Polarised Neutron Scattering for magnetism

Christy Kinane

IoP Winter School 2024

17/12/2024

Years of ISIS Neutron and Muon Source

Science and Technology Facilities Council

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Who am I?

- Christy Kinane

- Instrument scientist working on the POLREF Polarised Reflectometer (NR/PNR/PA) at ISIS.

- Work mainly on magnetic thin films and superconductors.

- If you have any questions, please contact me on: christy.kinane@stfc.ac.uk

Acknowledgements: (People I have stolen stuff from for this talk)

Andrew Caruana, ISIS

Sean Langridge, ISIS (HCM, Very big boss Human)

John Webster, ISIS (SM, Boss human)

Max Skoda, ISIS (SM, and furry human)

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Ross Stewart, ISIS (Very very Polarised neutron human)

Diego Alba-Venero, ISIS (HCM SANS human)

Dirk Honecker, ISIS (HCM SANS human)

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Alex Grutter NCNR, NIST

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Nina Juliane- Steinke, ILL

NIST

NEUTRONS FOR SOCIETY

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Introduction:

A full introduction not possible in 45 mins!

Neutrons are an epic tool for Materials science!

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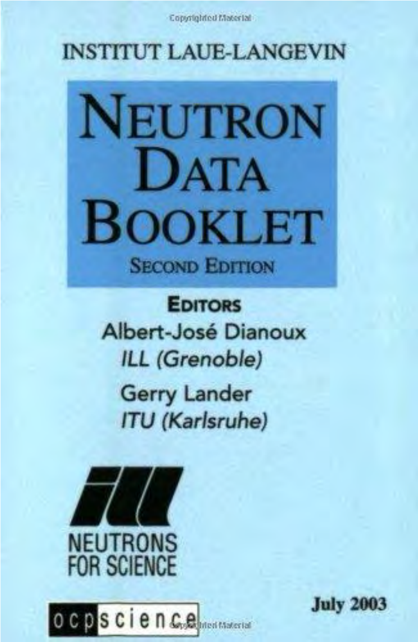
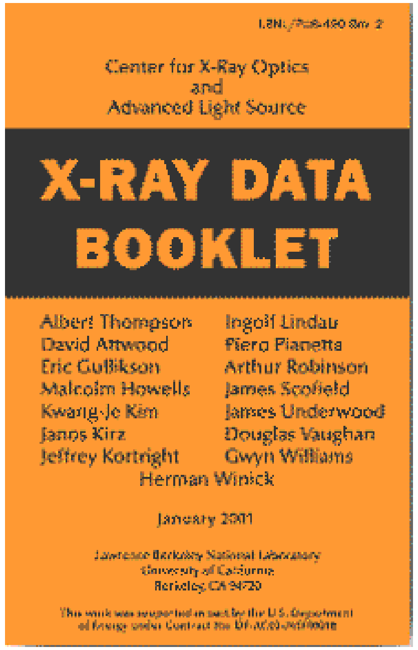
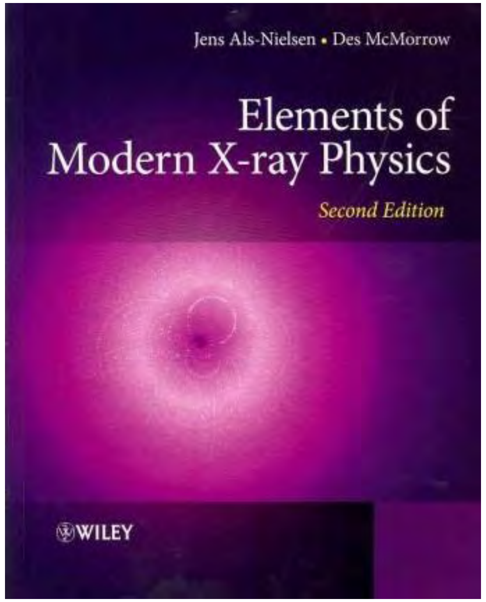
http://www.particlezoo.net/

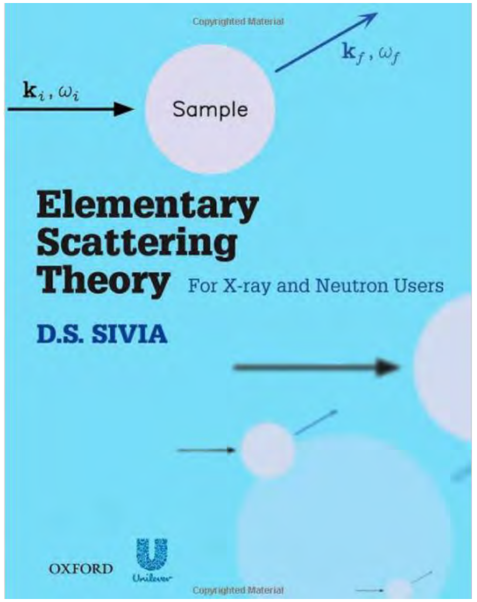
Useful books: These are truly helpful when doing experiments!

Free on web









Probably the most useful books I have ever owned

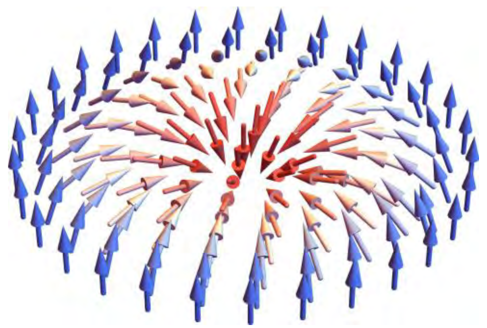
Covers both magnetic and non-magnetic x-ray scattering

Deals with all theory that's is needed for doing experiments.

Motivation: why neutrons?

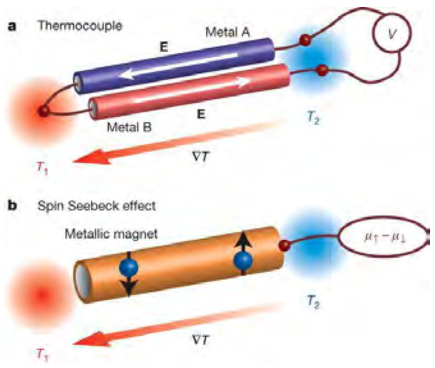
- The design of new magnetic materials and devices require a detailed microscopic understanding of their mechanisms and properties.
- Neutrons have a magnetic moment, and suitable wavelengths and energies to investigate a range of phenomena:

Skyrmions



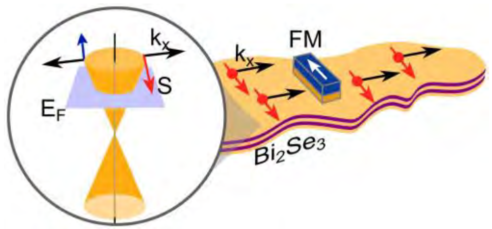
First experimental observation of skyrmion lattice

Spin Seebeck effect



First precise determination of magnon velocities and energies

Novel spintronics

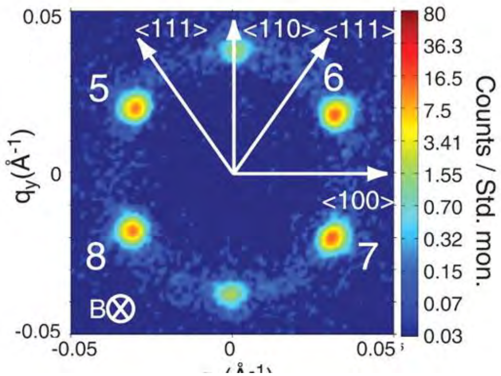


First magnetic structure of topological insulator multilayer

Motivation: why neutrons?

- The design of new magnetic materials and devices require a detailed microscopic understanding of their mechanisms and properties.
- But, neutrons are hard to produce and focus, so real space techniques are not easy, but still possible. Need to work in reciprocal space largely.

Skyrmions



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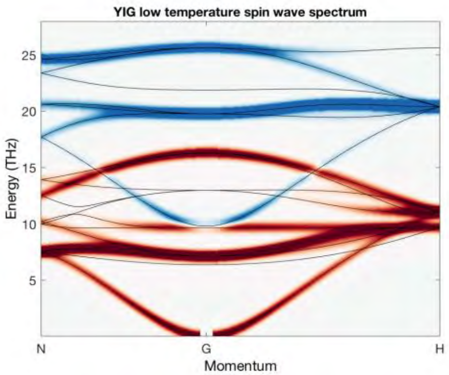

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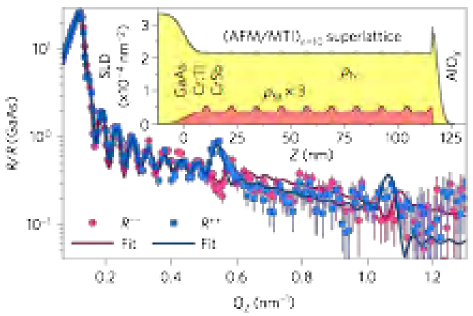

First magnetic structure of topological insulator multilayer

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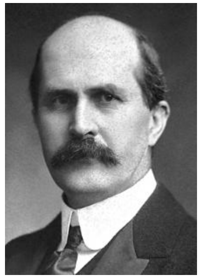
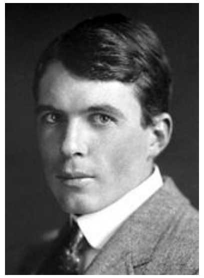
History: 'Scattering tells you where atoms ***are*** and what atoms ***do***'

- **Nobel Prize 1914: Max von Laue**
- **Prize motivation:** *"for his discovery of the diffraction of X-rays by crystals"*

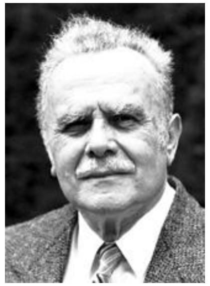




- **Nobel Prize 1915: Sir William Henry Bragg** and **William Lawrence Bragg**
- **Prize motivation:** *"for their services in the analysis of crystal structure by means of X-rays"*

University of Leeds

- **Nobel Prize 1924: Manne Siegbahn**
- **Prize motivation:** *"for his discoveries and research in the field of X-ray spectroscopy"*



- **Nobel Prize 1994: Bertram N. Brockhouse** and **Clifford G. Shull.**
- **Prize motivation:** *"for pioneering contributions to the development of neutron scattering techniques for studies of condensed matter" – in both fields of spectroscopy and diffraction.*

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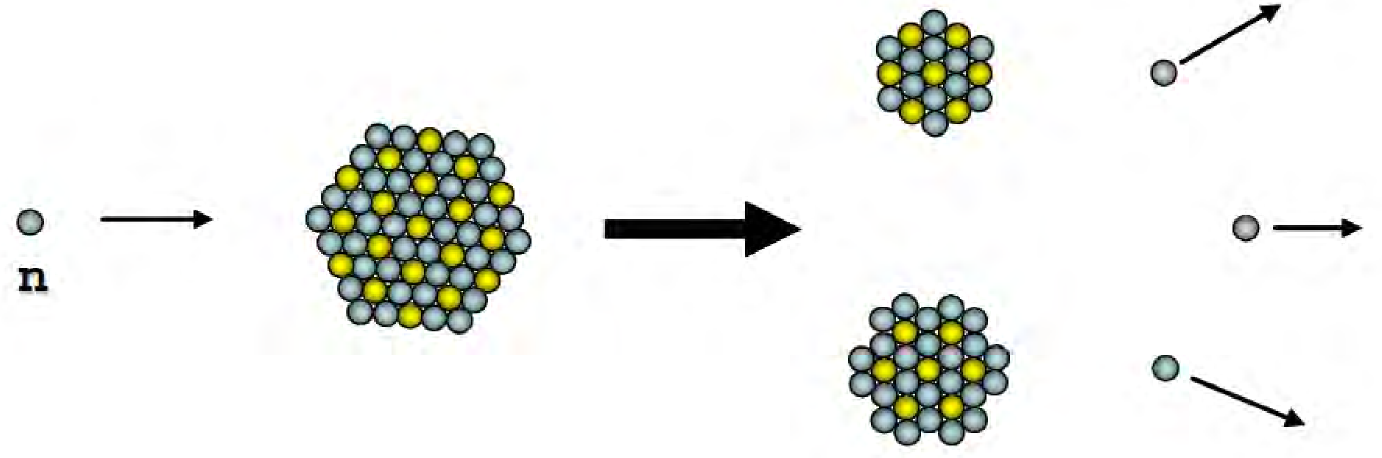
- Also check out the Nobel prizes given to Compton, de Broglie and Chadwick.
- Fortunately, a killer moustache is sadly no longer a requirement for scattering experiments!

