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Never Stand Still | Faculty of Science | School of Chemistry

Neutron Sources & Techniques: an overview

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Overview

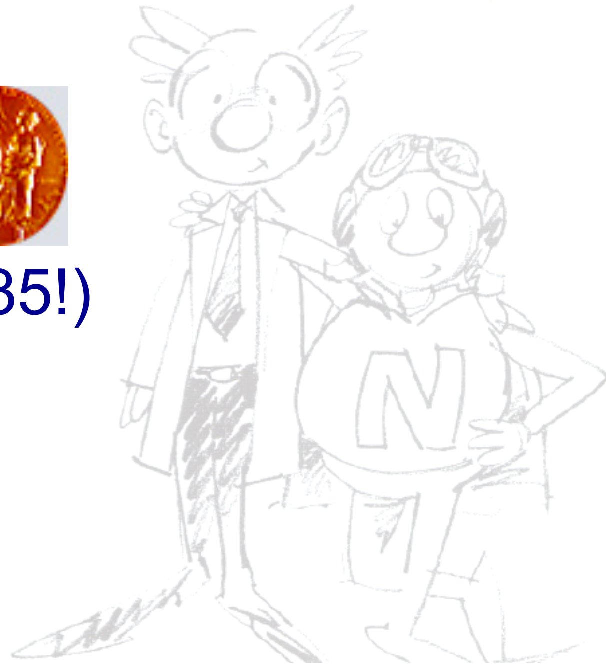
- Neutron scattering – catching the *bug*…

- Small science in big facilities with major impact!

- Today's talk:
  - A bit of historical perspective
  - Neutron sources
  - Techniques through examples…. and a few Nobels..

The neutron…..

- discovered in 1932 (James Chadwick, Cavendish Lab. - **Nobel Prize**, Physics 1935!)
    - Po source (alpha emitter) behind Be



- sub-atomic particle:
    - mass = 1.67493 x 10^{-27} kg ~ 1 amu
    - mean free lifetime ~ 880 s (~15 mins.)
    - charge = 0; spin = ½; magnetic moment = -0.966 x 10^{-26} JT^{-1}

Lise Meitner & Otto Hahn (Berlin) first observed fission in 1938 when bombarding uranium with neutrons….

(Hahn won **Nobel Prize**, Chemistry, in 1944).

