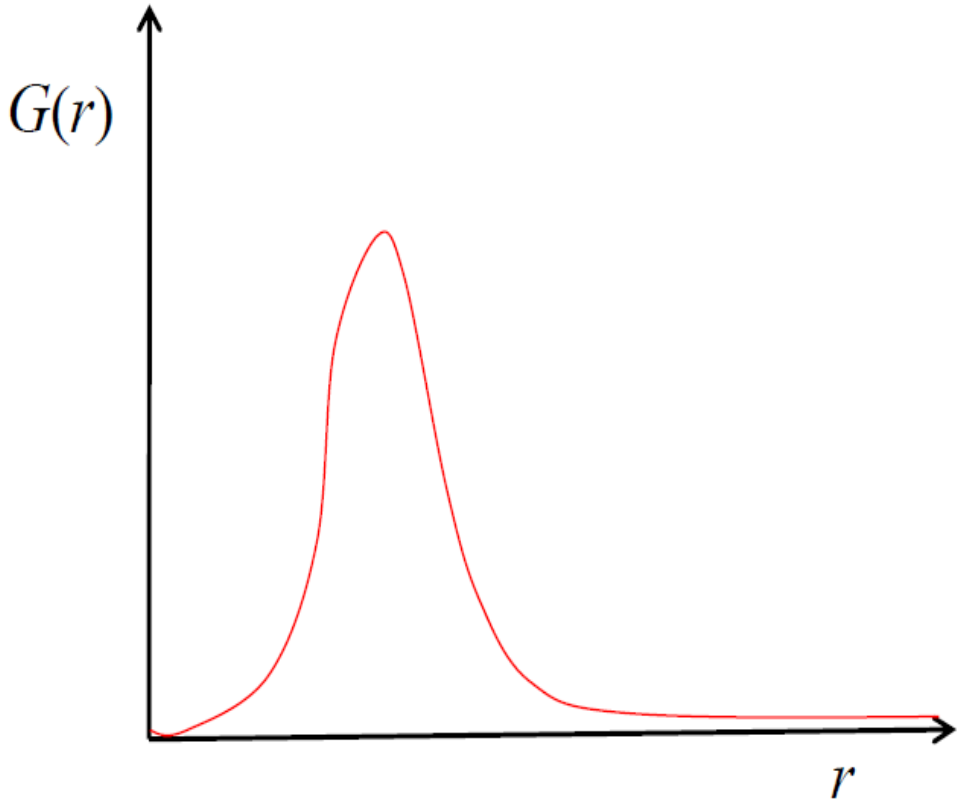
Neutrons and X-rays

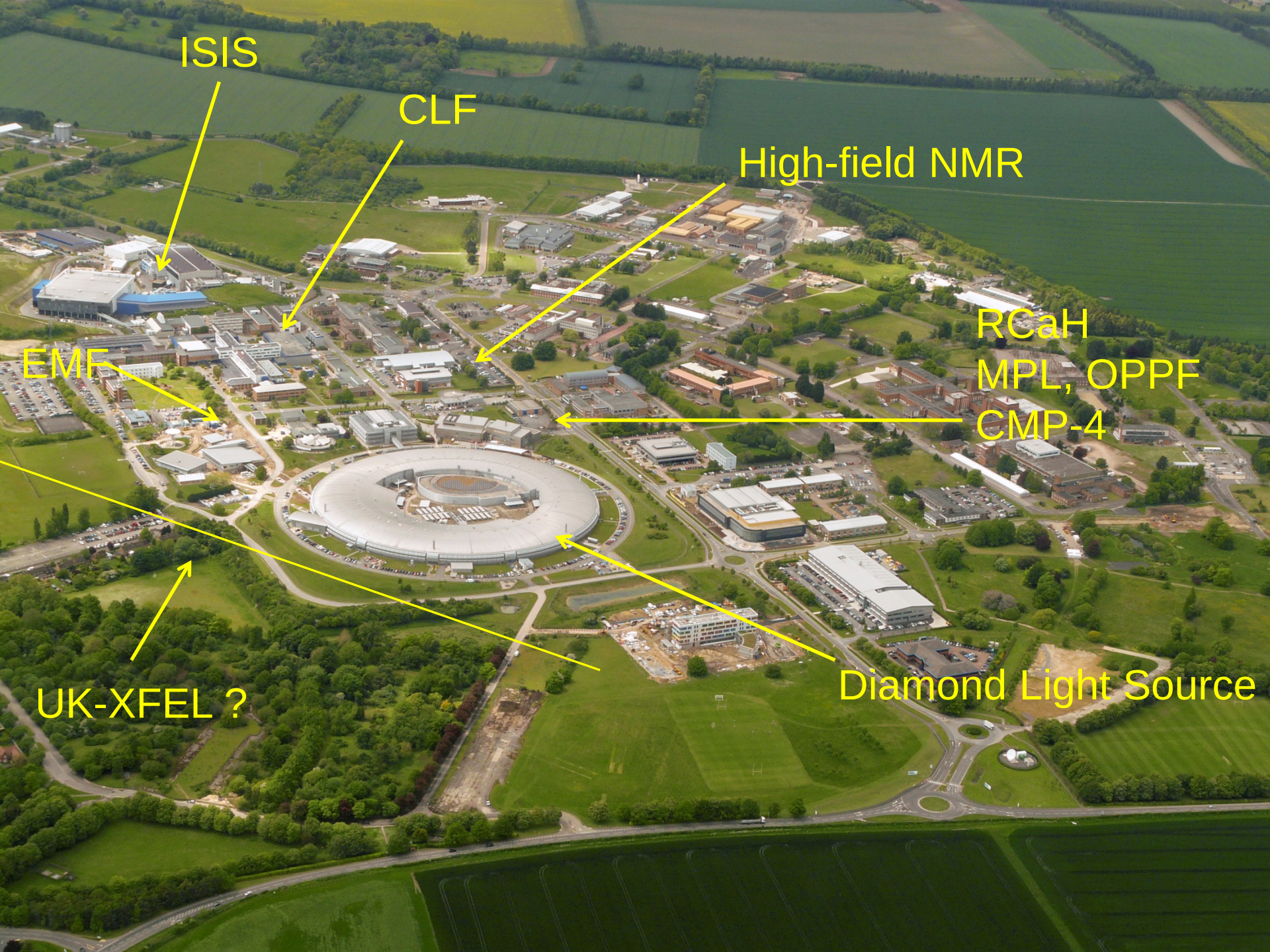
Andrew Harrison, Diamond Light Source

14th Oxford School on Neutron Scattering - 2015

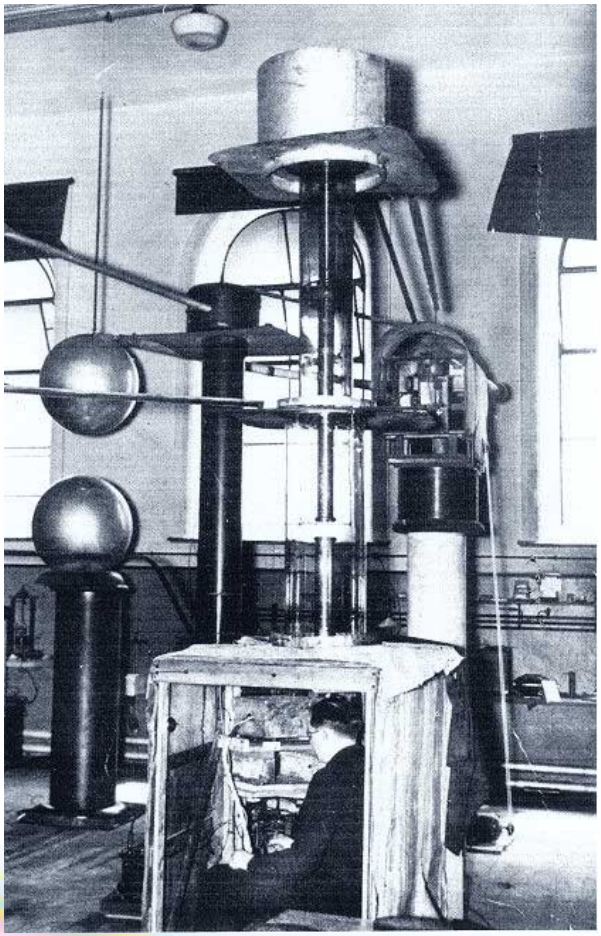
Neutron source – synchrotron distribution

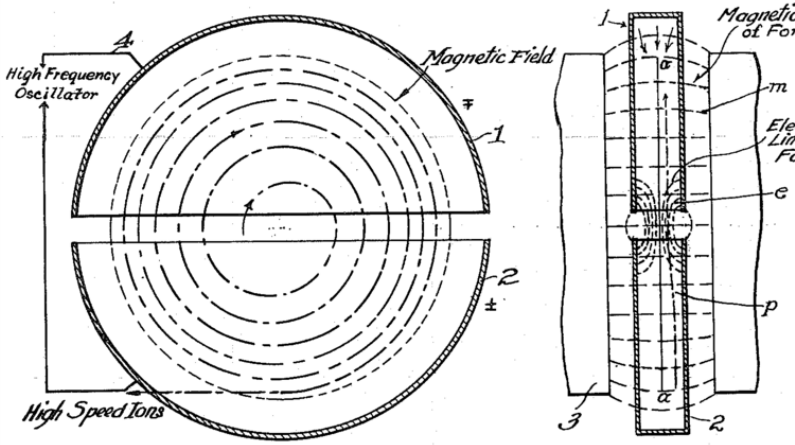


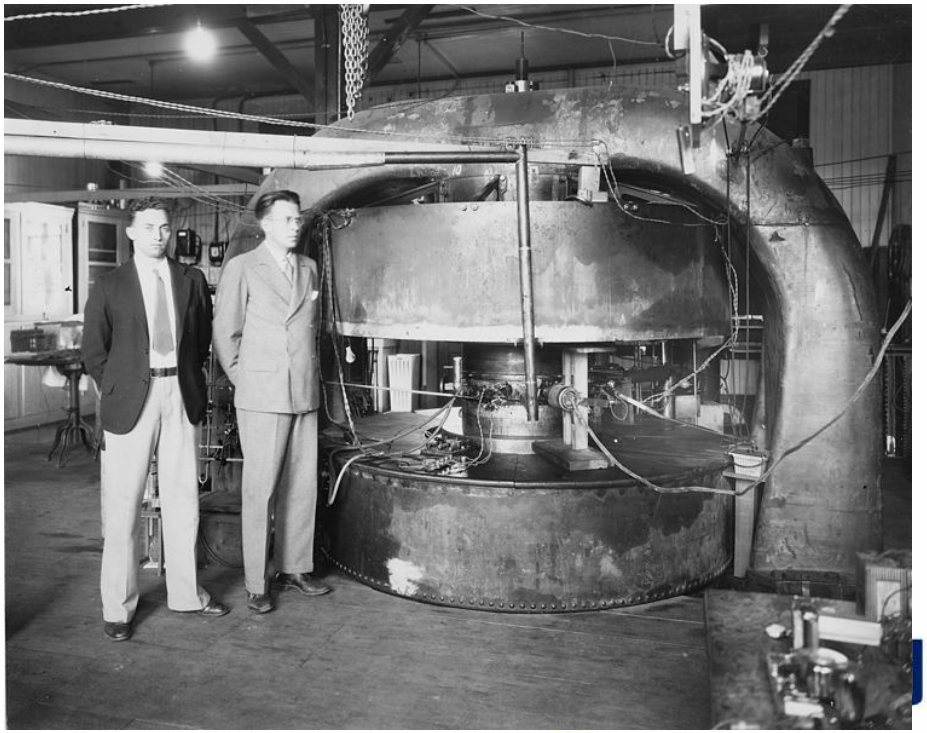
Acknowledgement: Jon Goff


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

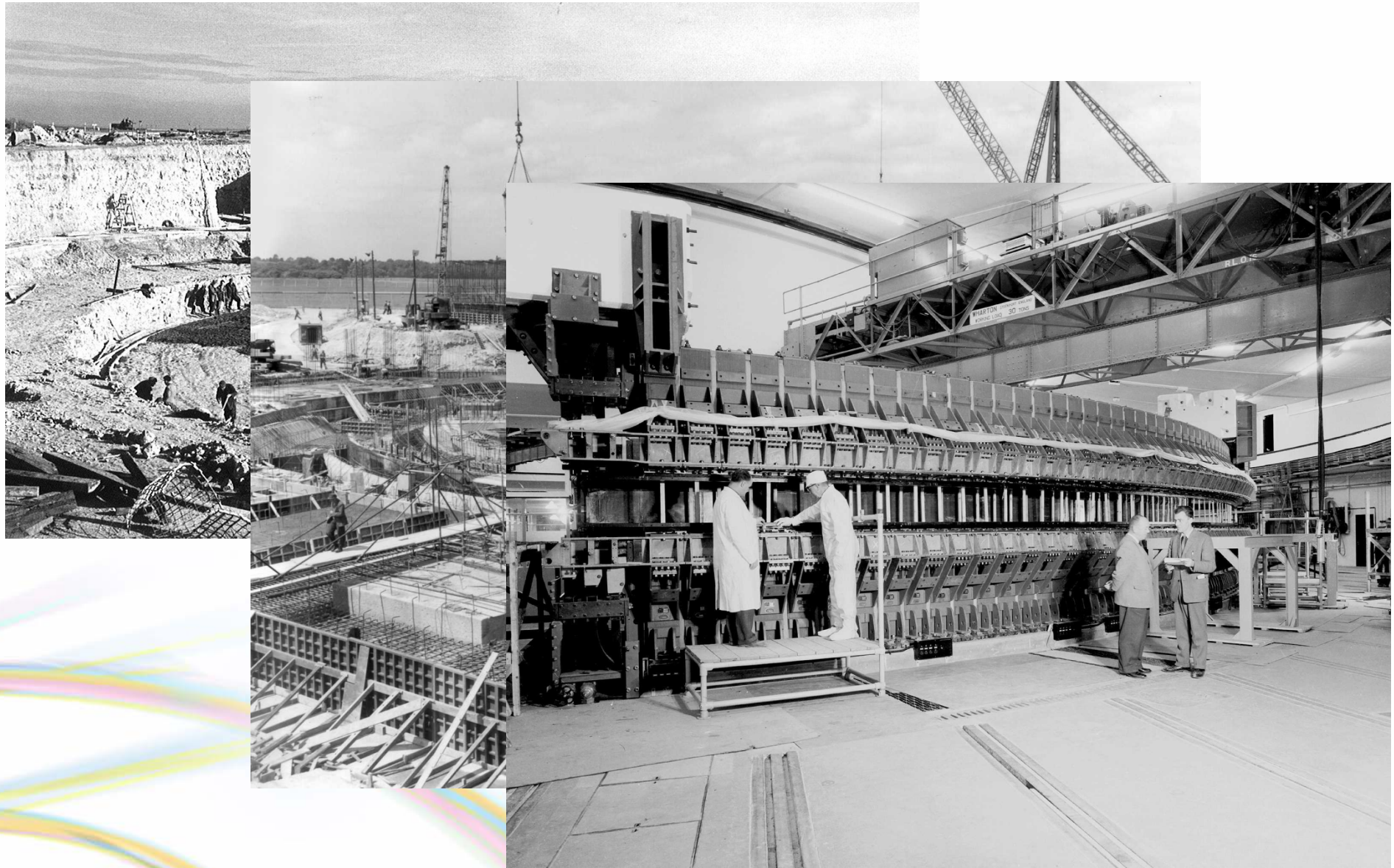
Origins




4
High Frequency Oscillator
Magnetic Field
1
2
High Speed Ions
1
m
e
p
3
2



Ever increasing circles



Ever increasing circles

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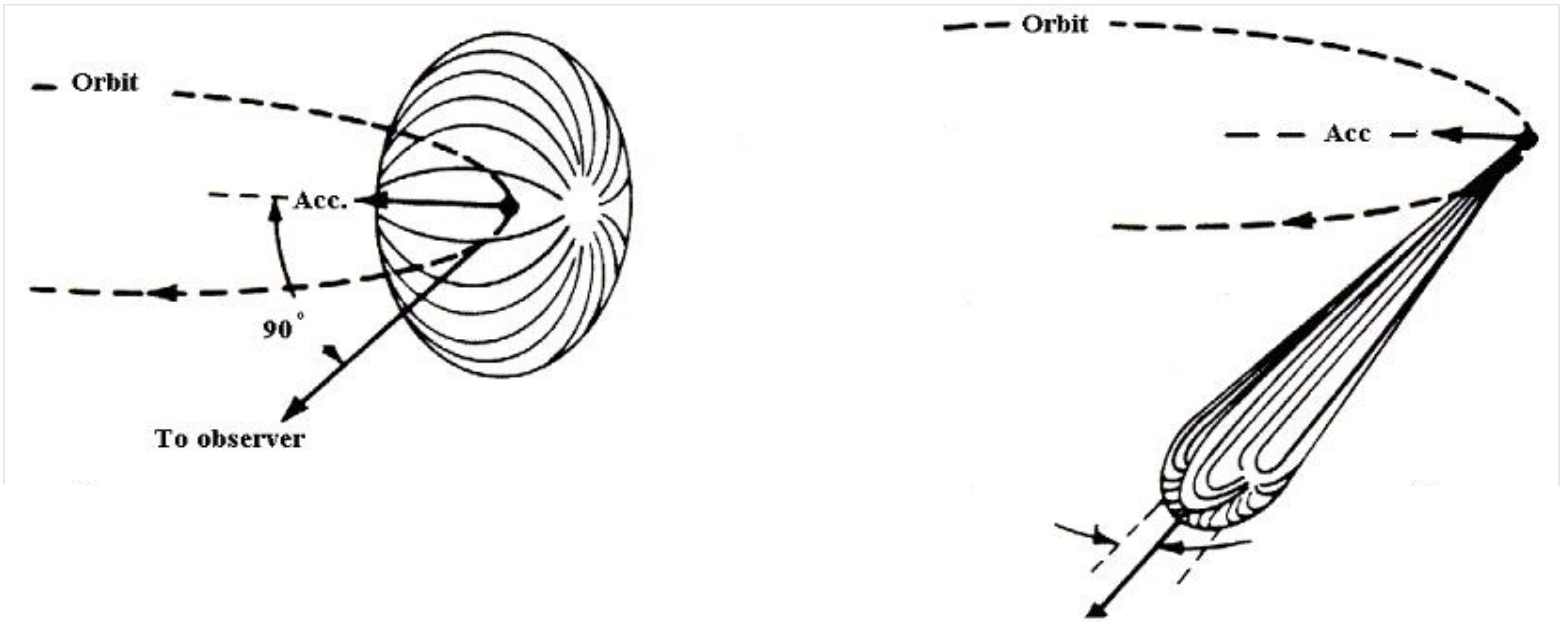
From Maxwell to synchrotrons



$v \sim c$

diamond

First observation ?

Radiation from Electrons in a Synchrotron

F. R. Elder, A. M. Gurewitsch, R. V. Langmuir, and H. C. Pollock
Research Laboratory, General Electric Company, Schenectady, New York
May 7, 1947

HIGH energy electrons which are subjected to large accelerations normal to their velocity should radiate electromagnetic energy.[1–4] The radiation from electrons in a betatron or synchrotron should be emitted in a narrow cone tangent to the electron orbit, and its spectrum should extend into the visible region. This radiation has now been observed visually in the General Electric 70-Mev synchrotron.[5] This machine has an electron orbit radius of 29.3 cm and a peak magnetic field of 8100 gausses. The radiation is seen as a small spot of brilliant white light by an observer looking into the vacuum tube tangent to the orbit and toward the approaching electrons. The light is quite bright when the x-ray output of the machine at 70 Mev is 50 roentgens per minute at one meter from the target and can still be observed in daylight at outputs as low as 0.1 roentgen.

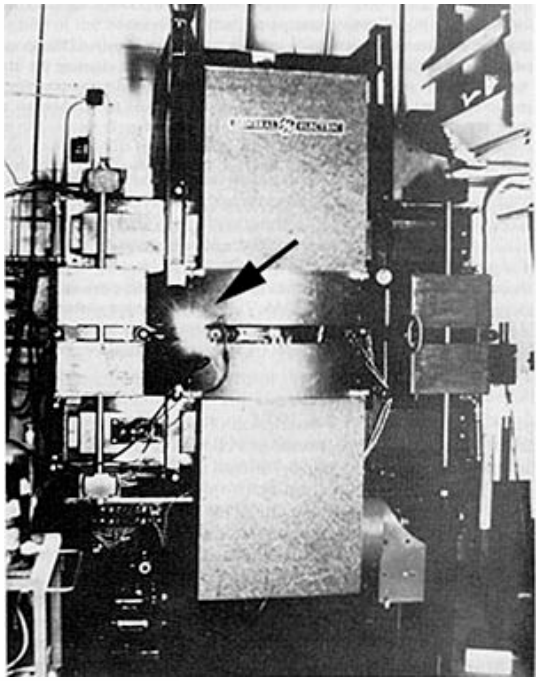
The synchrotron x-ray beam is obtained by turning off the r-f accelerating resonator and permitting subsequent changes in the field of the magnet to change the electron orbit radius so as to contract or expand the beam to suitable targets. If the electrons are contracted to a target at successively higher energies, the intensity of the light radiation is observed to increase rapidly with electron energy. If, however, the electrons are kept in the beam past the peak of the magnetic field and then expanded to a target, the intensity of the radiated light appears to be independent of the energy at which the electrons are removed from the beam. This is to be expected, for in a given machine the radiation is proportional to the fourth power of the electron energy. The light radiation is not observed if the beam is contracted before its energy is about 30 Mev. When the electron beam has been accelerated to the peak of the magnetic field and then decelerated to low energy, a rough measurement of the phase angle over which the light was visible gave a value of 90–100 degrees. The light was viewed through a slotted disk rotating at synchronous speed.

If the r-f resonator is turned off a short time before the peak of the magnetic field, the electron beam slowly contracts to a radius just larger than that of the interior target and then expands as the magnetic field decreases. In this case, the observer no longer sees a single point of light but a short line with extension in the plane of the orbit.

The light emitted from the beam is polarized with the electric vector parallel to the plane of the electron orbit. It disappears as the observer rotates a piece of Polaroid before the eye through ninety degrees. An investigation of the spectral distribution of the energy is in progress and will be reported.

This work has been supported by the Office of Naval Research under contract N5ori–178.

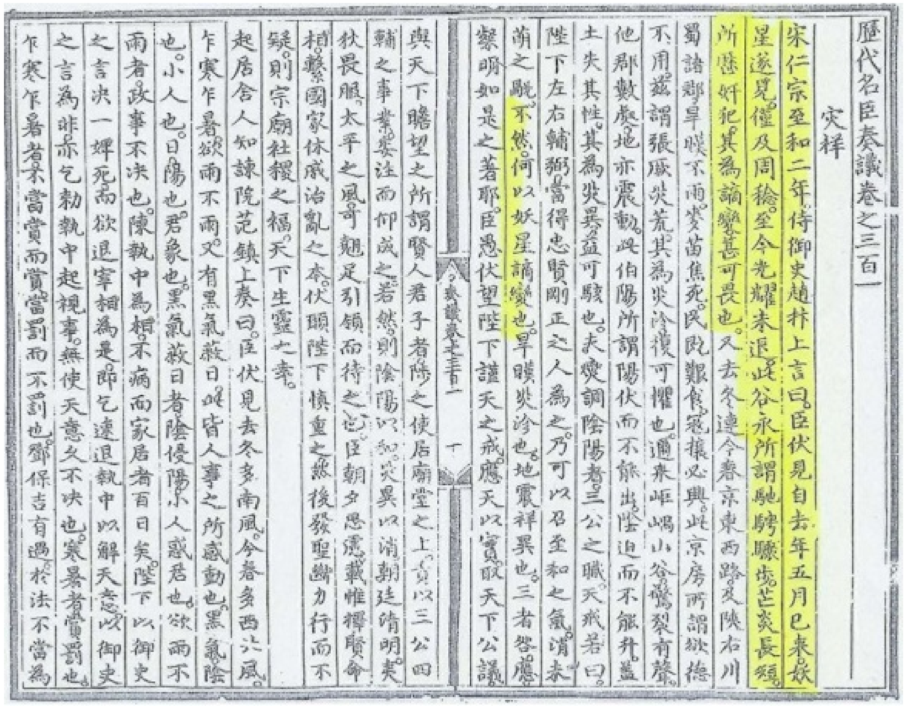
[1] D. Iwanenko and I. Pomeranchuk, Phys. Rev. **65**, 343 (1944).
[2] J. P. Blewett, Phys. Rev. **69**, 87 (1946).
[3] L. I. Schiff, Rev. Sci. Inst. **17**, 6 (1946).
[4] J. S. Schwinger, Phys. Rev. **70**, 798 (1946).
[5] H. C. Pollock *et al.*, Phys. Rev. **70**, 798 (1946).



1946

diamond

First observation ?



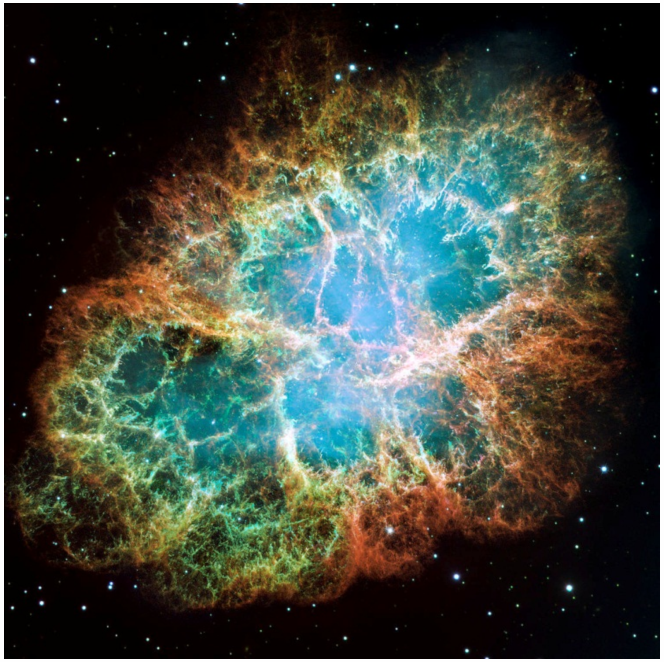
歷代名臣奏議卷之三百一

災祥

宋仁宗至和二年侍御史趙抃上言曰臣伏見自去年五月已來妖
星遂見僅及周歲至今光耀未退此谷永所謂馳騁驟步芒炎長緩
所懸奸犯其為譎變甚可畏也又去冬逹今春京東西路及陝右川
蜀諸郡旱暵不雨麥苗焦死民既艱食寇攘必興此京房所謂欲德
不用茲謂張厥災荒其為災沴復可懼也適來峄嵎山谷驚裂有聲
他郡數處地亦震動此伯陽所謂陽伏而不能出陰迫而不能升蓋
土失其性其為災異益可駭也夫燮調陰陽者三公之職天戒若曰
陛下左右輔弼當得忠賢剛正之人為之乃可以召至和之氣消未
萌之貺不然何以妖星譎變也旱暵災沴也地震祥異也三者咎應
黎明如是之著耶臣愚伏望陛下謹天之戒應天以實敢天下公議

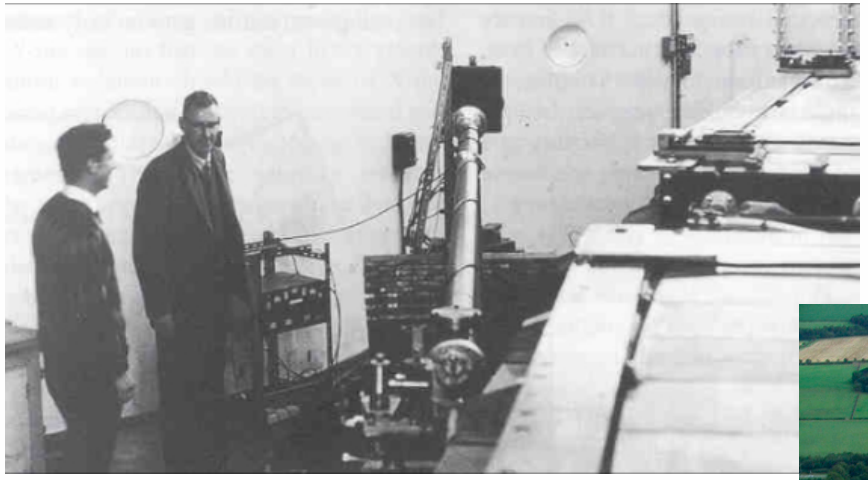
奏議卷三百一　十

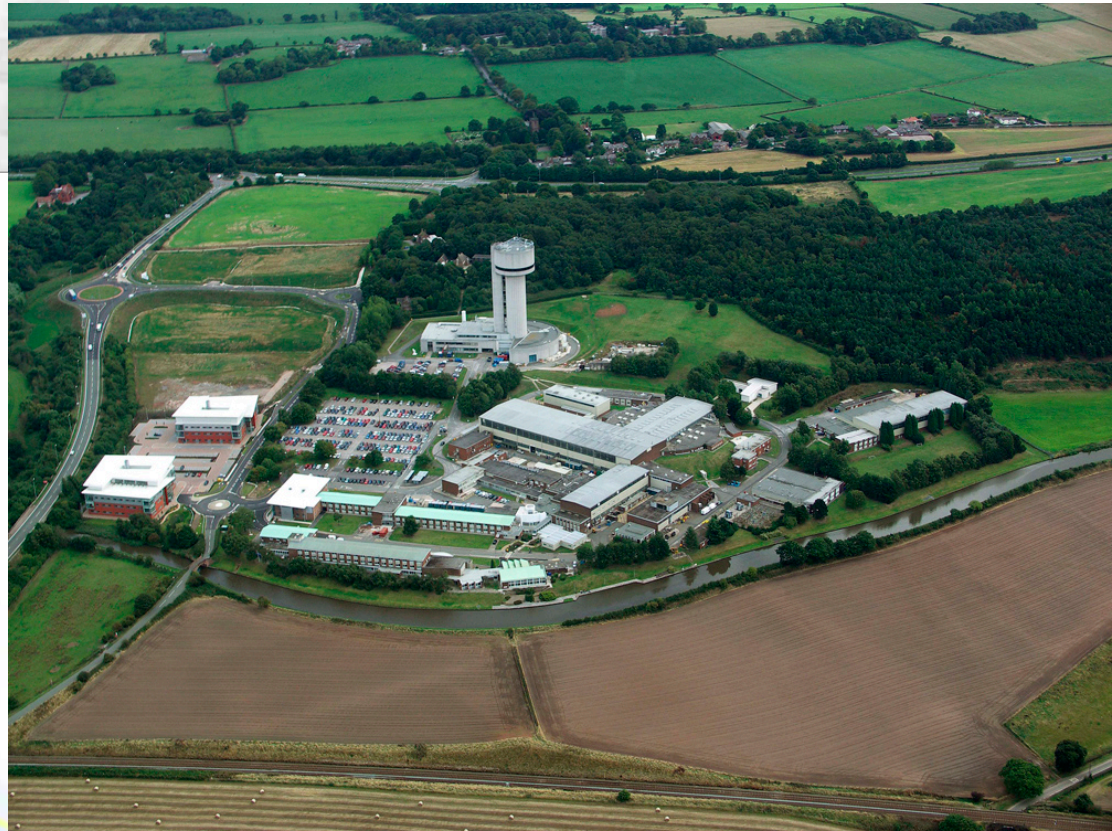
與天下瞻望之所謂賢人君子者陽之使居廟堂之上以三公四
輔之事業委注而仰成之若然則陰陽以和災異以消朝廷清明矣
狄畏服太平之風奇魁足引領而待之也臣朝夕思慮載惟擇賢命
相繫國家休戚治亂之本伏願陛下慎重之然後發聖斷力行而不
疑則宗廟社稷之福天下生靈之幸
起居舍人知諫院范鎮上奏曰臣伏見去冬多南風今春多西北風
乍寒乍暑欲雨不雨又有黑氣蔽日此皆人事之所感動也黑氣陰
也小人也日陽也君象也黑氣蔽日者陰侵陽小人惑君也欲雨不
雨者政事不決也陳執中為相不病而家居者百日矣陛下以御史
之言決一婢死而欲退宰相為是邪乞速退執中以解天意此御史
之言為非邪亦乞勅執中起視事無使天意久不決也寒暑者賞罰也
乍寒乍暑者不當賞而賞當罰而不罰也鄧保吉有過於法不當為