Fission neutron production

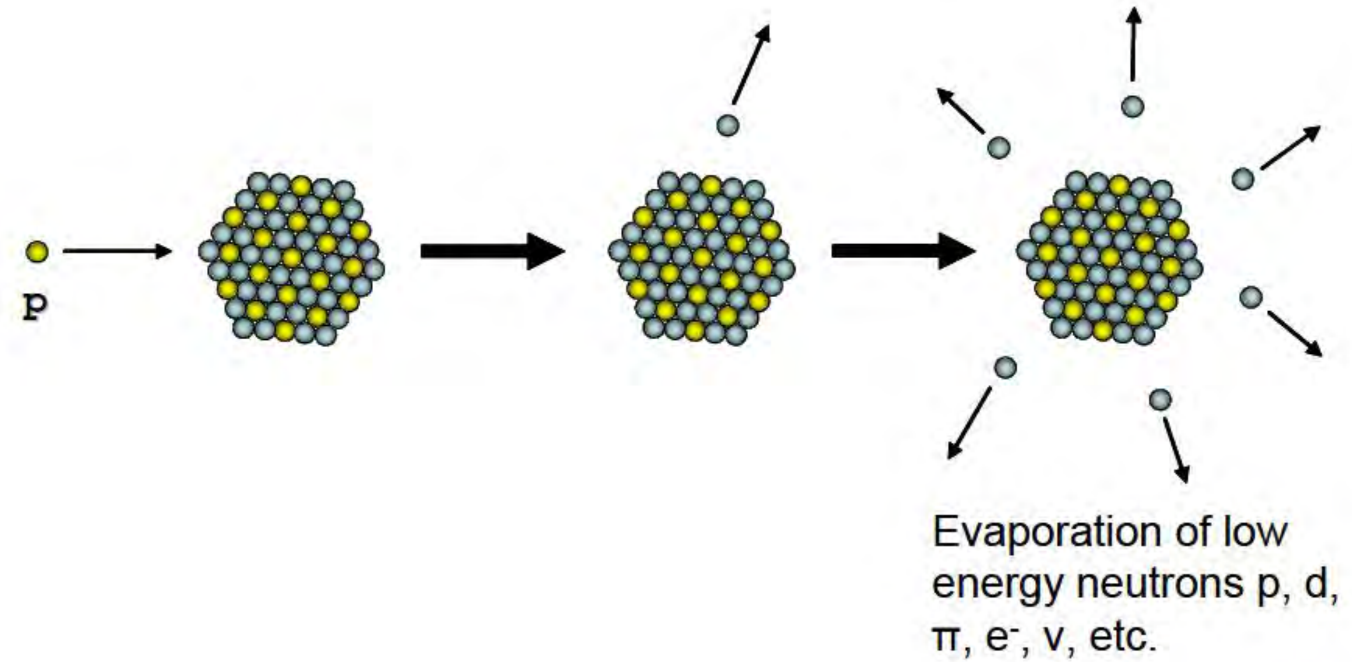
$$n_{Thermal} + U^{235} \longrightarrow \textit{2 fission fragments} + 2.5n_{fast} + 180\ MeV$$

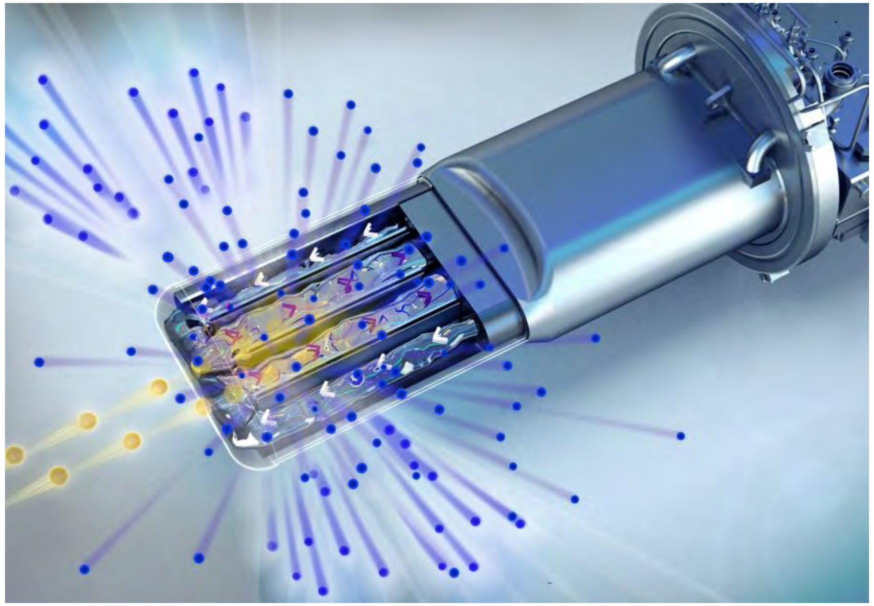


- **Constant supply of neutrons (good for monochromatic instruments)**
- **Needs no external power source.**
- **Requires a reactor, with the issues of waste disposal and fuelling, so politically unpopular)**
- **Have other uses for instance, power generation and making medical isotopes.**

https://www.ill.eu/about-the-ill/ill-at-a-glance

Spallation neutron production

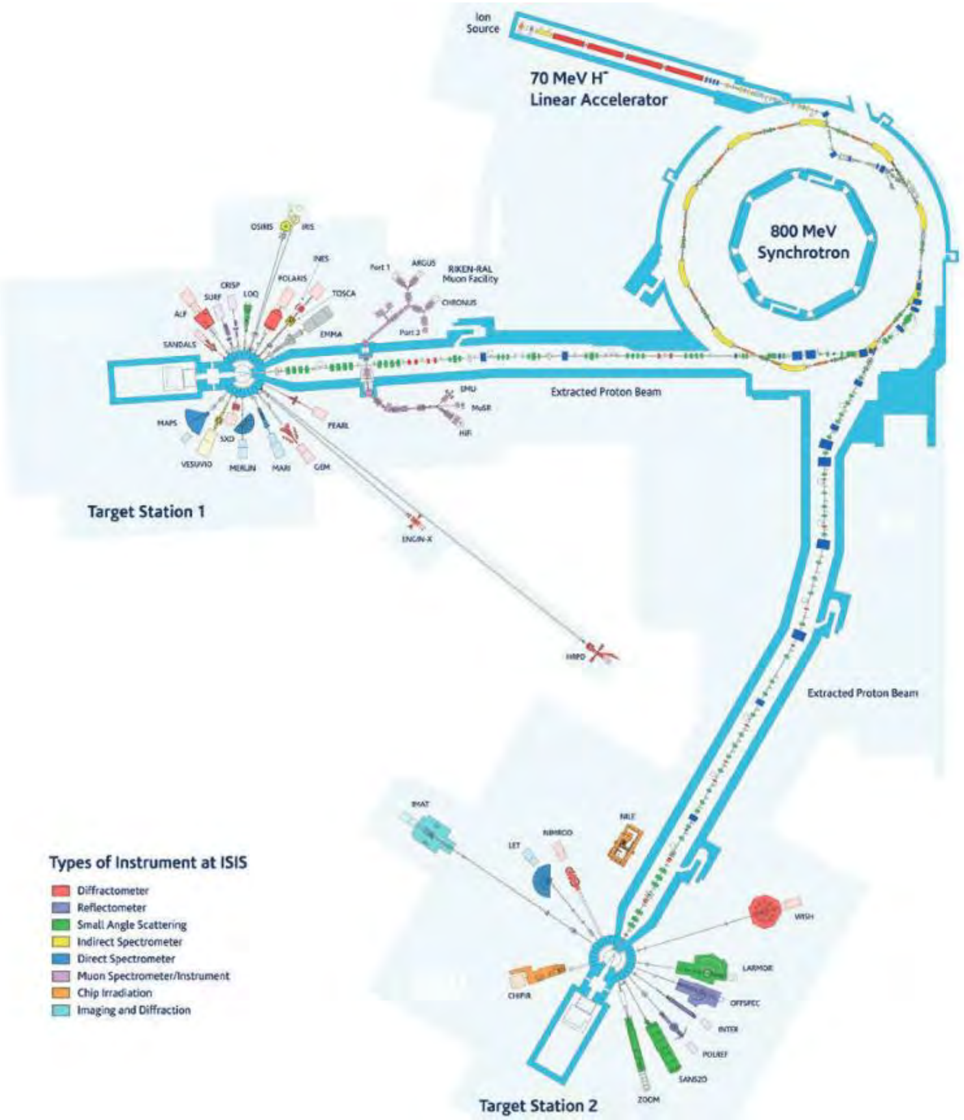




- **Pulsed sources only , giving white beams, so great for time of flight (TOF) instruments.**
- **Needs Power! You need a lot of electricity…….**
- **Very small amount of waste in that the target needs changing once every 5+ years.**
- **No power = No neutrons, hence no risk of criticality!**
- **Neutron flux limited by target cooling power on target.**

https://www.isis.stfc.ac.uk/Pages/What-does-ISIS-Neutron-Muon-Source-do.aspx

ISIS From Start to Finish



- H−ion source (17 kV)
- •665 kV H−RFQ
- •70 MeV H−linac
- •800 MeV proton synchrotron
- Extracted proton beam lines
- Targets
- Moderators
- Beamlines

The accelerator produces a pulsed beam of 800 MeV (84% speed of light) protons at 50 Hz, average beam current is 230 μA (2.9×1013ppp) therefore
184 kW on target (148 kW to TS-1 at 40 pps, 36 kW to TS-2 at 10 pps)

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https://www.isis.stfc.ac.uk/Pages/What-does-ISIS-Neutron-Muon-Source-do.aspx

Places to do neutron Science!

ISIS* , Oxfordshire

SNS, Oak Ridge TN

J-PARC, Japan

ILL, Grenoble,
reactor source

NCNR @ NIST ,Maryland
“Old nuclear sub reactor”

ESS, Lund, Sweden,
Under construction

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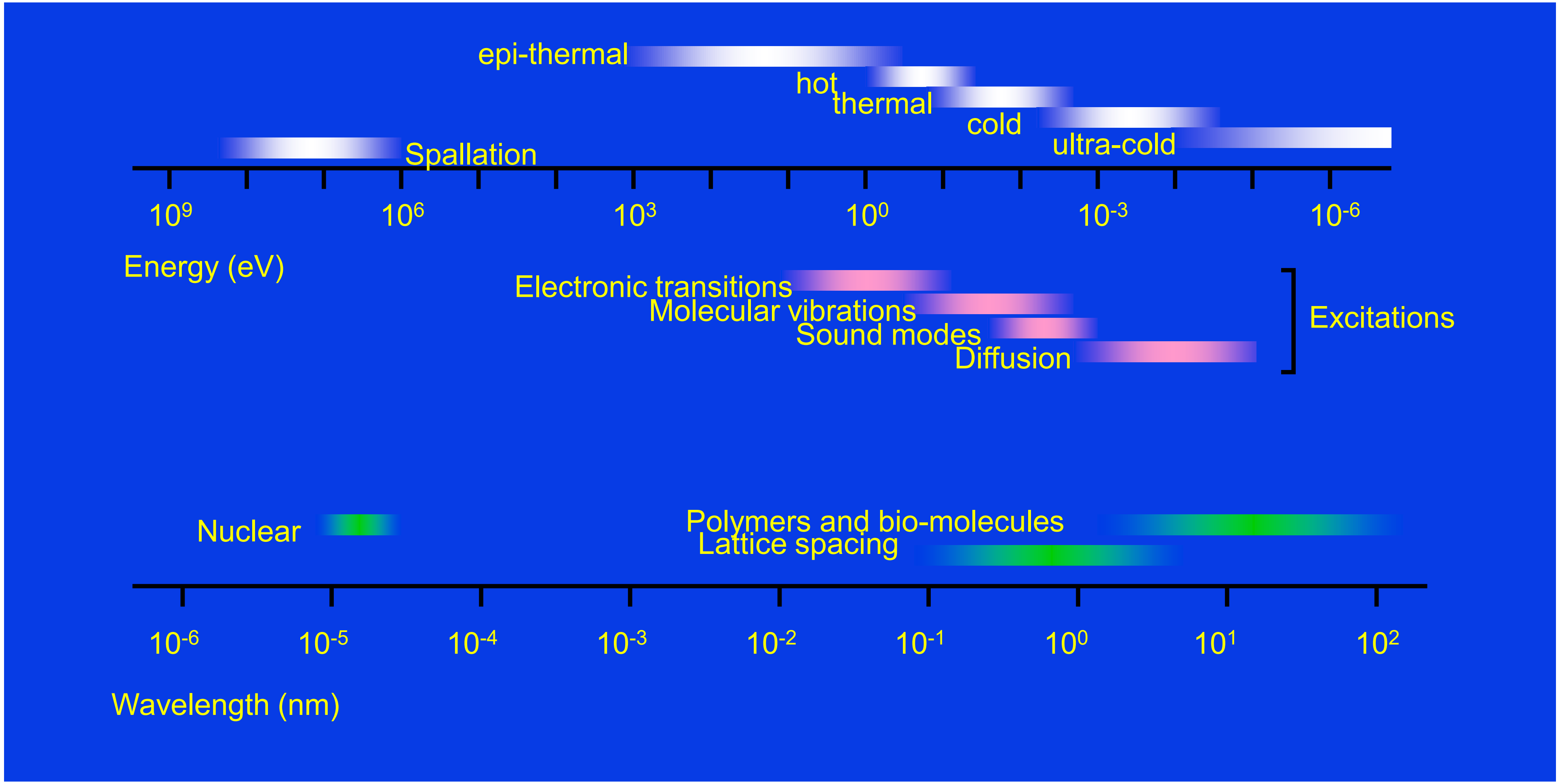
* I must stress we had the name ISIS first by 40 years, and it’s a reference to the Thames as it flows through Oxfordshire which for some reason is referred to as the ISIS (don’t ask me its Oxford).

Moderators: (Think Pinball with a Maxwell Boltzmann Distribution.)



- **But need meV (Cold neutrons) for condensed matter work, not MeV (epi-thermal)**
- **Moderation via elastic nuclear scattering (Basically a pin ball machine for neutrons)**
- **Liquid hydrogen (20K), Methane (100K), Water (300K) to get useful wavelengths.**

Basic concepts: The Neutron

Mass

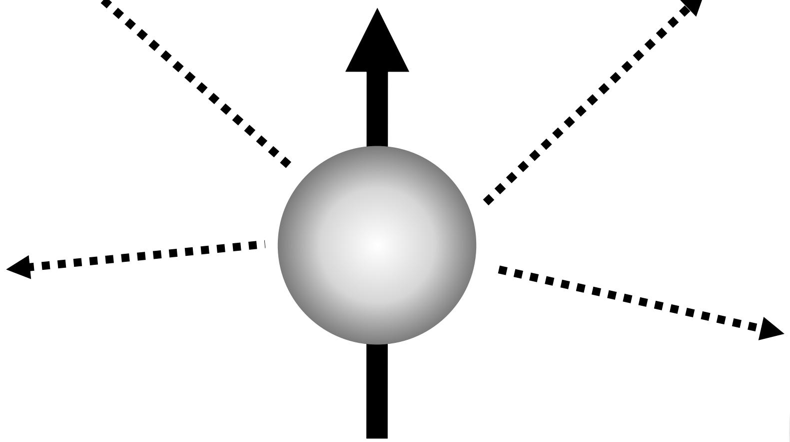
$$m = 1.67 \times 10^{-27}\text{kg}$$

Electrically neutral
(to within experimental error)

$$I = \frac{1}{2}$$

Magnetic moment

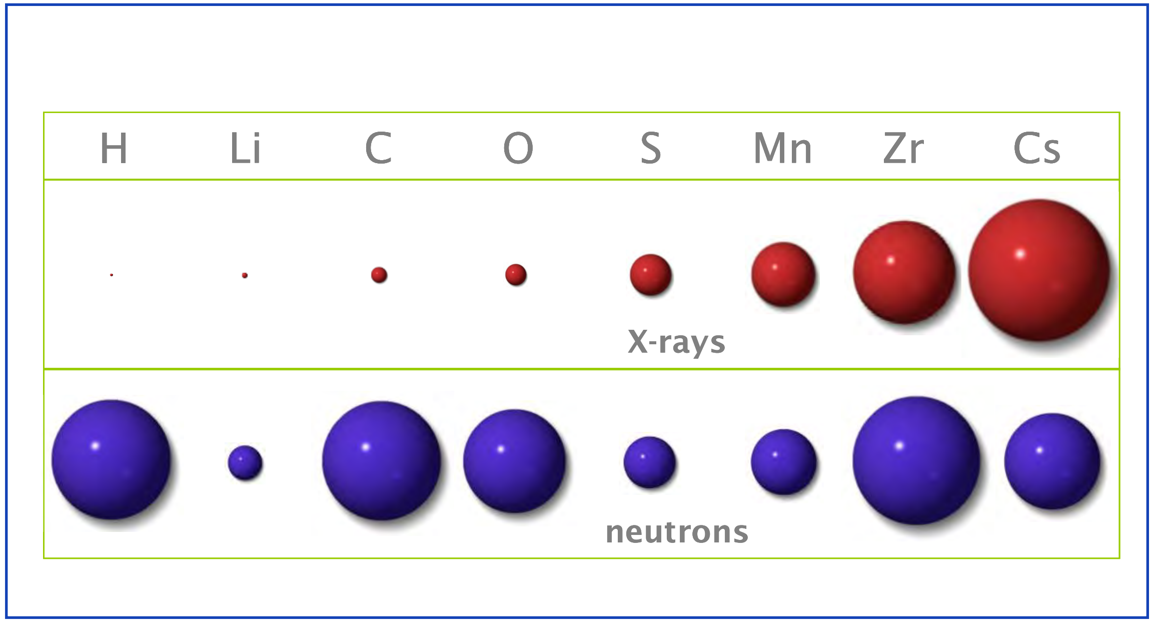
$$\mu = -\gamma \mu_N \sigma$$



- Electrically neutral
- Has an intrinsic magnetic moment
- Weakly interacting

Neutrons: Useful properties

- Neutron wavelength and energy 'just right' for condensed matter
  - *– structure and dynamics*
- Neutron cross-section
  - *–isotopic dependence*
- H/D contrast
  - *–nuclear form factor*
- Magnetic Moment
  - *–magnetic order & excitations*
- Weak probe
  - *–theoretical interpretation*
- Highly penetrating
  - *–bulk probe, complex SE*
- Has no charge!