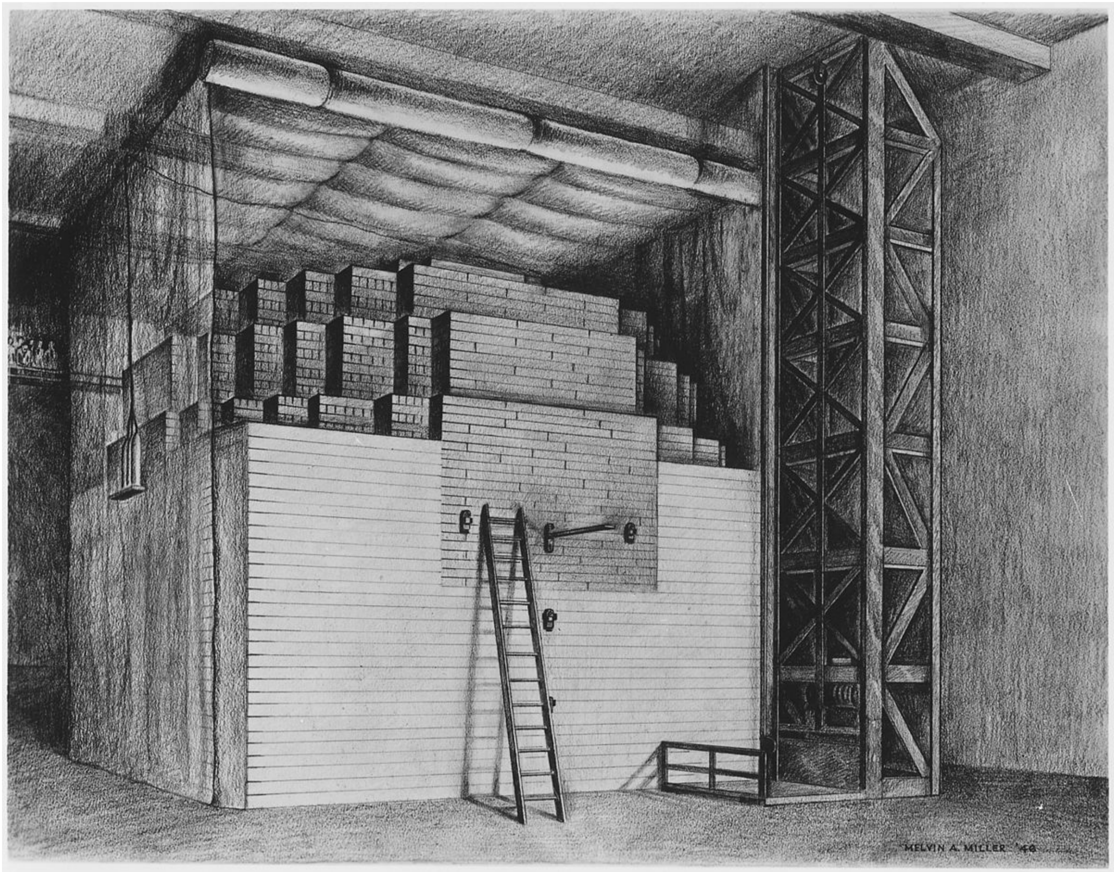
Neutron scattering

- Need a *machine* to provide **large numbers** of neutrons….
  - Chicago Pile 1 (CP-1); the world's first nuclear reactor (1942-3)

Enrico Fermi (**Nobel Prize**, Physics 1938), *et al.*

Built in an underground squash court at Stagg Field, U.Chicago…

SCRAM

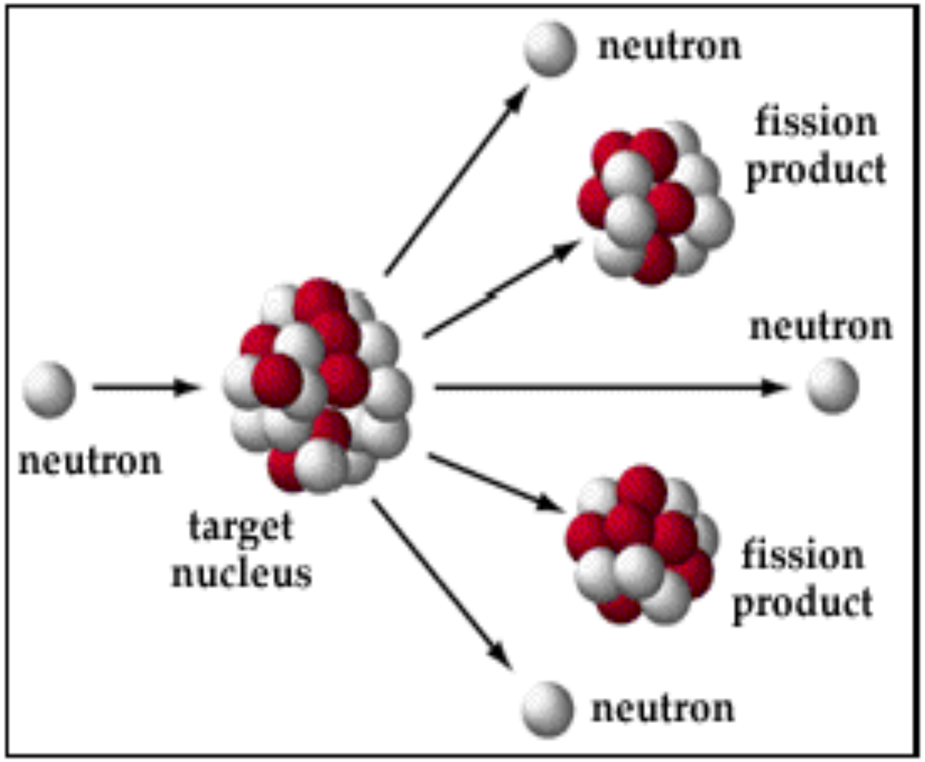


CP-1 (& existing nuclear reactors) sustain Nuclear Fission reactions

Fission

noun
1. division or splitting into two or more parts

verb
1. (chiefly of atoms) to undergo fission



- 2.5 neutrons per event
- 1 neutron consumed in sustaining reaction
- 0.5 absorbed
- high power load per neutron (~ 180 MeV)

