering

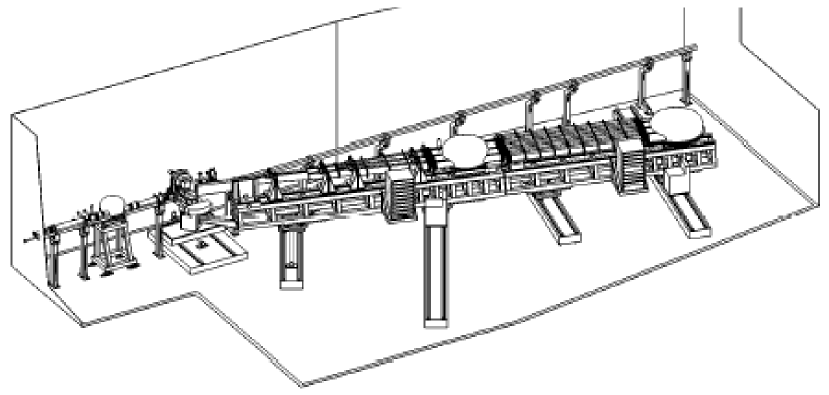
Optimise resolution with monochromator and analyser close to backscattering configurations:

$d\lambda/\lambda = dE/E = \cot\theta d\theta \rightarrow 0$ as $\theta \rightarrow \pi/2$




| Reflection | Energy[keV] | E [meV] | dE/E |
|---|---|---|---|
| Si(7,7,7) | 13.840 | 5.30 | $3.8{\cdot}10^{-7}$ |
| Si(8,8,8) | 15.817 | 4.40 | $2.8{\cdot}10^{-7}$ |
| Si(9,9,9) | 17.794 | 2.20 | $1.2{\cdot}10^{-7}$ |
| Si(11,11,11) | 21.747 | 0.83 | $4.7{\cdot}10^{-8}$ |
| Si(12,12,12) | 23.725 | 0.73 | $3.1{\cdot}10^{-8}$ |
| Si(13,13,13) | 25.704 | 0.50 | $1.9{\cdot}10^{-8}$ |