- Sadly neutron facility sources are not very bright.

ISIS TS1 1/10th of a foot candle in brightness
ISIS TS2 1 foot candle! (the sun is 3.8×10^{27}candles approx.)
Diamond light source x 10^6 the sun!

https://en.wikipedia.org/wiki/Candlepower

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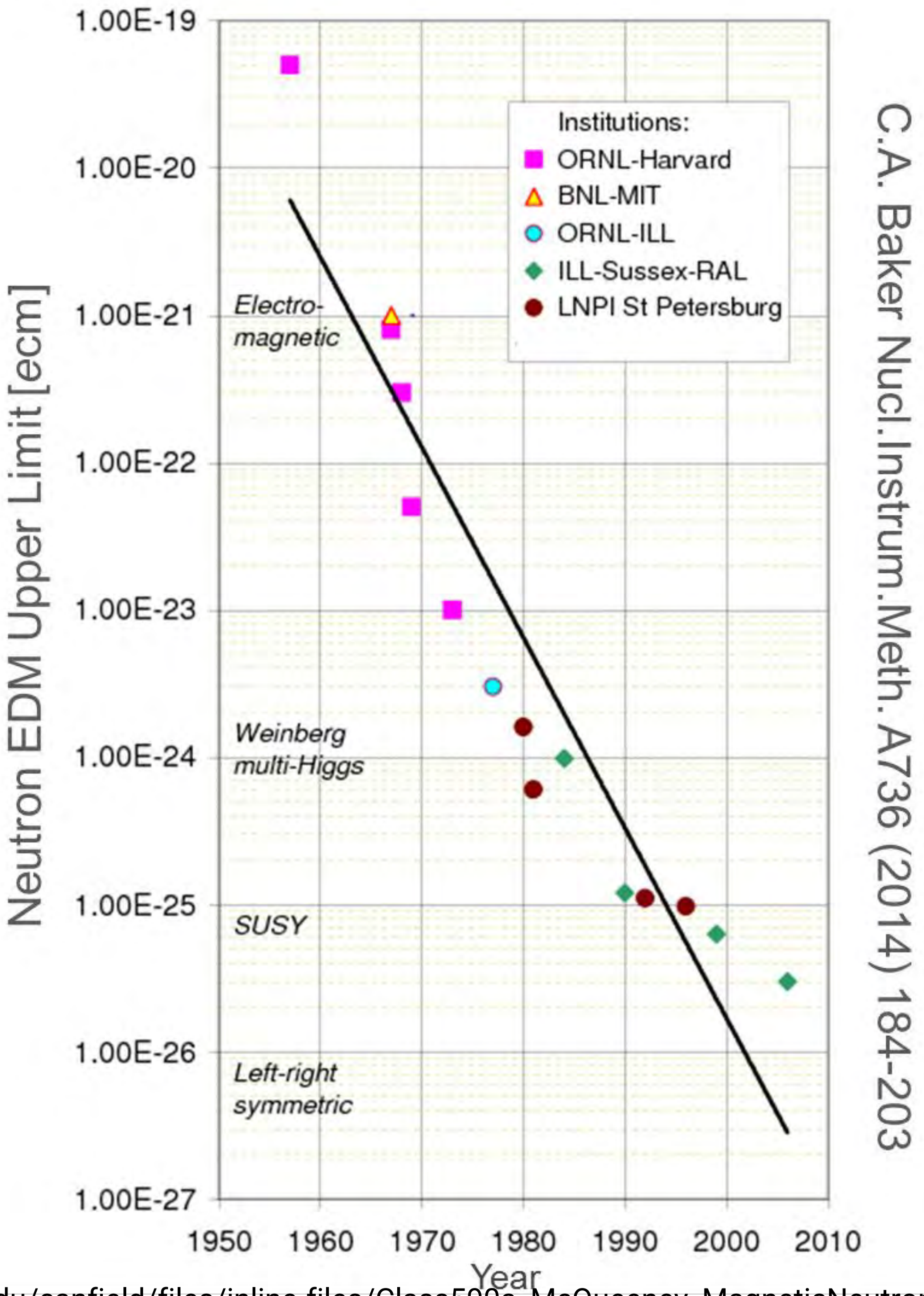
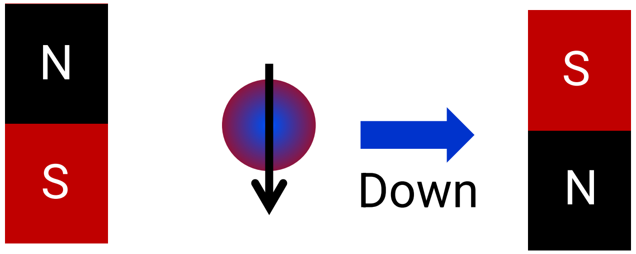
Neutrons: Magne

- Neutrons have a magnetic c
- Each neutron is effectively a
- This means that neutrons sc diffraction.
- Neutrons also allow vectoria some manner. i.e. make all spins align in the same direction
- Spin "up" and spin "down" e have different refractive indices.
- This is the basis of Polarisec

V = (I





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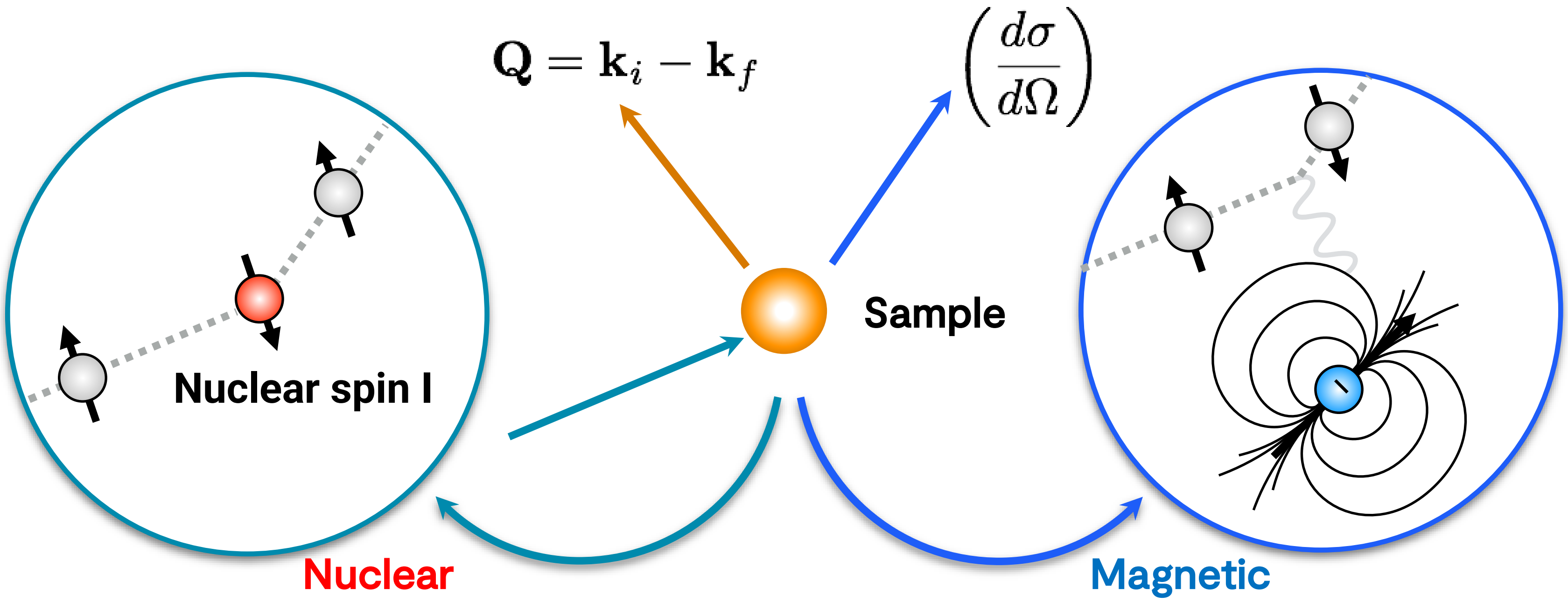
https://faculty.sites.iastate.edu/canfield/files/inline-files/Class590a_McQueeney_MagneticNeutronDiffraction_180919.pdf

Neutron-electron interaction

The neutron has magnetic moment which can interact with sample moments:



Interaction is between dipolar fields of neutron and unpaired electron(s) → directional dependence.

Neutrons scatter magnetically for the B field of the magnetic material!

Strength of magnetic interaction similar to that neutron and nucleus (unlike X-rays).

A neutron is a spin ½ particle with a magnetic dipole moment, effectively it's a little bar magnet. (like a fridge magnet only smaller)

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Thanks to G Nilsen for this slide

Basic concepts:

Cross section "σ" :

- Defined as a measure of the probability of an interaction of some kind happening.

Can you hit this barn door?

- Measured in the non-SI unit of area the *barn* (10^{-28} m^2) (Atomic barn doors).

$$\sigma_{\text{total}} = \sigma_{\text{scat.}} + \sigma_{\text{abs}}$$

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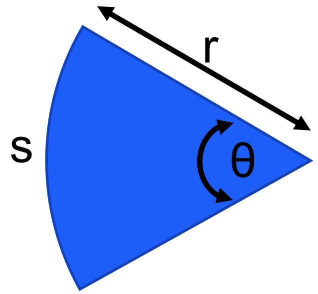
https://en.wikipedia.org/wiki/Barn_(unit)

Basic concepts:

Solid Angle “Ω”:

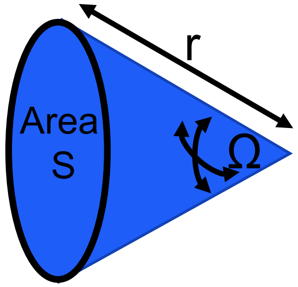
- Defined as the two-dimensional angle in three-dimensional space that an object subtends at a point.:
    - ***For a simple angle => the “transverse distance” at a distance.***
    - ***For a solid angle => the “transverse area” at a distance.***
- It is a measure of how large an object appears to an observer looking from that point.

$$\theta = \frac{s}{r}$$



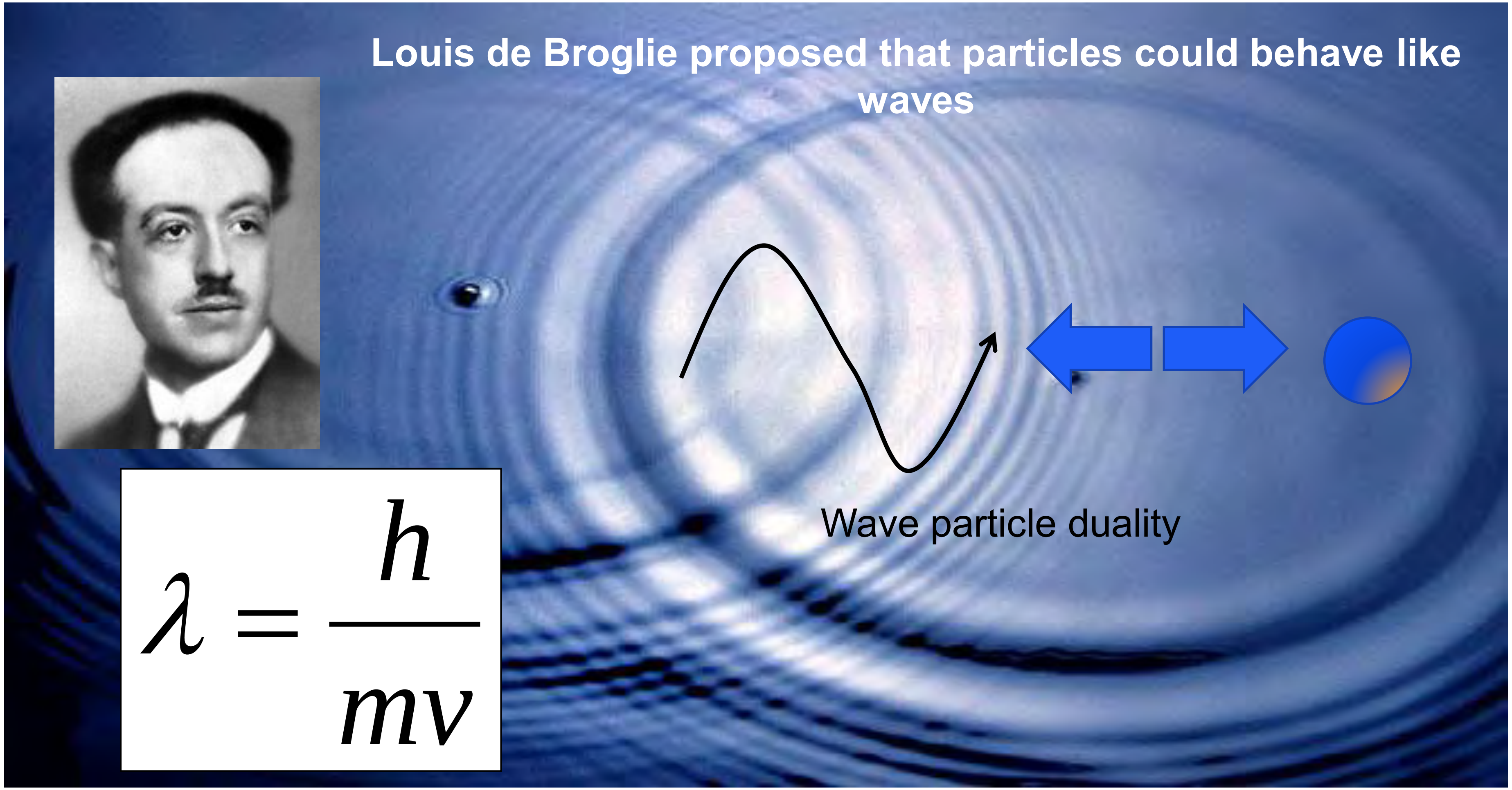
Arc of a circle (in radians)
Circle = 2π radians

$$\Omega = \frac{S}{r^2}$$



Cap of sphere
Sphere = 4π steradian

Basic concepts: Wave-particle duality



$$\lambda = \frac{h}{mv}$$

- We can treat both neutrons and X-rays as waves.