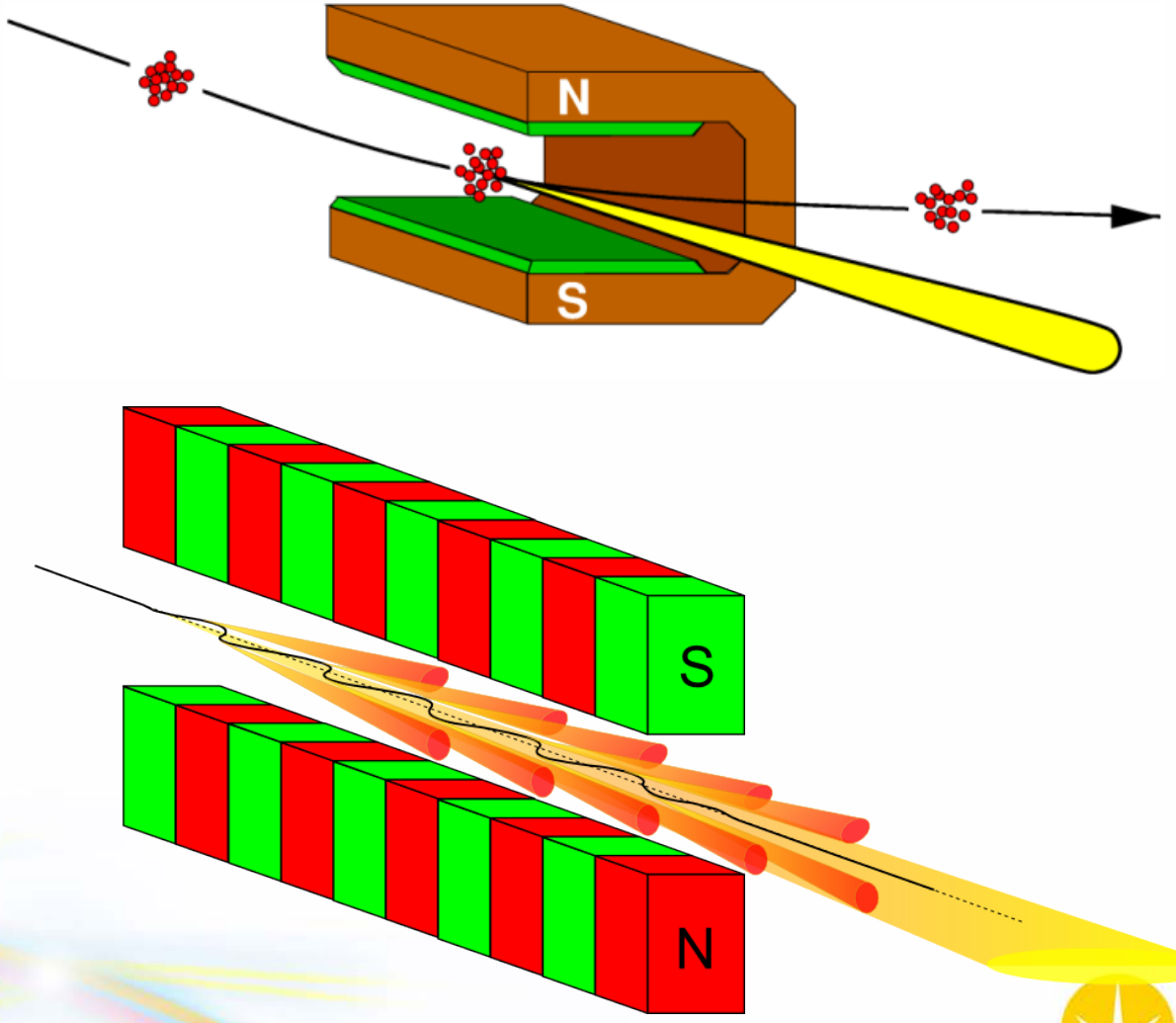
1054

Jumping a generation (2nd incarnation)

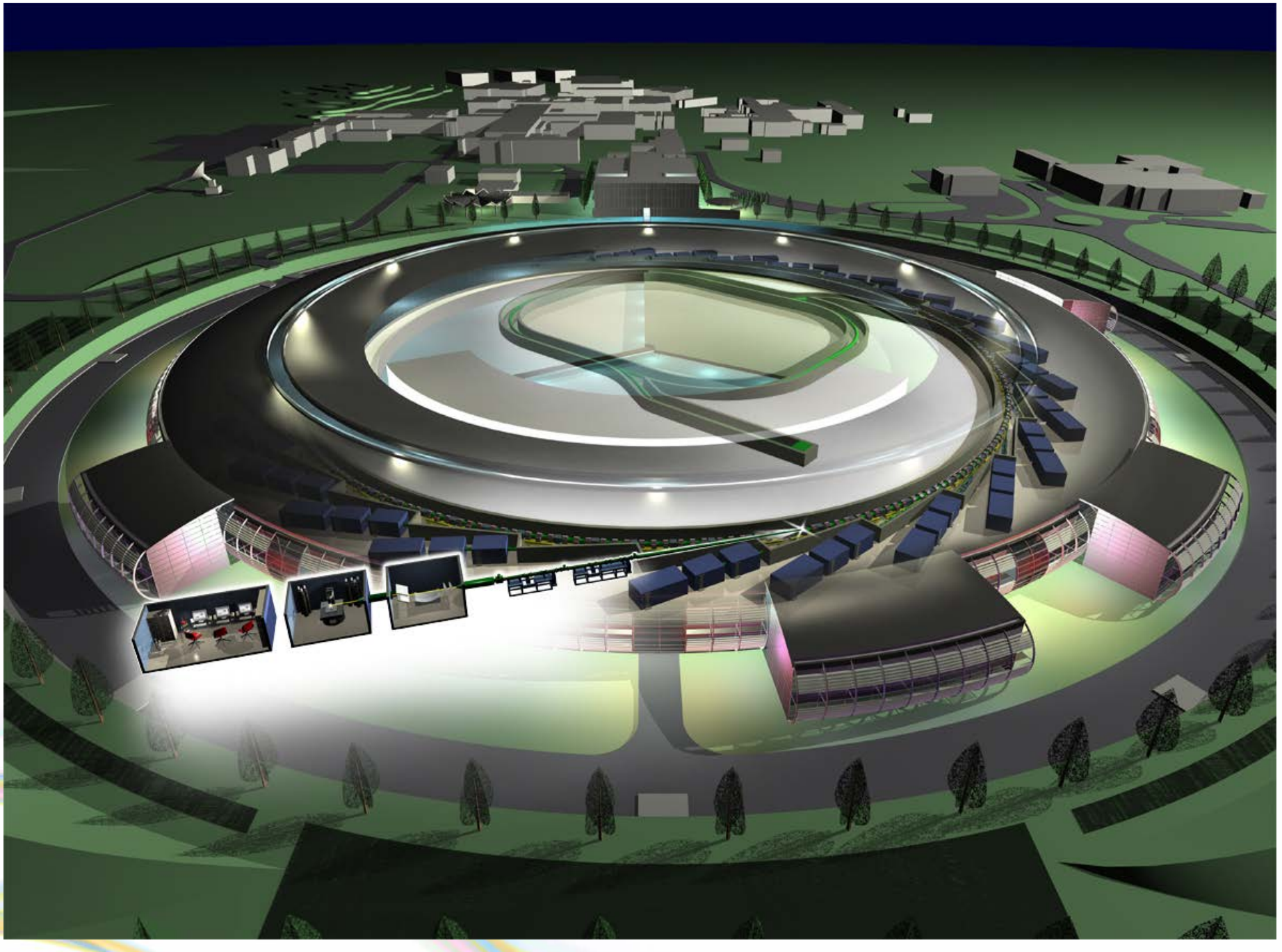




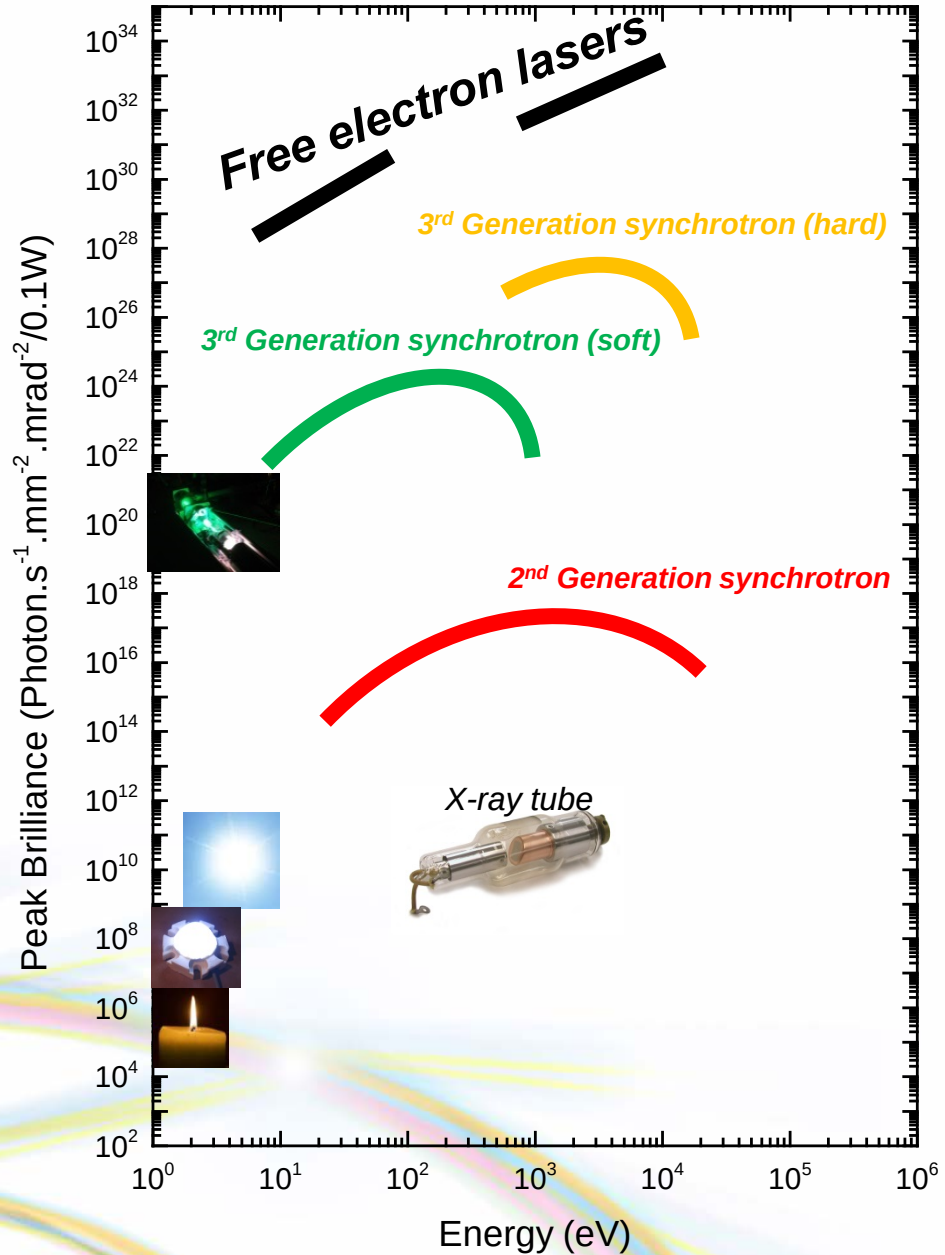
2nd and 3rd generation sources



diamond



Not just ordinary light…

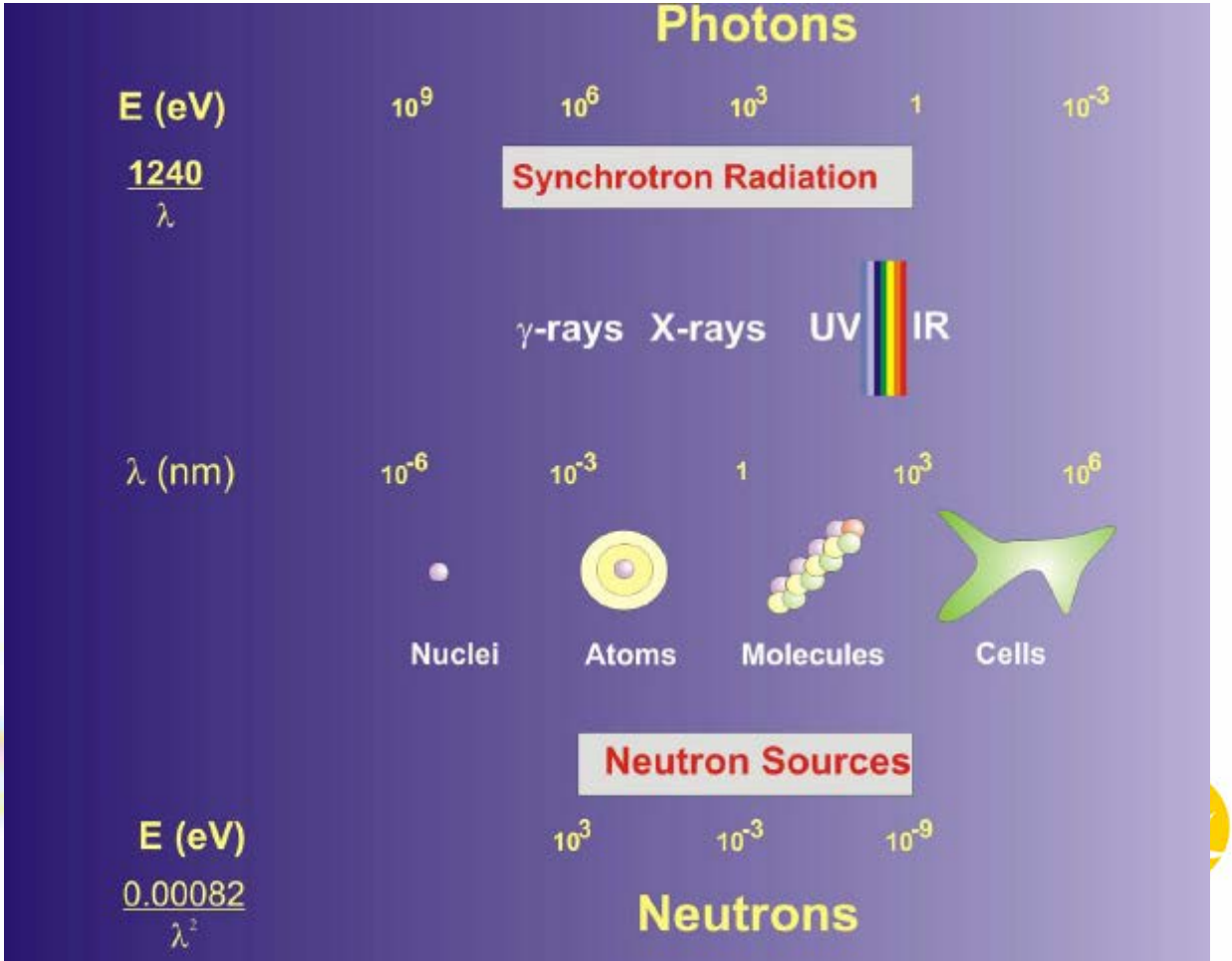


- Brilliant
- Tuneable energy – from IR to X-ray
- Focussing without loss of intensity – 100 mm to 10 nm
- High degree of coherence

diamond

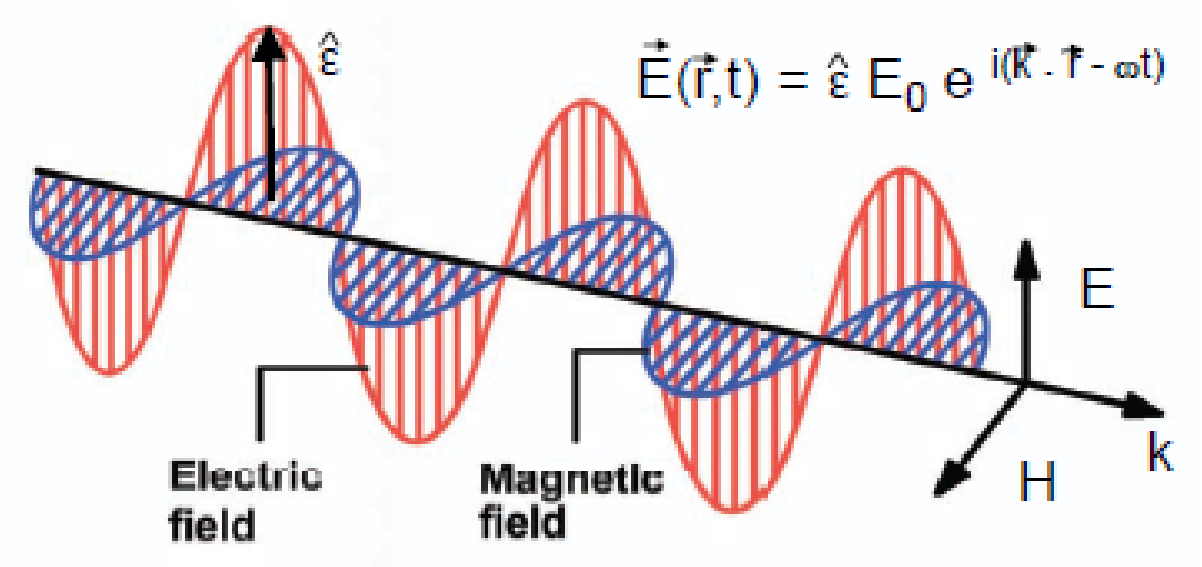
Properties of neutrons and X-rays

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



diamond

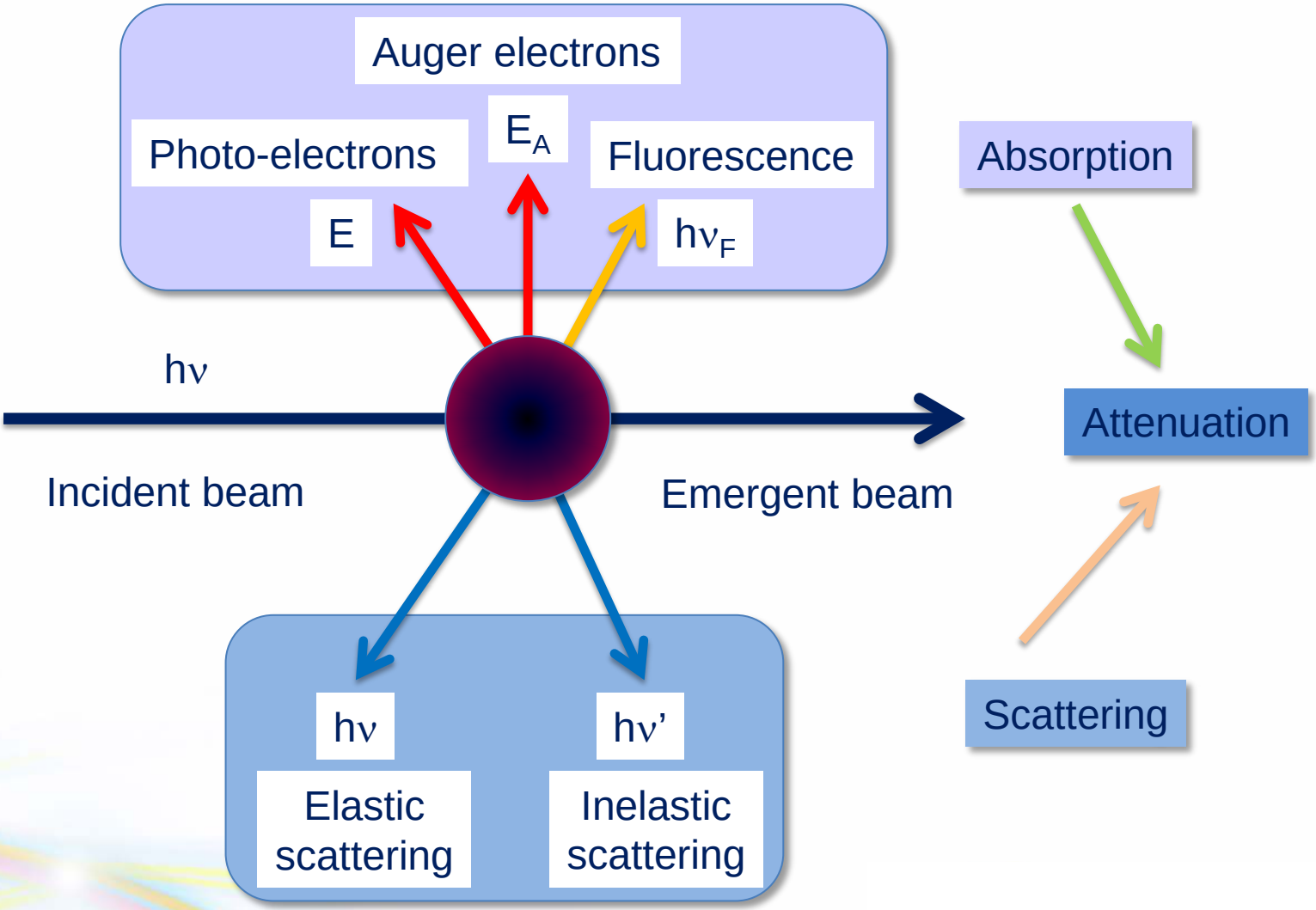
EM wave characteristics



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

diamond

X-ray – matter interactions



X-ray absorption/emission processes

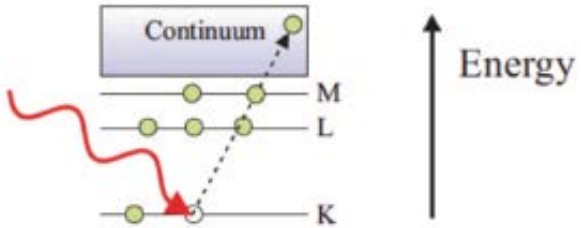
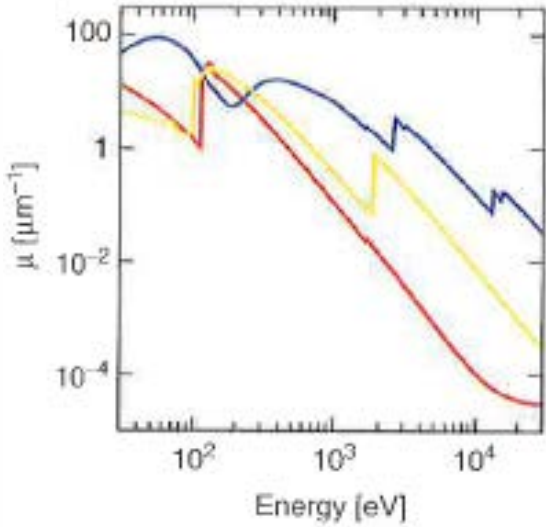
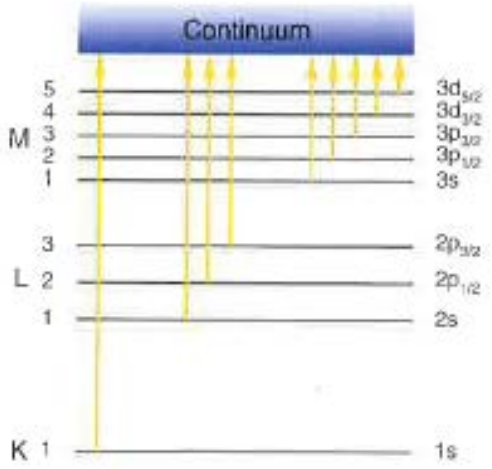
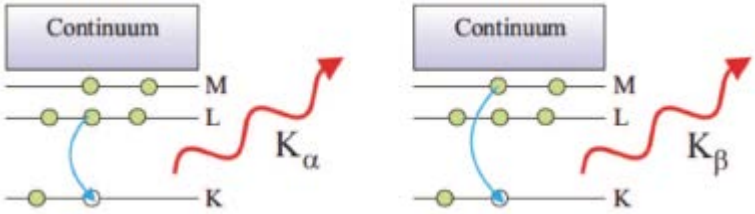


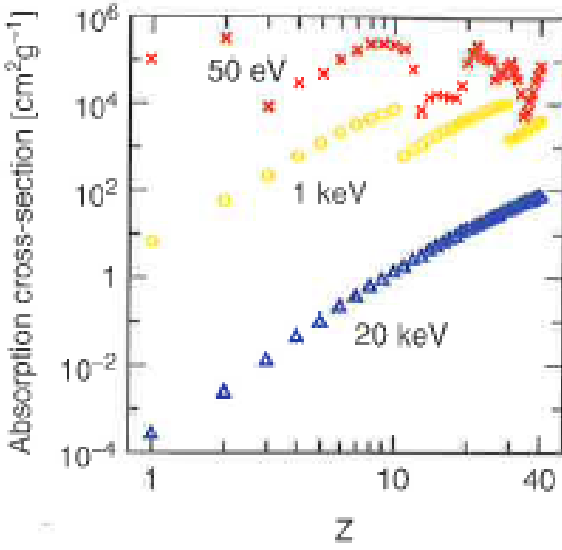
Photo-electric absorption

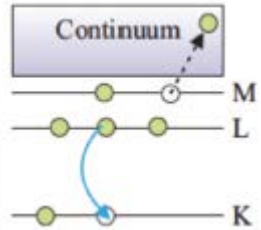






Fluorescent emission

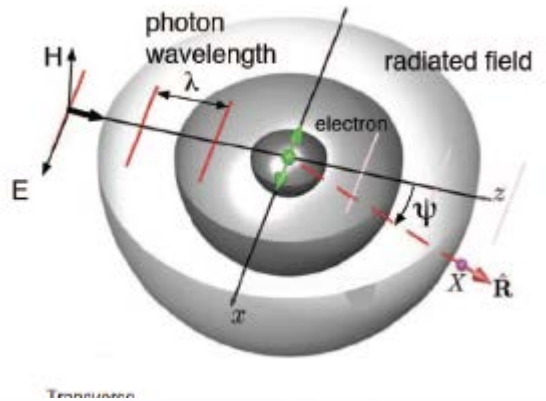


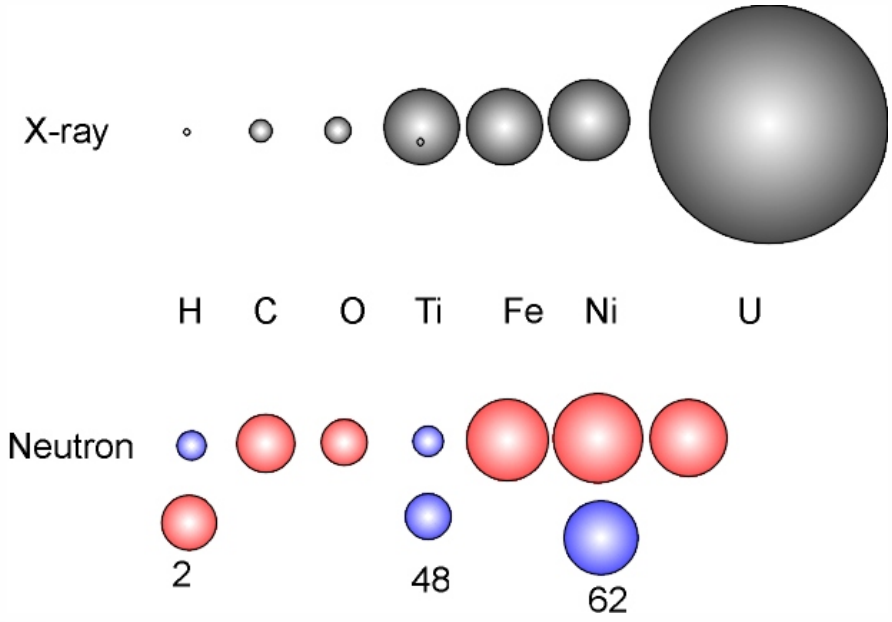


Auger electron emission

X-ray scattering - elastic

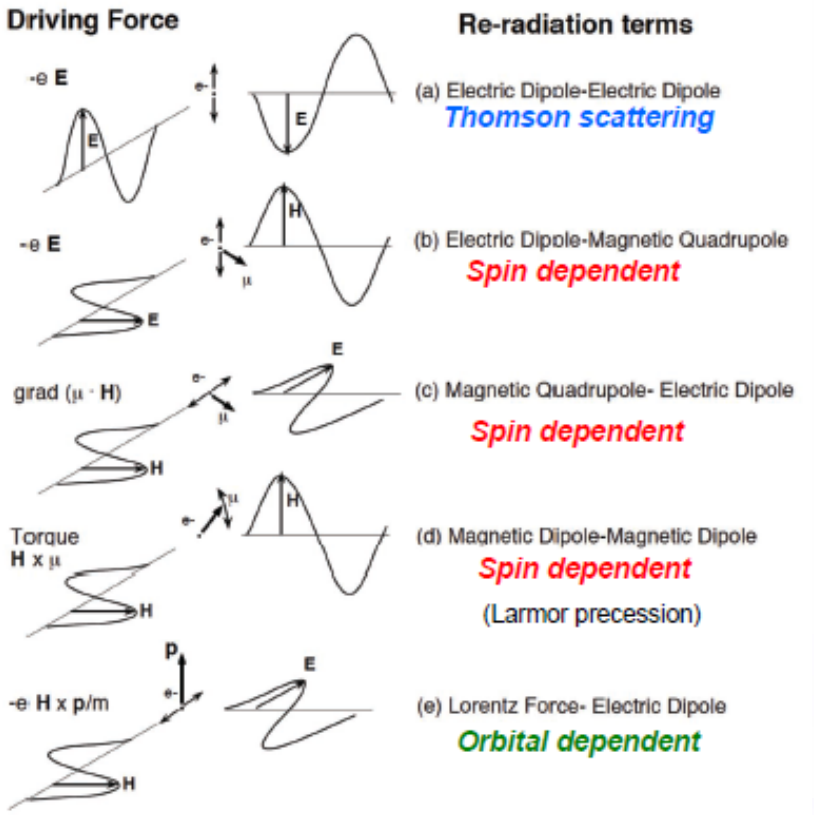
- Leading term is the (Thomson) scattering by electrons
  - The electric field of the incident EM wave drives motion of the electrons and produces spherical waves of the same energy which may interfere to produce a diffraction pattern
  - Elastic scattering intensity is proportional to Z^2