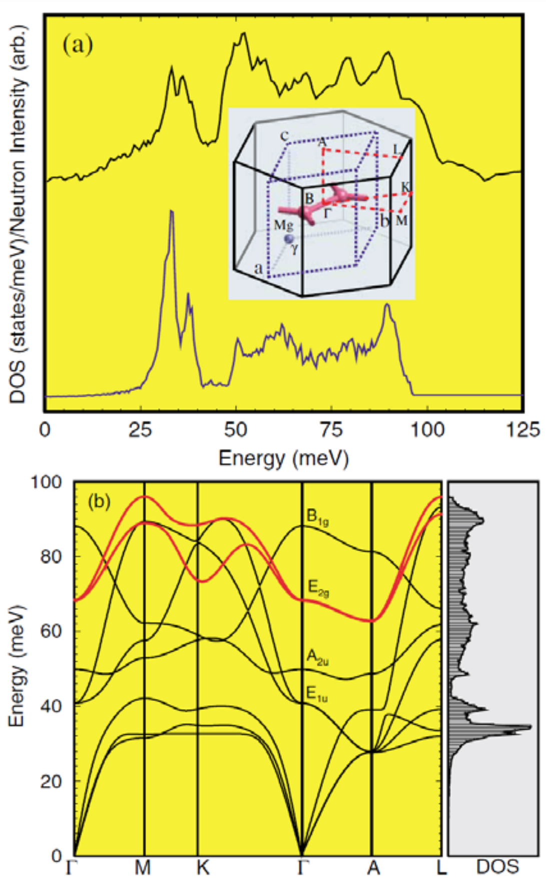
***9- analyser crystal spectrometer***

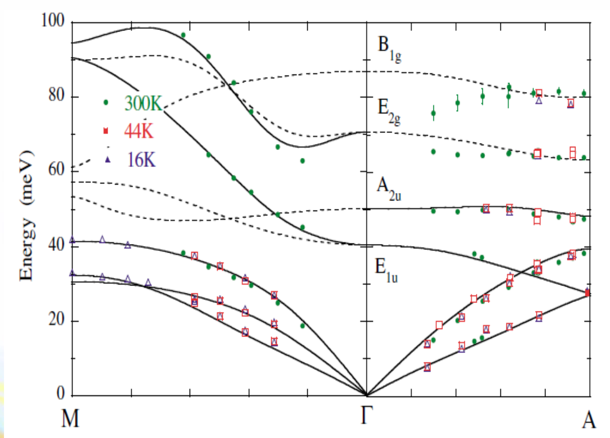


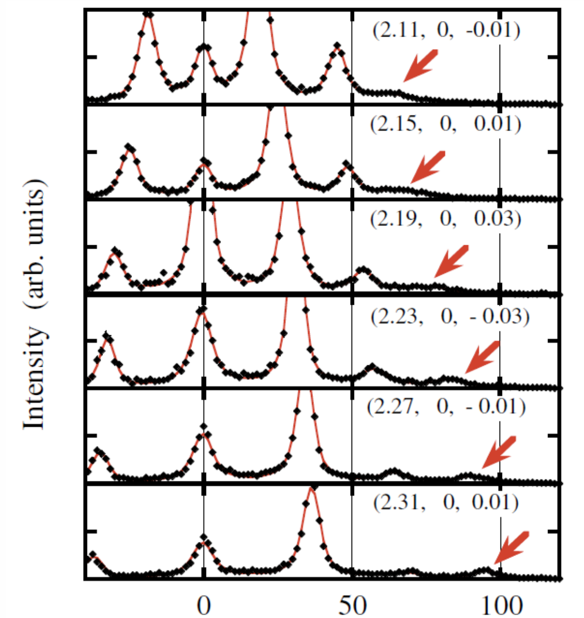
diamond

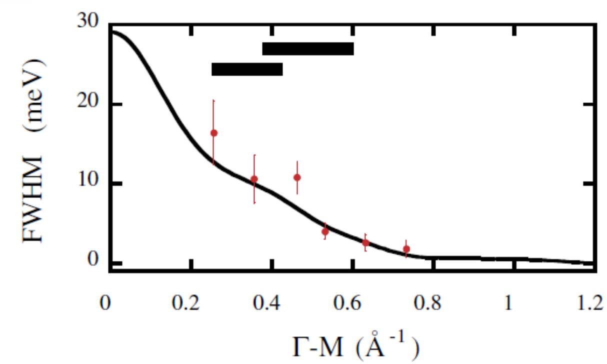
# IXS: applications

- Role of phonons in superconductivity in $MgB_2$ – DOS from lattice dynamics and neutron scattering measurements from powders then IXS on 50 x 70 $\mu^2$ crystals



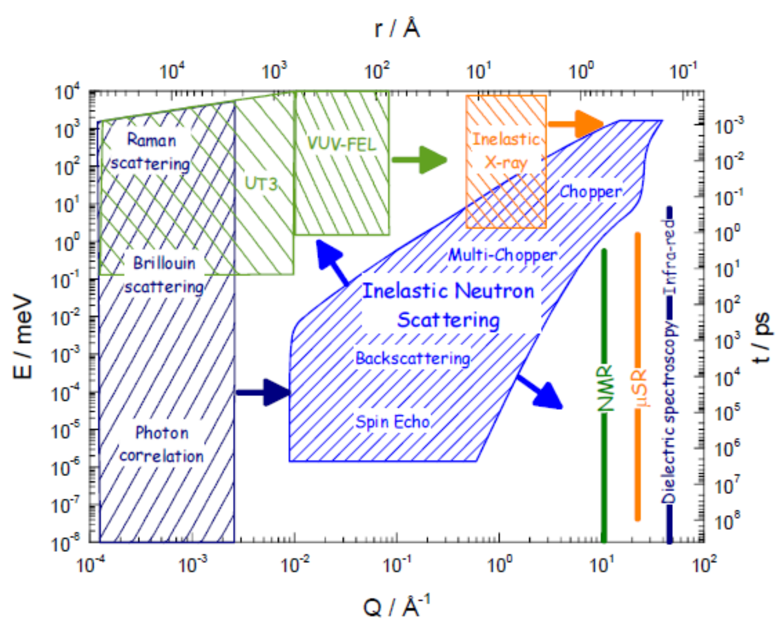



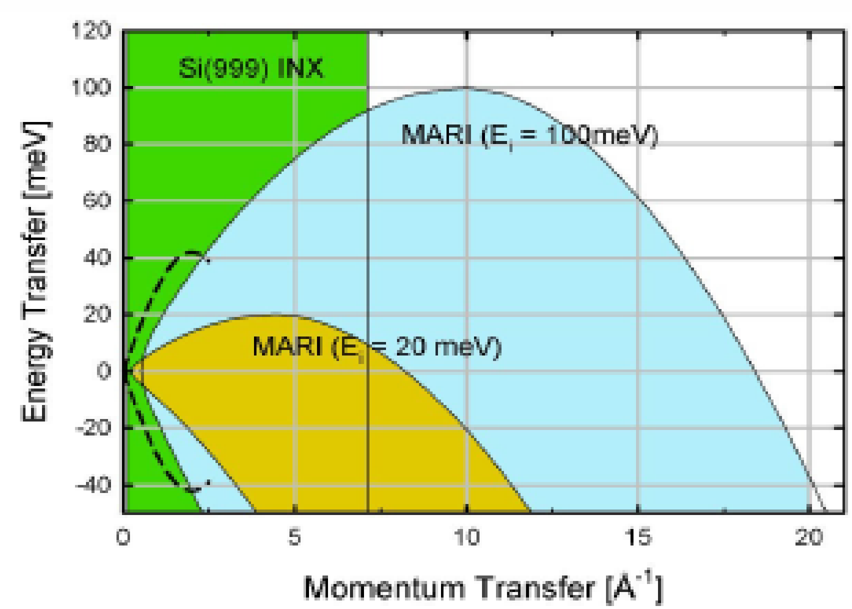




*Yildirim et al, PRL 87 (2001) 37001*
*Baron et al, PRL 92 (2004) 197004*

# IXS vs INS

- Advantages of IXS
  - Fine beams - ~ 10 μm - work with very small samples
  - Resolution is independent of energy transfer as ΔE and Q are decoupled (below)
  - Open up new kinematical window in ΔE, Q at small Q and high ΔE –useful for disordered systems
  - No incoherent scattering
- Disadvantages of IXS
  - Very challenging instrumentation to achieve resolution ΔE/E ~$10^{-7}$
  - Cross-section dominated by photoelectric absorption - less sensitive to light atoms
  - Resolution more complex than for neutrons (non Gaussian)