Again with the moustaches!

Basic Concepts: What do we measure

Thanks to Andy Boothroyd for this slide, which I got when he gave a similar lecture to me at this course a long long time ago now.

(This is the hardest slide of the lot! Gets easier after this)

- **Total cross-section**

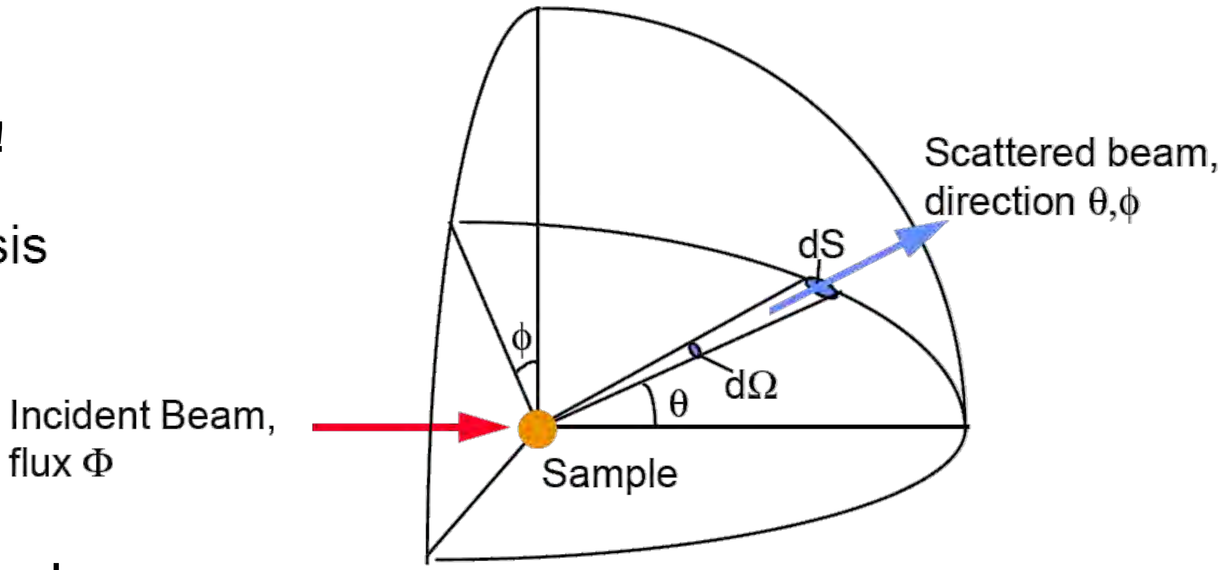
  Imaging!!

$$\sigma = \int_{4\pi} \frac{d\sigma}{d\Omega} d\Omega \qquad \sigma_{total} = \sigma_{scat.} + \sigma_{abs}$$

- **Differential cross-section** **DIFFRCTOMETERS!!**

$$\frac{d\sigma}{d\Omega} = \int_0^\infty \frac{d^2\sigma}{d\Omega dE'} dE'$$

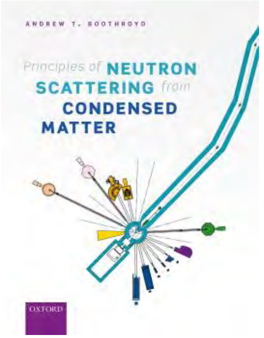
: no energy analysis



- **Double differential cross-section** **SPECTROMETERS!**

$$\frac{d^2\sigma}{d\Omega dE'} = \text{(number of neutrons scattered into } d\Omega \text{ per second with final energy between E' and E'+dE)}/\Phi d\Omega dE'$$

Principles of Neutron Scattering from Condensed Matter: Andrew T Boothroyd



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Scattering length

- Scattering power of an atom in the sample expressed as a scattering length b

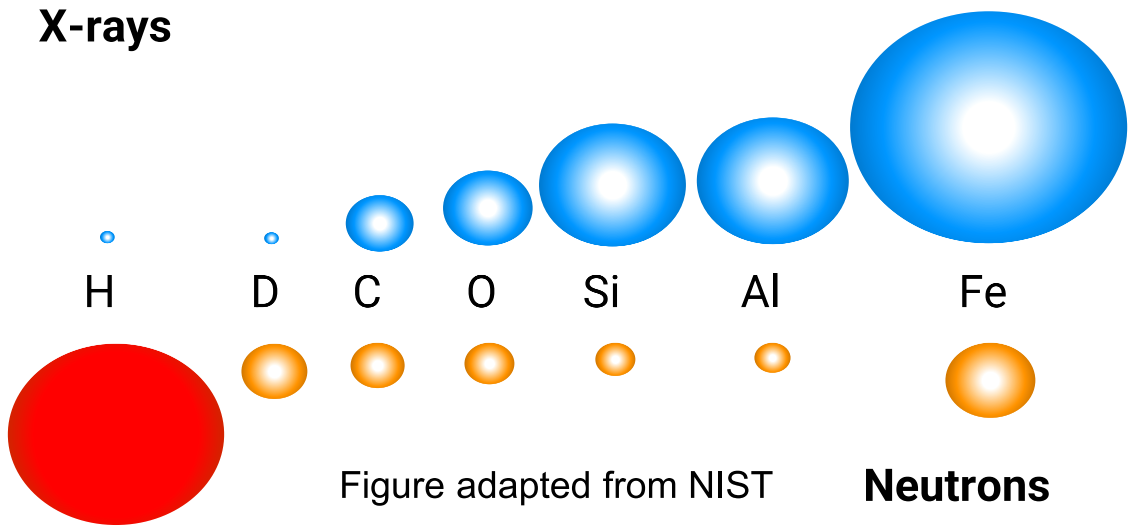
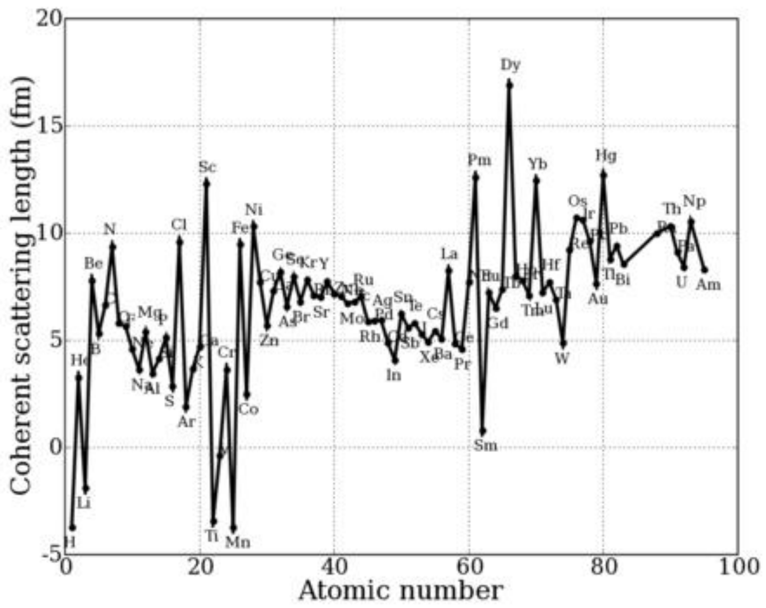


Figure adapted from NIST



- For neutrons, ***b*** varies randomly through the periodic table - light atoms more visible, larger contrast between adjacent elements.
  - For instance, Lead (Pb) will stop x-rays, but is an open window to neutrons.
- Isotopes of the same element scatter differently unlike x-rays.
- X-rays scattering factor ***f*** scales as Z^4

Neutron imaging and radiography

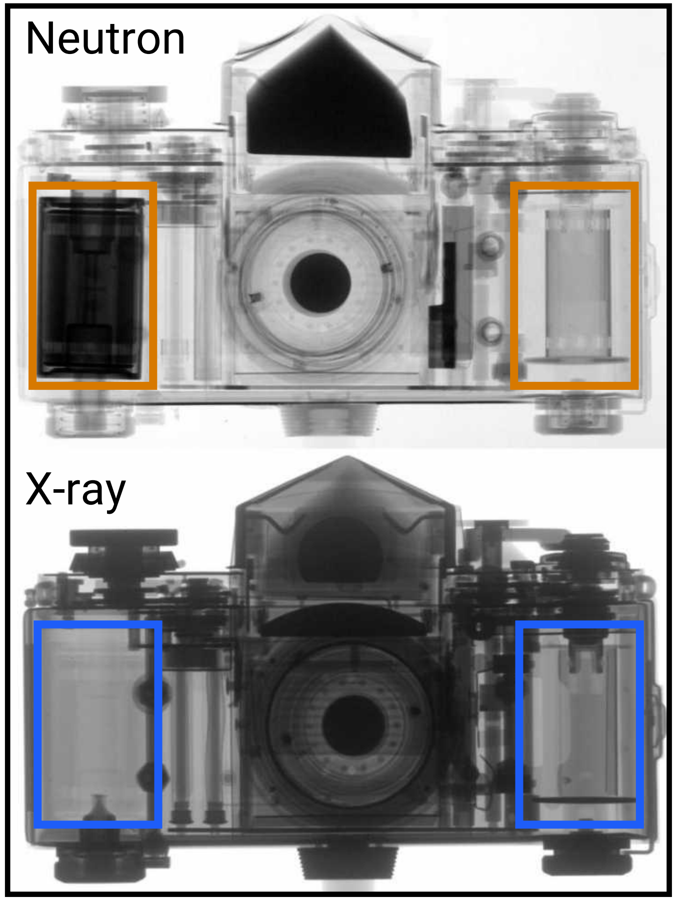
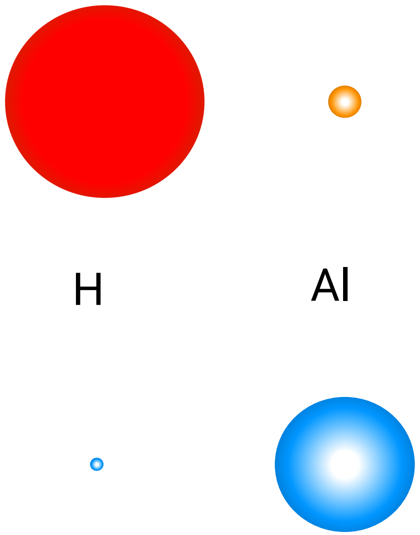


Image: Paul Scherrer Institute

Scattering power varies randomly over periodic table:



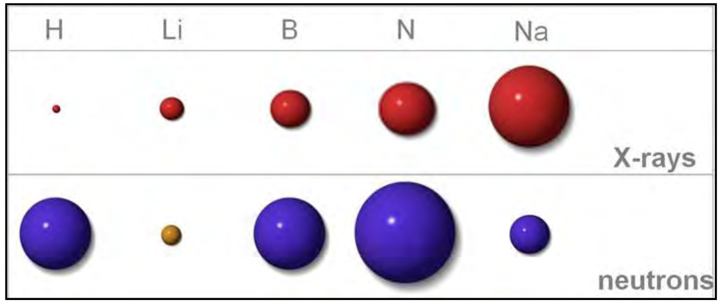
Strong contrast for H-containing components (e.g. plastics)

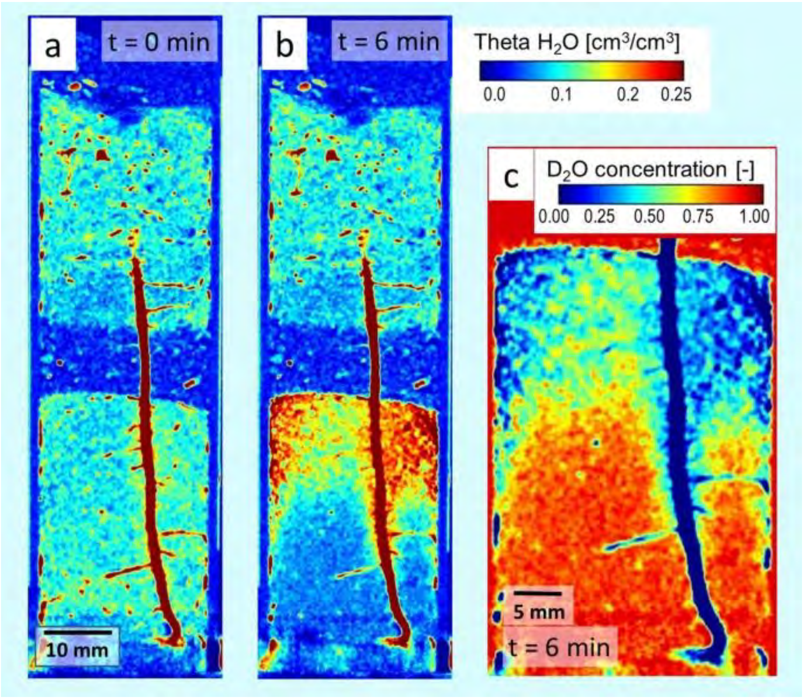
Magnetic imaging by polarised neutrons is a growth area.

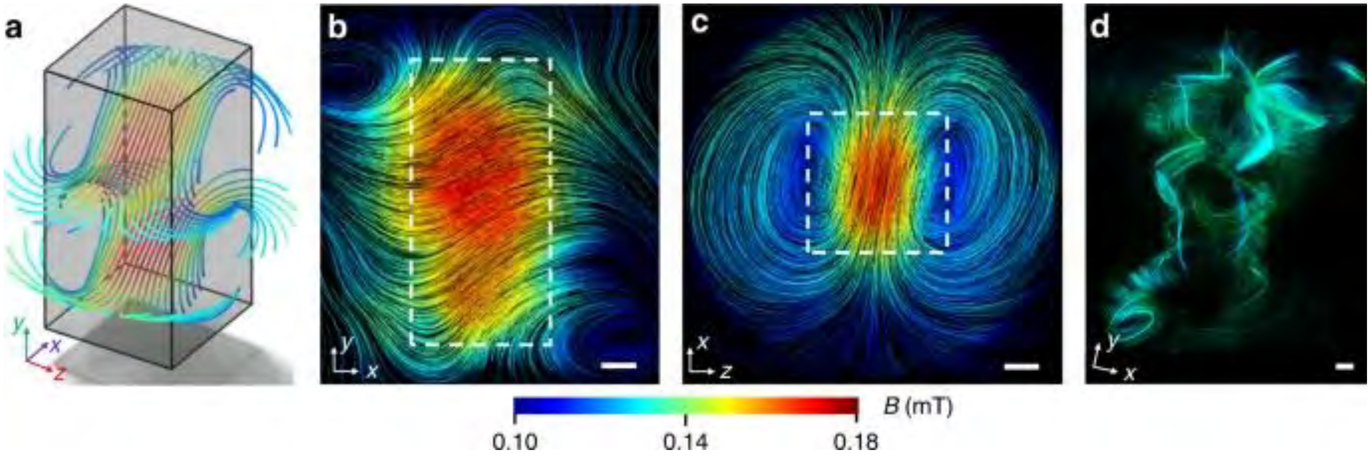
Basic concepts:

• Absorption/Emission: Imaging

- Measurements made using direct beam.
- Material absorbs incoming particle probes via some mechanism or scatters particles out of the Transmitted beam that appear as Bragg dips.