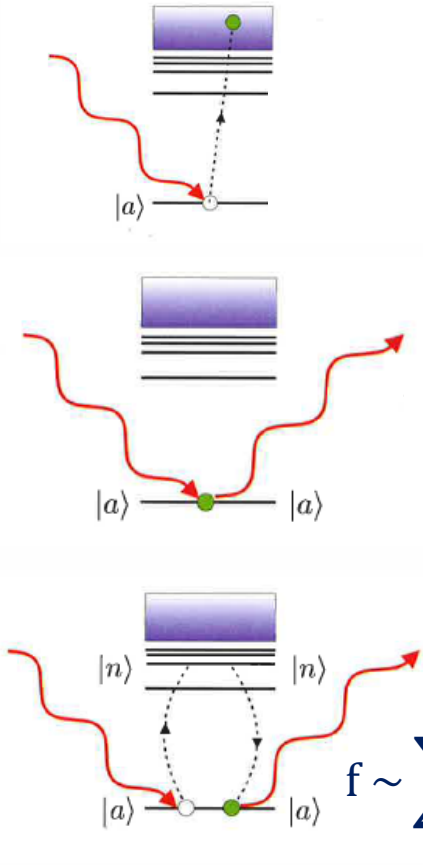




X-ray scattering - elastic

- 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

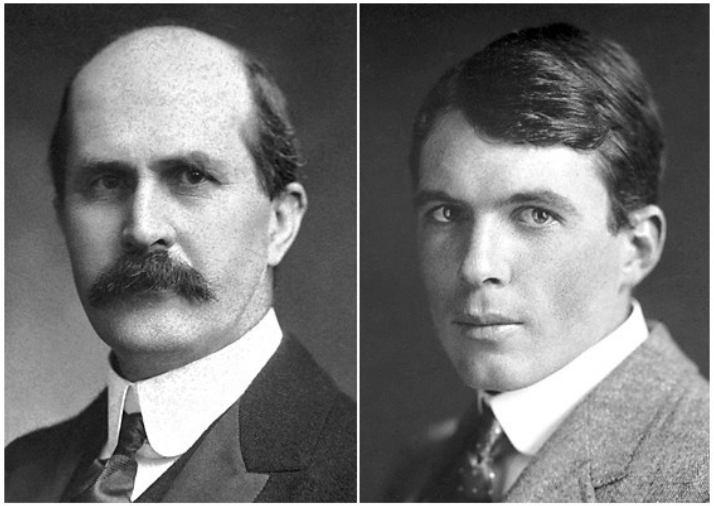
Resonant scattering

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

Diversity - imaging, diffraction, spectroscopy

Wilhelm Röntgen

Max von Laue

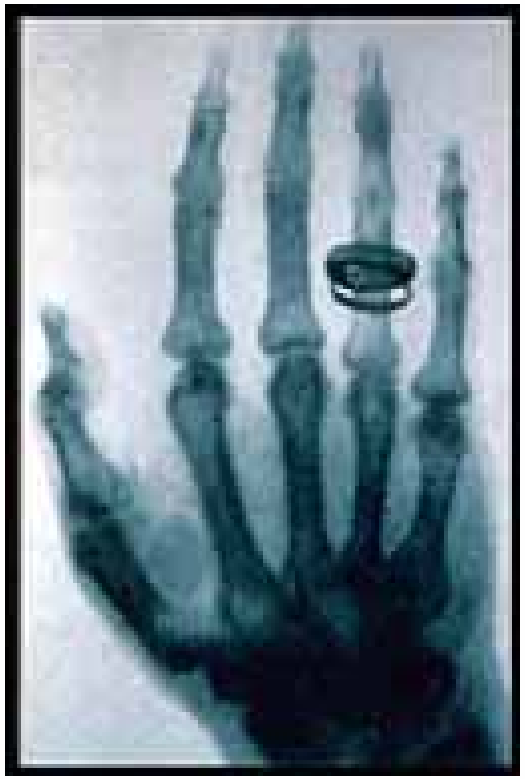


William and Lawrence Bragg

Henry Moseley

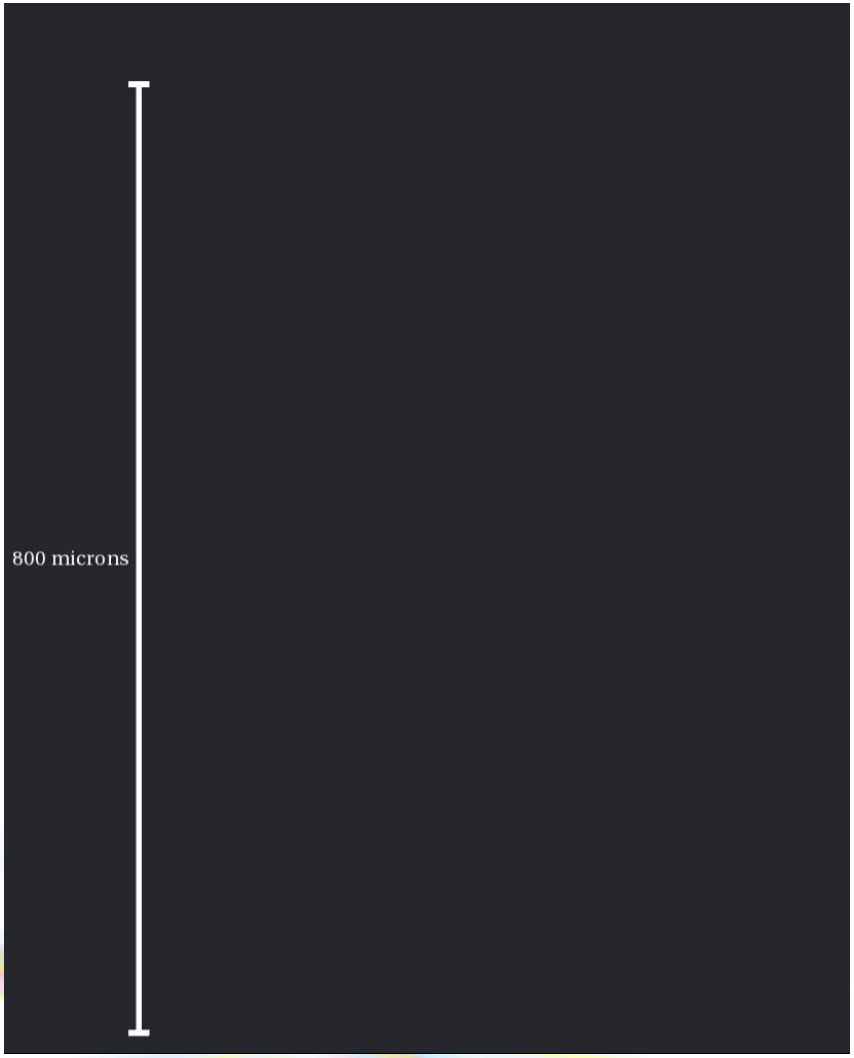
diamond

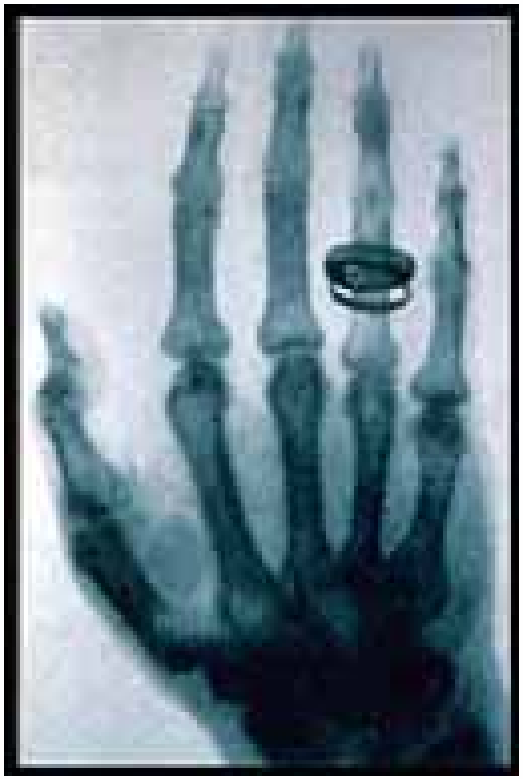
Origins of imaging



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

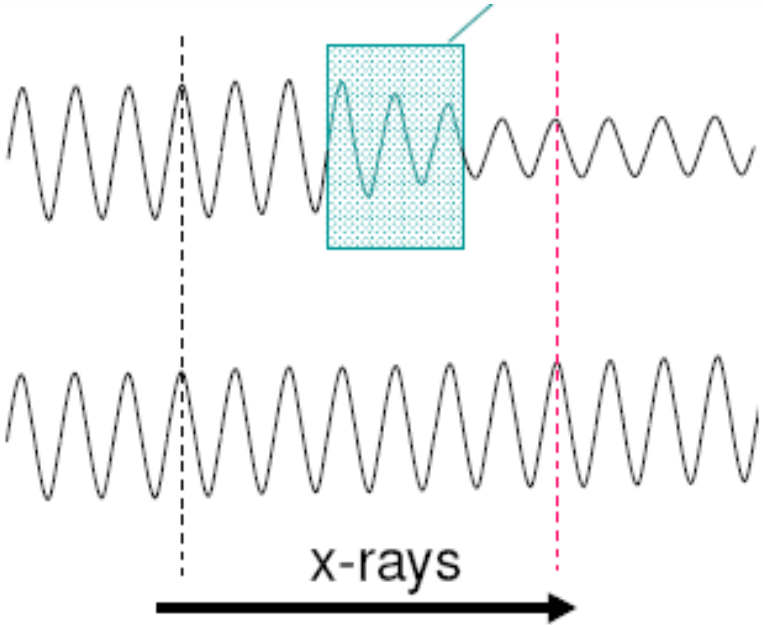
Finer, faster, three dimensional …

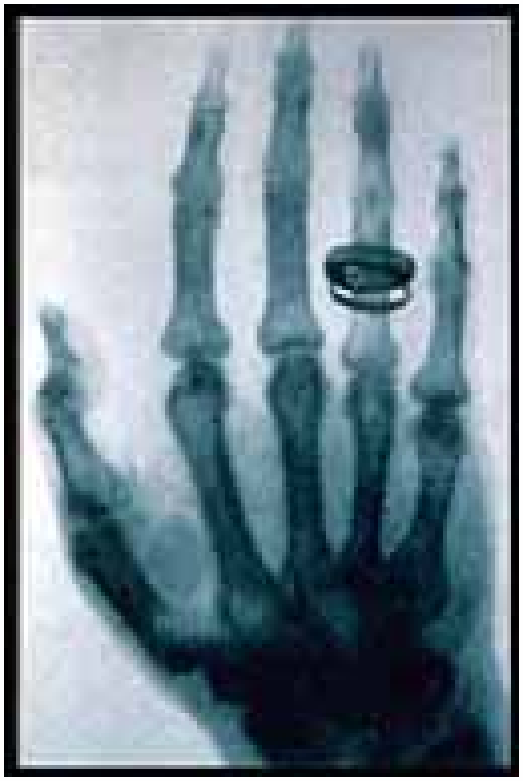




…more subtle

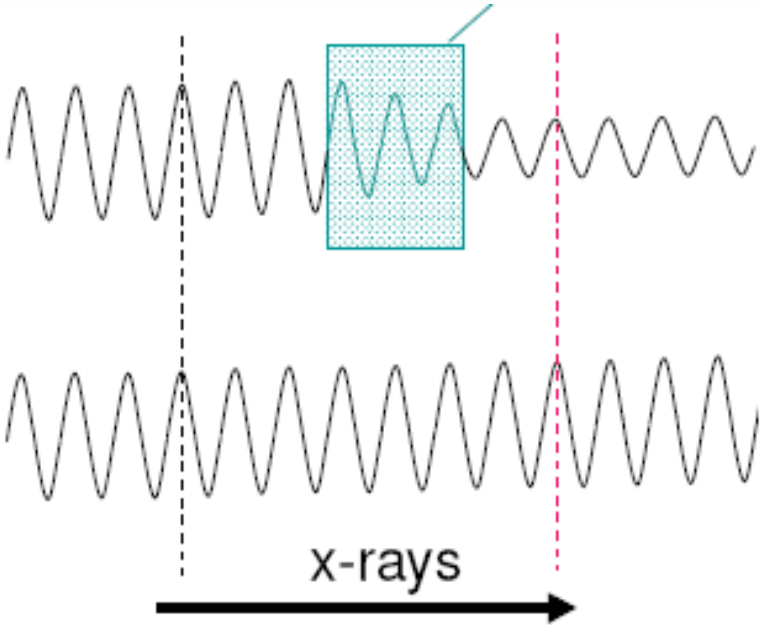
Contrast very poor
if absorption
contrast low



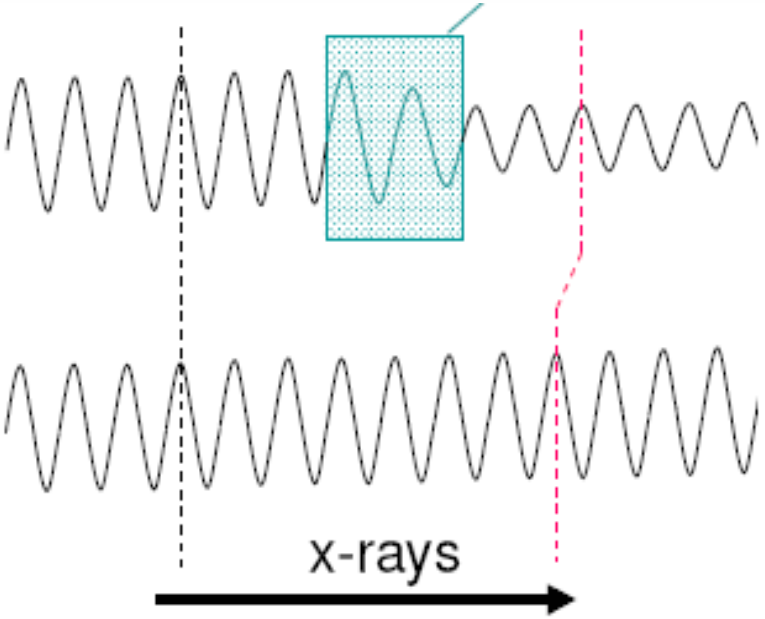


…more subtle

Contrast very poor if absorption contrast low

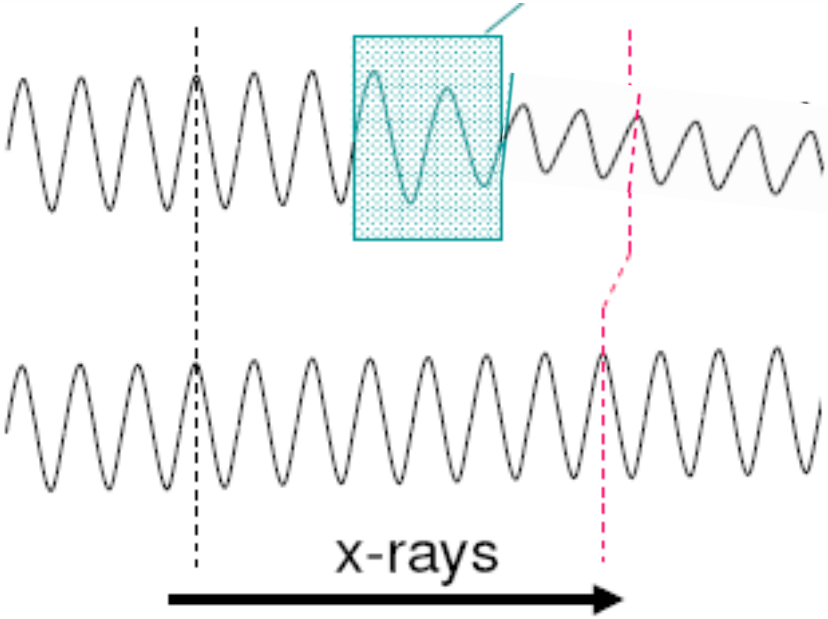


If refractive index different, phase shift occurs

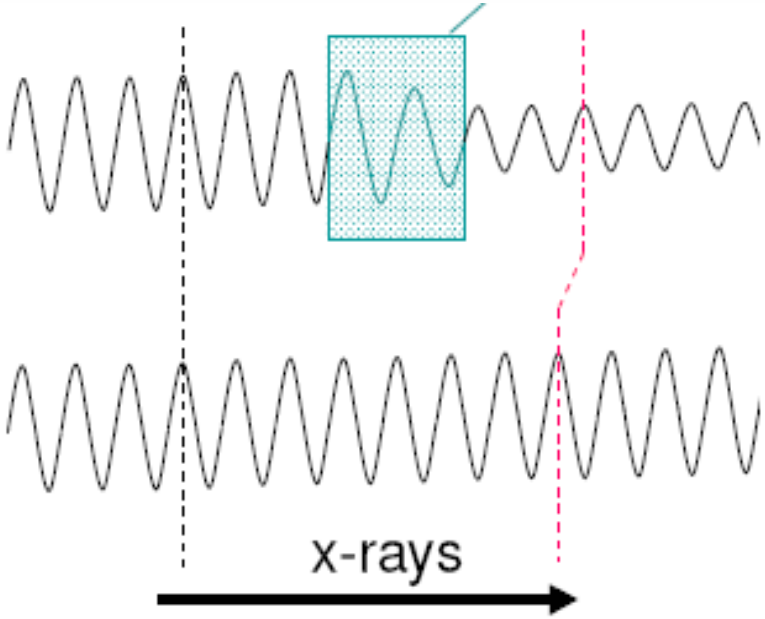


…more subtle

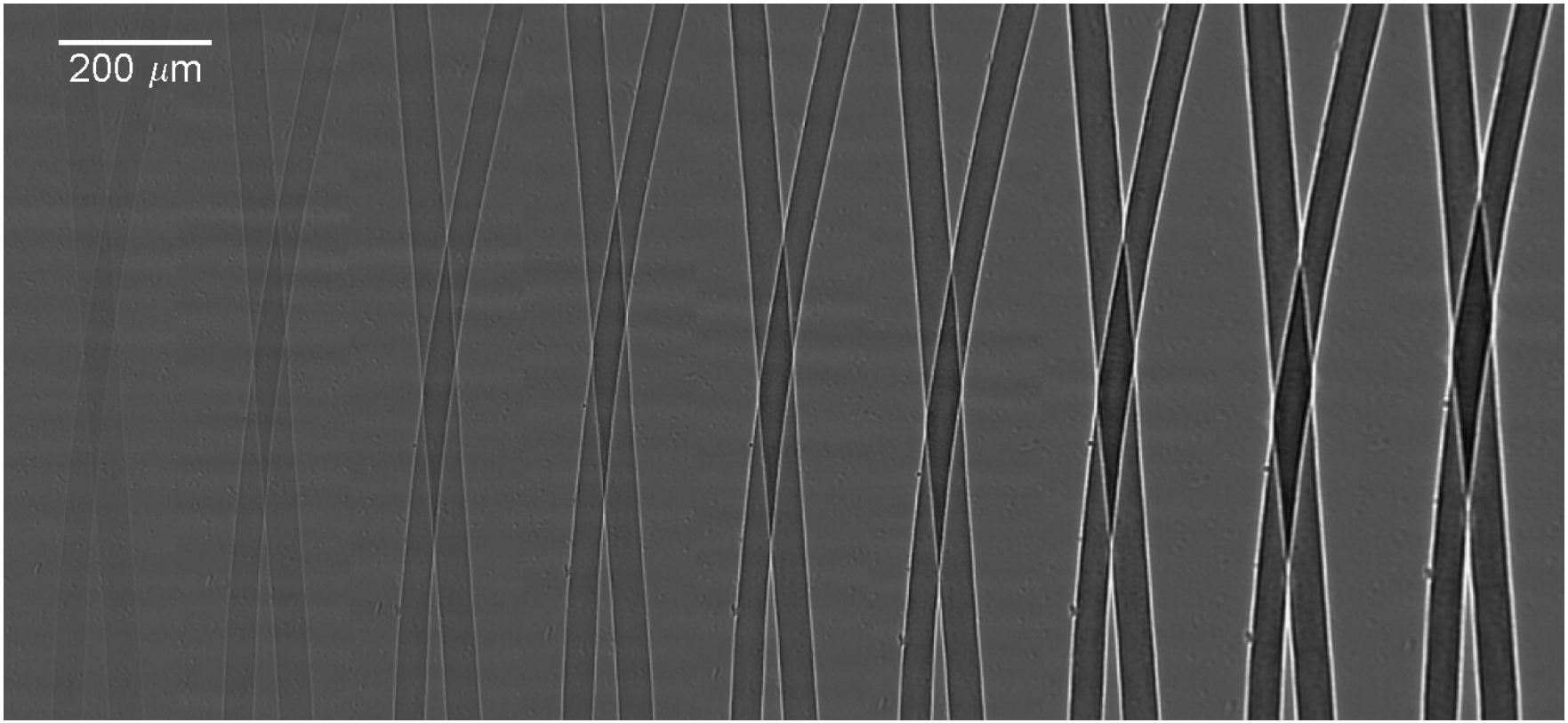
Some diffraction may also occur



If refractive index different, phase shift occurs



…more subtle

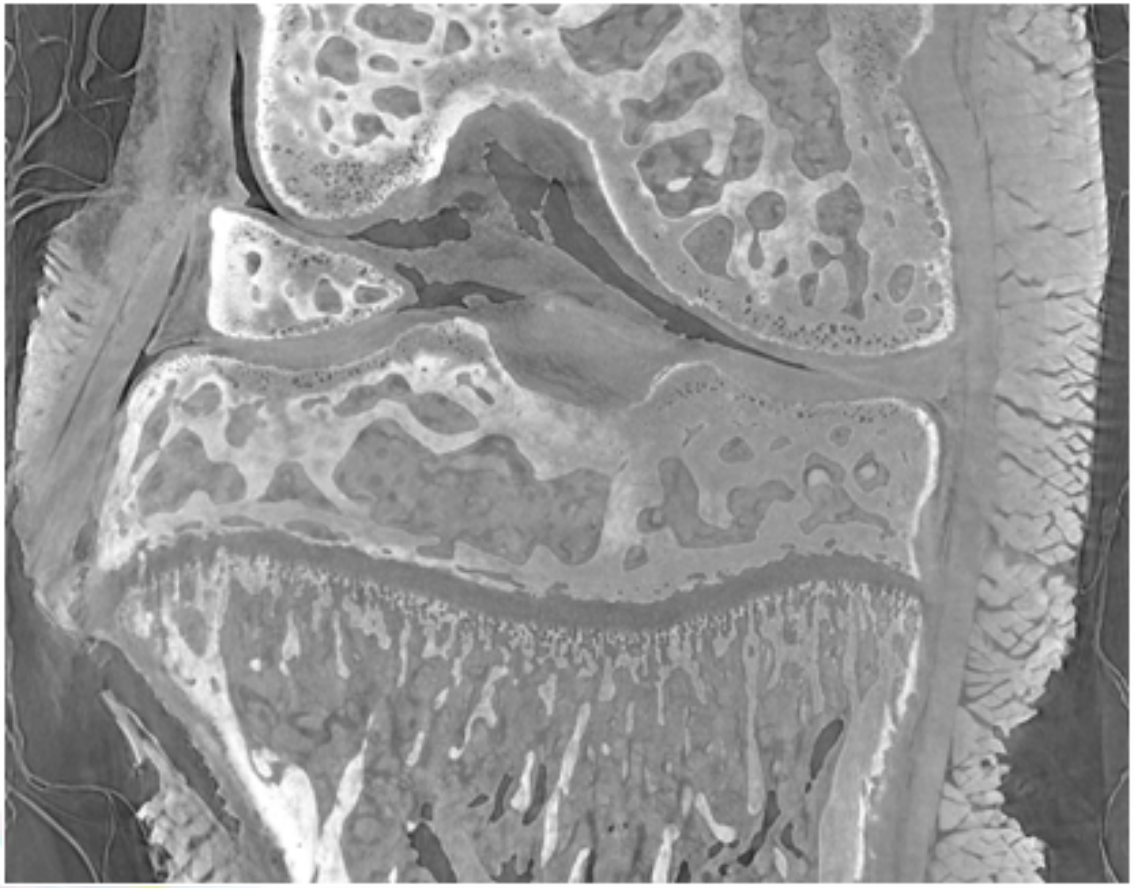


20mm → *Distance* → *1140mm*

E= 20keV

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

Low Z, soft tissue

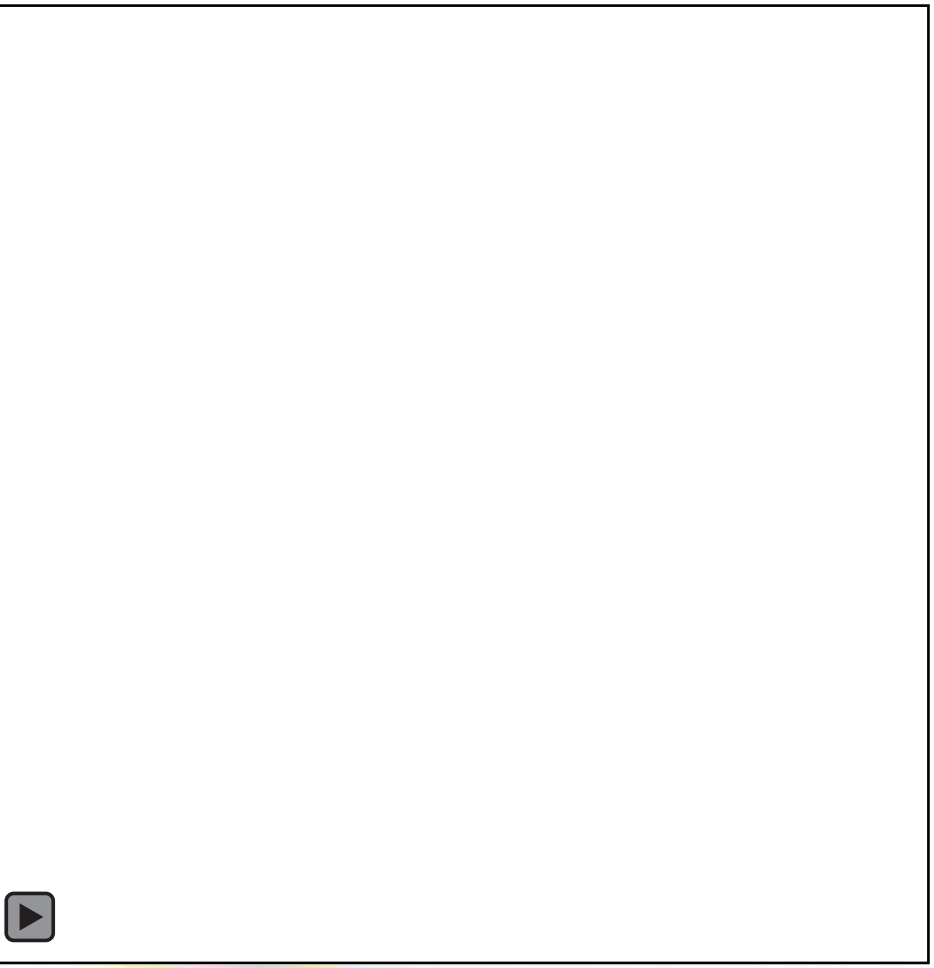


Reconstructed slice of mouse knee

Results courtesy
K. Maadi

Image processed with support of A. Bodey

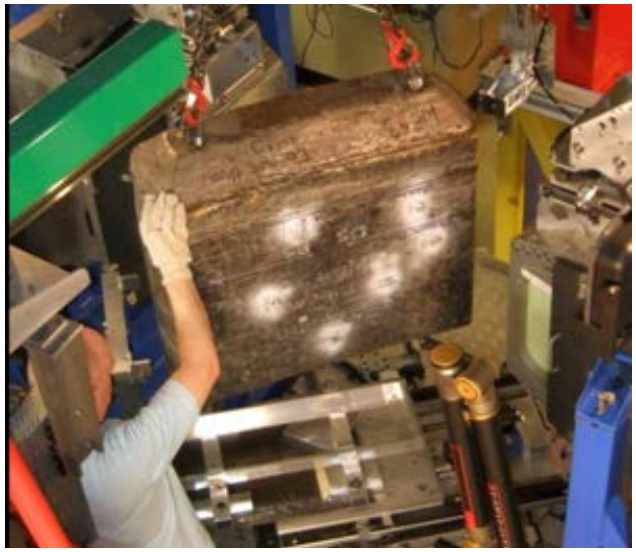
Complementarity of neutron imaging

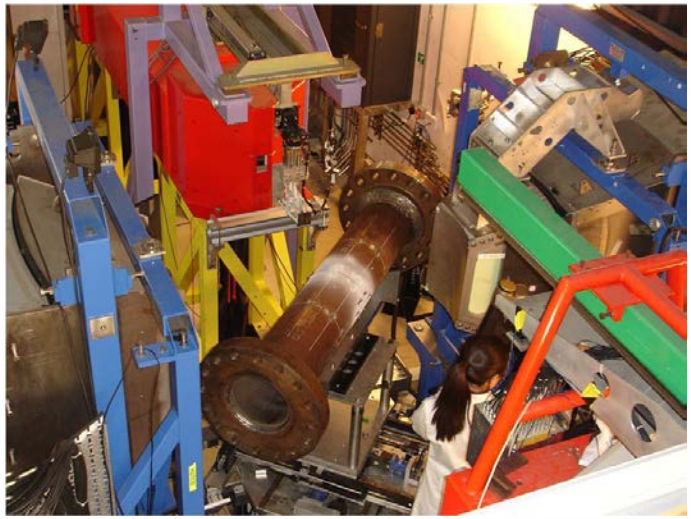


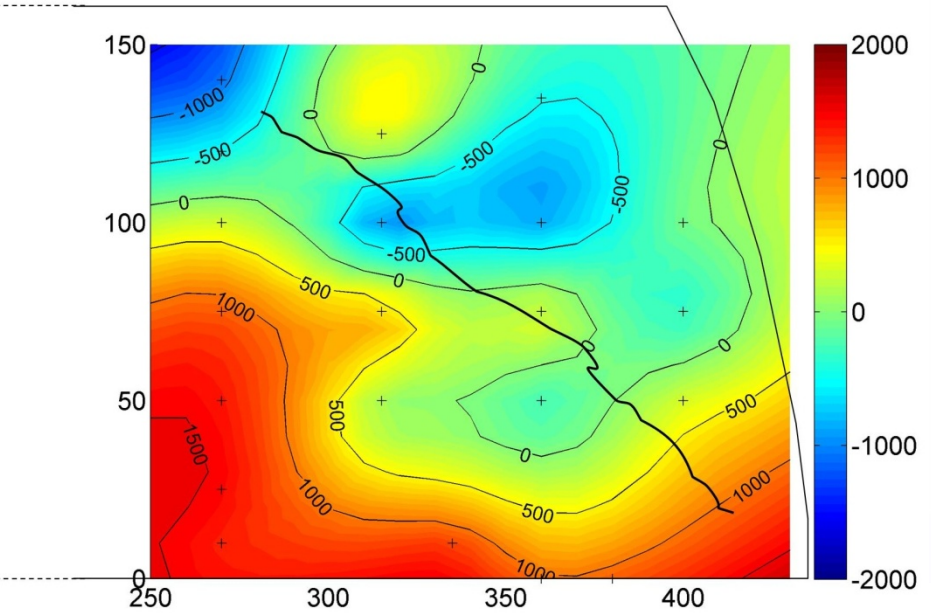
	Neutrons	Synchrotron X-rays
Penetration •Steels	40-60 mm	20-25 mm
Spatial resolution	0.5 mm^3 (cubic gauge volume)	0.1 mm lateral to incident beam; 1 mm parallel to beam (elongated gauge volume)
Stress complete tensor	Yes	With problems
Coarse grain problem	No	Yes
Counting time	10-30 minutes	Seconds-minutes

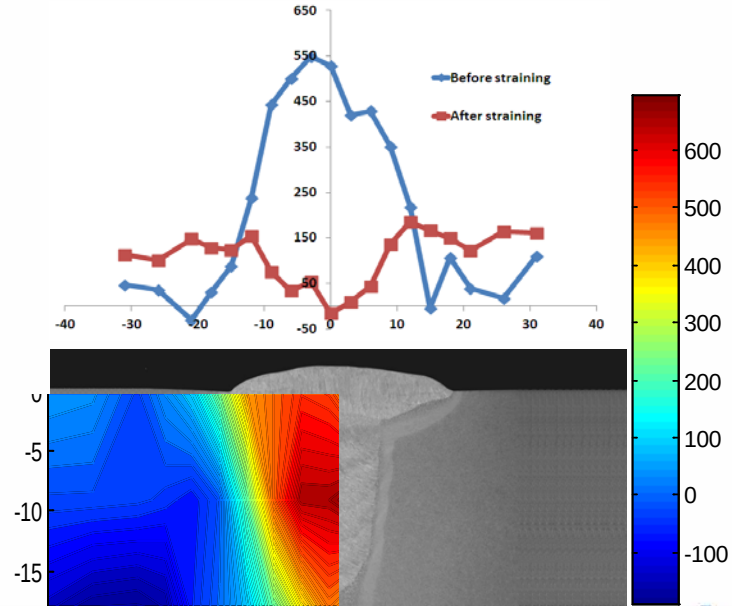
With thanks to Shu Yan Zhang, ISIS

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.