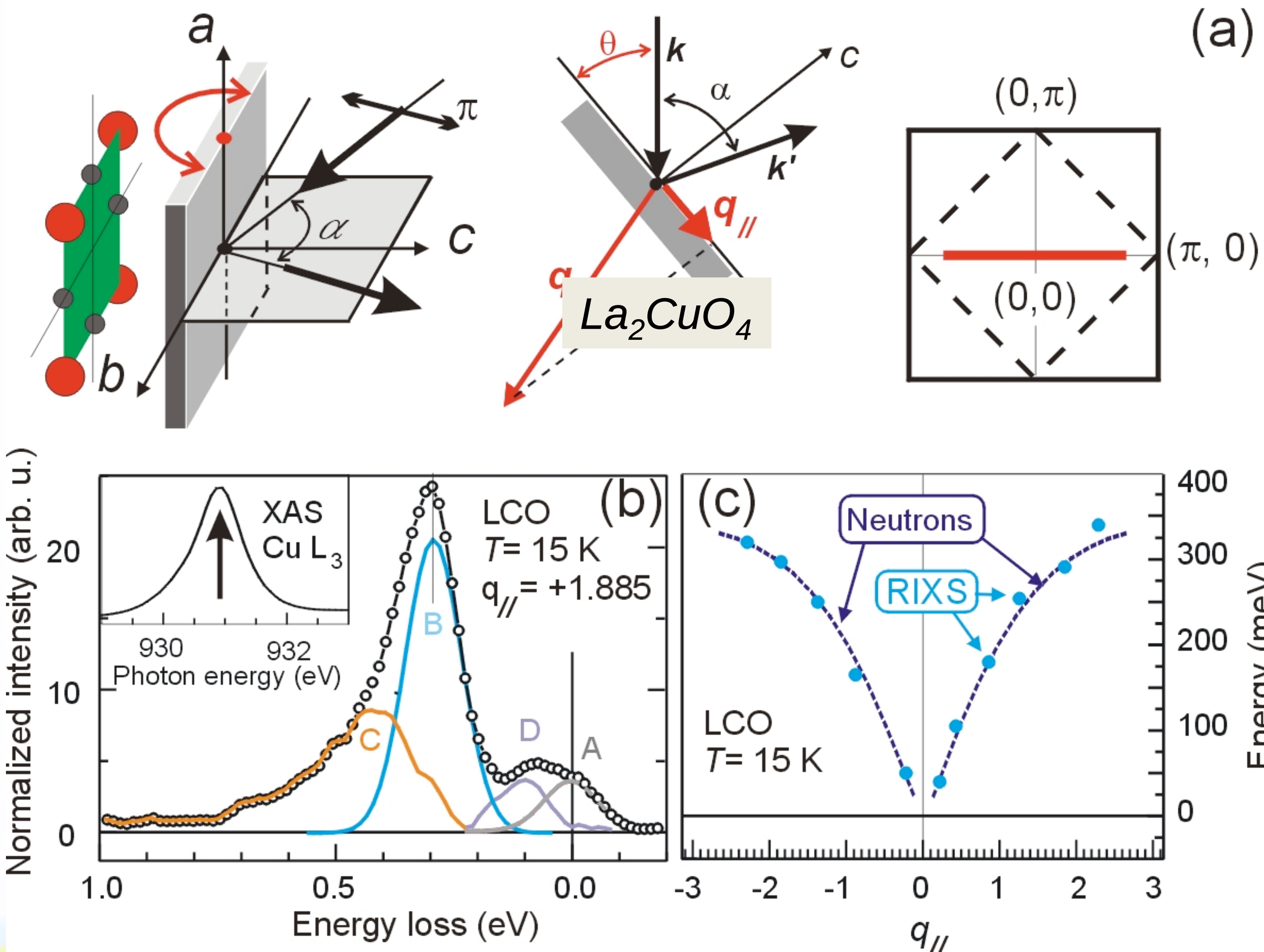



# IXS +R → RIXS: a complementary method to neutrons for studying magnetic excitations

High sensitivity of soft x-rays allows study of small sample volumes.