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TOPICAL REVIEW: Polarization measurements in neutron imaging: M Strobl *et al* 2019 J. Phys. D: Appl. Phys. 52 123001.

Hilger, *et al*. Tensorial neutron tomography of three-dimensional magnetic vector fields in bulk materials. *Nat Commun* **9**, 4023 (2018)

C. Tötzke, *et al*. Three-dimensional in vivo analysis of water uptake and translocation in maize roots by fast neutron tomography. *Sci Rep* **11**, 10578 (2021). https://doi.org/10.1038/s41598-021-90062-4

Imaging: Effectively which atoms are present or the strength and shape of any magnetic fields.

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Take home point!

Basic Concepts: What do we measure

(This is the hardest slide of the lot! Gets easier after this)

- **Total cross-section**

  Imaging!!

$$\sigma = \int_{4\pi} \frac{d\sigma}{d\Omega} d\Omega$$

$$\sigma_{total} = \sigma_{scat.} + \sigma_{abs}$$

- **Differential cross-section** **DIFFRACTOMETERS!!**

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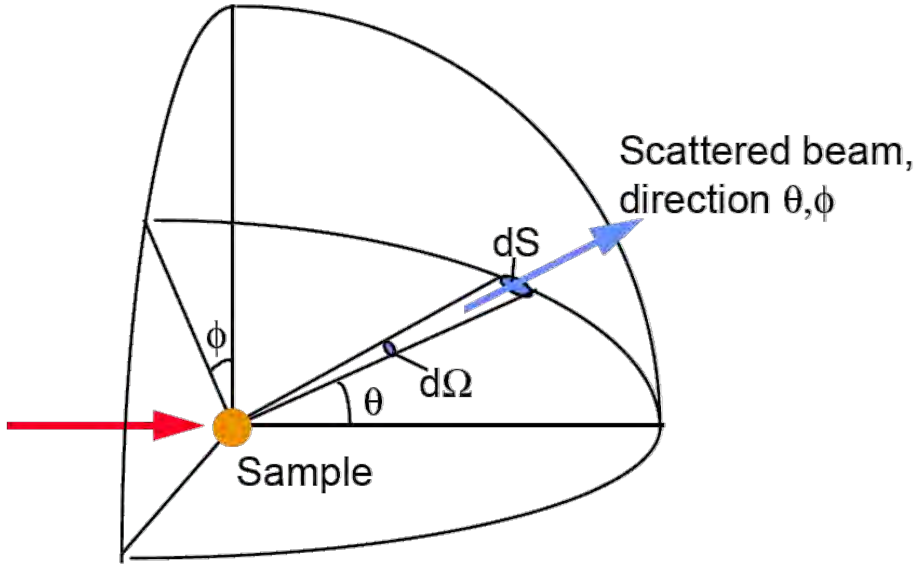

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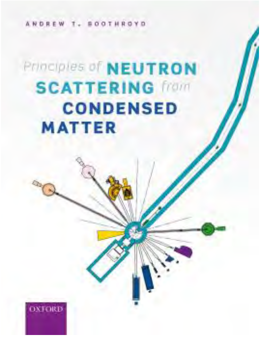
$\frac{d^2\sigma}{d\Omega dE'}$ = (number of neutrons scattered into dΩ per second with final energy between E' and E'+dE)/ΦdΩdE'

Principles of Neutron Scattering from Condensed Matter: Andrew T Boothroyd



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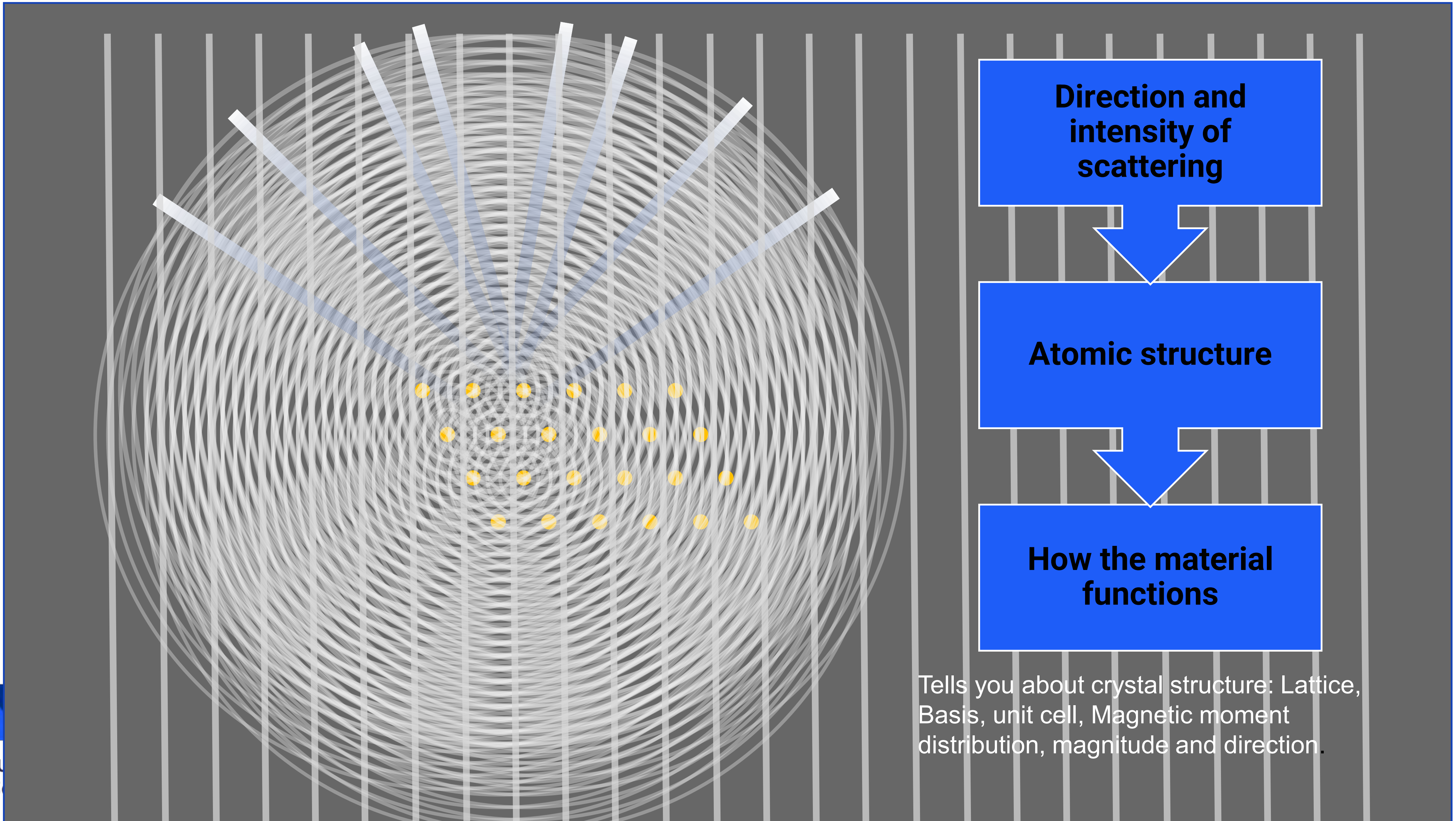
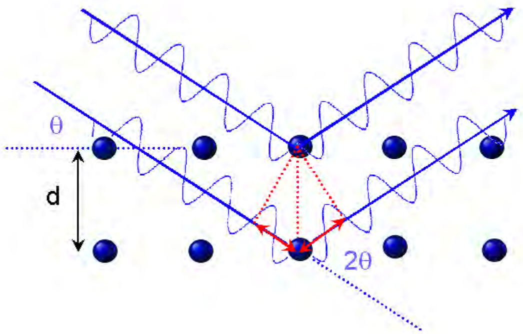
Diffraction: Where atoms are …

No energy change only direction upon scattering: Scattering is **ELASTIC!**



Basic concepts: Elastic scattering

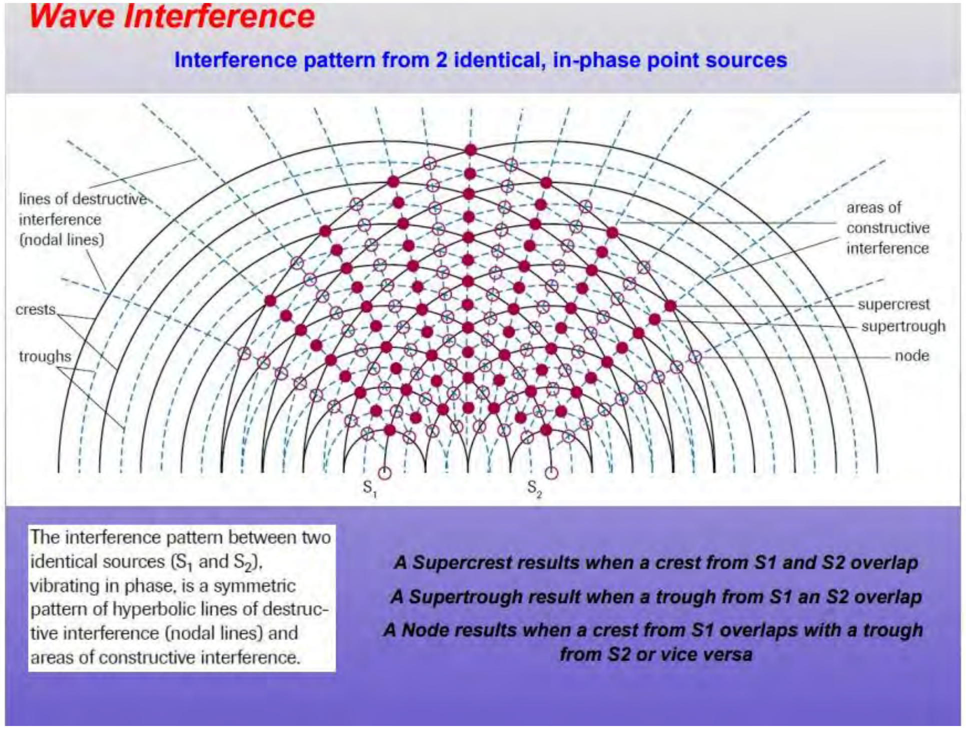
- Majority of the techniques you're possibly already familiar with fall under this heading:
  - Small angle scattering (SANS) - sees big things μm to nm scale
  - Reflectivity (NR)- sees intermediate stuff on the 100s nm to 1nm scale
  - Diffraction - see tiny knee-high to a grasshopper stuff sub 10 nm scale



- **All scattering is elastic – so momentum changes but the energy stays constant!**

- Tells you:
  - **What things are**
  - **Where they are.**
  - **How big they are**

- Information mainly comes from destructive and constructive interference once the basic scattering interactions are accounted for



Bragg's Law: $\lambda = 2d \sin(\theta)$

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Non Polarised Magnetic Neutron diffraction

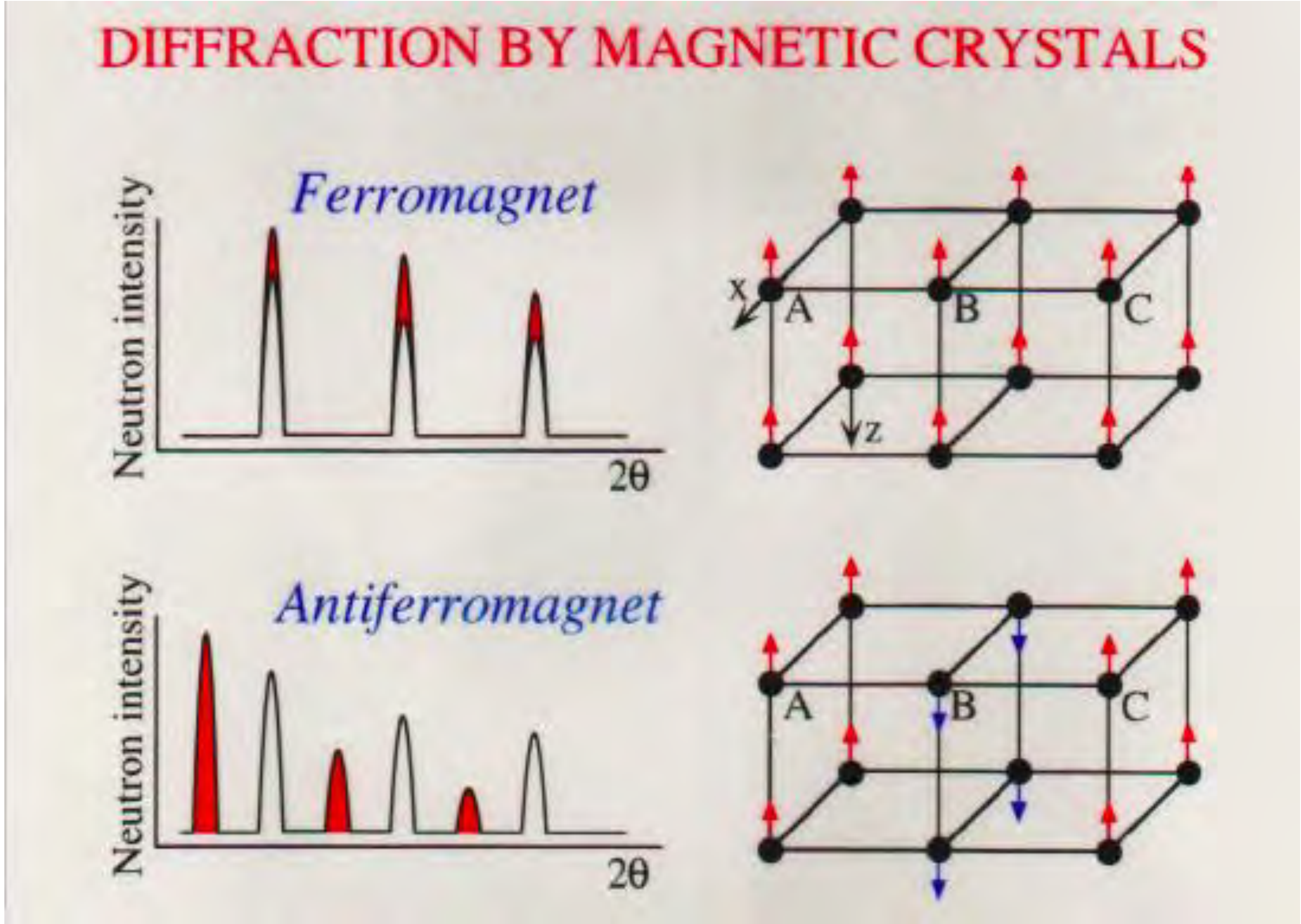

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Figure courtesy of C Majkrzak, NIST

First Confirmation of Antiferromagnetic structure:

- In 1949, Clifford Shull Observed additional magnetic reflections in MnO using Neutron Diffraction, which led to the confirmation of antiferromagnetism.
- Up until this point it was still an unproven theoretical idea!