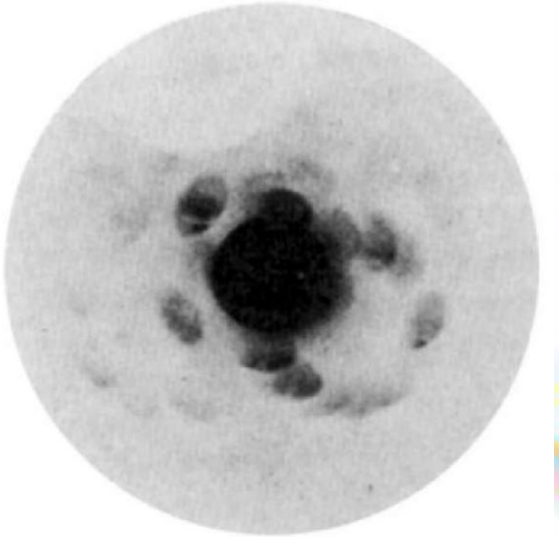
M Turski, et al, Metall Mater Trans A 43A (5) 1547-1557 (2012)

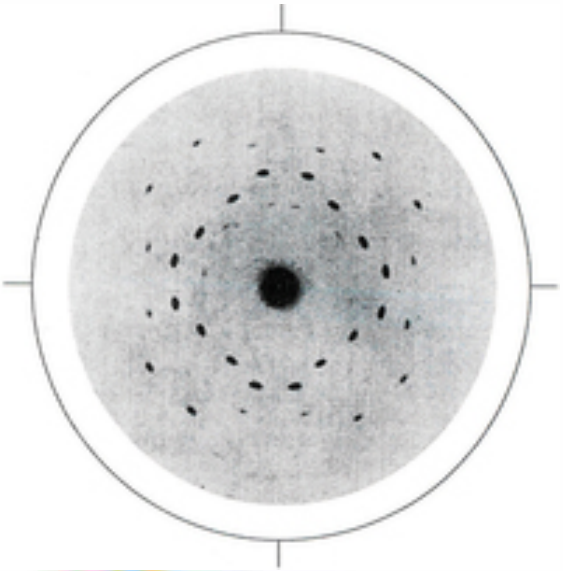
Measure welding residual stresses in pipeline for on-offshore transport of oil and gas

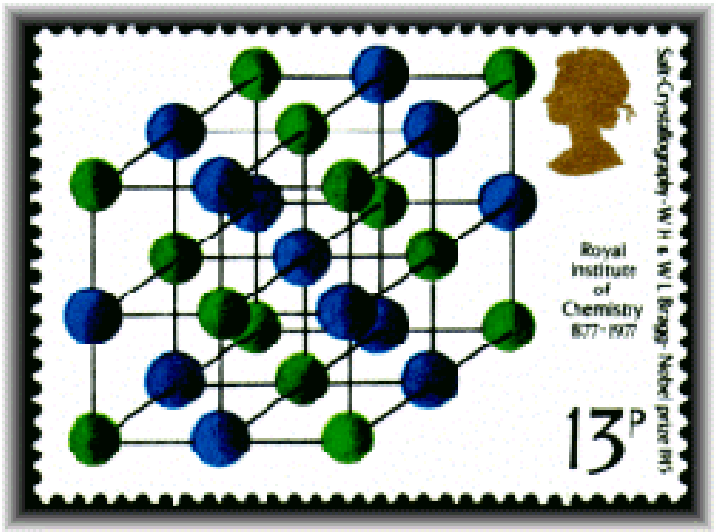
With thanks to Shu Yan Zhang, ISIS

Origins of diffraction at atomic scales

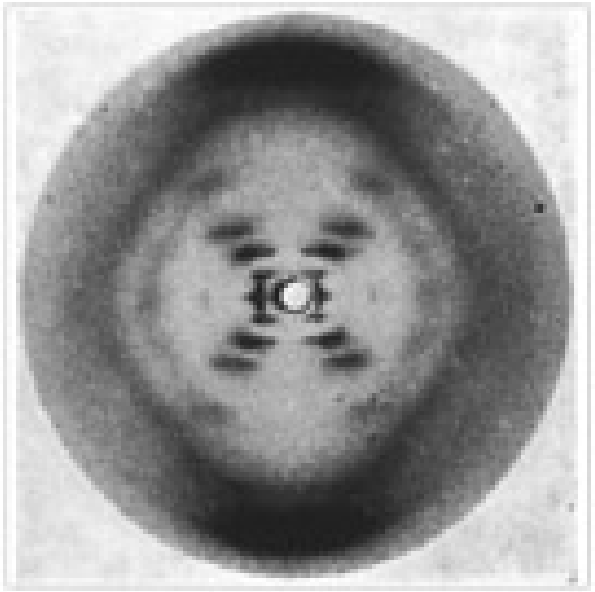
$$n\lambda = 2d.\sin\theta$$

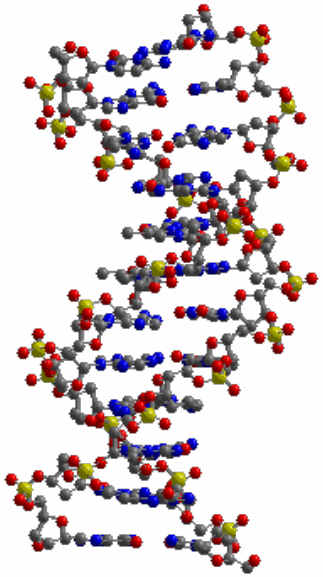


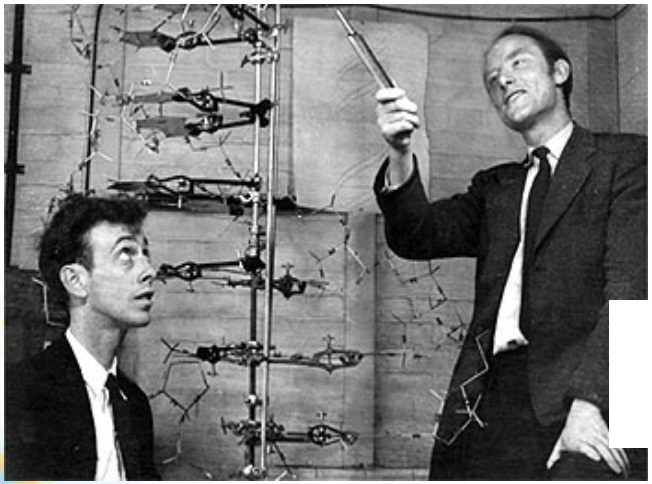




40 years on…

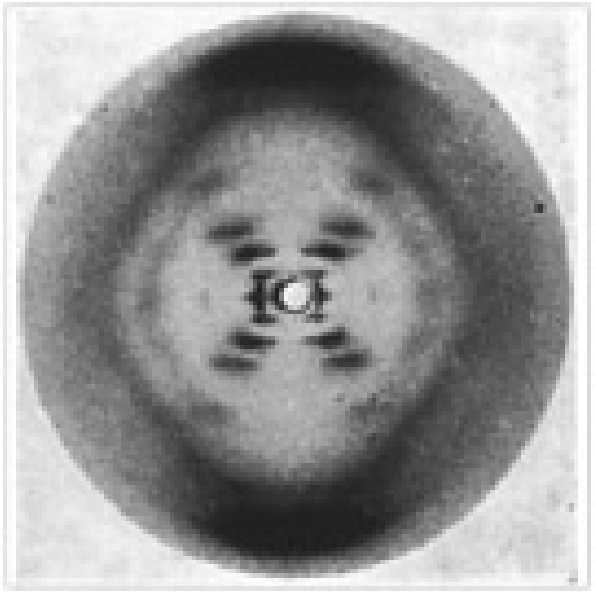


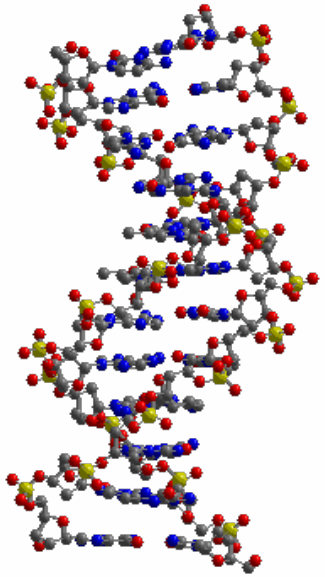


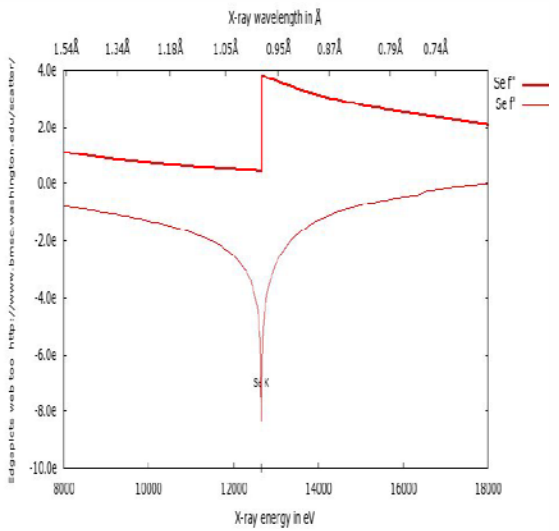


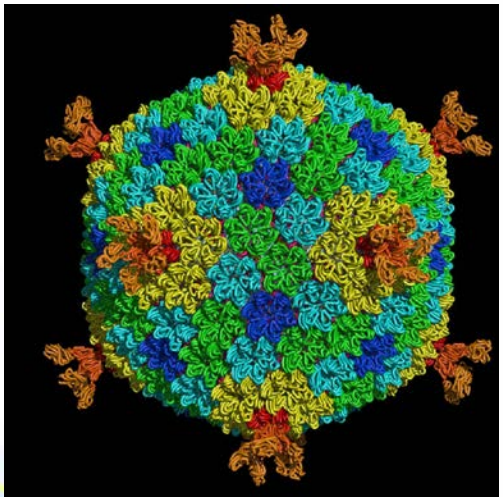
Macromolecular crystallography today

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



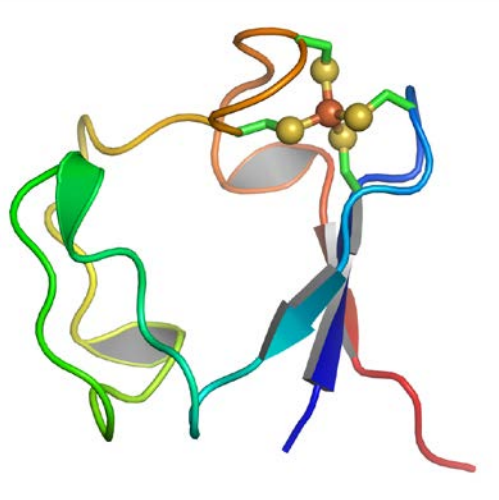


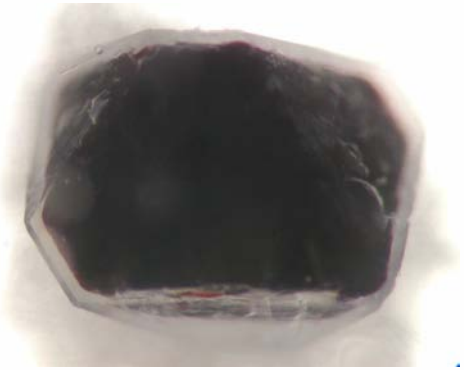




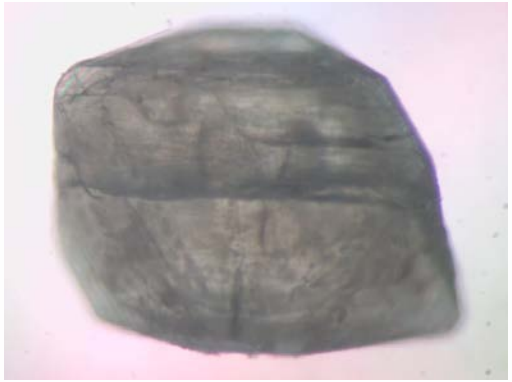
Complementarity: neutrons

- 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+}

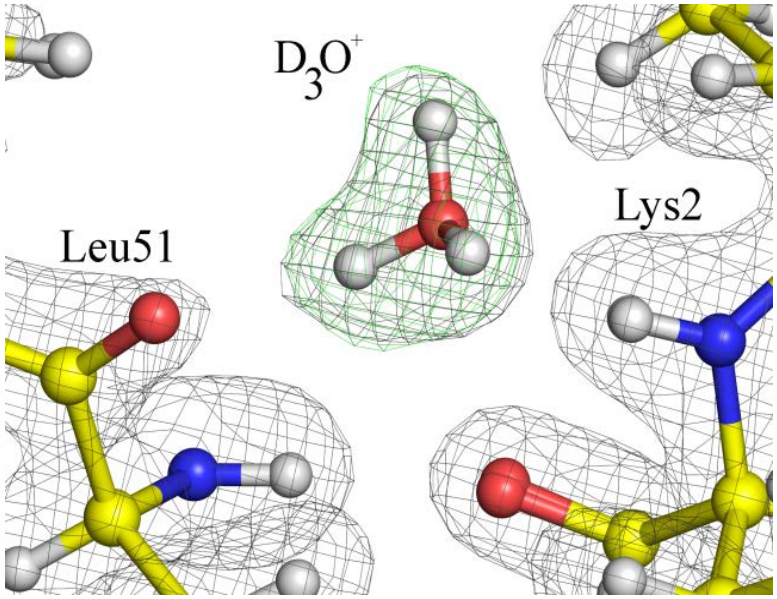


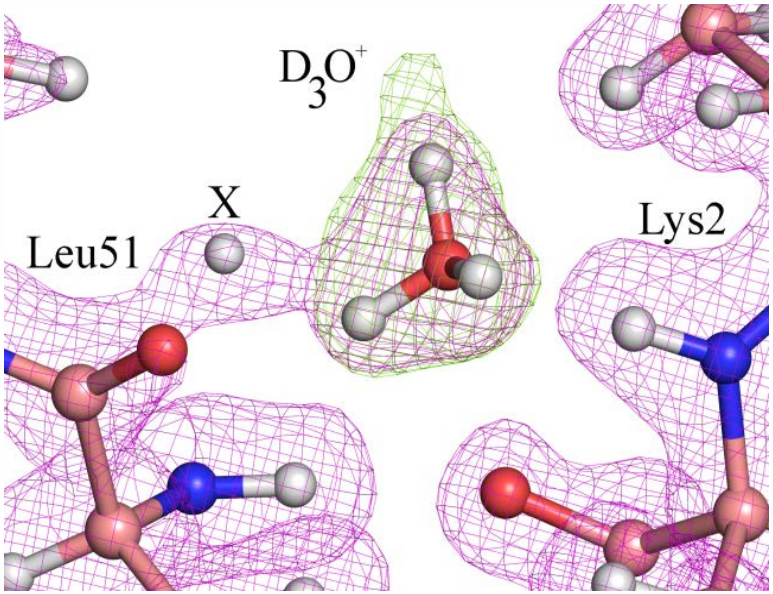
Fe^{2+}

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

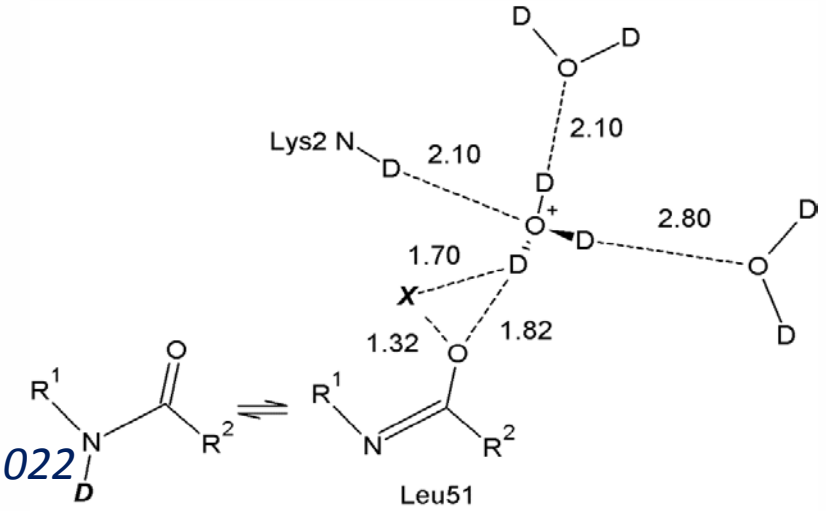
Complementarity: neutrons

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





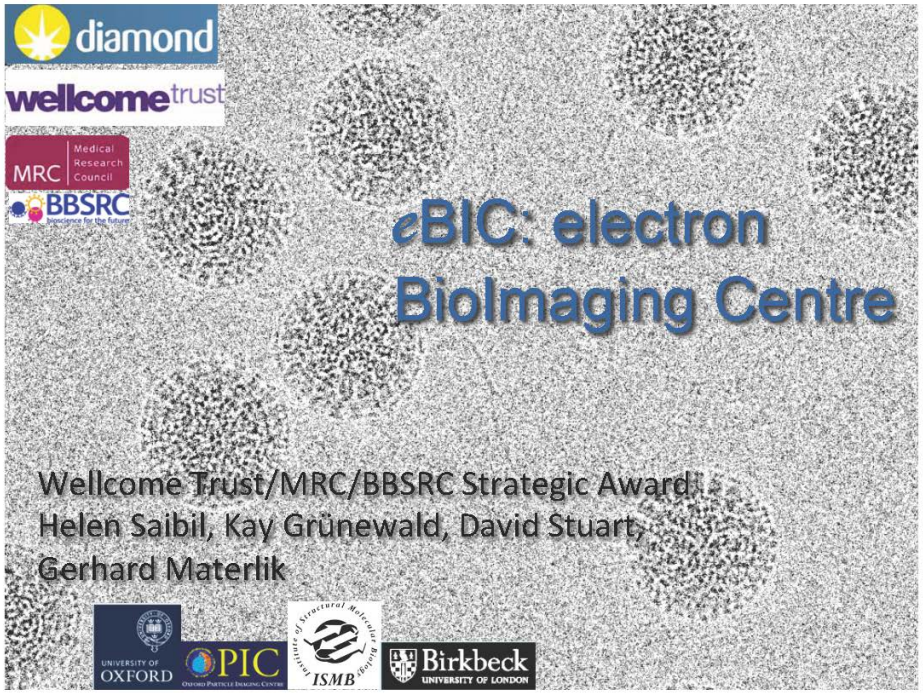
Observation of hydronium (D) ions and of tautomeric shifts following the change from the oxidised form to the reduced form

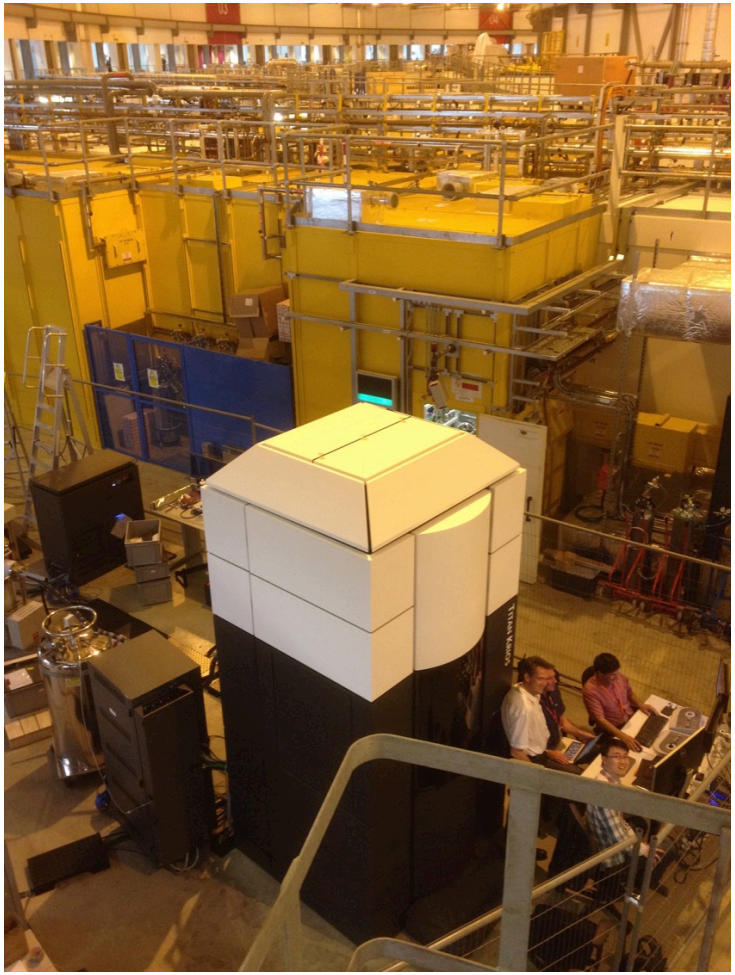


Max Cuypers et al, Angewandte Chemie 52 (2013) 1022

Complementarity: the EM revolution

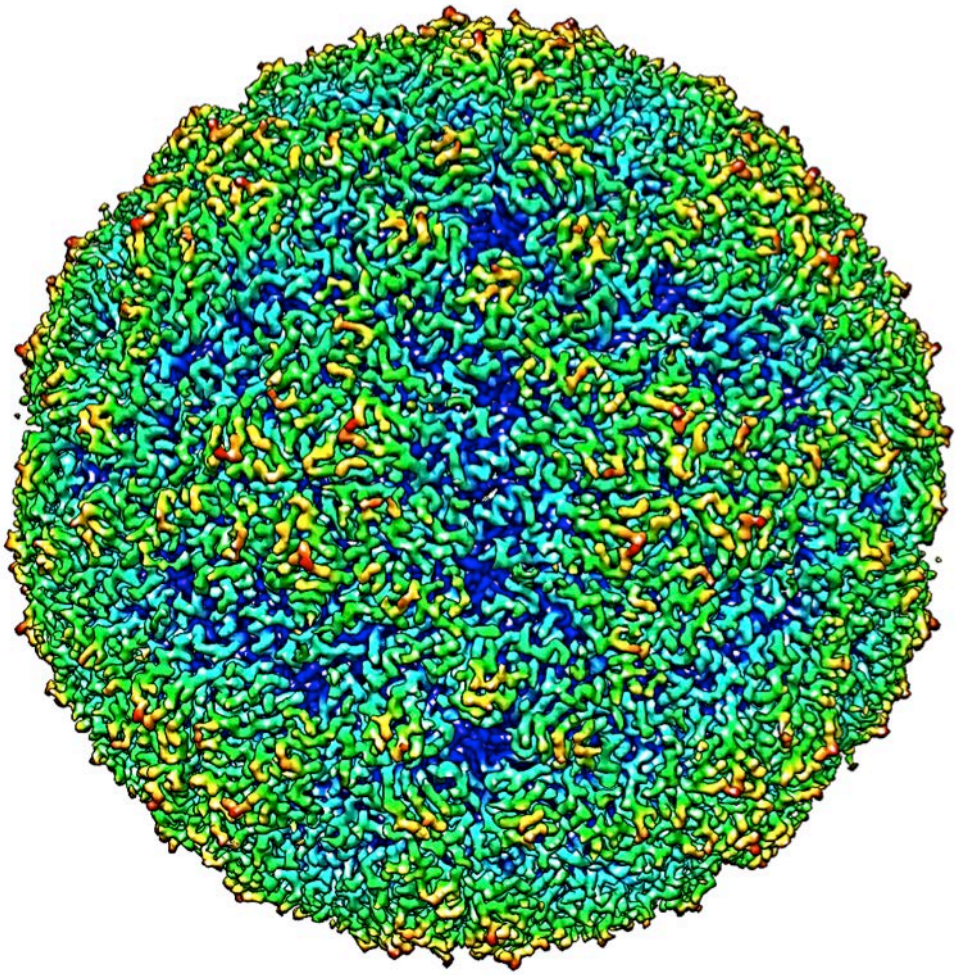
- Wonderful new detectors that collect movies and allow particle movements to be tracked in software to reveal atomic detail
- Native particles frozen in a water ‘glass’