Tune to specific transition – element and orbital selective

***$La_2CuO_4$***
***100nm films***




*Braicovich et al. PRL **104**, 077002 (2010)*

# RIXS: Analysing spectrometer at the ESRF

ESRF
ERIXS@ID32

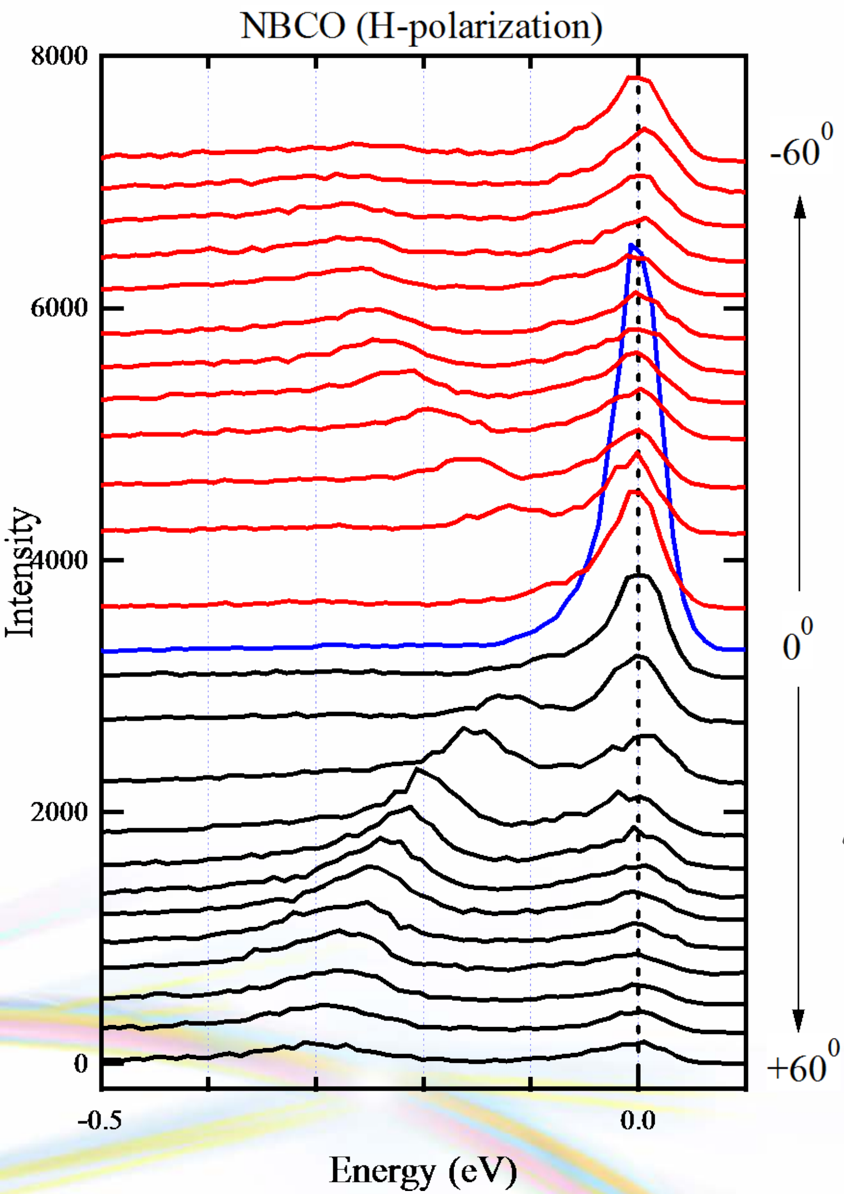
***2015: ERIXS, ID32 – ESRF/Milano***

***L = 12.0 m***

***Design: E/ΔE = 42000 at Cu-$L_3$***

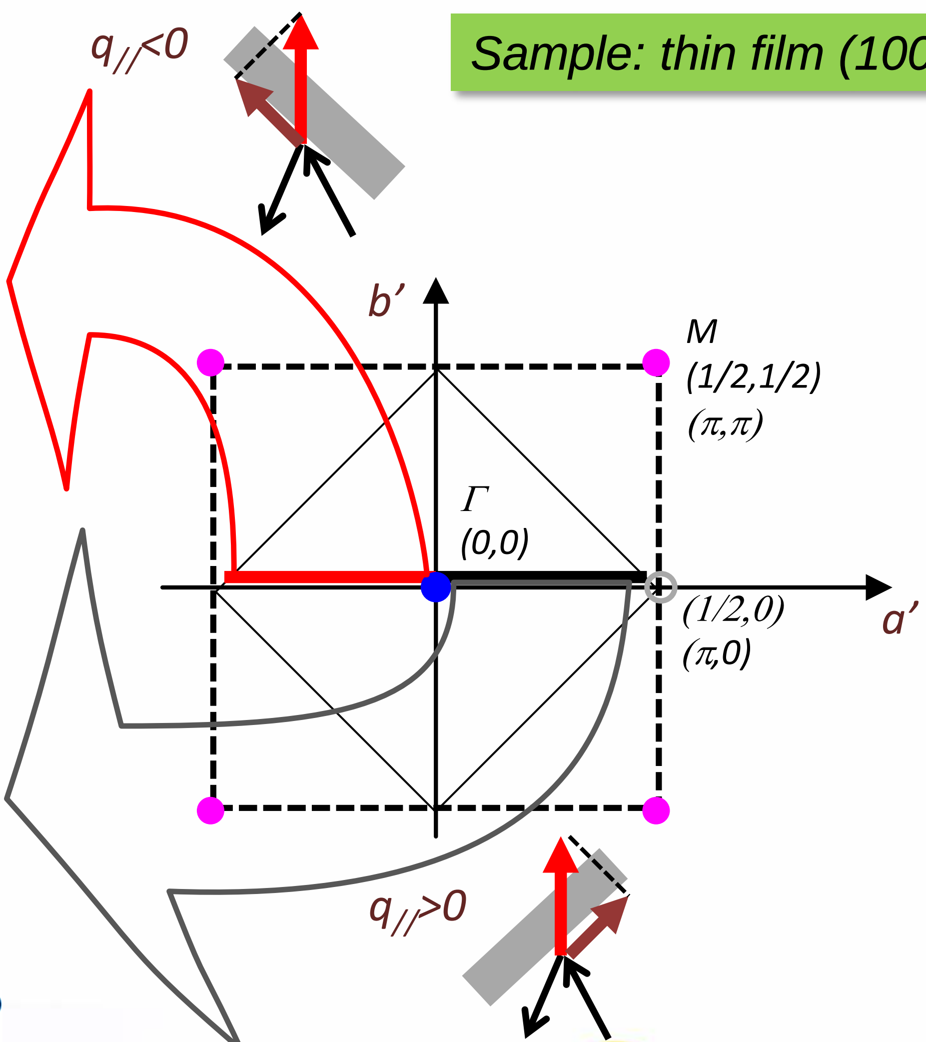
# Antiferromagnetic $NdBa_2Cu_3O_{6+\delta}$






*"Low" resolution set up at ID32 – July 2015*

Sample: thin film (100 nm)