TABLE I. Comparison between the observed antiferromagnetic reflection intensities for MnO and those calculated for the magnetic structure model of Fig. 5.

Magnetic reflection	Observed integrated intensity	Calculated intensity (neutrons/min)
(111)	1072	1038
(311)	308	460
(331)	132	129
(511) (333)	70	54

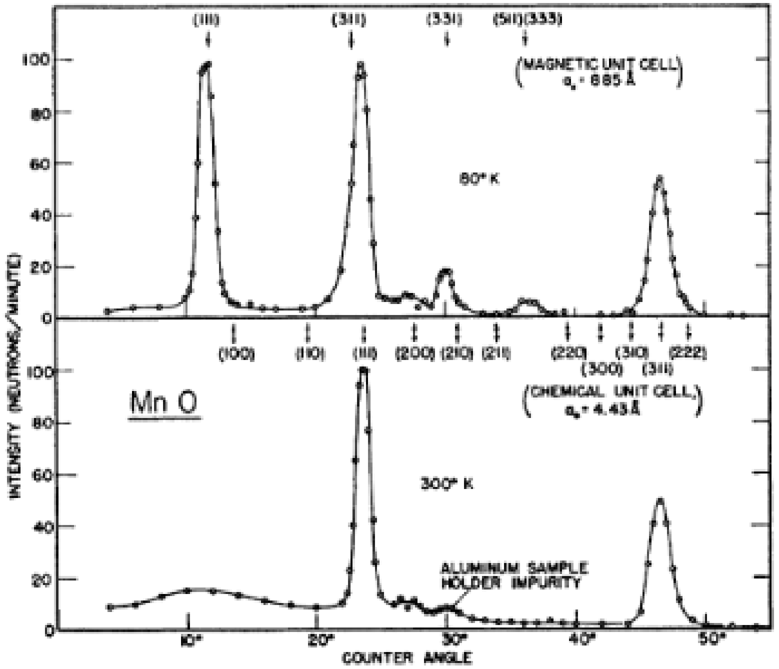

FIG. 1. Neutron diffraction patterns for MnO at room temperature and at 80°K.

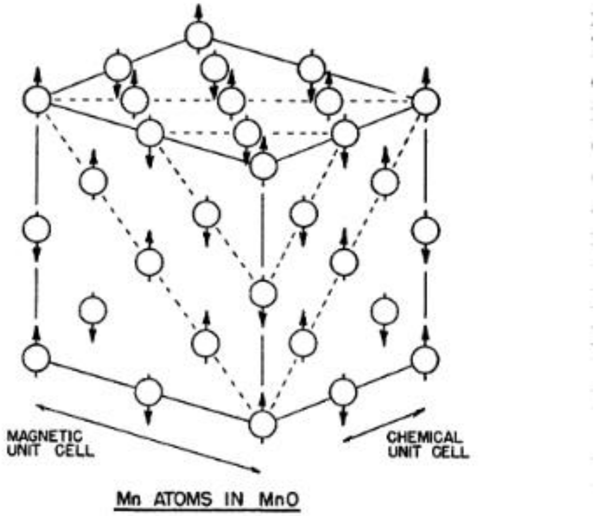

FIG. 5. Antiferromagnetic structure existing in MnO below its Curie temperature of 120°K. The magnetic unit cell has twice the linear dimensions of the chemical unit cell. Only Mn ions are shown in the diagram.

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Shull got the Noble Prize for this work

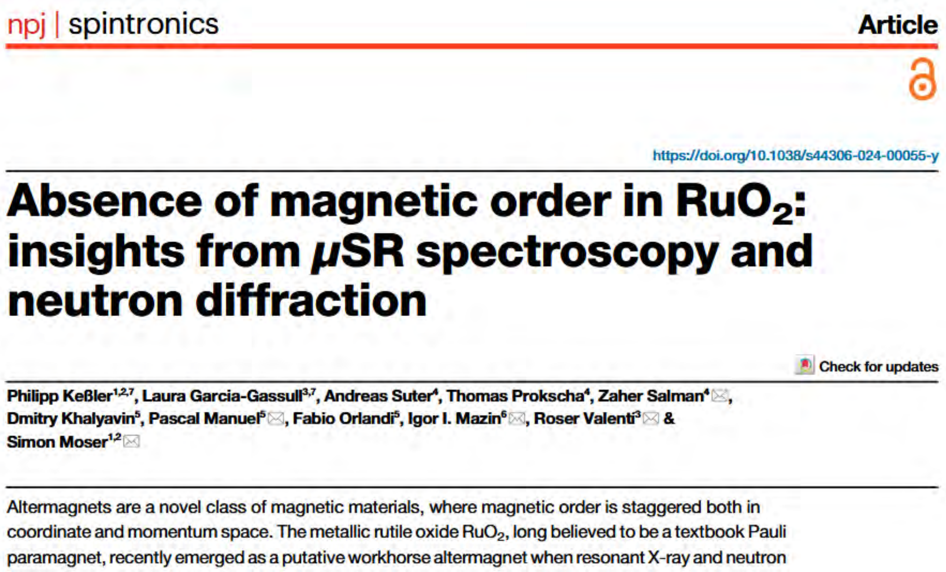
- Shull and J. S. Smart, Phys Rev 76, 1256 (1949).
- C. G. Shull et al., Phys. Rev. 83, 333 (1951).

More Topical Magnetic Diffraction example: Is RuO_2 an Altermagnet??

- Example of the power of combining facilities techniques.

  Muons, Neutron Diffraction and DFT calculations

- Is Altermagnetism real or just another theoretical idea????



npj | spintronics — Article

https://doi.org/10.1038/s44306-024-00055-y

Absence of magnetic order in RuO_2: insights from μSR spectroscopy and neutron diffraction

Check for updates

Philipp Keßler[1,2,7], Laura Garcia-Gassull[3,7], Andreas Suter[4], Thomas Prokscha[4], Zaher Salman[4], Dmitry Khalyavin[5], Pascal Manuel[5], Fabio Orlandi[5], Igor I. Mazin[6], Roser Valentí[3] & Simon Moser[1,2]

Altermagnets are a novel class of magnetic materials, where magnetic order is staggered both in coordinate and momentum space. The metallic rutile oxide RuO_2, long believed to be a textbook Pauli paramagnet, recently emerged as a putative workhorse altermagnet when resonant X-ray and neutron

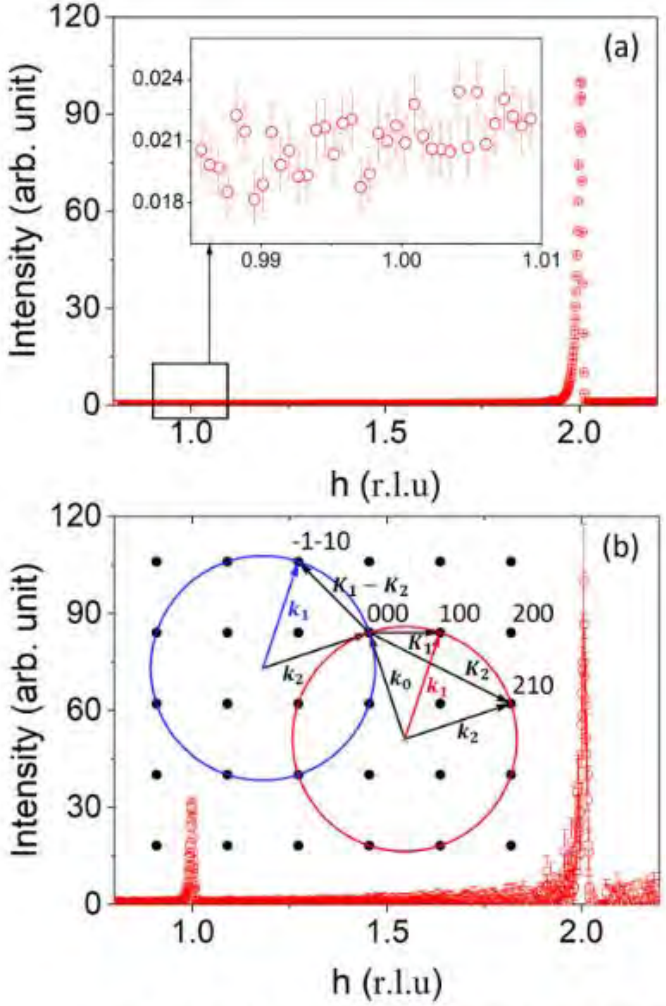


Fig. 1 | Neutron diffraction of a RuO_2 single crystal. The data was collected at T = 1.5 K with the $(h, 0, 0)$ reflections at the scattering angle $2\Theta = 71°$ (a) and $2\Theta = 32°$ (b). The counting time was 10 hours and 15 minutes, respectively. The data demonstrate

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Sadly nope it's not! At least for these samples!

Keßler, P., Garcia-Gassull, L., Suter, A. *et al.* Absence of magnetic order in RuO_2: insights from μSR spectroscopy and neutron diffraction. *npj Spintronics* **2**, 50 (2024).

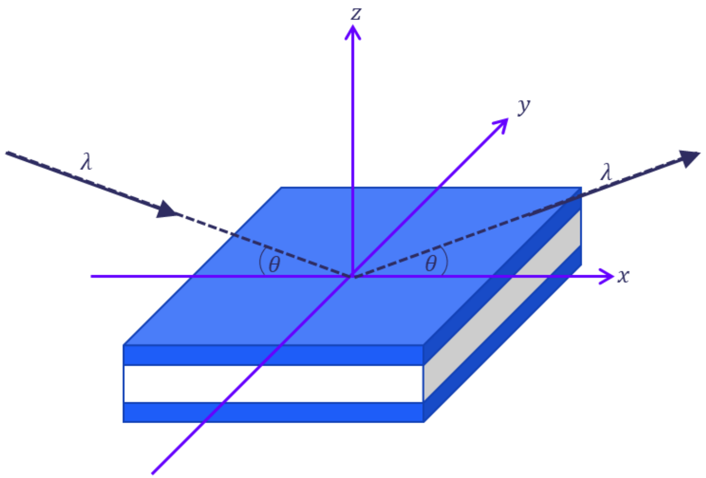
Polarised Neutron Reflectometry (PNR/PA):

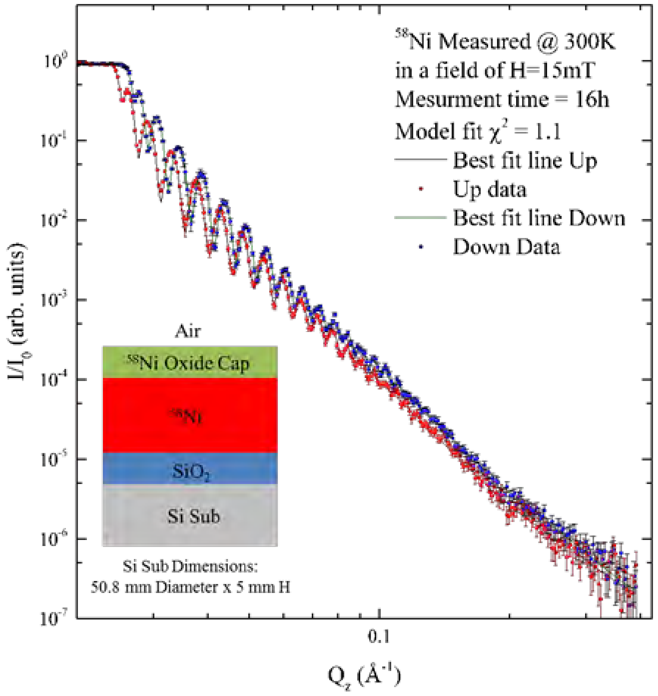
- **Specular neutron reflection provides information about the density profile normal to the sample surface**

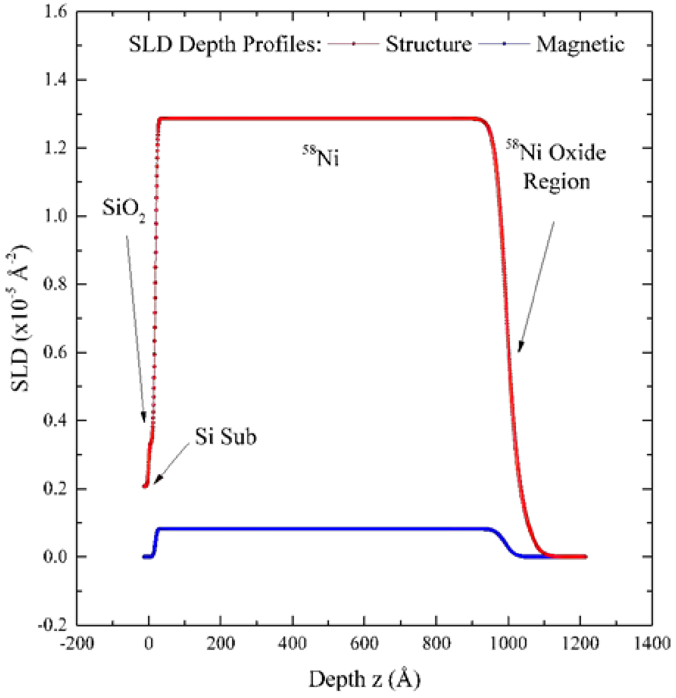
$$k^{\pm} = \sqrt{k - 4\pi N(b \pm cB)}$$

- The magnetic information comes by measuring the two neutron spin states labelled +/- or up/down
- Obtain the magnitude and orientation of atomic magnetic moments.

- <u>PNR gives magnetisation *depth* profile.</u>







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What kind of PRACTICAL scientific information can be obtained PNR:

Polarised Specular Reflectivity provides both the chemical/nuclear (isotopic) and the Magnetic scattering length density depth profile along the surface normal with a spatial resolution approaching half a nanometer (nm).