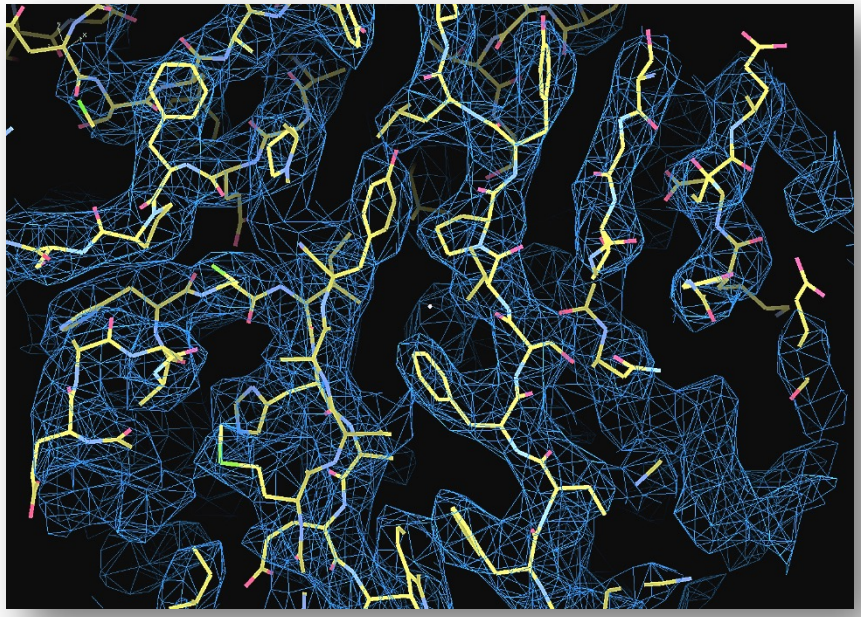


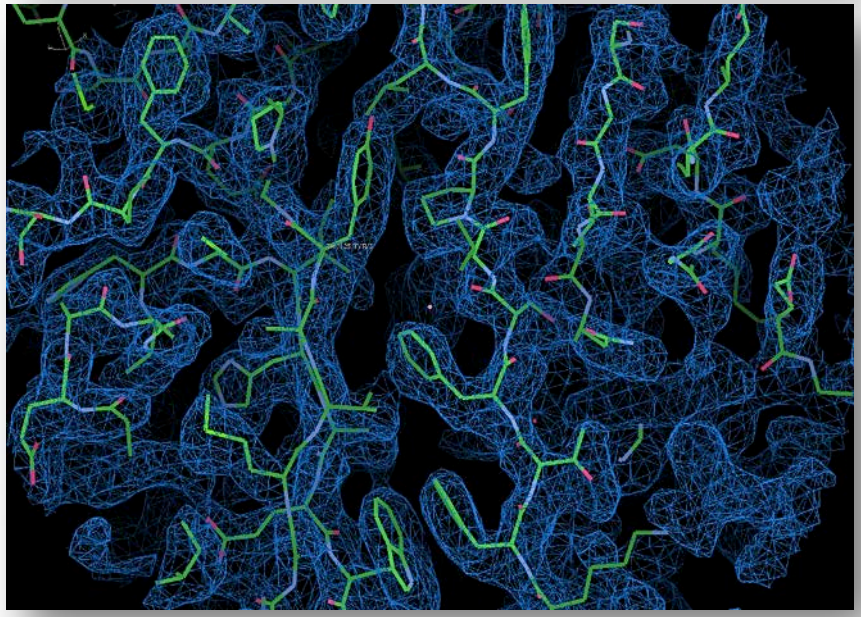


Complementarity: the EM revolution

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

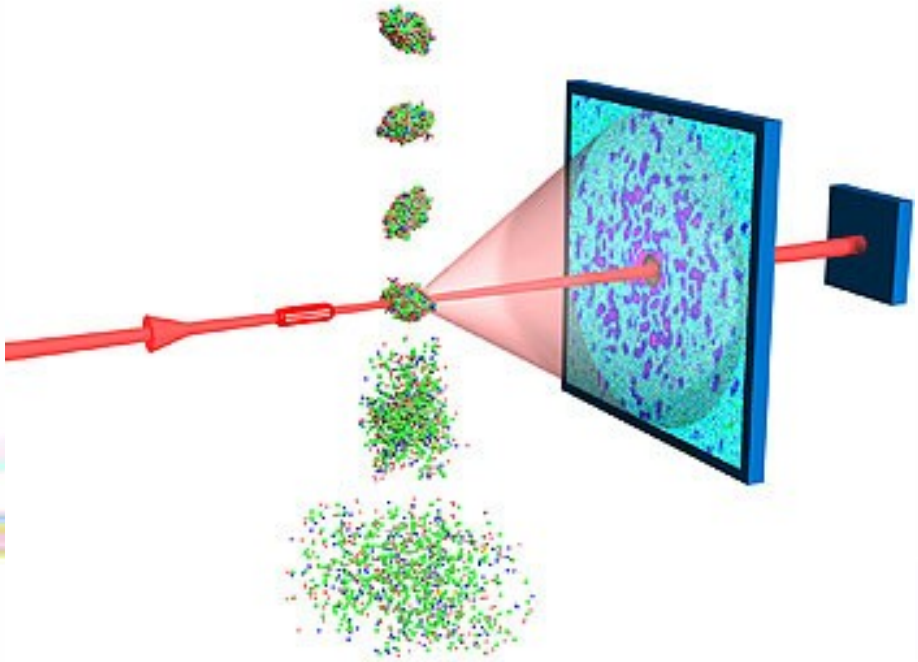






Complementarity: FELs

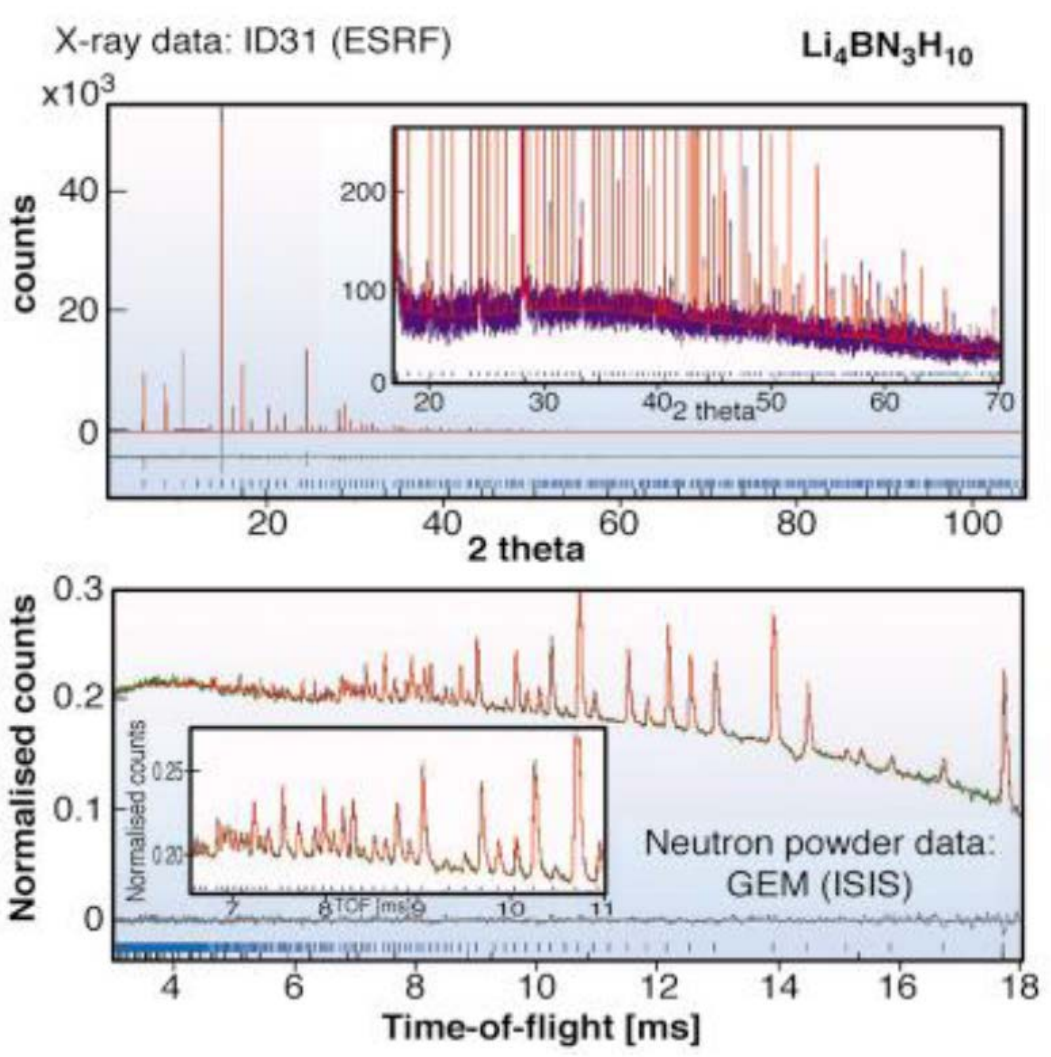
- X-rays pulses in a free electron laser (FEL) are ultrafast (fs) and ultrabright (x 10^{10}) and capture structure before the sample is perturbed by the beam – ‘diffract and destroy’ !
- Challenge of sample injection, image processing…



‘Hard’ materials

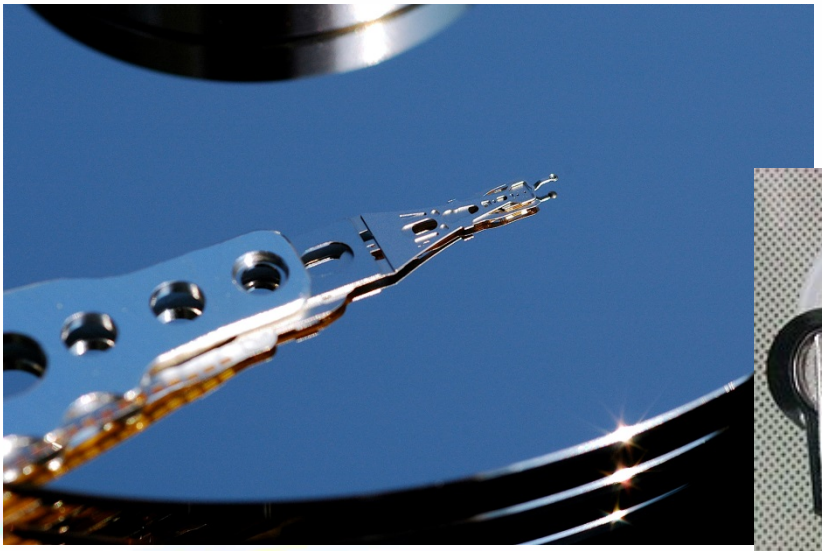
- Many important materials contain hydrogen or other light atoms that are crucial to function – e.g. for hydrogen storage

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



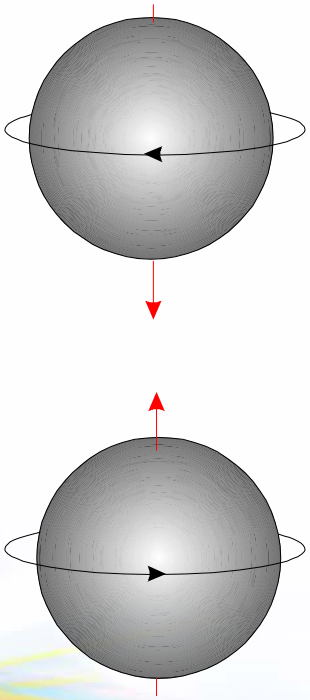
Magnetic structures

- 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

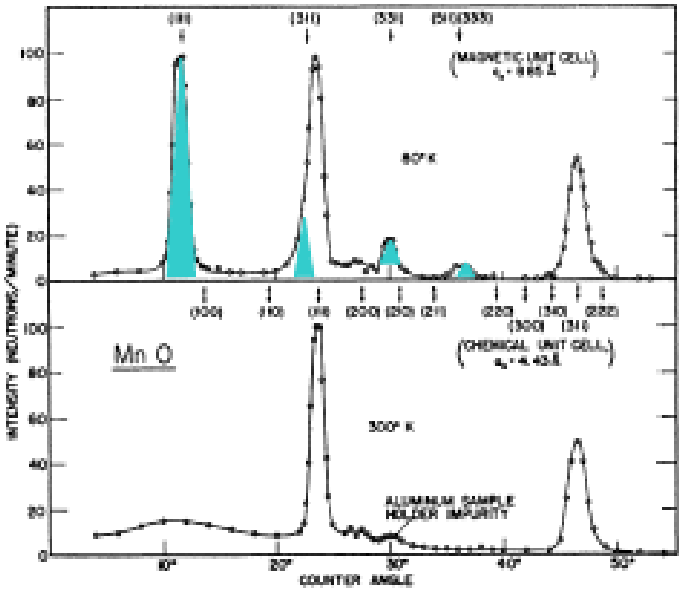
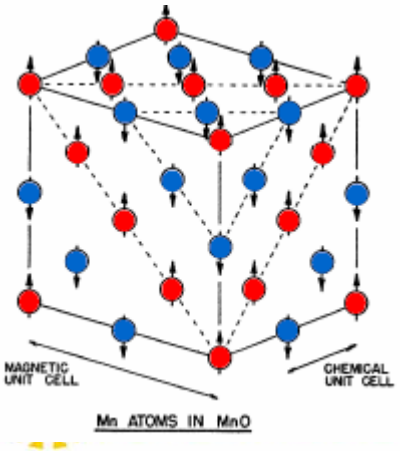
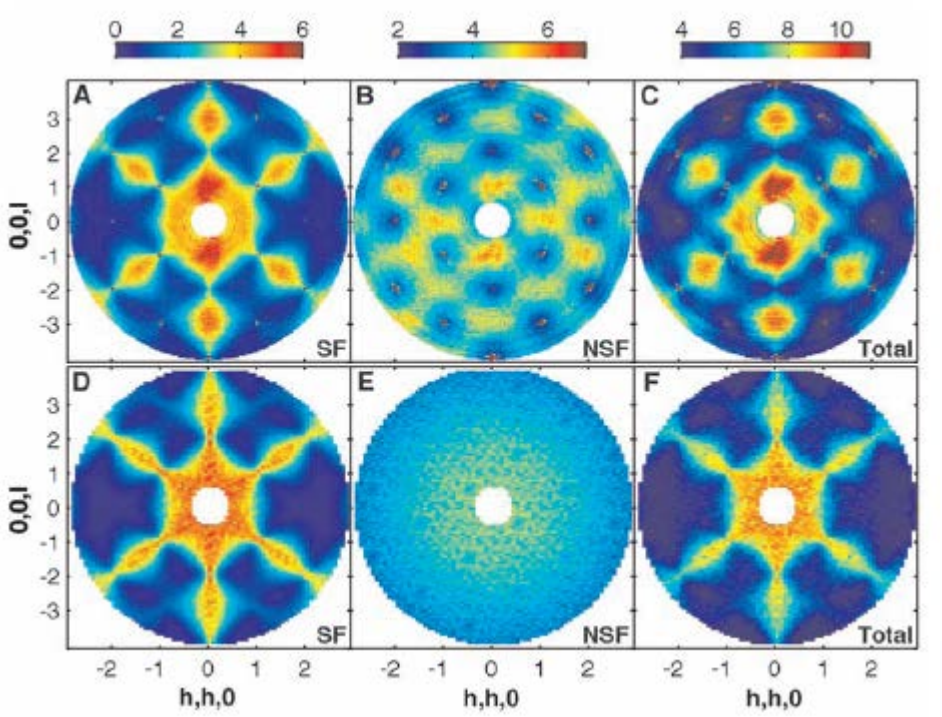


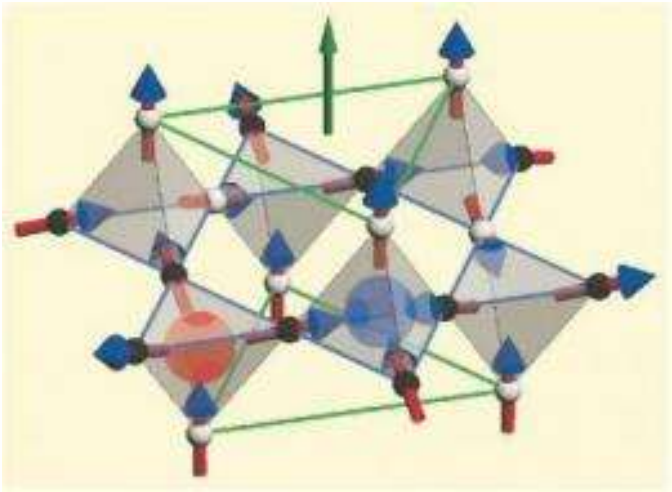
FIG. 1. Neutron diffraction patterns for MnO at room temperature and at 80°K.



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





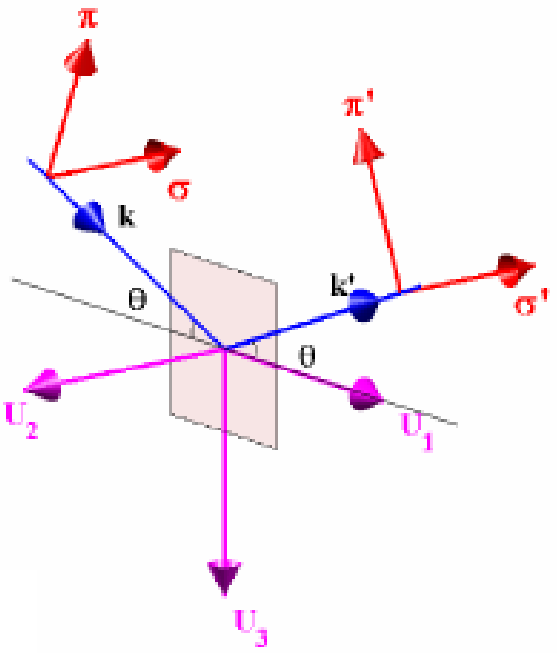
diamond

Magnetic structures: X-rays

- Scattering cross-section generally very weak – typically 10^6 weaker than the nuclear (Thomson) terms ($(\hbar\omega/mc^2)\sim10^{-2} \rightarrow I^{mag}/I^{at} \sim10^{-6}$)
- Polarisation analysis can be used to separate spin (S) and orbital (L) contributions
- Magnetic scattering amplitude $f^{mag}(Q)$:

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

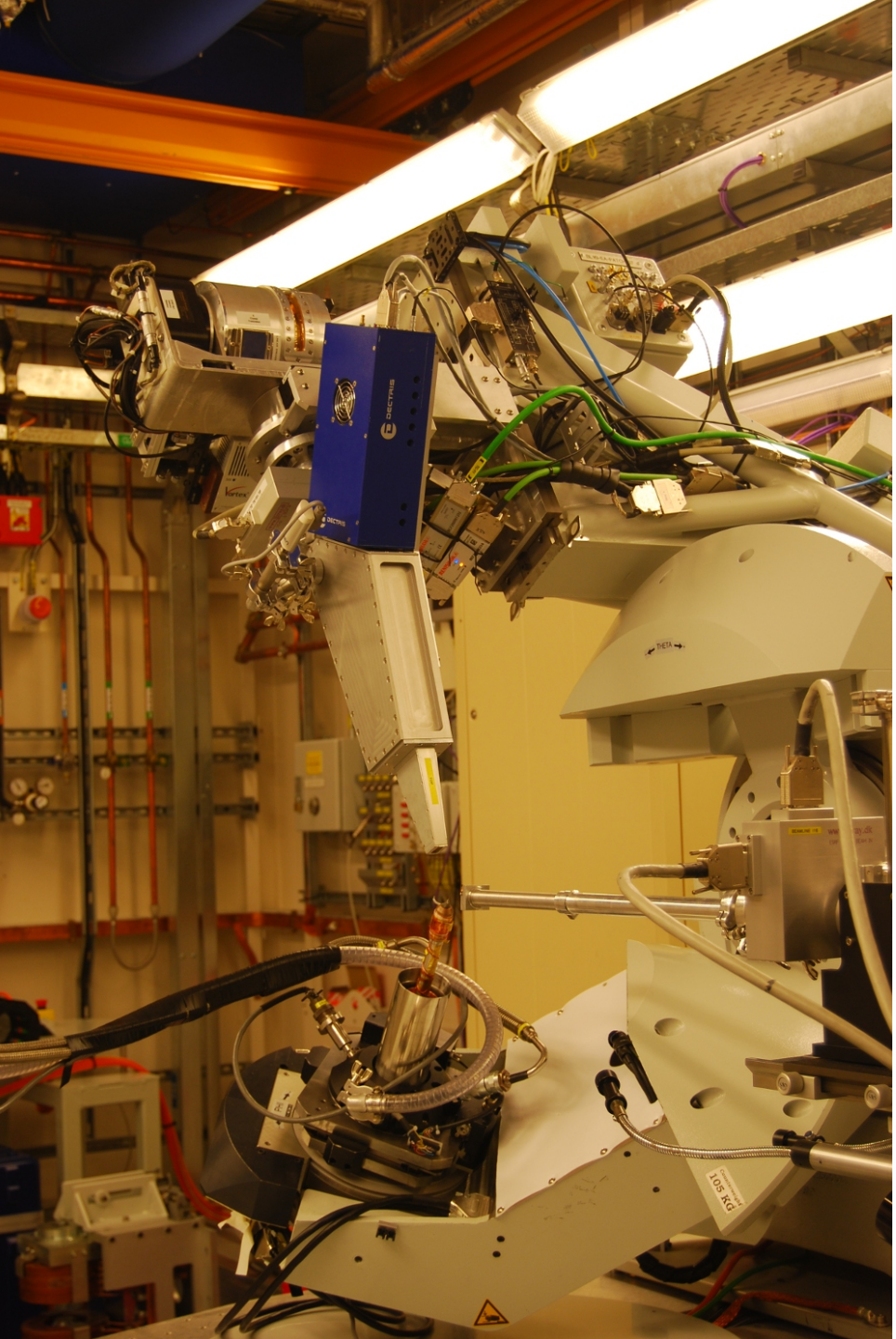
L(Q) and S(Q) are the Fourier transforms of the atomic and spin magnetisation densities; A" and B depend on the relative orientation of k, k', ε, ε'



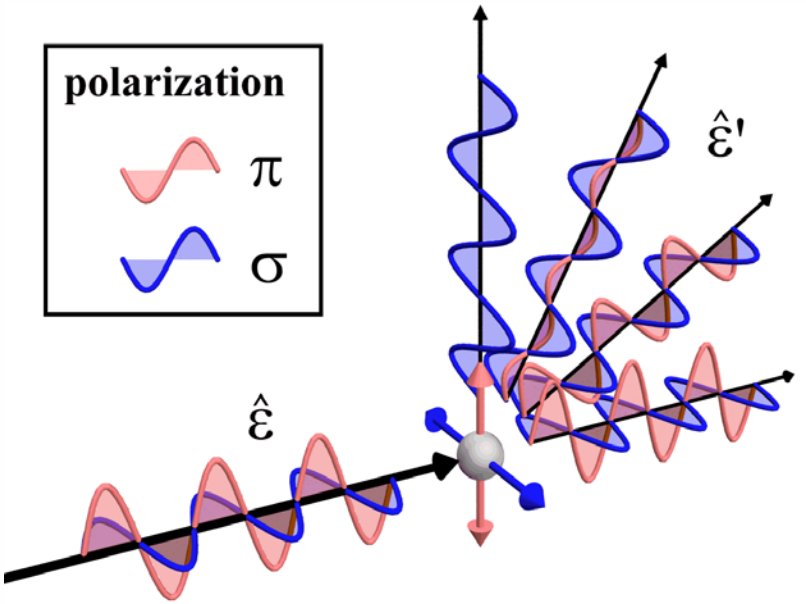
$$f^{mag}(\boldsymbol{Q}) = i\,r_0\left(\frac{\hbar\omega}{mc^2}\right) \times$$

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

diamond



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



diamond

Magnetic structures: X-rays

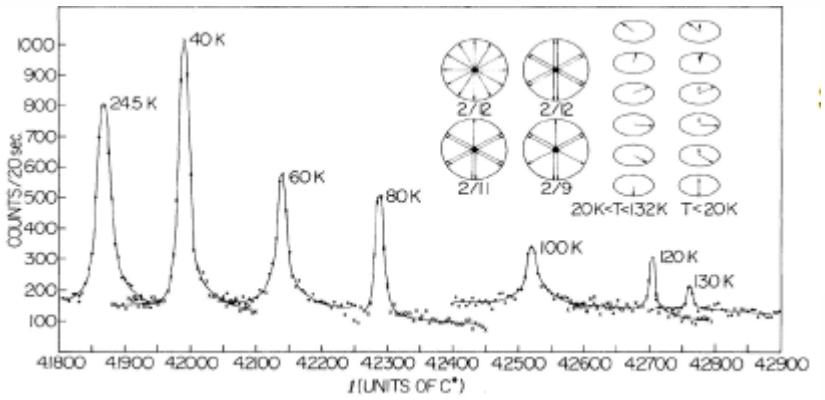
- First magnetic structures studied at a synchrotron in 1985 - spiral spin structure of Ho mapped out as a function of temperature

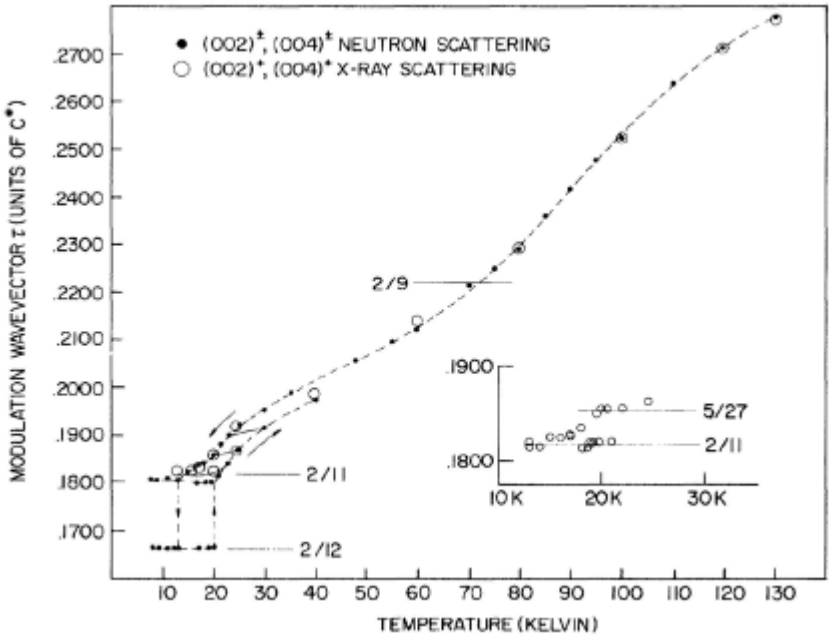
VOLUME 55, NUMBER 2 PHYSICAL REVIEW LETTERS 8 JULY 1985

Magnetic X-Ray Scattering Studies of Holmium Using Synchrotron Radiation

Doon Gibbs, D. R. Moncton, K. L. D'Amico, J. Bohr,[a] and B. H. Grier[b]
Department of Physics, Brookhaven National Laboratory, Upton, New York 11973
(Received 26 March 1985)

(1985) First Synchrotron Radiation Studies of Magnetism



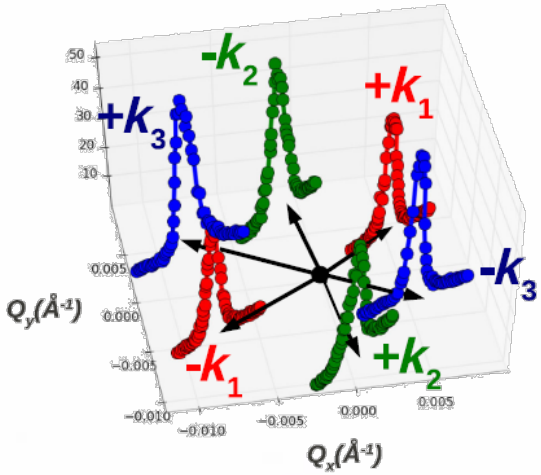


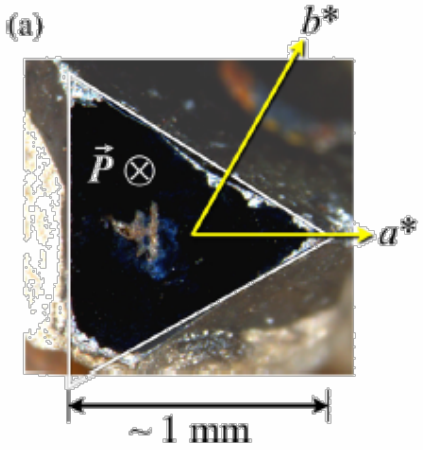
- Advantages of (non-resonant) X-ray magnetic scattering
  - Orbital and spin magnetisation density may be separated
  - Highly-focused beams enable very small samples to be studied
  - High resolution in Q – e.g. for subtle changes in spin structure
  - ***However***, cryogenics are challenging !