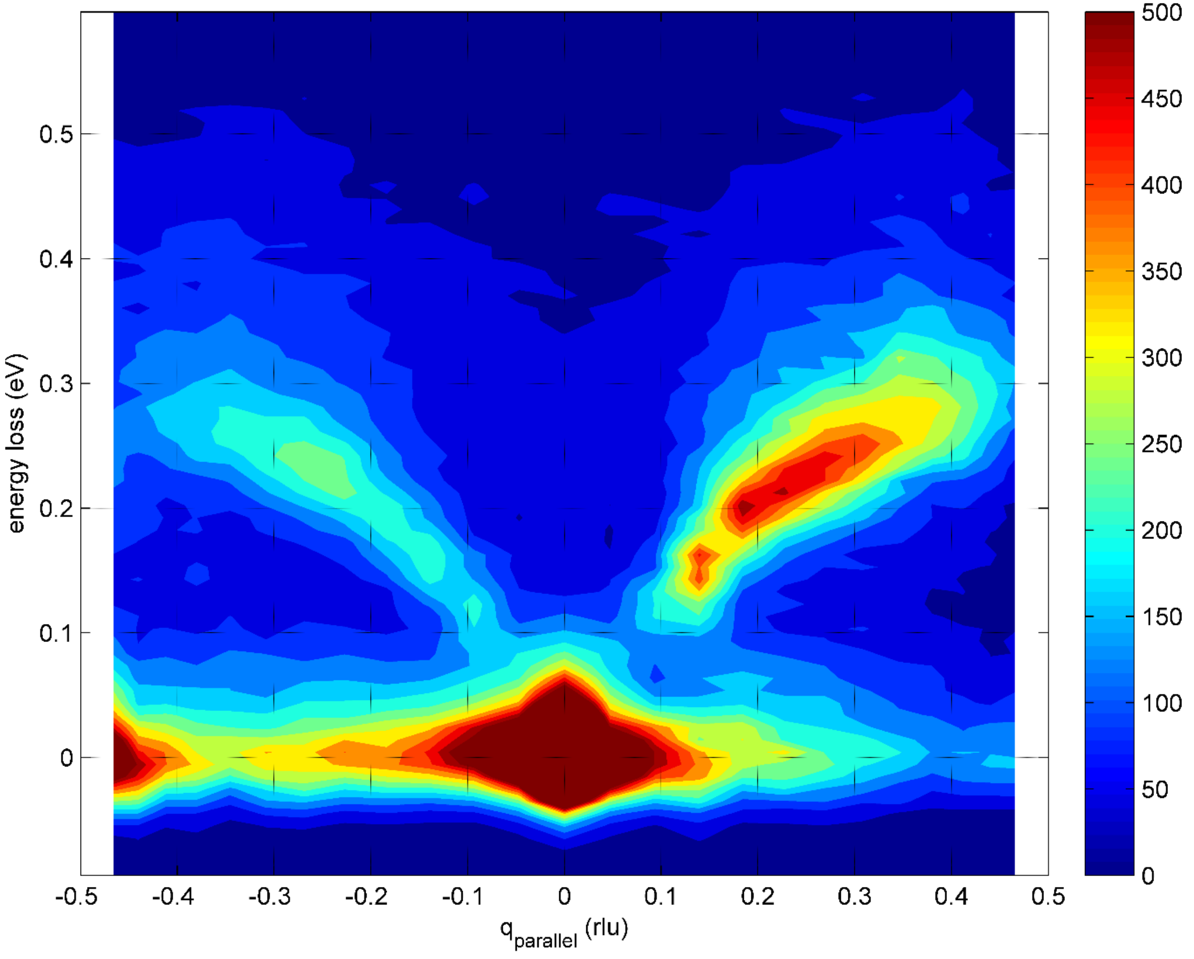

π pol. – spin-flip

Combined BW ≈ 55 meV

# AF $NdBa_2Cu_3O_{6+\delta}$

ESRF
ERIXS@ID32

Sample: thin film (100 nm)

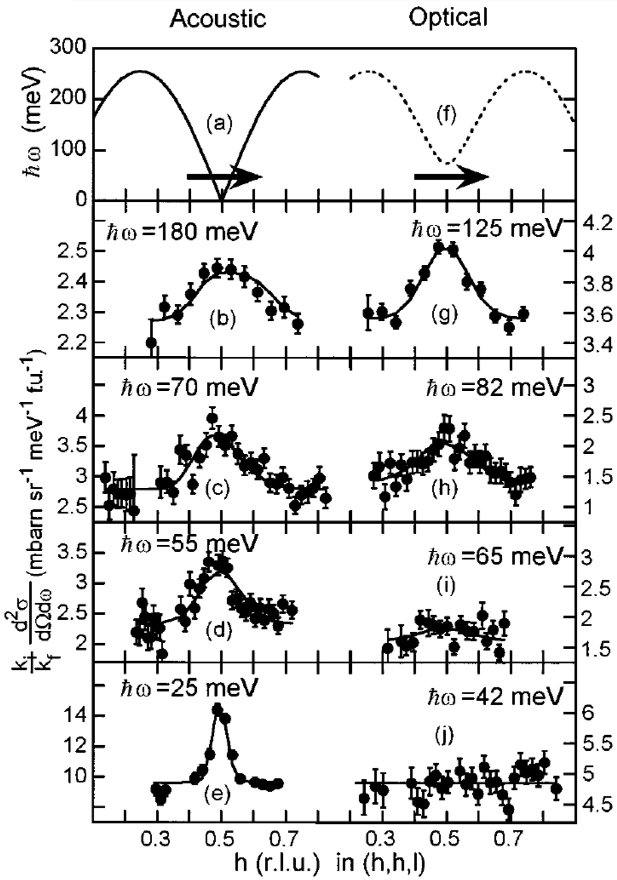



diamond

*ESRF ID32 – July 2015*

# AF YBCO and NBCO: magnon optical branch

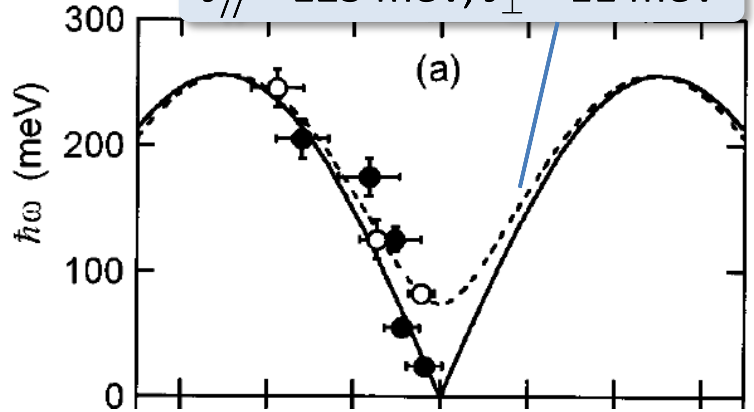
INS: BW ≈ 2 meV




*The sample used in the present investigation was a single crystal of $YBa_2Cu_3O_{6.15}$ with mass 96 g.*