1. In Neutron Reflectivity (NR) you get three basic parameters assuming a box model is used to construct the SLD profile:

=> essentially **thickness, roughness, density (nSLD).**

2. In Polarised Neutron Reflectivity (PNR) these are joined by three more magnetic parameters:

=> essentially **magnetic thickness, magnetic roughness and magentic density (mSLD).**

a) Magnetic thickness: Magnetic dead layers, magnetic proximity effects, topological insulators.

b) Magnetic roughness: Canting, spirals, coupling, superconducting vortices.

c) Magnetic Scattering Length Density: Total moment, interlayer coupling (RKKY AF coupled layers), inhomogeneities, magnetic transitions (AF/FM/P).

3. In Polarisation Analysis (PA) you get all of the above, but also the in-plane vector direction- but it's really really hard work!!!!

- Note: PNR is NOT sensitive to inter-atomic magnetic order like antiferromagnetism!

Polarised Neutron Reflectivity (PNR example)

- Can you d
  Torque de

E

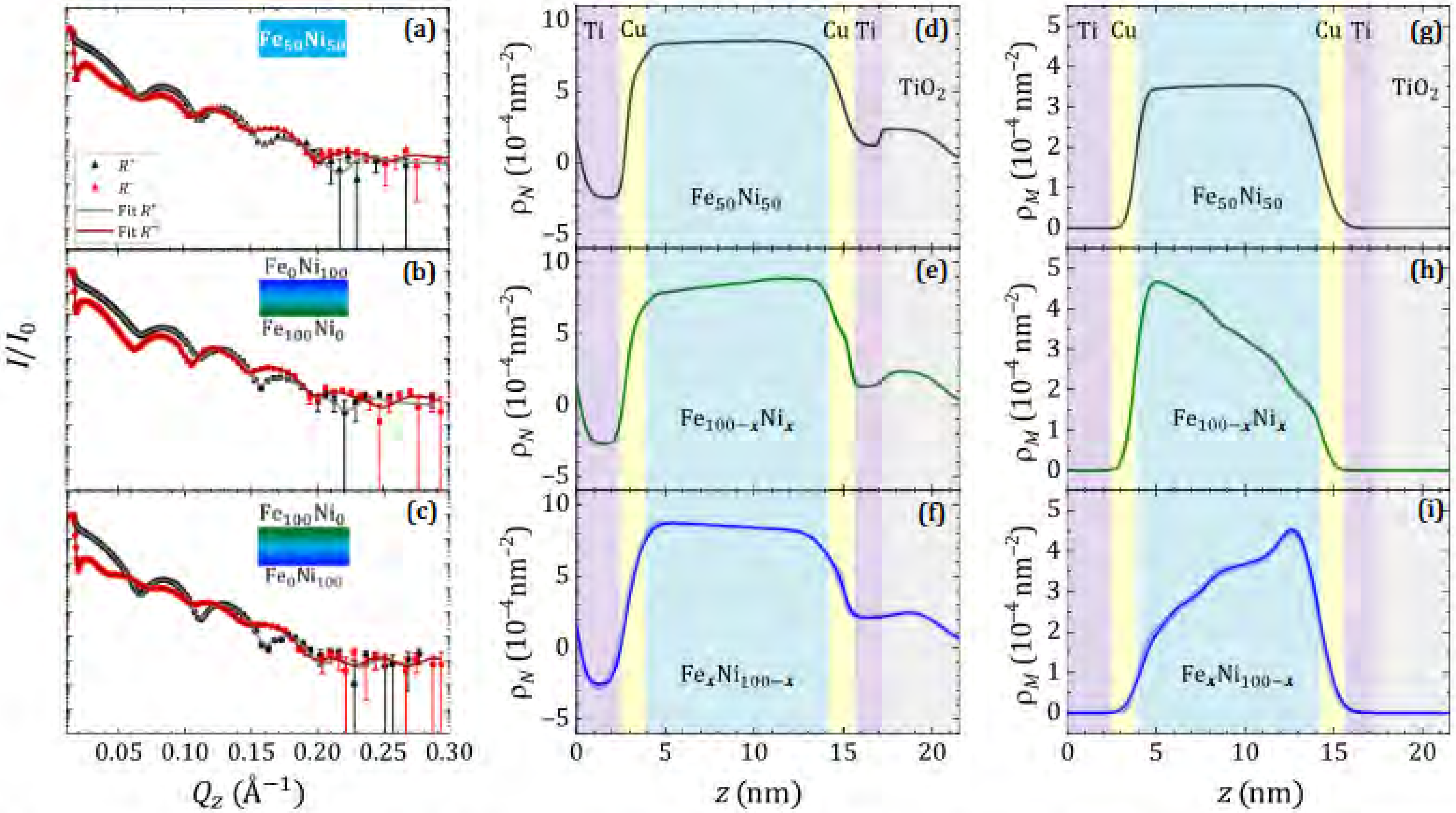

FIG.
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FIG. 8. (a)–(c) The normalized reflectivity in reciprocal space for polarized neutrons spin up, R^+, or spin down, R^-, (closed symbols) for (a) $Fe_{50}Ni_{50}$, (b) $Fe_{100-x}Ni_x$, and (c) Fe_xNi_{100-x}. The theoretical fits are shown by the solid gray (R^+) and maroon (R^-) lines. (d)–(f) The nuclear-scattering-length density ρ_N with the film thickness z for (d) $Fe_{50}Ni_{50}$, (e) $Fe_{100-x}Ni_x$, and (f) Fe_xNi_{100-x}. (g)–(i) The magnetic-scattering-length density ρ_M and the corresponding magnetization M (1 kA/m = 2.91 × 10^{-7} nm^{-2}) with the film thickness z, for (g) $Fe_{50}Ni_{50}$, (h) $Fe_{100-x}Ni_x$, and (i) Fe_xNi_{100-x}. The error bars represent ±1 standard deviation. The shaded bands indicate the 95% confidence bands of the best-fit depth profiles, determined by Markov-chain Monte Carlo calculations.

2 (2024)

izable spin-orbit torque

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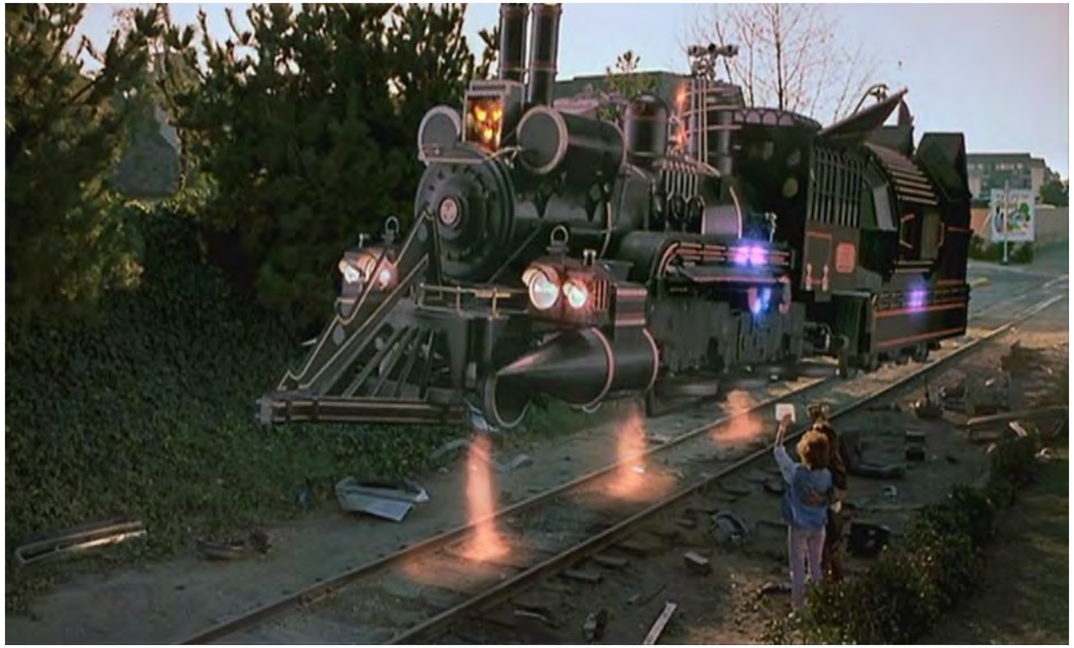
2024; published 21 October 2024)

T) and low damping to excite
et bilayers, reducing the ferro-
ally increases damping. Here,

Yes, you can !!

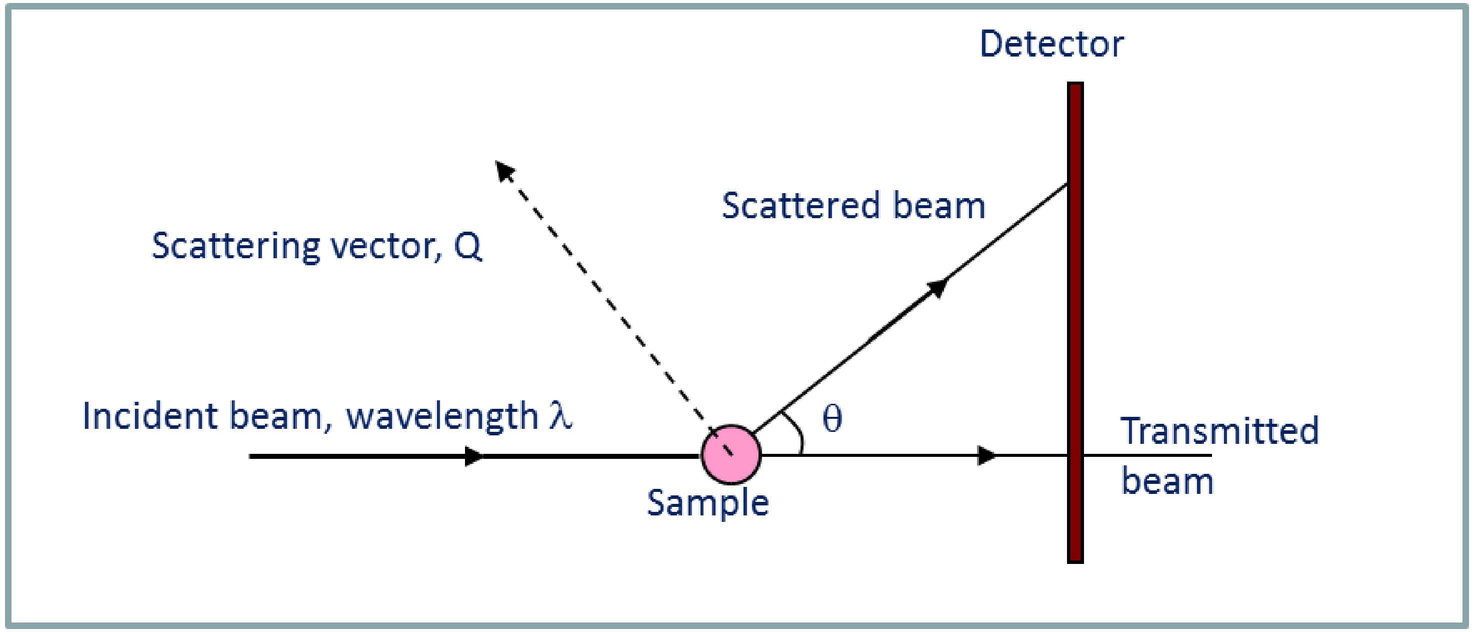
ISIS POLREF BEAMLINE: Some blatant self-promotion

- TOF wavelength band 1Å – 16Å
- Vertical and horizontal geometry.
- Non-polarised (NR), polarised (PNR) and polarisation analysis (PA) modes.
- Sample point goniometer capable of moving 900kgs.
- Experimental Setups:
  - GMW Magnet (± 0.7T with 2 samples), HTS Magnet (± 3T with one sample) with 3K -300K
  - Vacuum furnace (300K – 800K in ± 0.7T)
  - All with in-situ electrical connections
  - Helmholtz setup for Very low fieldwork sub 5 Oe.
  - RT 6 position sample changer for 0.7 and 3T magnets.



What is SANS? Well its diffraction at very small angles which means big things!

- Non-Magnetic materials studied include surfactants, polymers, liquid crystals, nanoparticles, lipids and fibres.
- Magnetic materials studied include things like superconducting vortexes, Nanoparticles, ferrofluids, skyrmions, magnetic helixes as well as steels and high entropy alloys.
- Length scales probed range from 10s to 100s nm even into the micron range



Allows the bulk properties of a material:

- Size
- Polydispersity
- Structure
- Particle Interaction

$$I(Q) = (r_p - r_m)^2 N_p V_p^2 P(Q) S(Q)$$

$$Q = \frac{4\pi \sin(\theta/2)}{\lambda}$$

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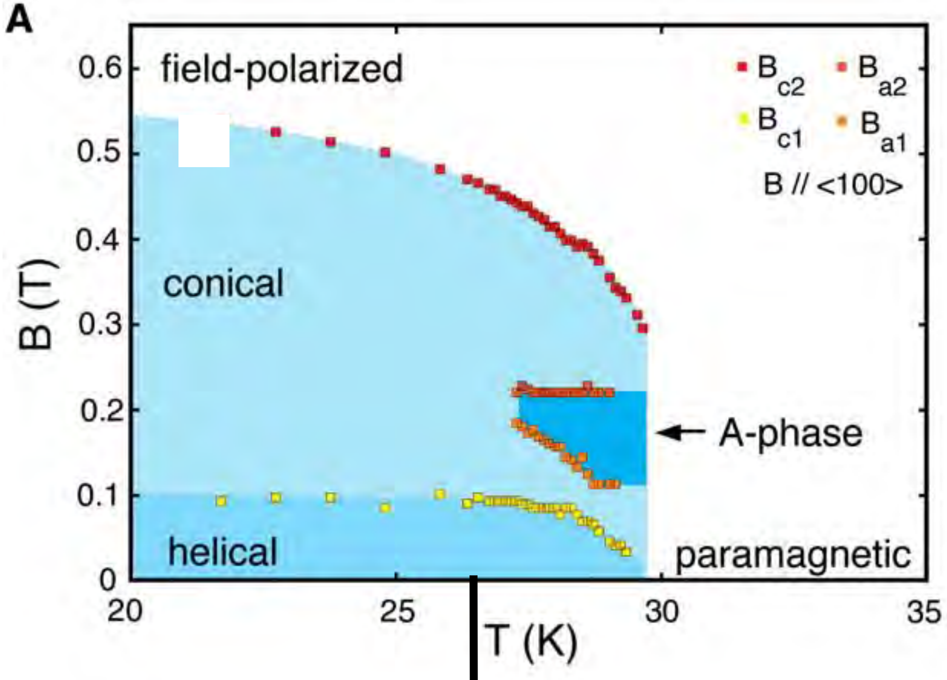
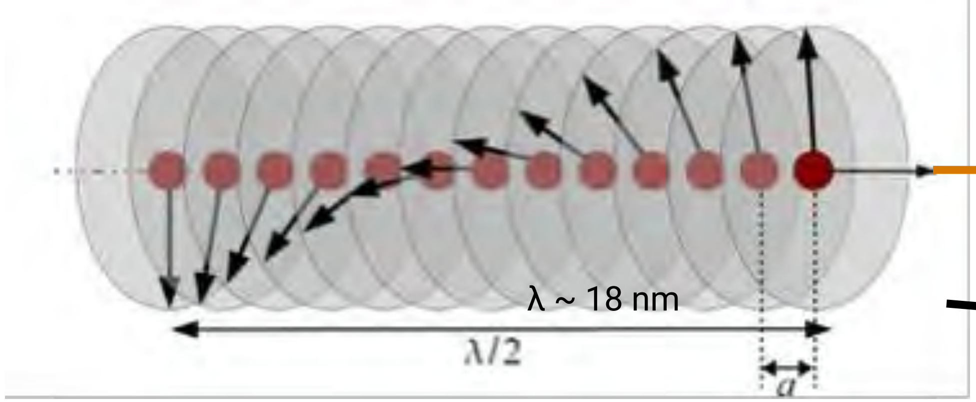
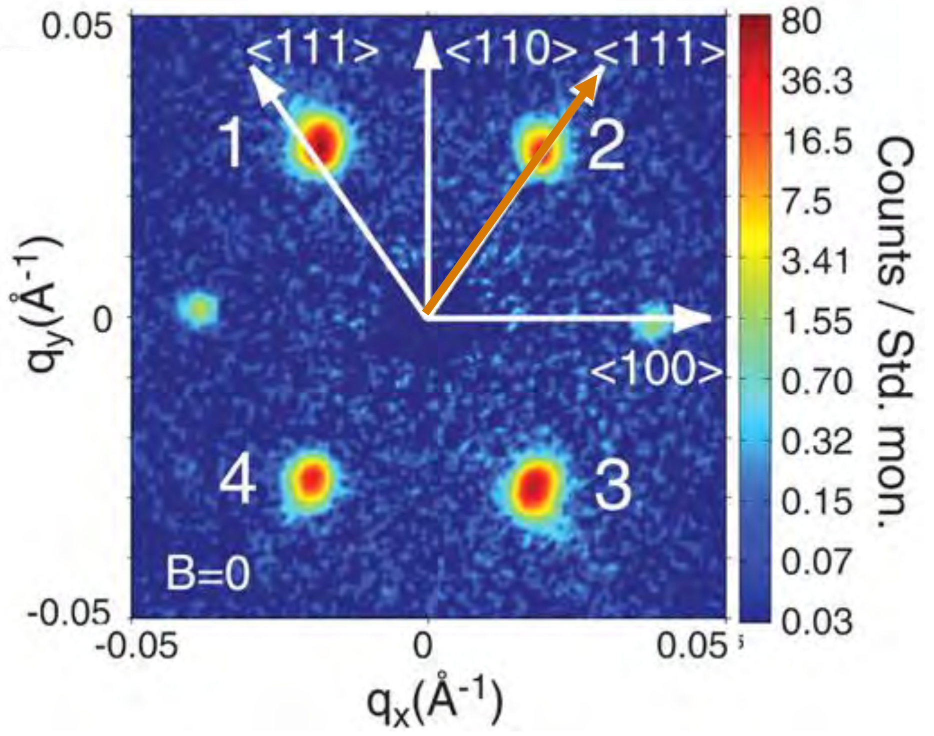
Magnetic SANS: Example: skyrmion lattice in MnSi





Neutron diffraction



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S. Mühlbauer *et al.* Skyrmion Lattice in a Chiral Magnet.*Science* **323**,915-919(2009)

Basic Concepts: What do we measure

(This is the hardest slide of the lot! Gets easier after this)

- **Total cross-section**

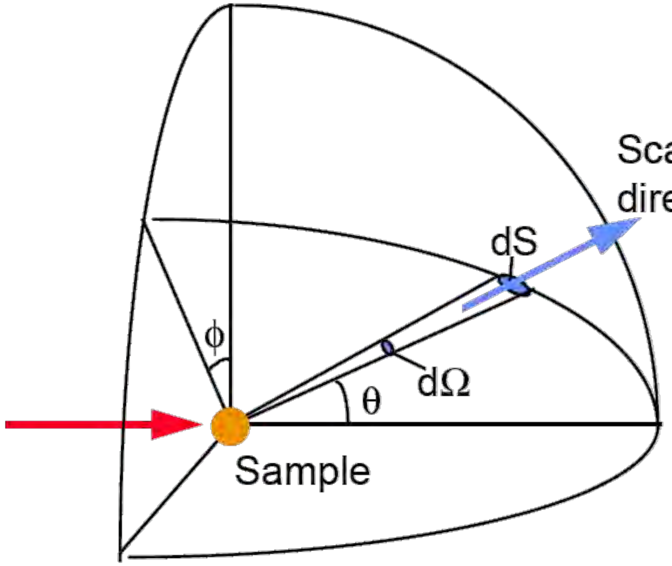
Imaging!!

$$\sigma = \int_{4\pi} \frac{d\sigma}{d\Omega} d\Omega$$

$$\sigma_{total} = \sigma_{scat.} + \sigma_{abs}$$

- **Differential cross-section** **DIFFRCTOMETERS!!**

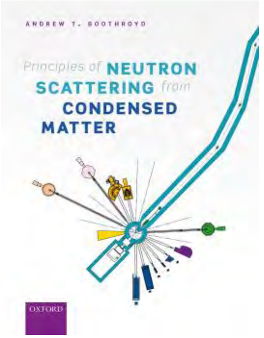
$$\frac{d\sigma}{d\Omega} = \int_0^\infty \frac{d^2\sigma}{d\Omega dE'} dE'$$: no energy analysis



- **Double differential cross-section** **SPECTROMETERS!**

$$\frac{d^2\sigma}{d\Omega dE'} =$$ (number of neutrons scattered into dΩ per second with final energy between E' and E'+dE)/ΦdΩdE'

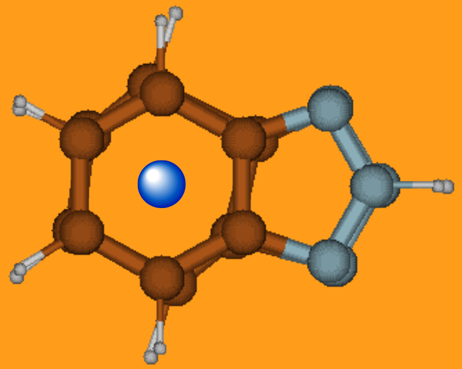
Principles of Neutron Scattering from Condensed Matter: Andrew T Boothroyd

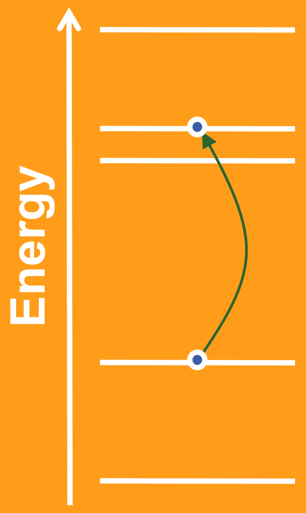


Spectroscopy: What atoms do…

There is both an energy change and a vector change upon scattering: INELASTIC!

2H **- Benzotriazole**





Vibrational energies

↓

Bond strengths

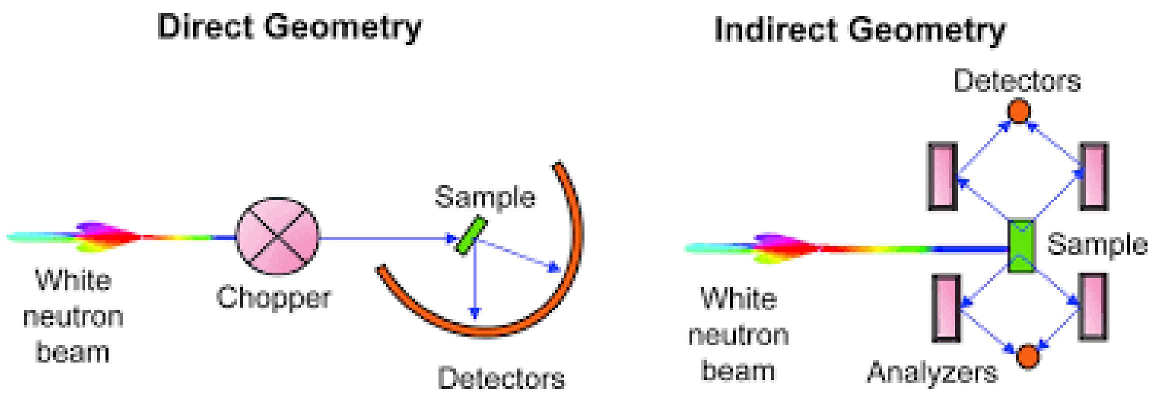
↓

Thermodynamics

Tells you about excitations: Phonons, Vibrations, Rotations, Magnon's.

Basic concepts in-elastic Scattering

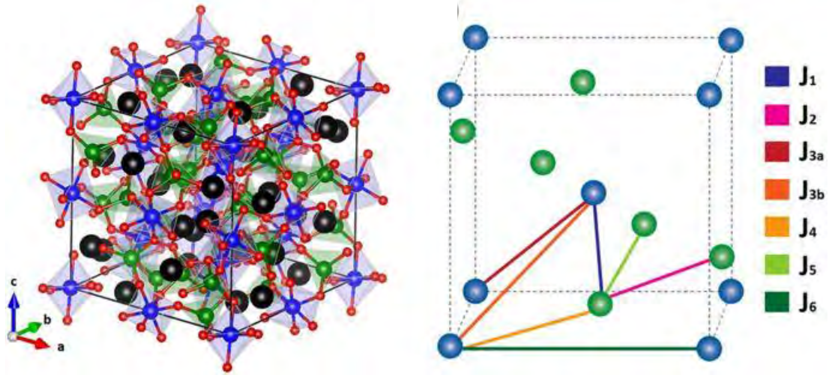
- Both direction (momentum transfer) and energy (wavelength) can change on interaction with the sample.
- Neutrons can both give up energy to the sample and gain energy from the sample.
- Can provide information on vibrational modes in the sample e.g.
  - Phonons,
  - Rotational and other vibrational excitations.
  - Including magnetic excitations – Magnons
  - Can also look at how Bonds behave.



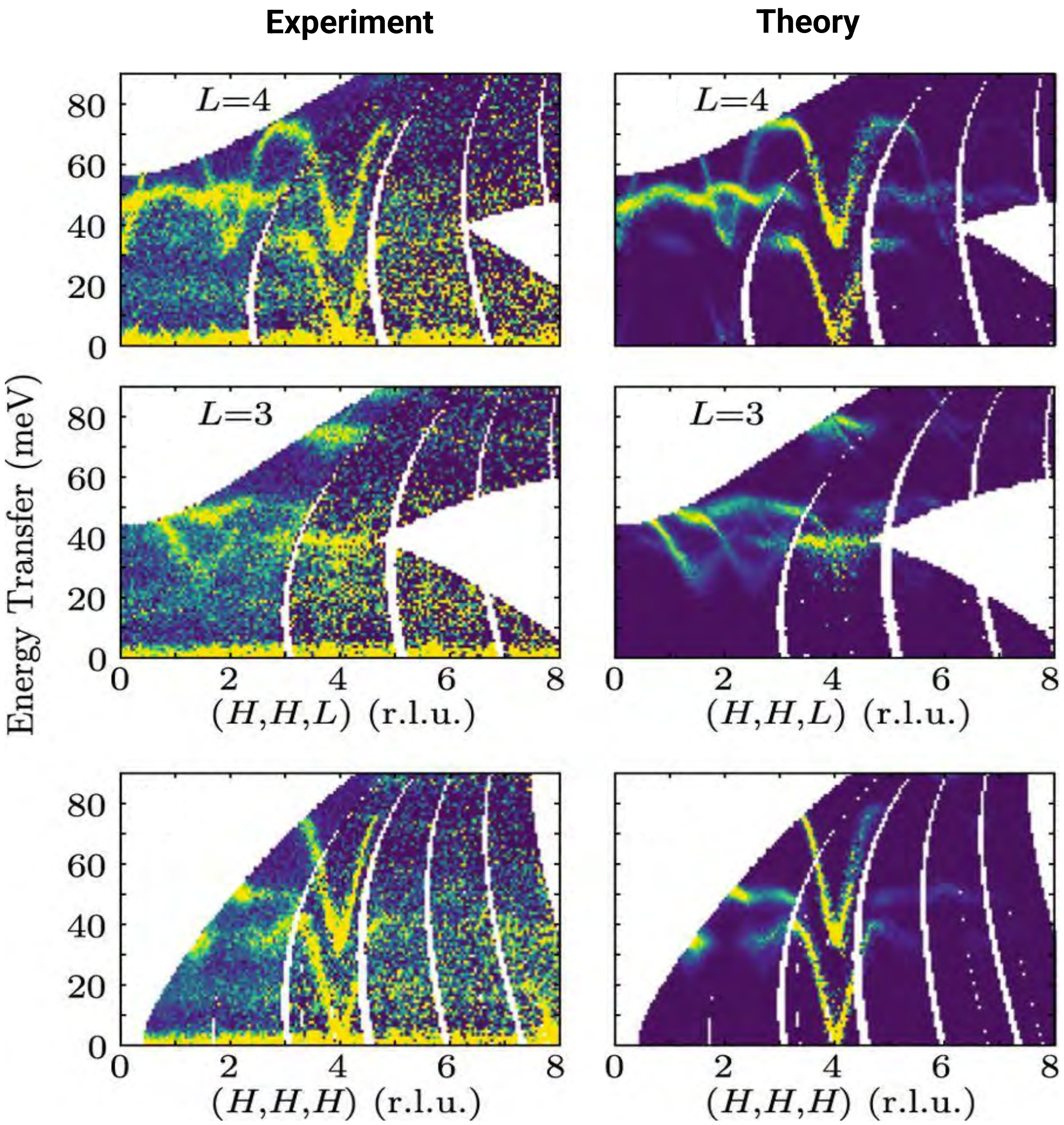
LET Direct Spectrometer at ISIS

https://www.isis.stfc.ac.uk/Pages/let.aspx

Example: magnons in YIG



$$\mathcal{H} = \sum_{i,j} J_{ij} \mathbf{S}_i \cdot \mathbf{S}_j$$



- Ferrimagnetic insulator with 2 magnetic sites, complex structure
- Used as the spin-current carrying material in devices which exploit the spin-Seebeck effect…

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A. J . Princep. *et al.* The full magnon spectrum of yttrium iron garnet. *npj Quant Mater* **2**, 63 (2017).

Thank you for listening and please don’t forget these conferences coming up in the UK.

Also, the next ISIS proposal deadline is 3rd of March 2025
https://www.isis.stfc.ac.uk/Pages/For-Users.aspx

But Express/Rapid access also available! See the URL for details!

- Please email me if you have any questions about using neutrons.
- If I can't help, the will I will forward you to someone who hopefully can.
- Email: christy.kinane@stfc.ac.uk

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