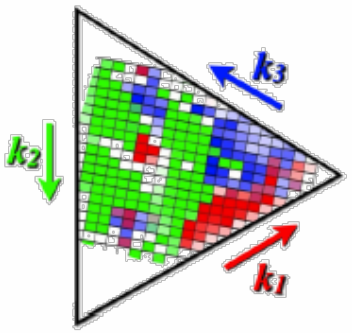
diamond

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



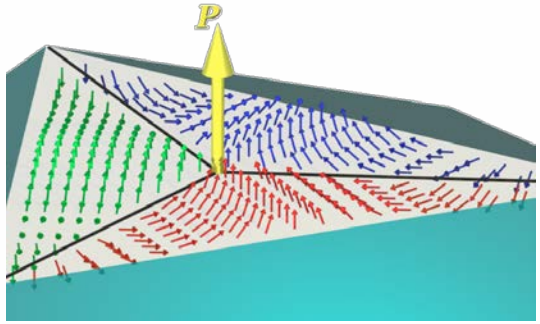




PRL 110, 217206 (2013) PHYSICAL REVIEW LETTERS week ending 24 MAY 2013

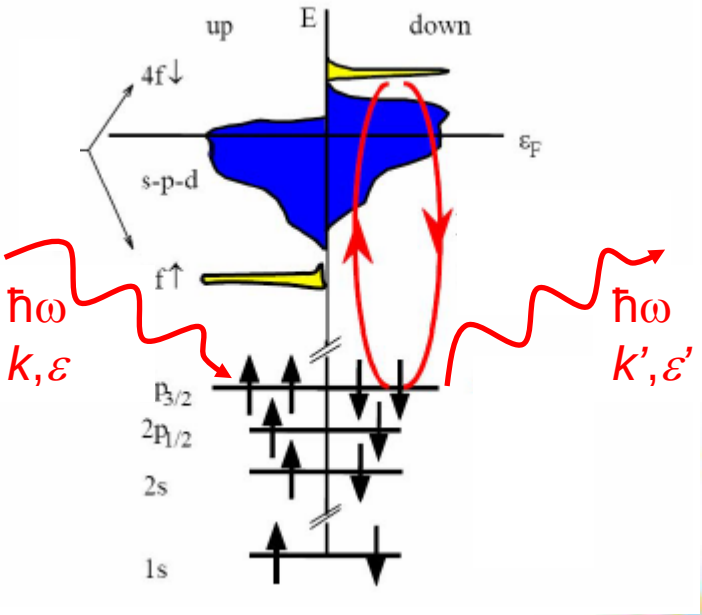
X-Ray Imaging and Multiferroic Coupling of Cycloidal Magnetic Domains in Ferroelectric Monodomain $BiFeO_3$

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]



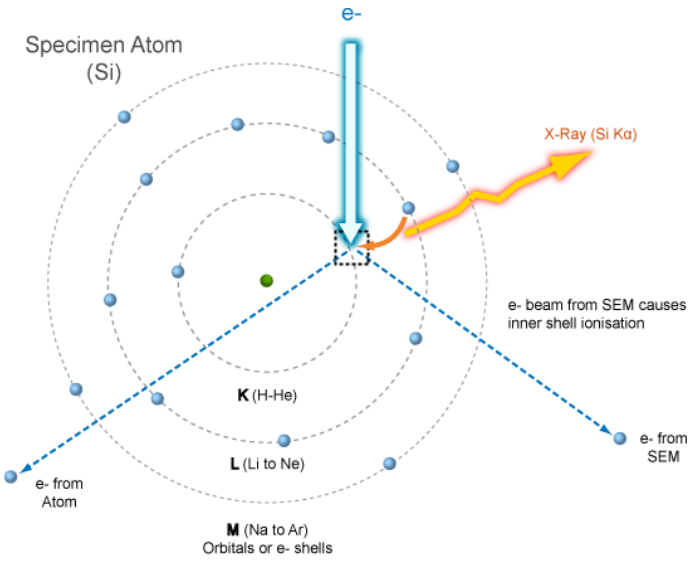
Magnetic structures: resonant scattering

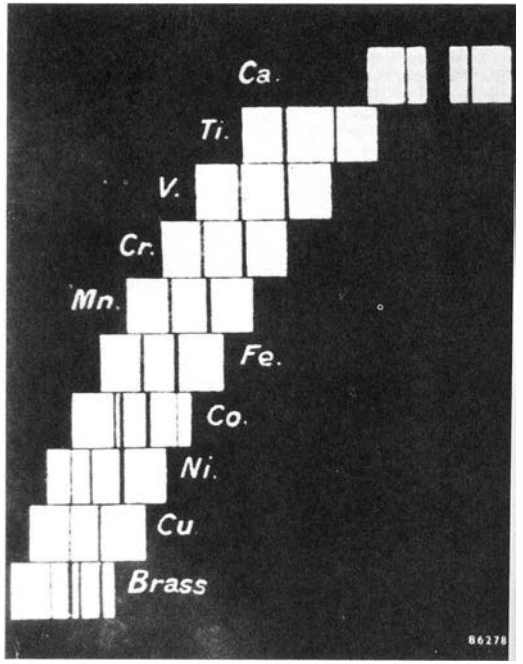
- Tune energy to a core-(empty) valence state transition and exploit resonant scattering to greatly enhance intensity (x10^3)
  - Sensitive to specific element and oxidation state
  - Dipole transitions ($\Delta L = 1$) generally stronger than quadrupole transitions ($\Delta L = 2$) and these will also 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 2p → 4f	6 - 10	medium	dipolar quadrupolar
4f	M_I	3s → 5p	1.4 - 2.5	weak	small overlap
4f	M_{II}, M_{III}	3p → 5d 3p → 4f	1.3 - 2.2	medium to strong	dipolar 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



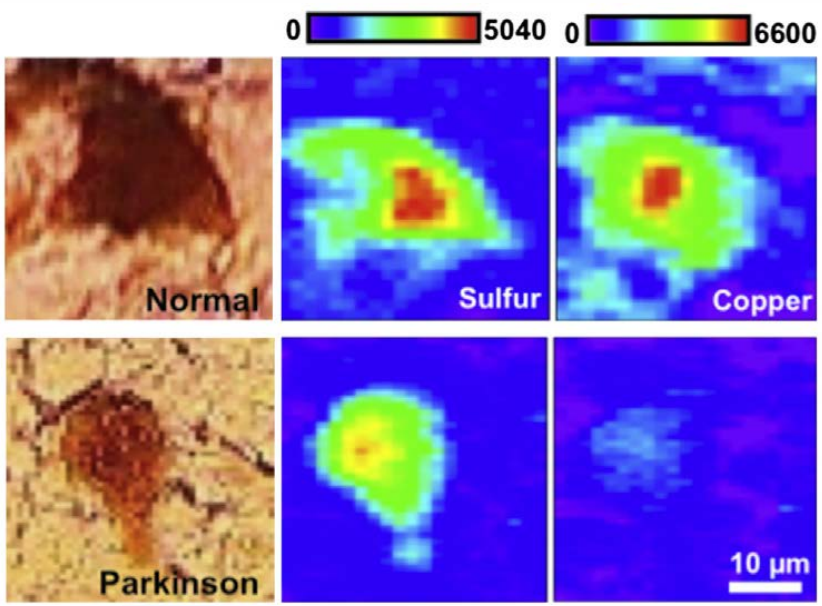


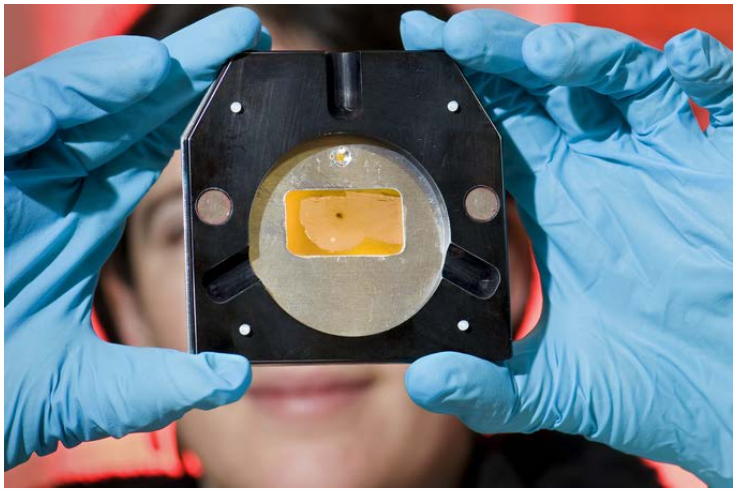
H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo		Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La		Ta	W		Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po		Rn
	Ra	Ac	Th		U												

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

diamond

Mapping elements – but what about chemistry ?

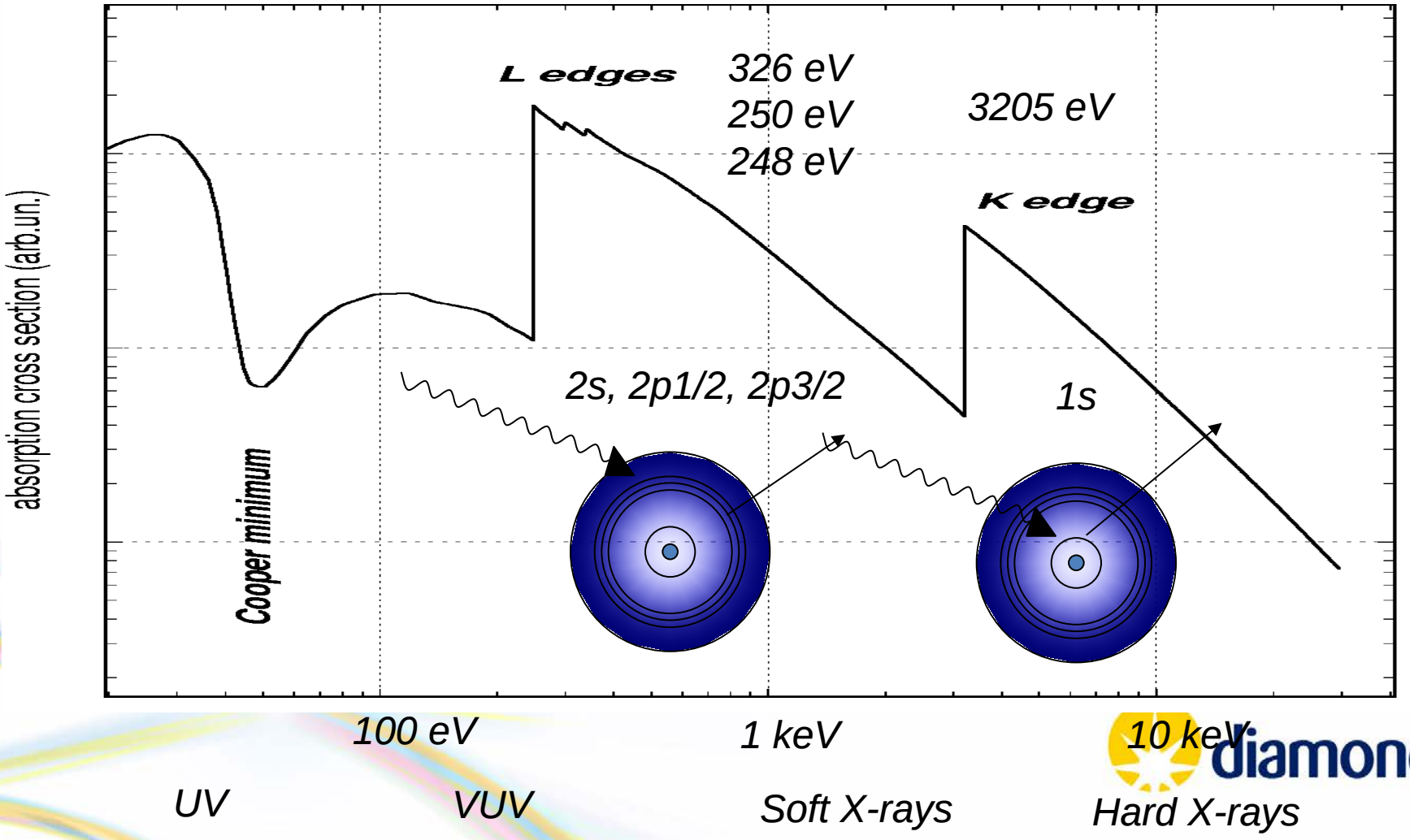




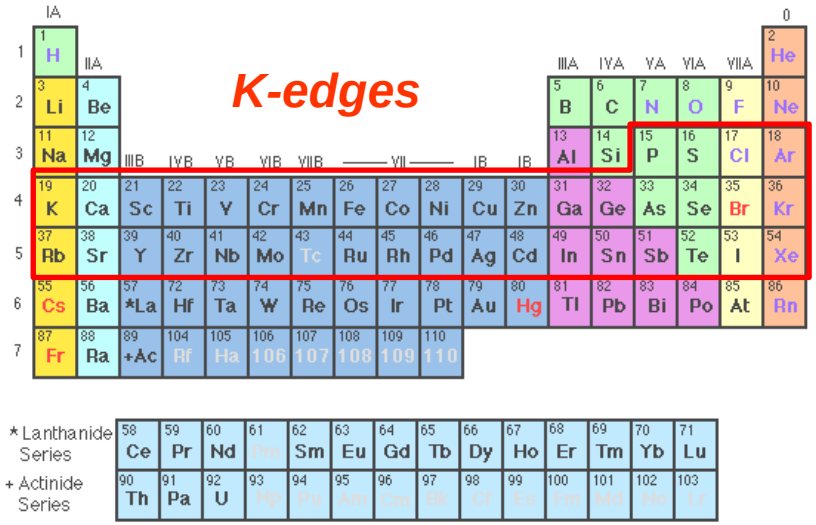
diamond

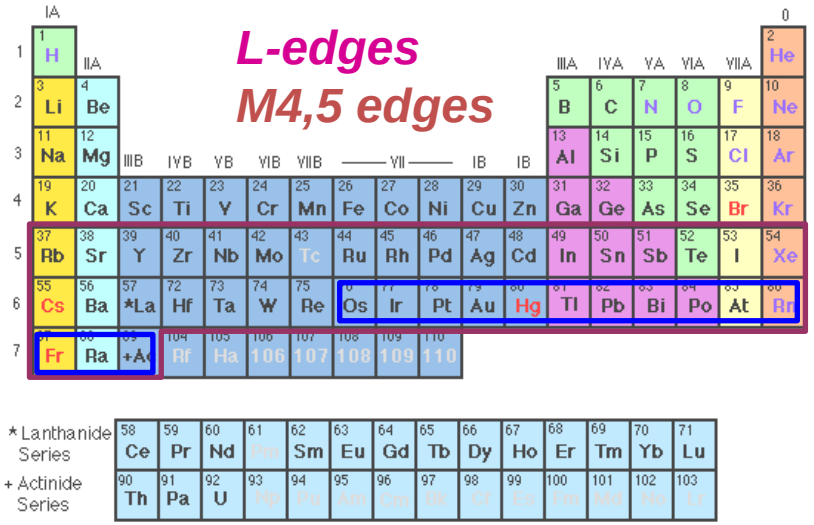
Exploring electronic structure

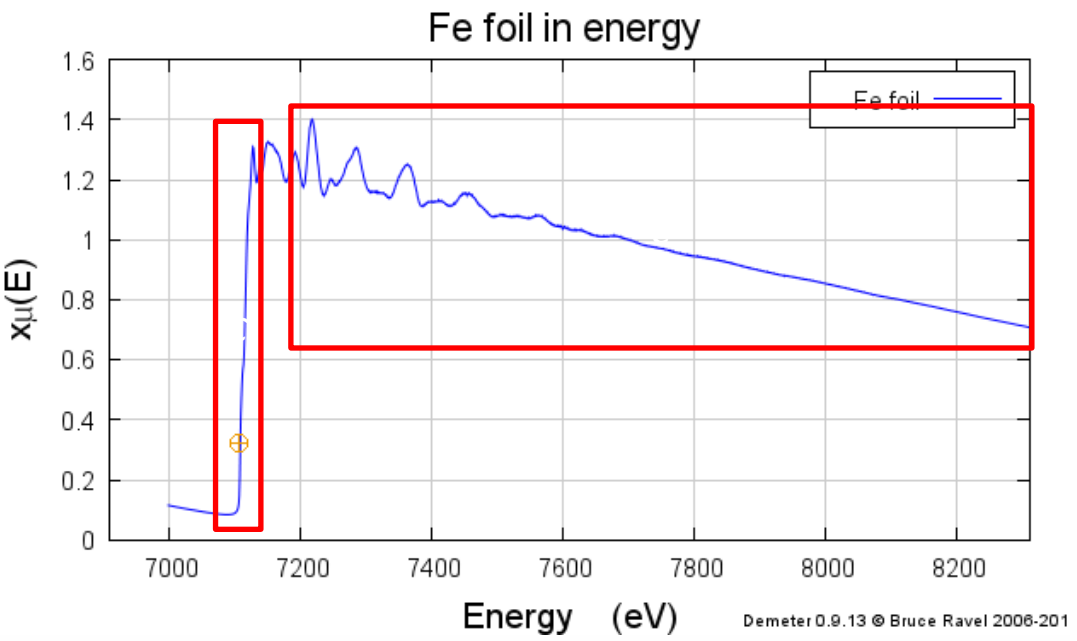
- Atomic levels - e.g. Argon – $1s^2\ 2s^2 2p^6\ 3s^2 3p^6$



Sensitivity to chemical environment



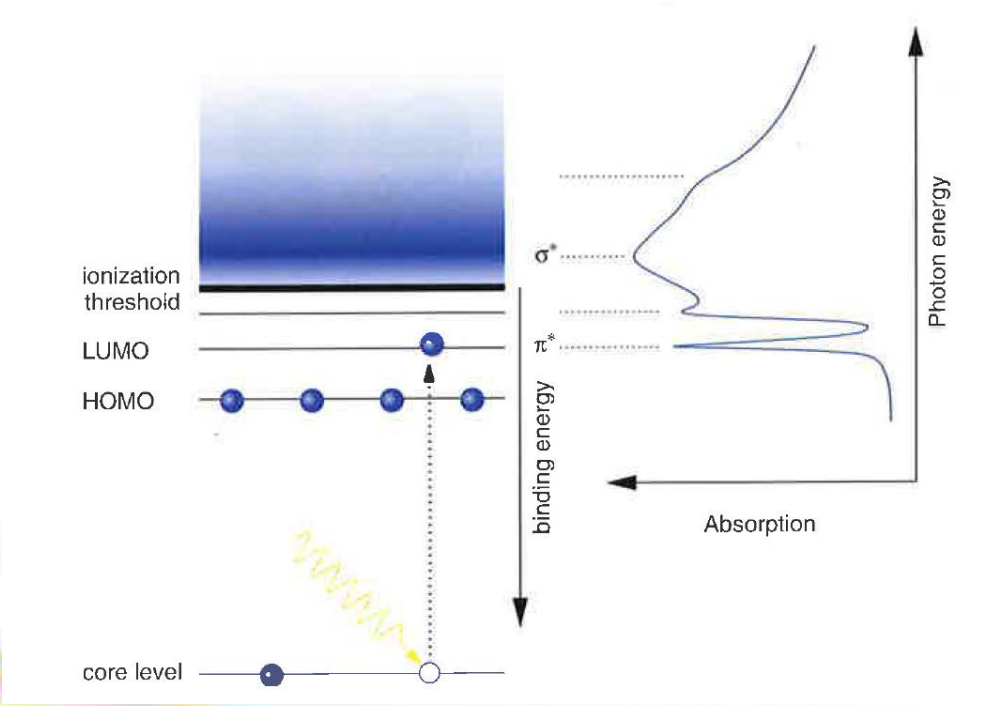


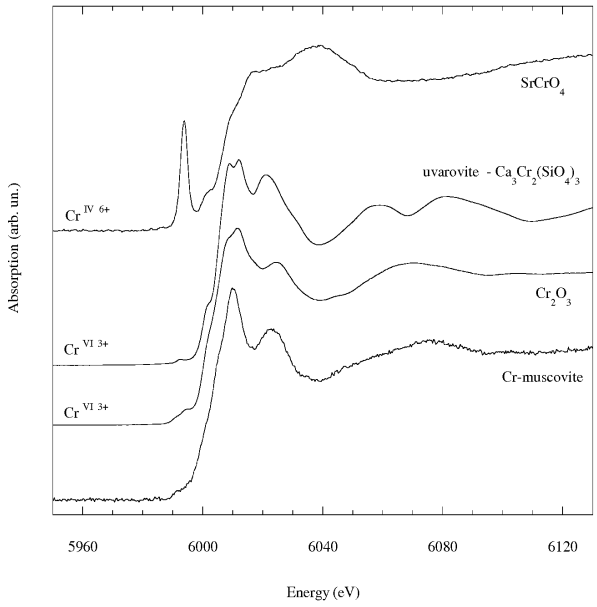


diamond

XANES

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

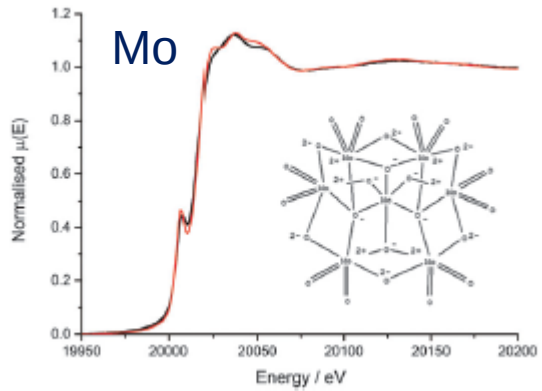


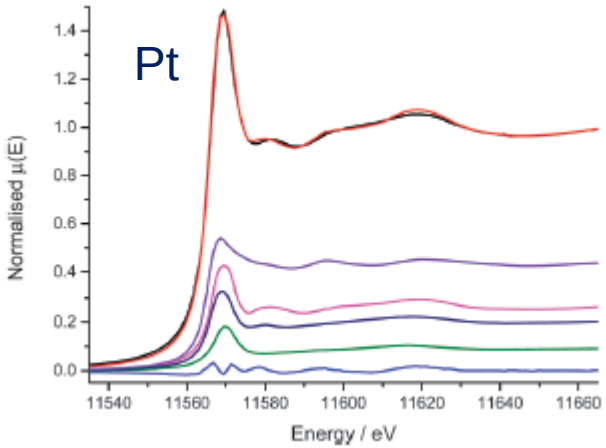


Cr K absorption edge

diamond

Unravelling chemical clusters

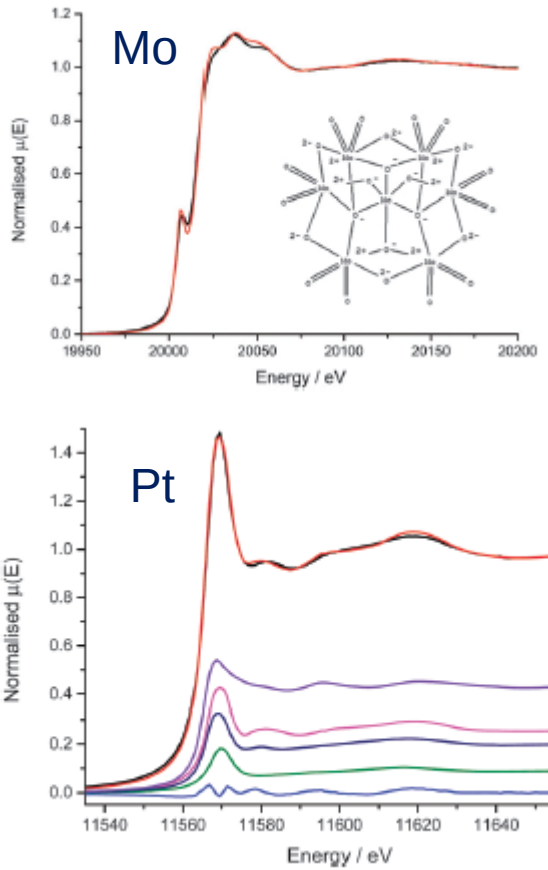




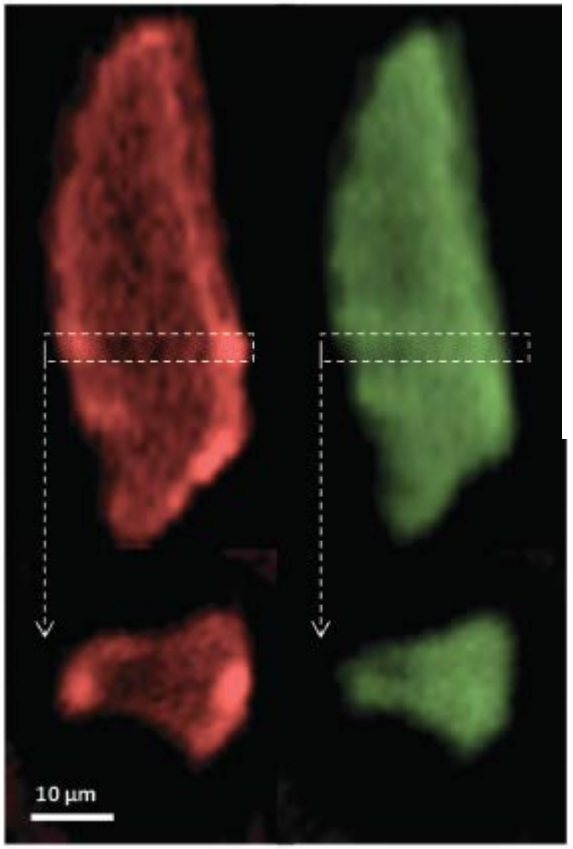
Species identification

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

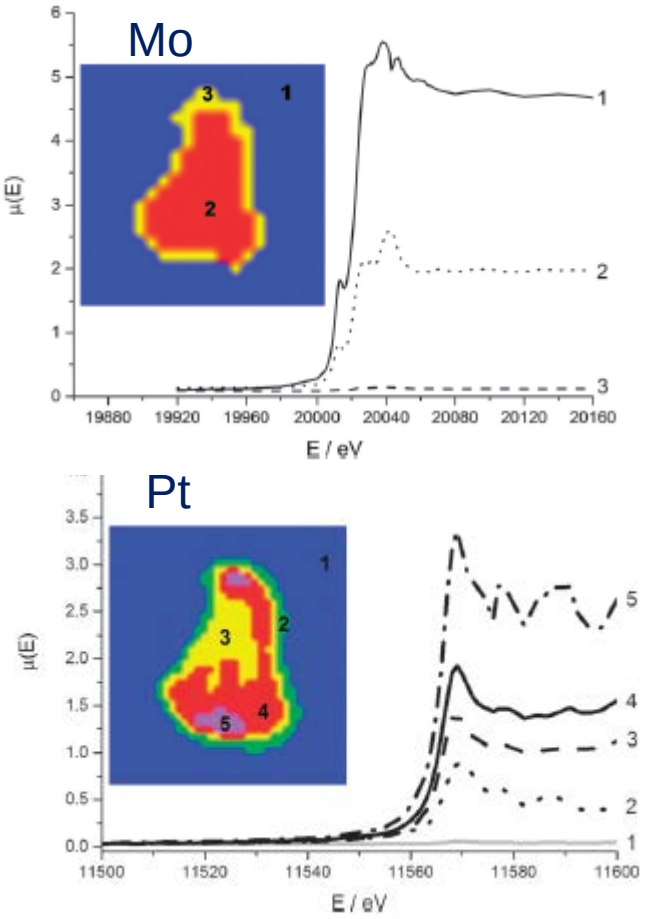
…and mapping where they are



Species identification



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

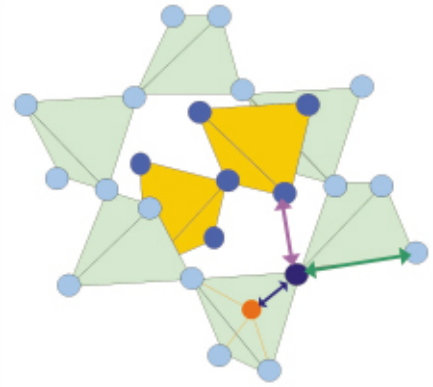


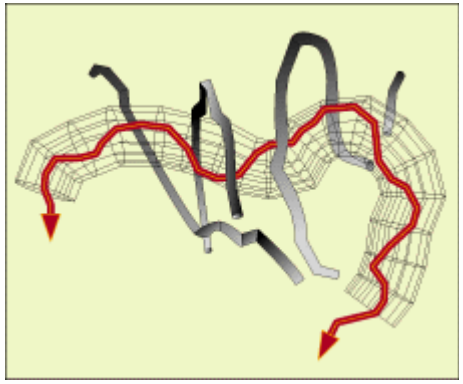
Species distribution

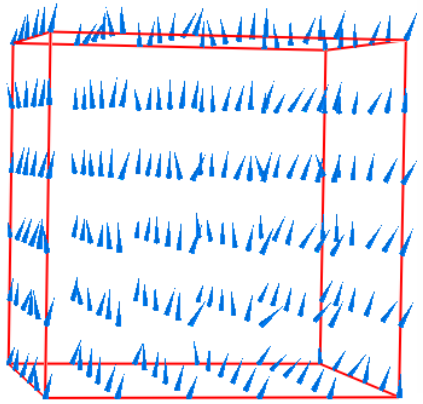
Price *et al*, PCCP, Phys. Chem. Chem. Phys., 17 (2015) 521

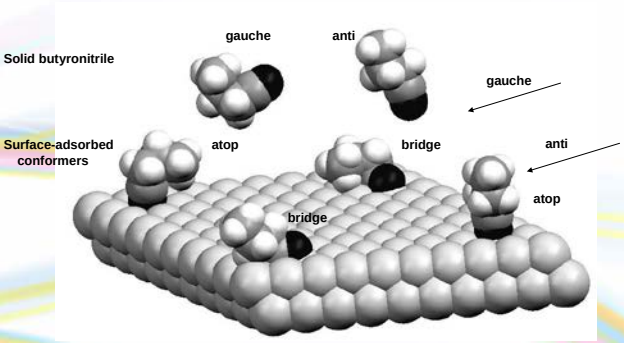
Higher resolution spectroscopy: atomic and molecular motion in materials

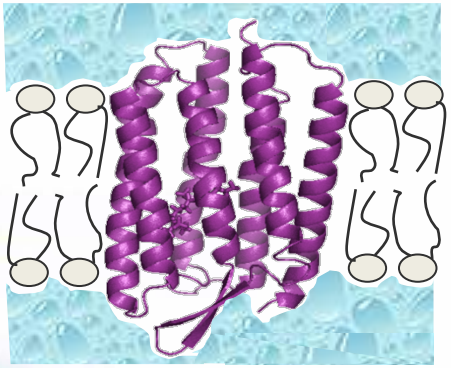
- Vibrations and magnetic excitations in molecules and solids are essential to key processes e.g. thermal conductivity and expansion, polymer processing and properties, biochemical processes, magnetic storage media, superconductors –

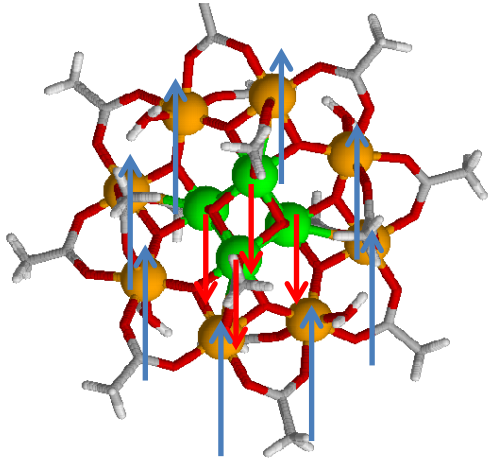








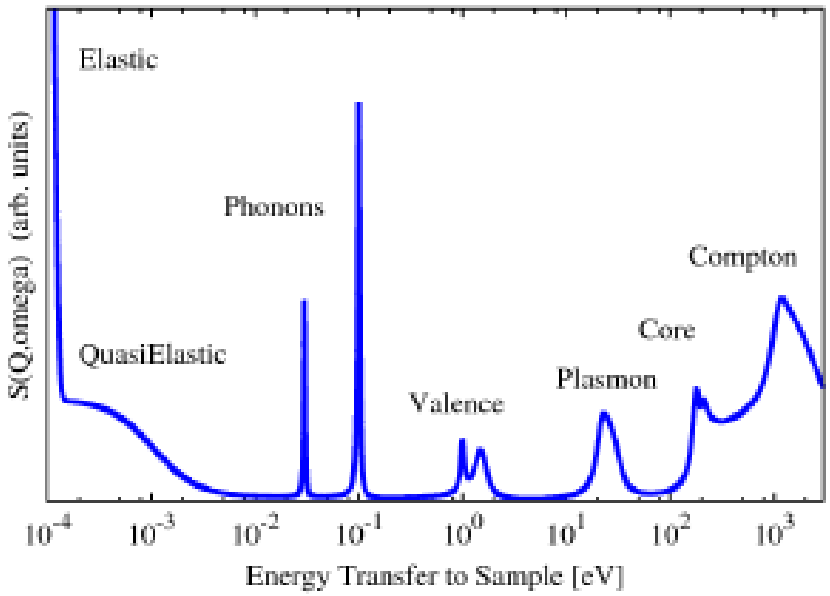


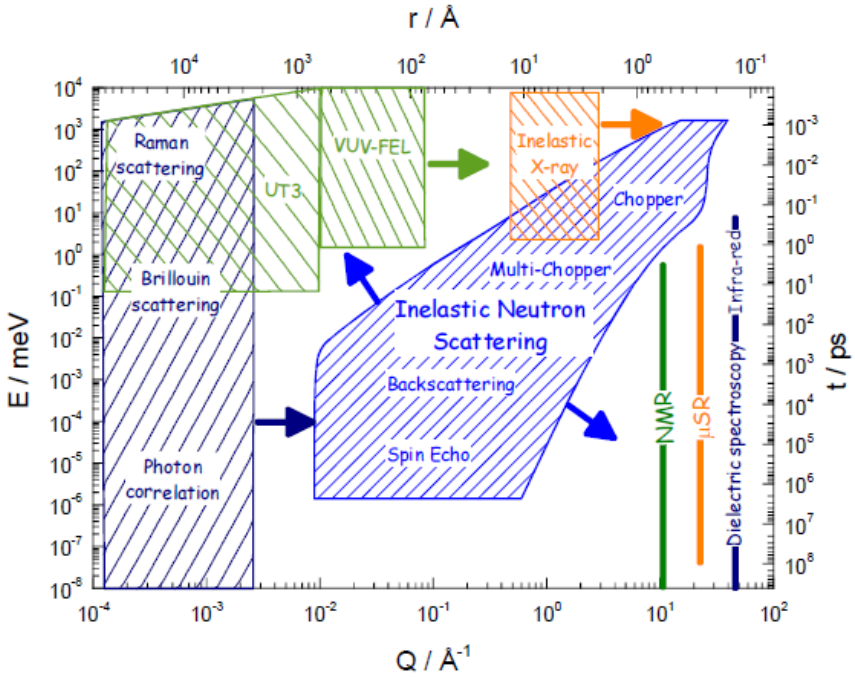


Atomic and molecular motion in materials

"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.