*S. M. Hayden et al, PRB 54 6905 (1996)*

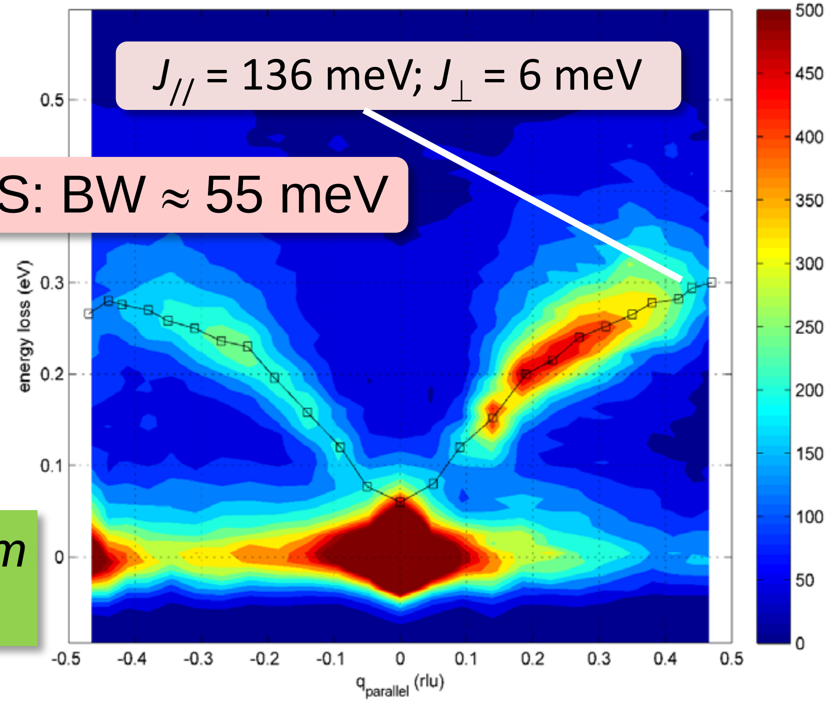
$J_{//}$ = 125 meV; $J_\perp$ = 11 meV




ESRF
*ERIXS@ID32*

$J_{//}$ = 136 meV; $J_\perp$ = 6 meV

RIXS: BW ≈ 55 meV




*Sample: thin film (100 nm)*

*Y.Y. Peng, G. Dellea, M. Minola, G.M. De Luca, M. Salluzzo, M. Le Tacon, B. Keimer, L. Braicovich, N.B. Brookes, and G. Ghiringhelli, unpublished*

# Further reading

- Introductions
  - P. Willmott, *'An introduction to synchrotron radiation',* Wiley 2011
  - J. Als-Nielsen and D.McMorrow, *'Elements of modern X-ray physics'*, Wiley 2011
- Magnetic X-ray scattering
  - S.W.Lovesey and S.P. Collins, '*X-ray scattering and absorption by magnetic materials*', Oxford University Press, 1996
  - G. van der Laan, C.R. Physique 9 (2008) 570
  - Look out the Paolasini review
  - T. Bruckel review
- Inelastic X-ray scattering
  - *A.Q.R. Baron, Introduction to High-Resolution Inelastic X-Ray Scattering, arXiv:1504.01098 [cond-mat.mtrl-sci*
  - Rev. Mod Phys. review

diamond

# Neutrons and X-rays

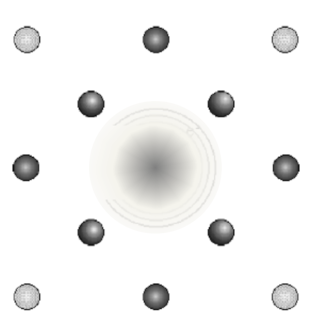
Andrew Harrison, Diamond Light Source

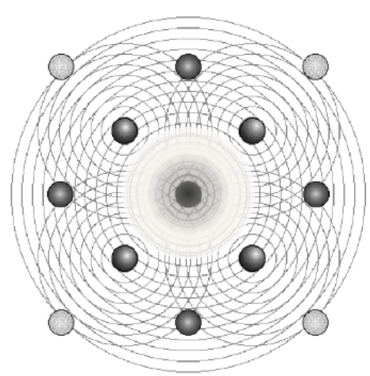
15th Oxford School on Neutron Scattering - 2017