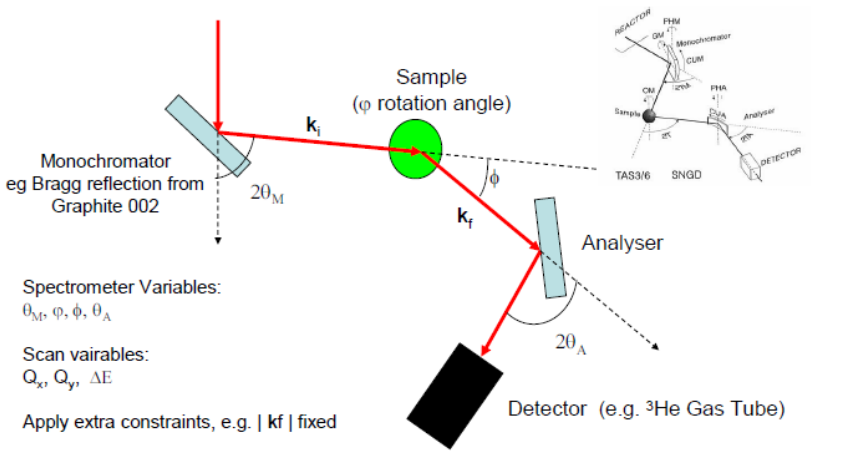
- Typically wish to measure vibrations and magnetic excitations in the range 1-100 meV as a function of q ($1/\lambda$) so the wavelength of the neutrons or X-rays should typically be of the order of 1-2 Å (0.1-0.2 nm) [Q – measured momentum transfer; q – wavevector of the excitation]
- 1 Å corresponds 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)

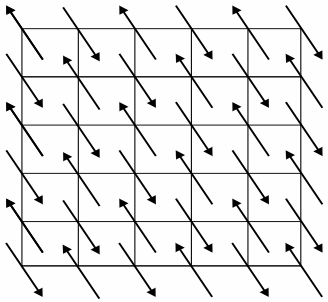


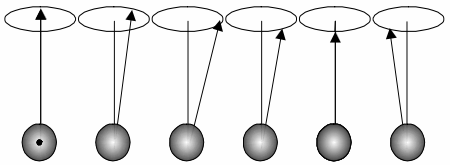


INS: experimental methods

- 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 !)

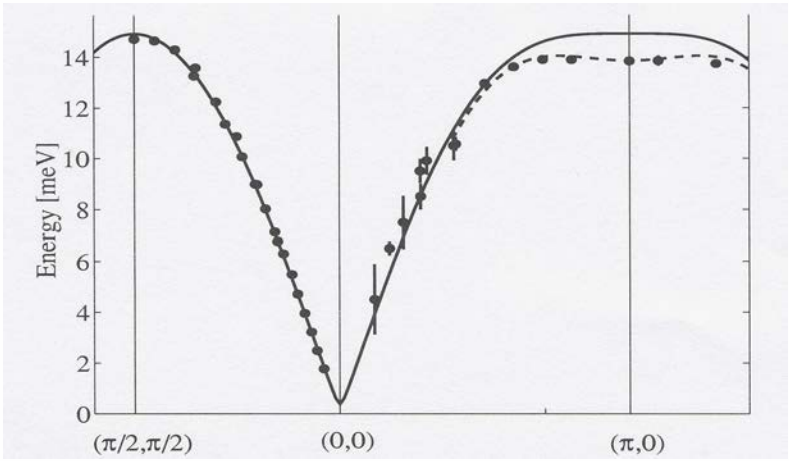






$Q = k_f - k_i$ $\hbar\omega = E_f - E_i$ $E_{f,i} = \hbar^2k^2/2m_n$

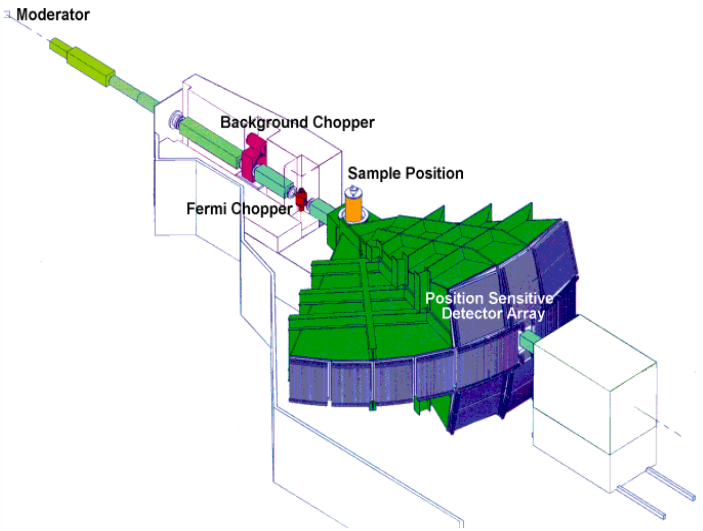
$\lambda=\infty$ $\lambda=2a$

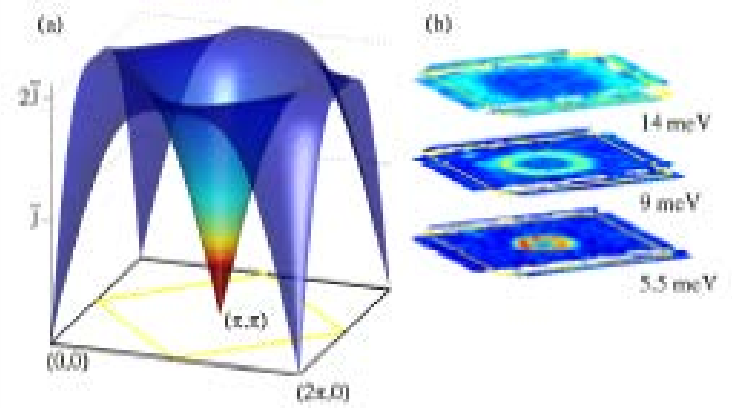


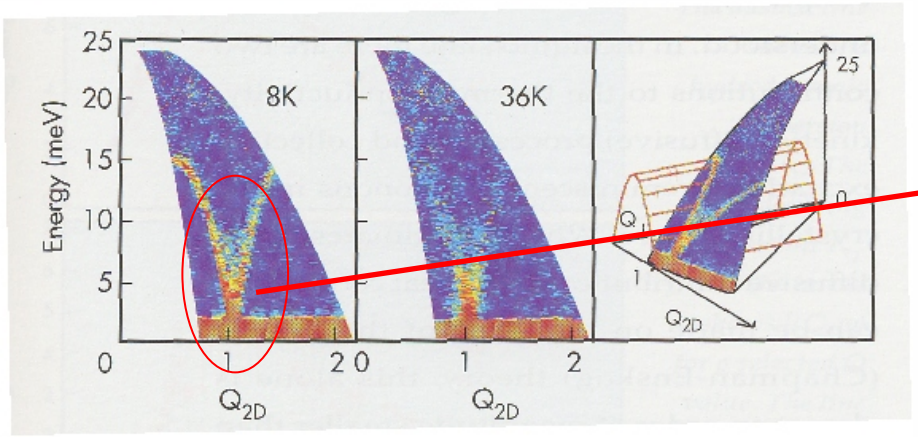
qa

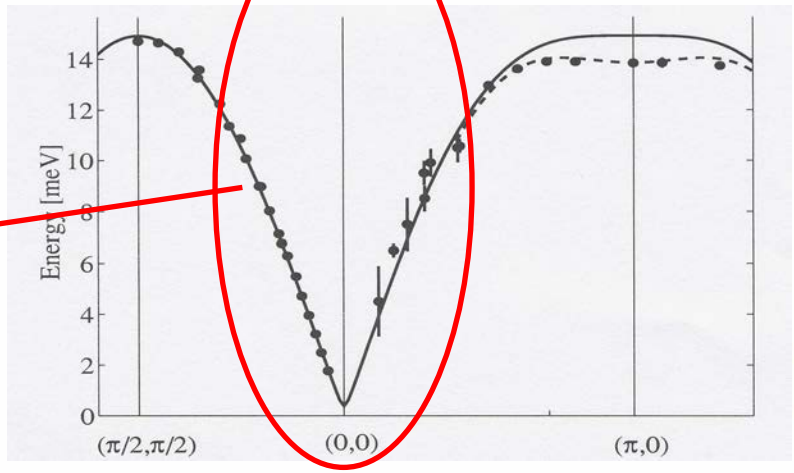
INS: experimental methods

- 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







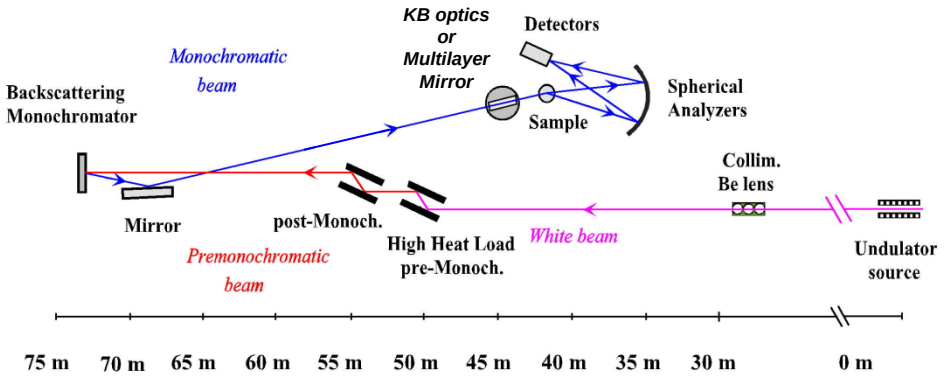


IXS: experimental methods

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

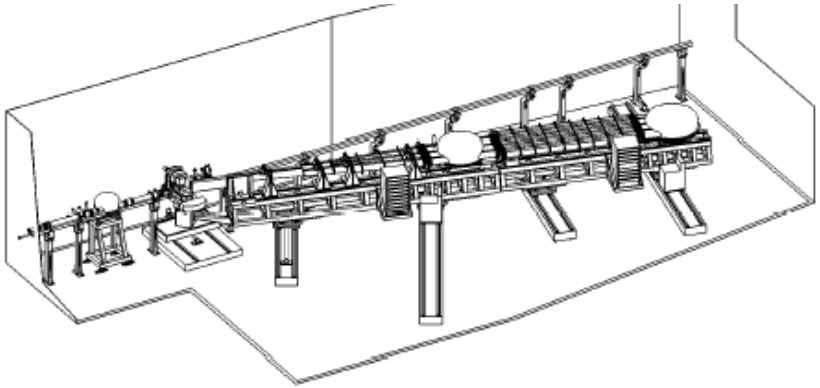
Optimise resolution with monochromator and analyser close to backscattering configurations:

$d\lambda/\lambda = dE/E = \cot\theta d\theta \rightarrow 0$ 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	$1.9 \cdot 10^{-8}$

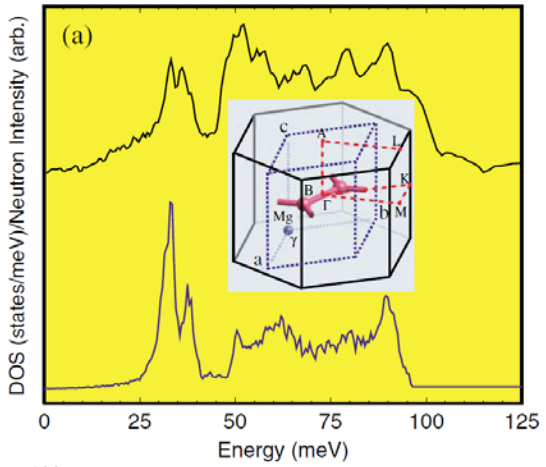
9- analyser crystal spectrometer

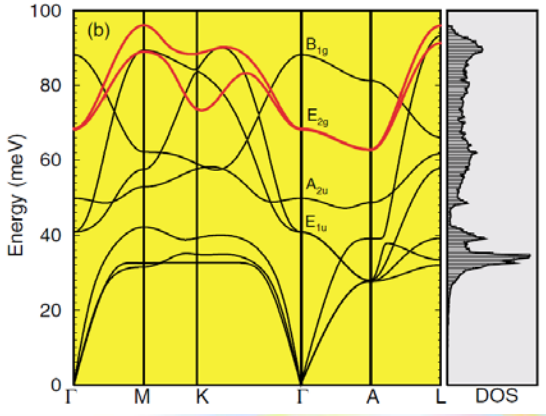


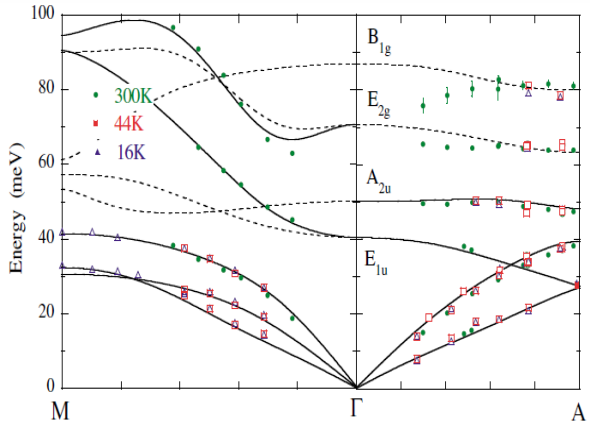
diamond

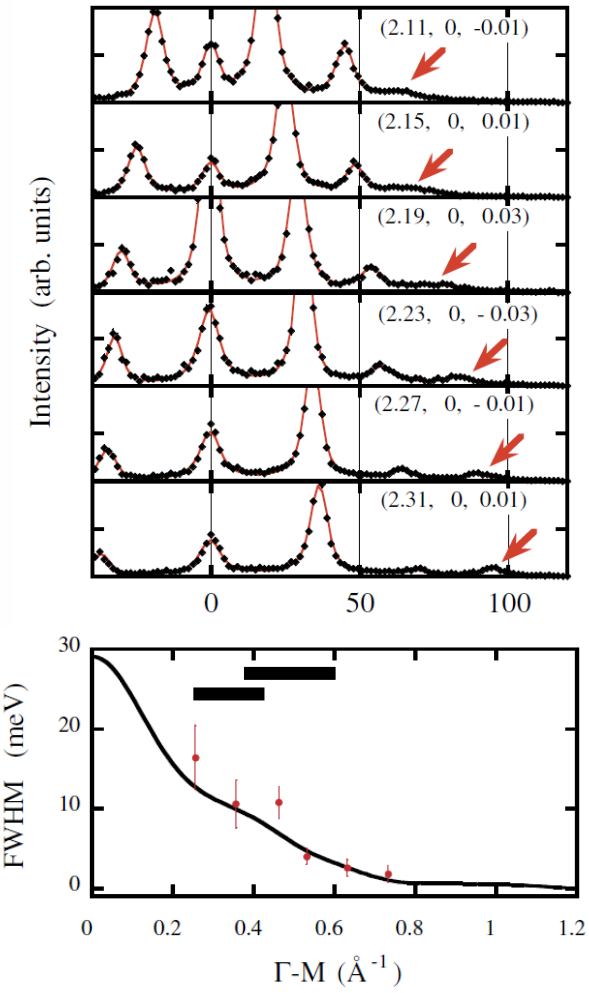
IXS: applications

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





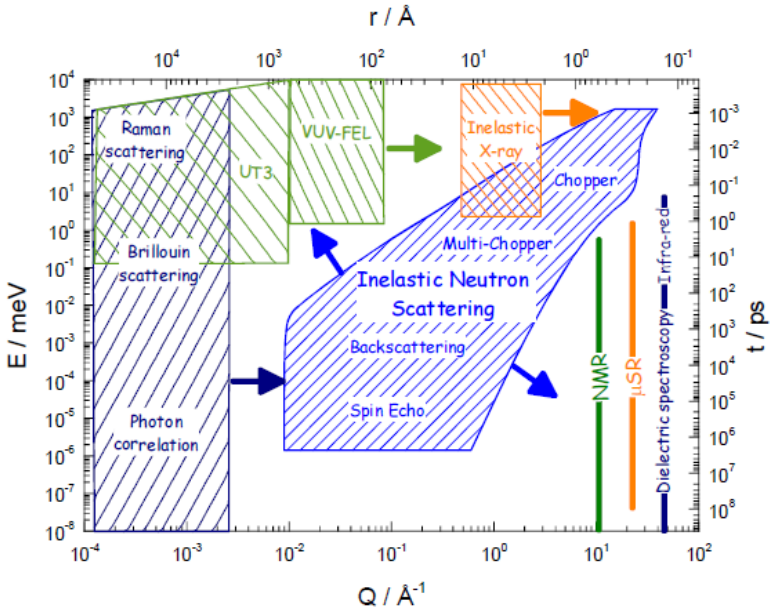


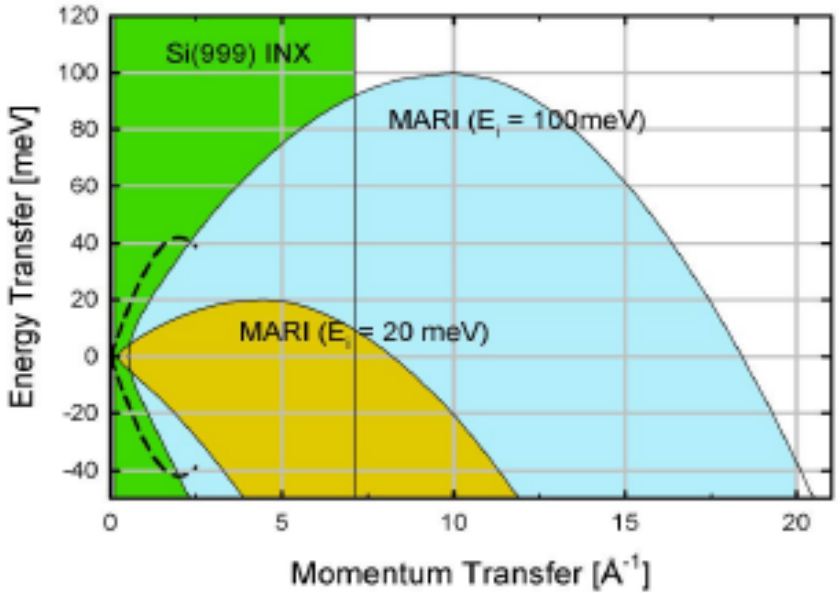


Yildirim et al, PRL 87 (2001) 37001
Baron et al, PRL 92 (2004) 197004

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 $\Delta E/E \sim 10^{-7}$
  - Cross-section dominated by photoelectric absorption - less sensitive to light atoms
  - Resolution more complex than for neutrons (non Gaussian)



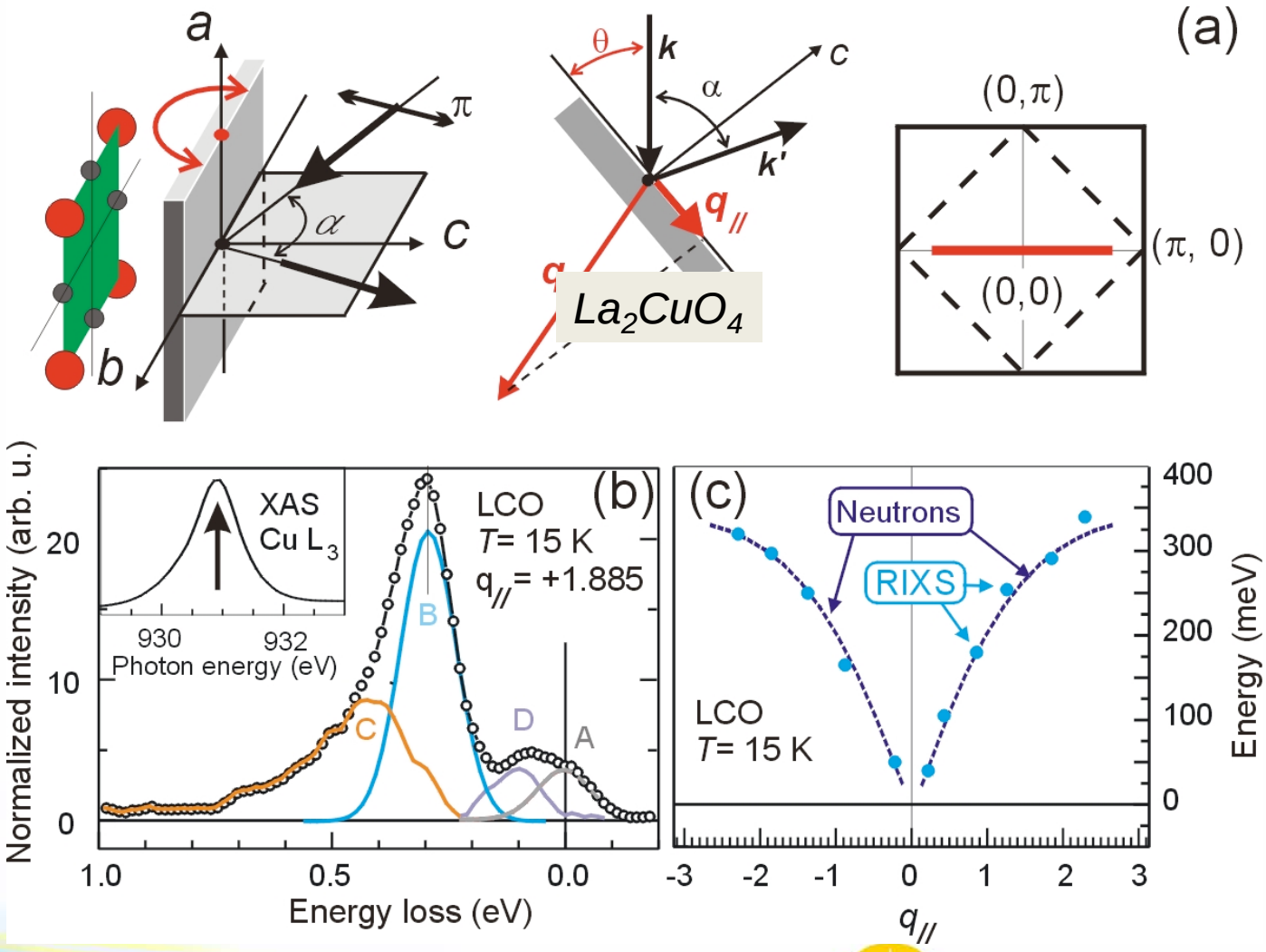


IXS +R → RIXS: a complementary method to neutrons for studying magnetic excitations

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

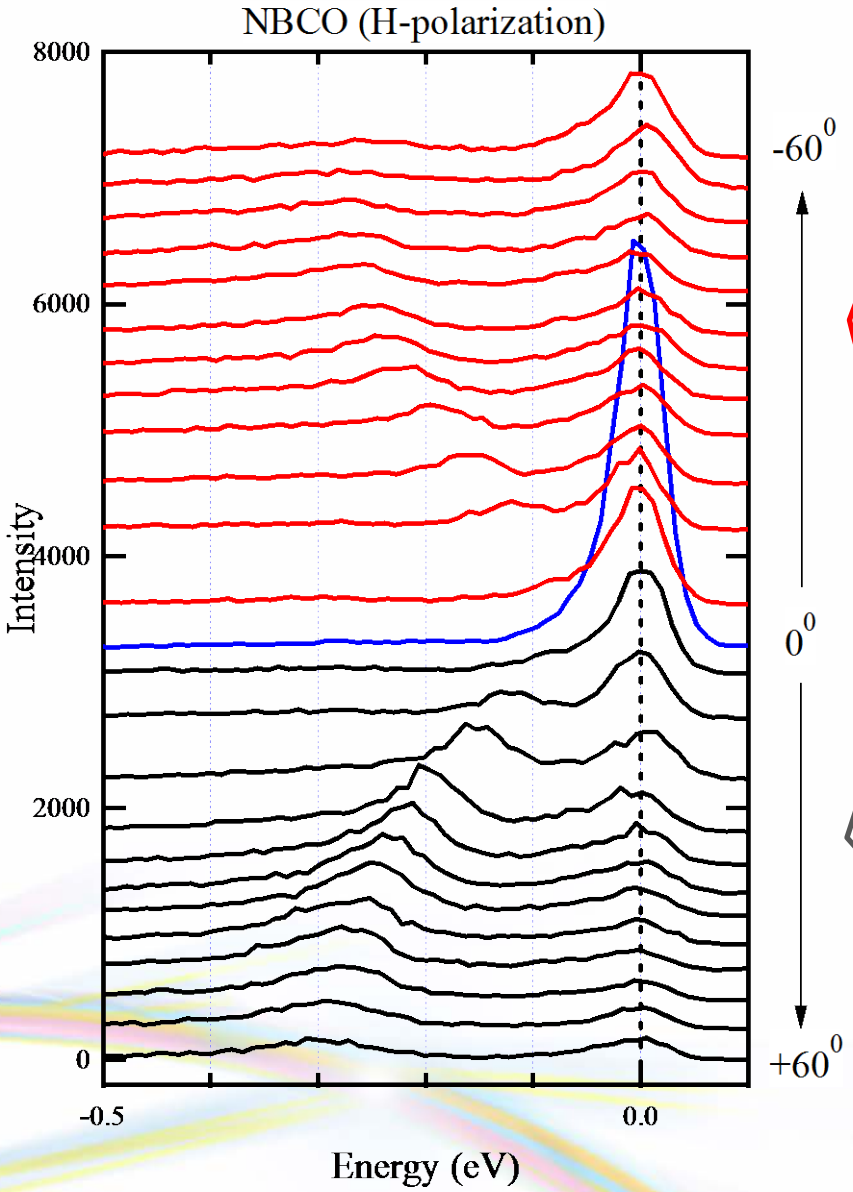
2015: ERIXS, ID32 – ESRF/Milano

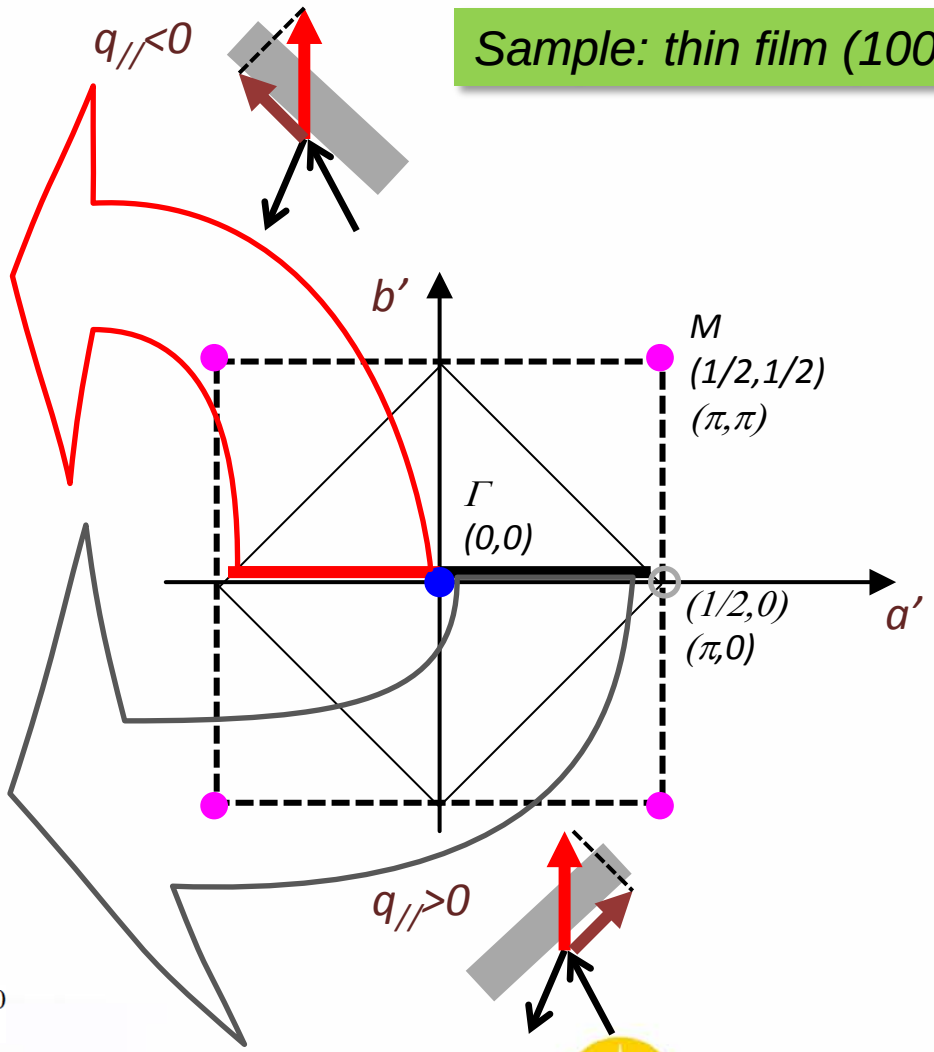
L = 12.0 m

Design: E/ΔE = 42000 at Cu-L_3

Antiferromagnetic $NdBa_2Cu_3O_{6+\delta}$

ESRF
ERIXS@ID32





Sample: thin film (100 nm)

π pol. – spin-flip

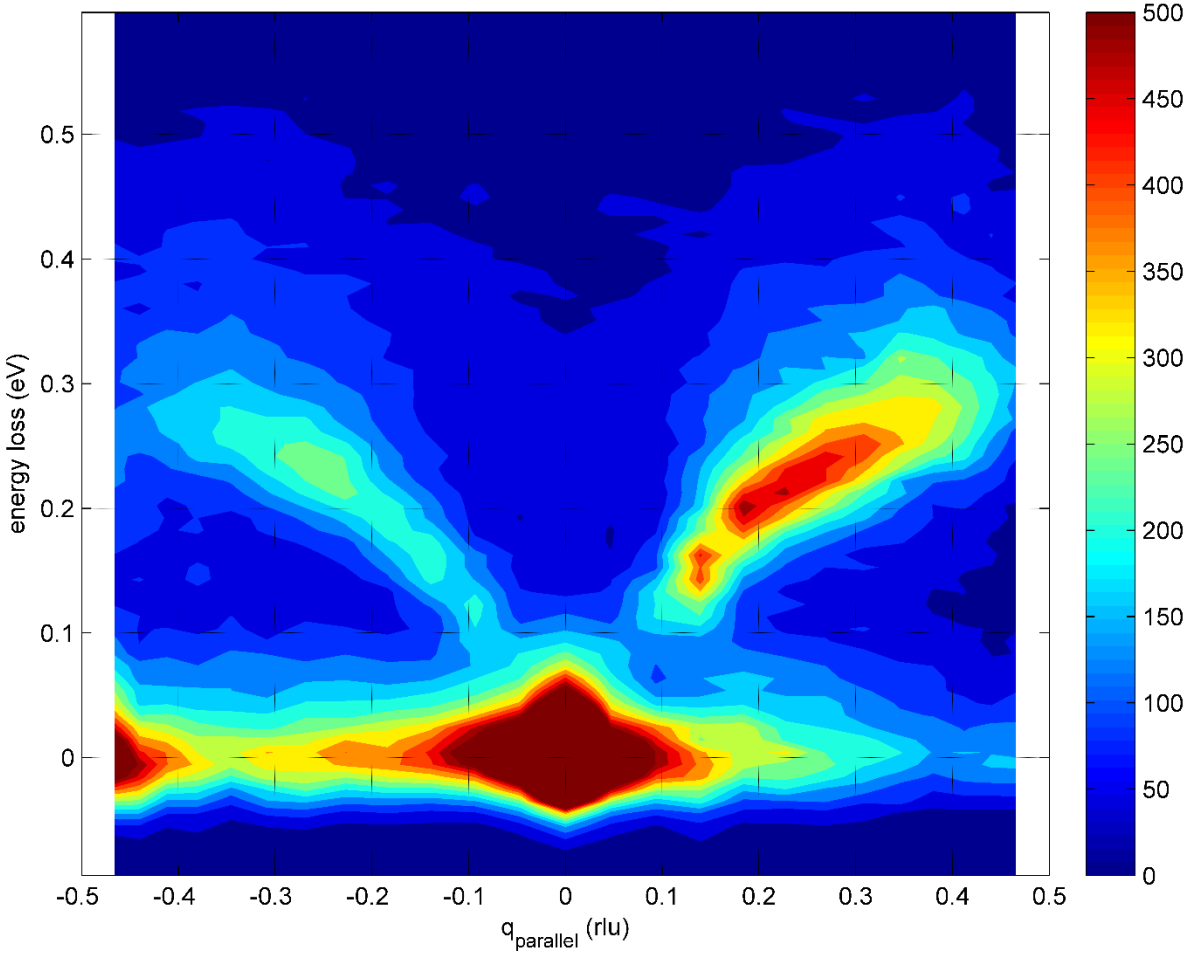
Combined BW ≈ 55 meV

"Low" resolution set up at ID32 – July 2015

AF $NdBa_2Cu_3O_{6+\delta}$

ESRF
ERIXS@ID32

Sample: thin film (100 nm)

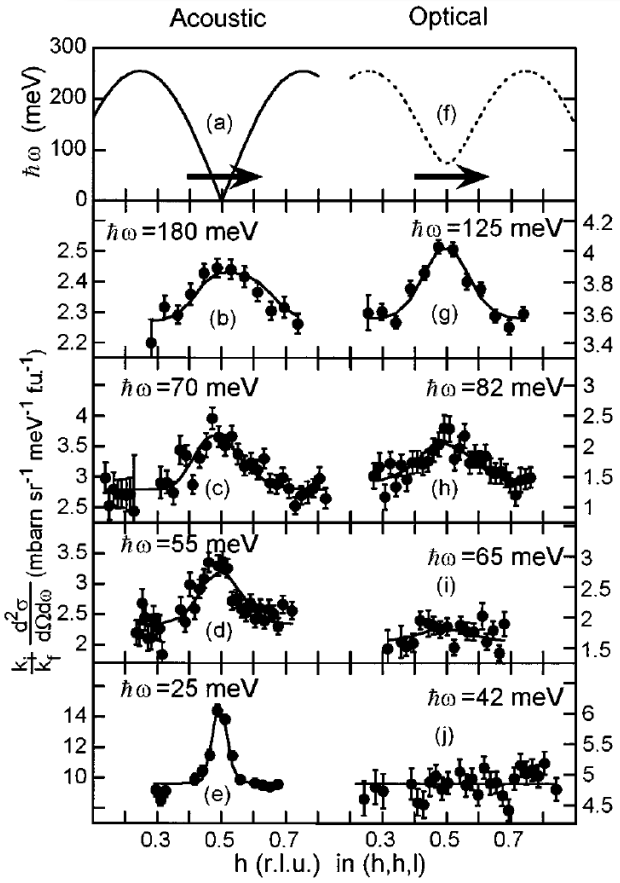


diamond

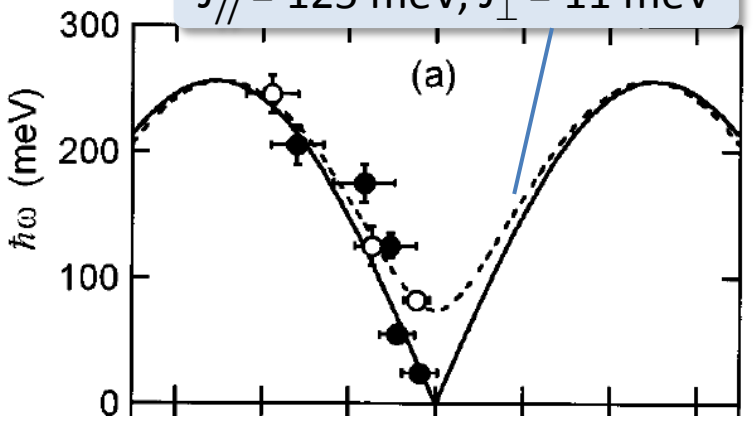
ESRF ID32 – July 2015

AF YBCO and NBCO: magnon optical branch

INS: BW ≈ 2 meV

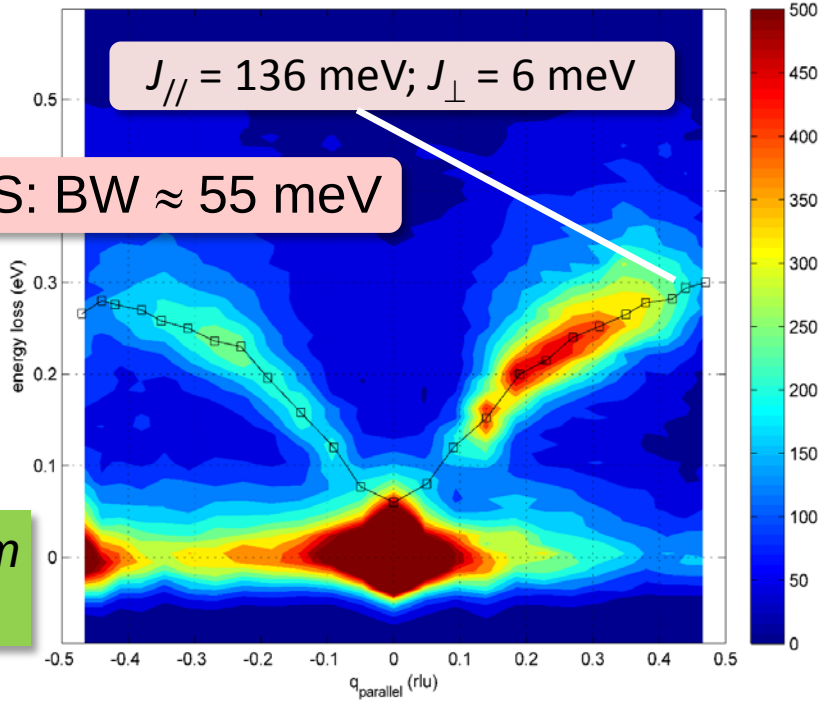


$J_{//}$ = 125 meV; $J_{\perp}$ = 11 meV



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

RIXS: BW ≈ 55 meV



ESRF
ERIXS@ID32

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

Sample: thin film (100 nm)

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

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

Neutrons and X-rays

Andrew Harrison, Diamond Light Source

14th Oxford School on Neutron Scattering - 2015

Further reading

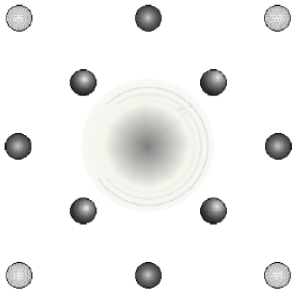
- Introductions
  - P. Willmott, *'An introduction to synchrotron radiation',* Wiley 2011
  - J. Als-Nielsen and D.McMorrow, *'Elements of modern X-ray physics'*, Wiley 2011
- Magnetic X-ray scattering
  - S.W.Lovesey and S.P. Collins, *'X-ray scattering and absorption by magnetic materials*', Oxford 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

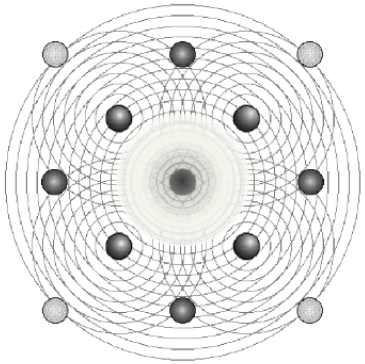
diamond

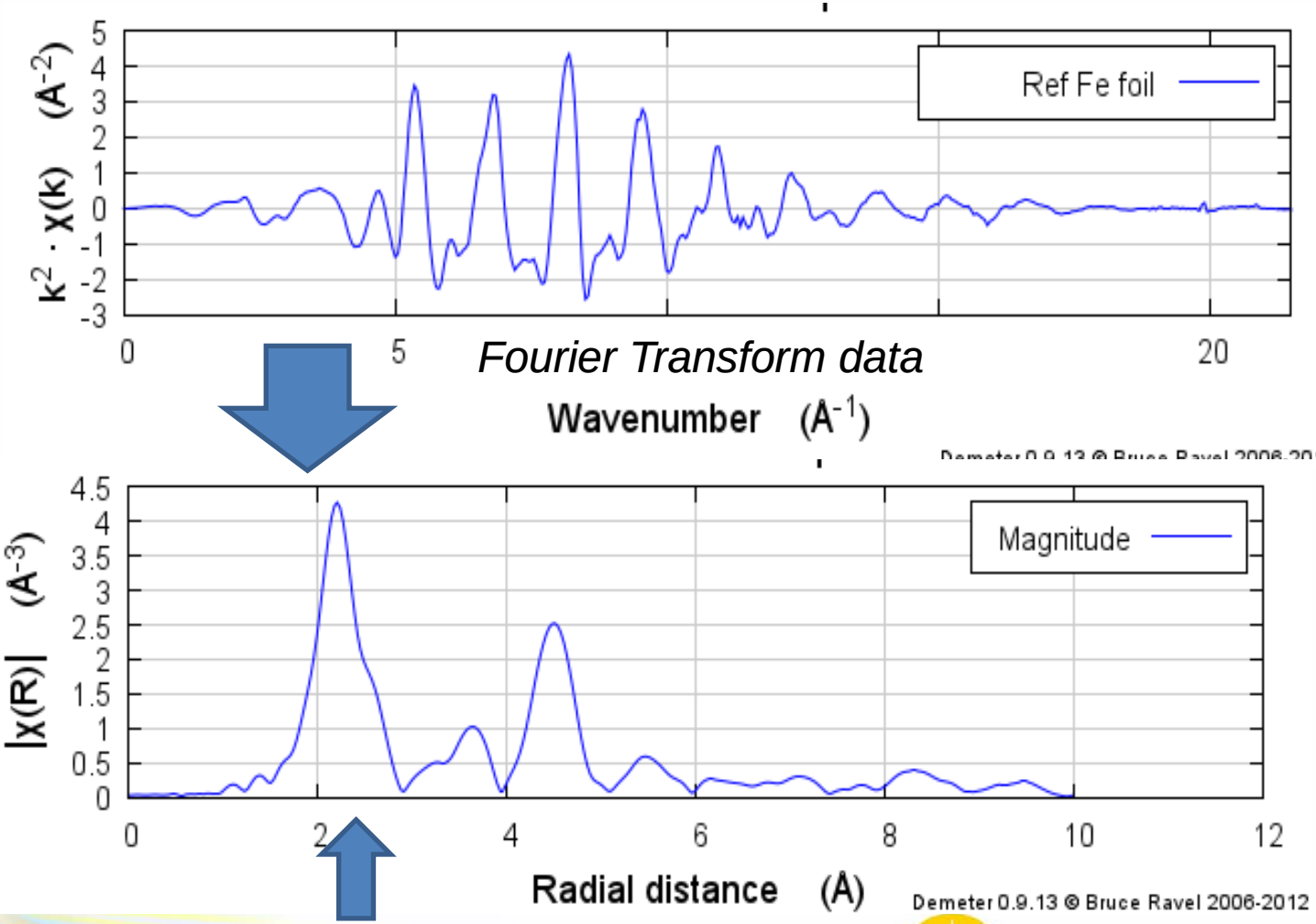
EXAFS

- Extended X-Ray Absorption Fine Structure

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





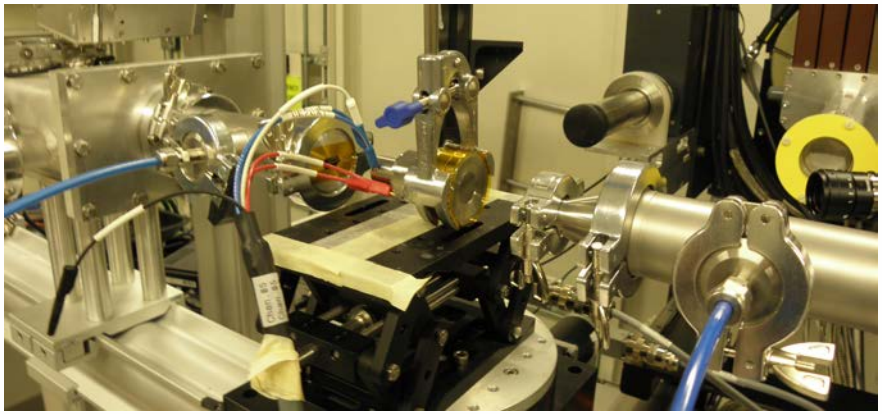


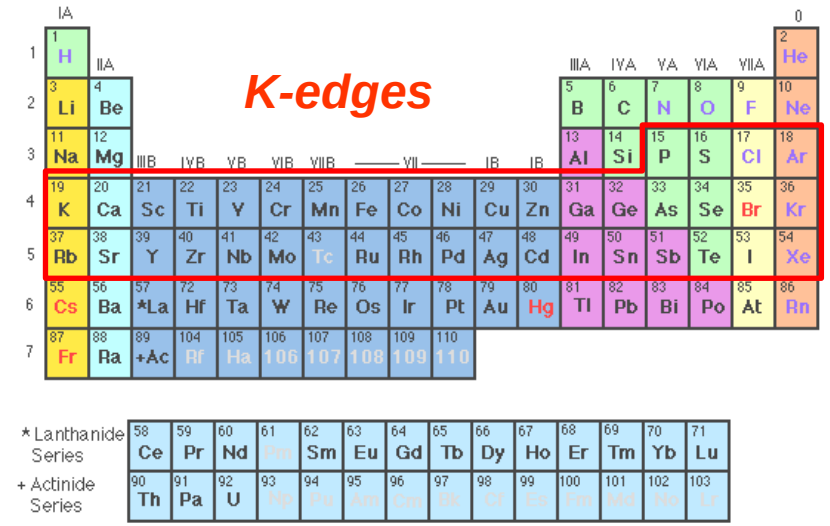
Detecting Fe atoms AROUND absorber Fe
Position: At about 2.5 Angstrom
Coordination: 8 next neighbours

diamond

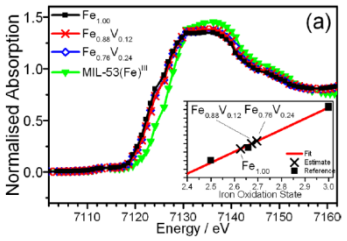
B18 – general purpose XAS

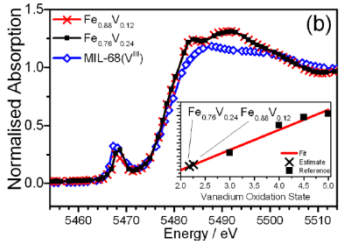
- 2.05 – 35 keV – covers absorption edge from P to U with 200 x 250 μm spot size

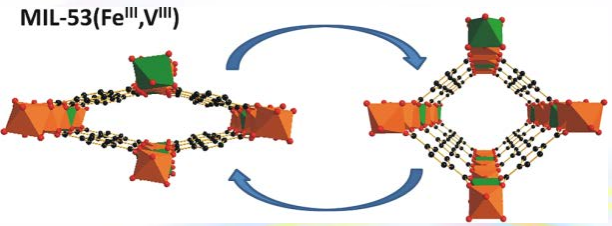


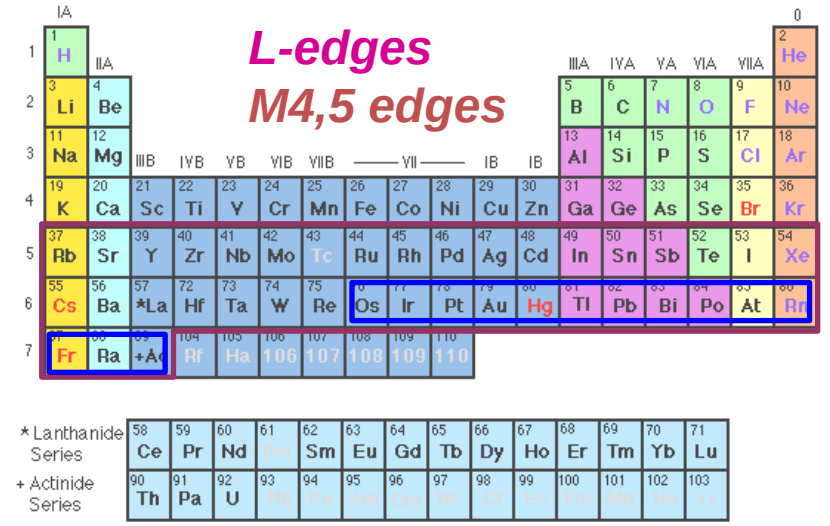


Determining oxidation states of Fe and V in flexible mixed valent MOF by XANES



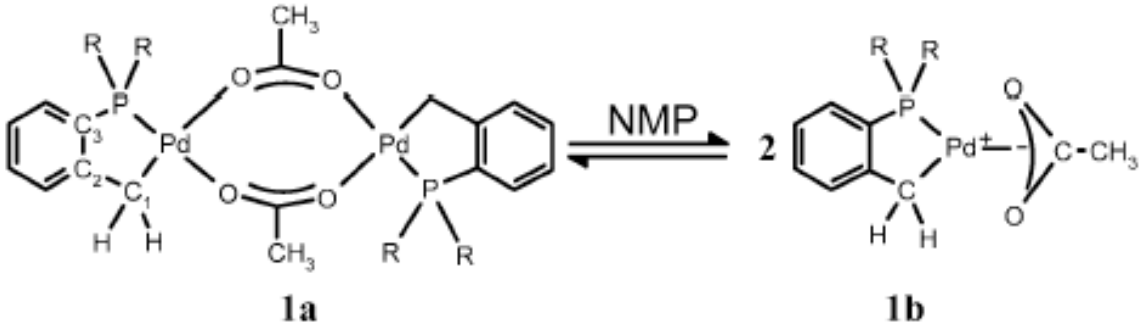


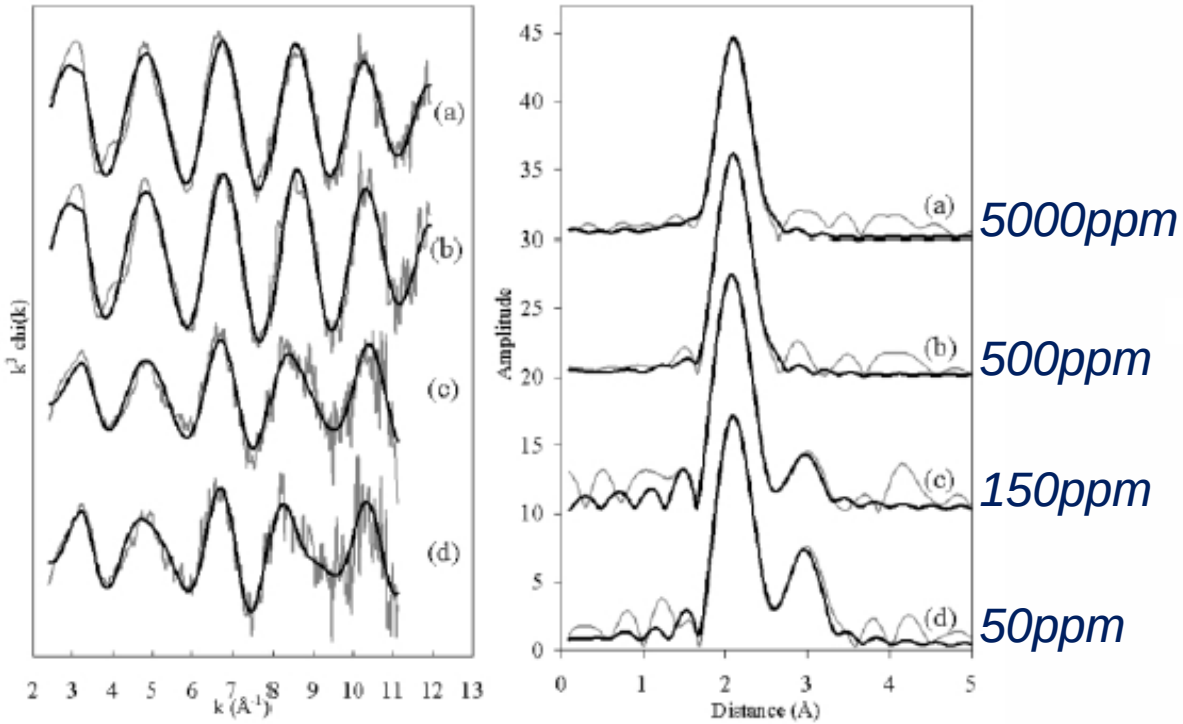




Breeze et al, Inorganic Chemistry, 52 (2013) 8171

What the Heck ?





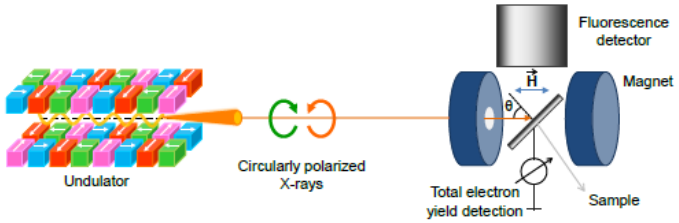
Future limits ? I20 at Diamond will give ppb of chemical species

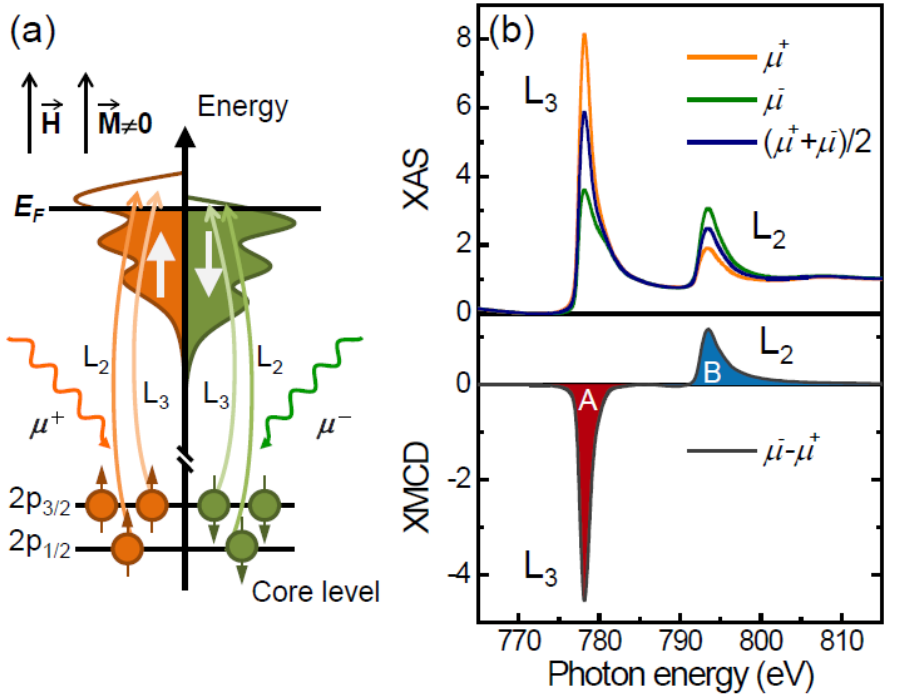
diamond

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

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





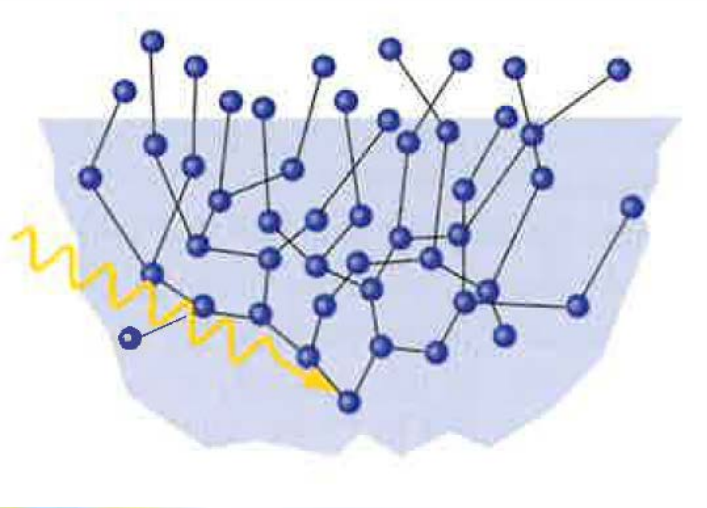
- XAS with LP radiation: $\Delta (\perp - //) \sim \langle M^2 \rangle \rightarrow$ XMLCD

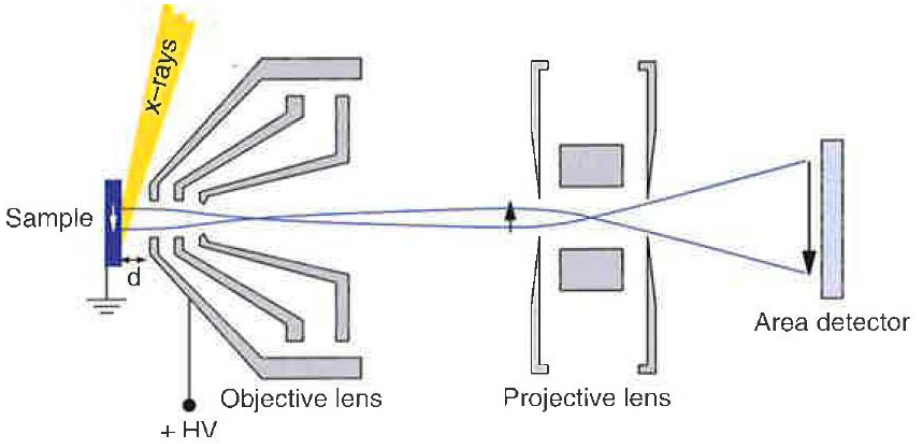
G van der Laan and A.I. Figueroa,
Coord. Chem. Reviews 277-278 (2014) 95

diamond

Mapping nanomagnetism

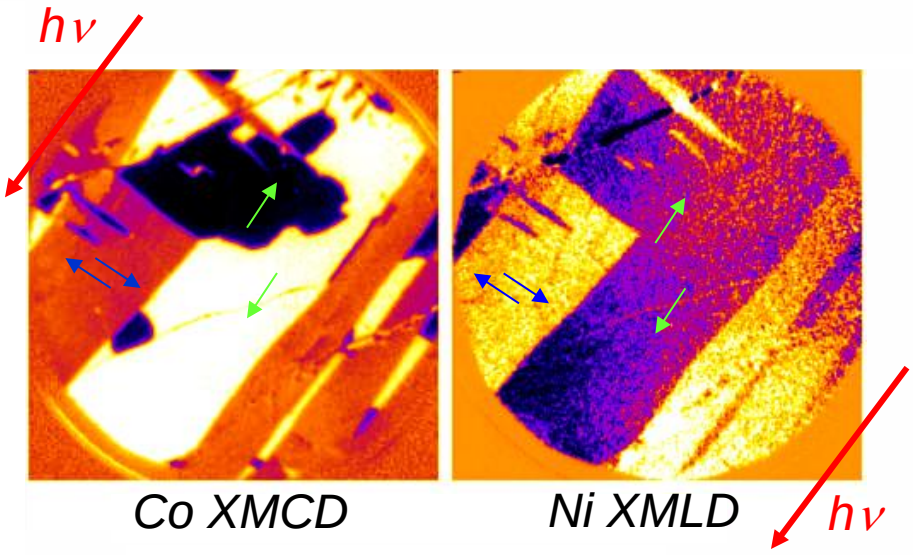
- Resolution to 50 nm with Photoemission Electron Microscopy (PEEM)
- XAS in XANES region produces photoelectrons which create secondary, low-energy (eV) photoelectrons with longer mean-free path. Spatial resolution for imaging these e^- is 10's of nm

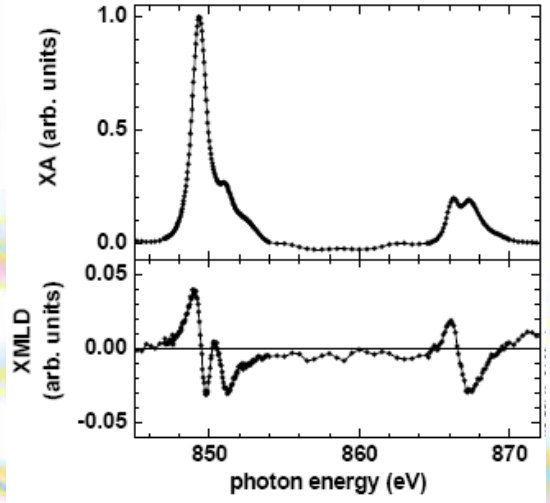




Mapping nanomagnetism

- 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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