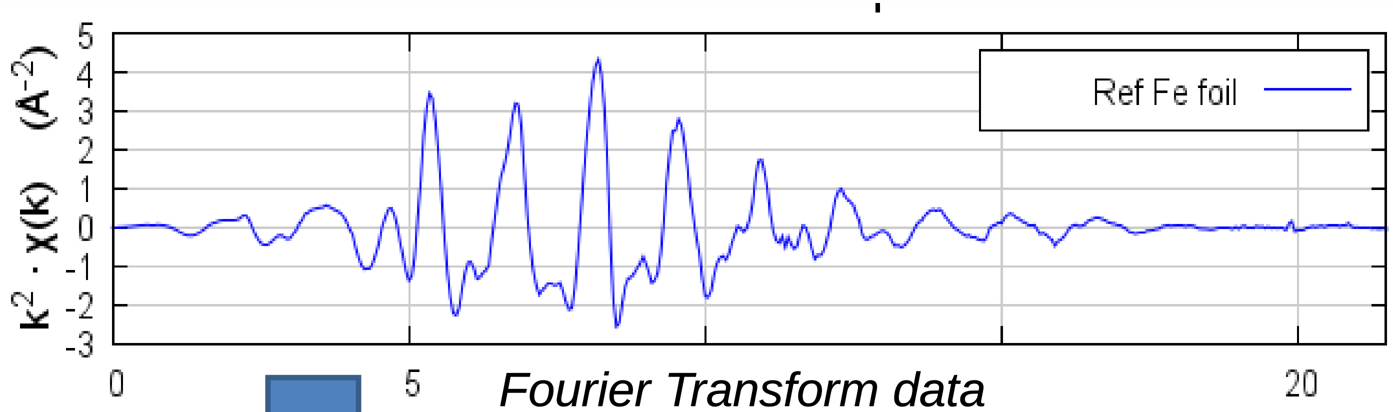
# EXAFS

- Extended X-Ray Absorption Fine Structure

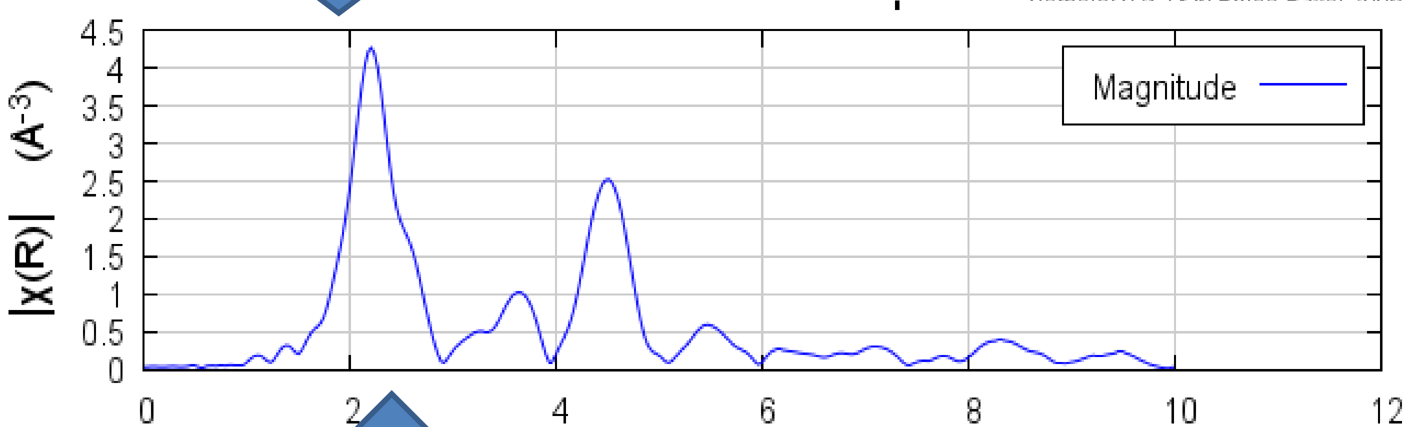
*EXAFS Oscillations come from photo-electron scattering and interference in the material*








*Fourier Transform data*

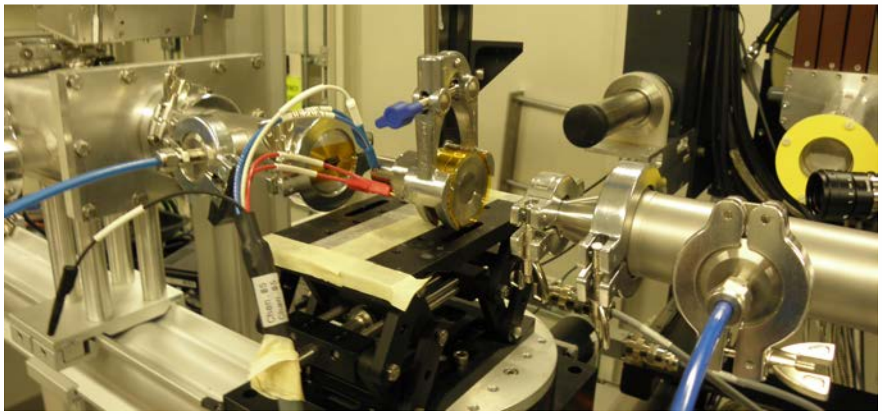


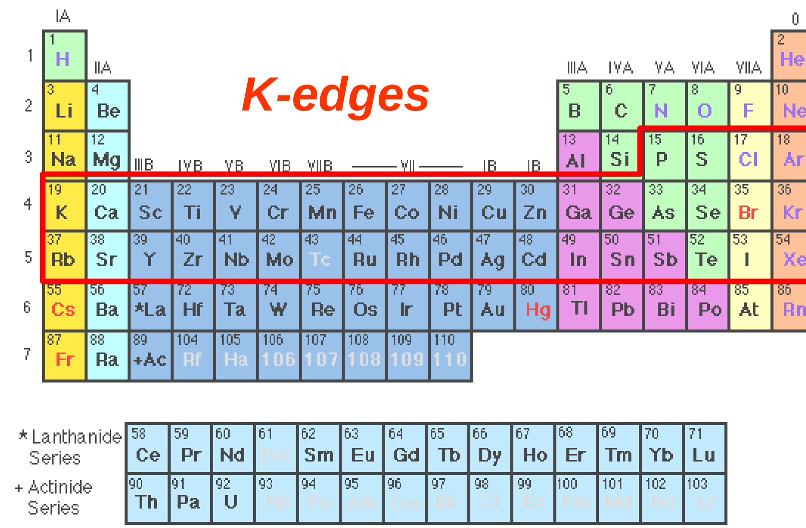

*Detecting Fe atoms AROUND absorber Fe*
*Position: At about 2.5 Angstrom*
*Coordination: 8 next neighbours*

diamond

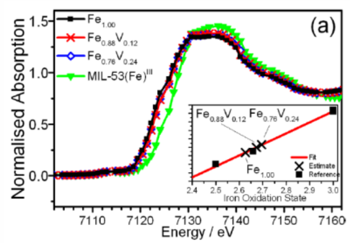
# B18 – general purpose XAS

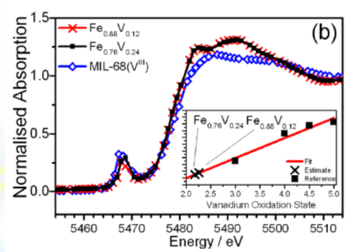
- 2.05 – 35 keV – covers absorption edge from P to U with 200 x 250 μm spot size



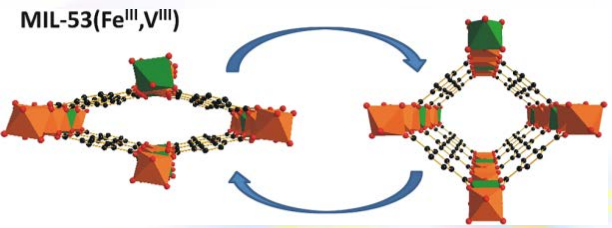


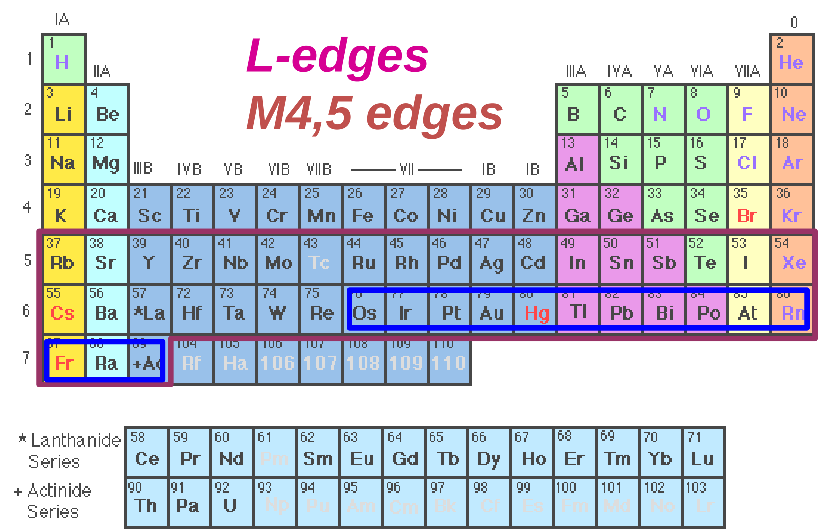

*Determining oxidation states of Fe and V in flexible mixed valent MOF by XANES*



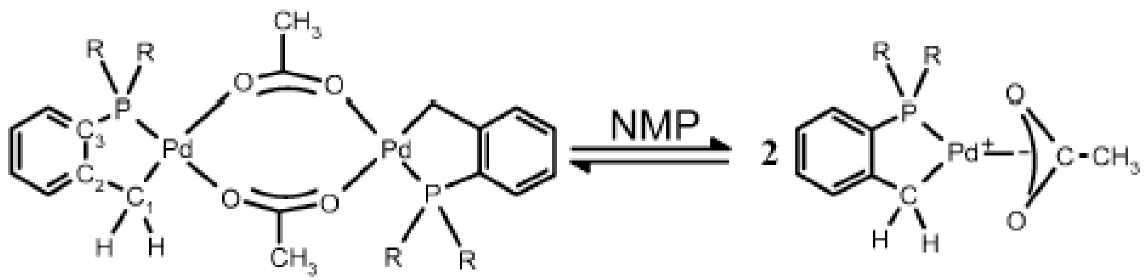

MIL-53($Fe^{III}$,$V^{III}$)



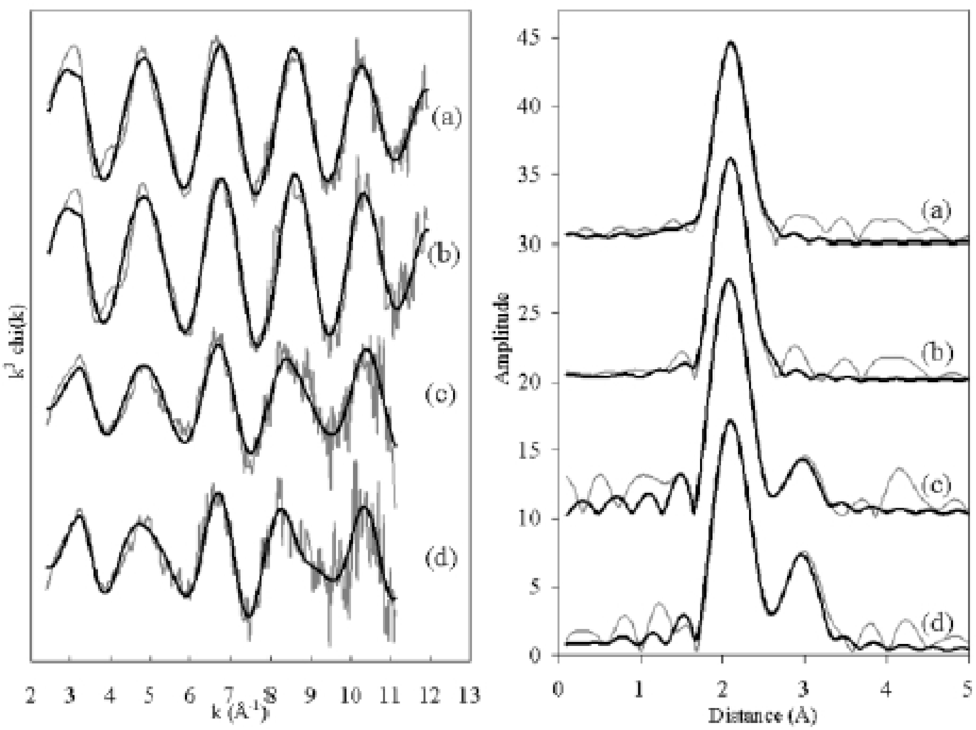



*Breeze et al, Inorganic Chemistry, 52 (2013) 8171*

# What the Heck ?




**1a** **1b**




*5000ppm*

*500ppm*

*150ppm*

*50ppm*

*Future limits ?*
*I20 at Diamond*
*will give ppb*
*of chemical*
*species*

diamond

*S. G. Fiddy et al, Chem. Commun., 2682 (2003)*