Neutron beams

de Broglie's wavelength:

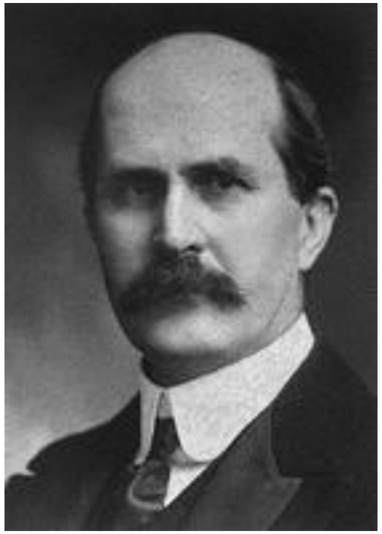
$$\lambda = \frac{h}{p} = \frac{h}{mv}\sqrt{1-\frac{v^2}{c^2}} \approx \frac{h}{mv}$$

Paris, **Nobel Prize**, Physics, 1929

$$E = \frac{3}{2}kT = \frac{1}{2}mv^2$$

$$\rightarrow \lambda = \frac{h}{mv} = \frac{h}{m\sqrt{\frac{3kT}{m}}}$$

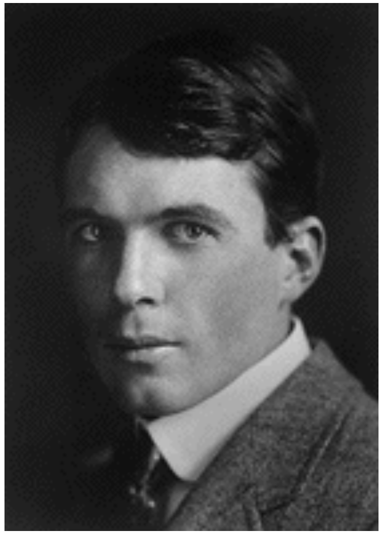
For $T = 300\text{K}$; $\lambda = 1.45 \times 10^{-10}\,\text{m}$



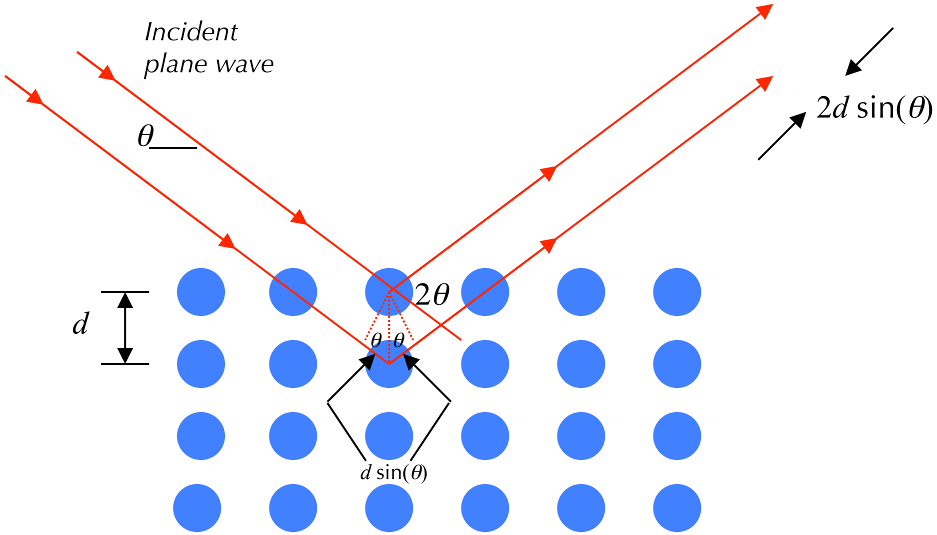
William Henry Bragg
1862-1942

Bragg's Law

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



William Lawrence Bragg
1890-1971

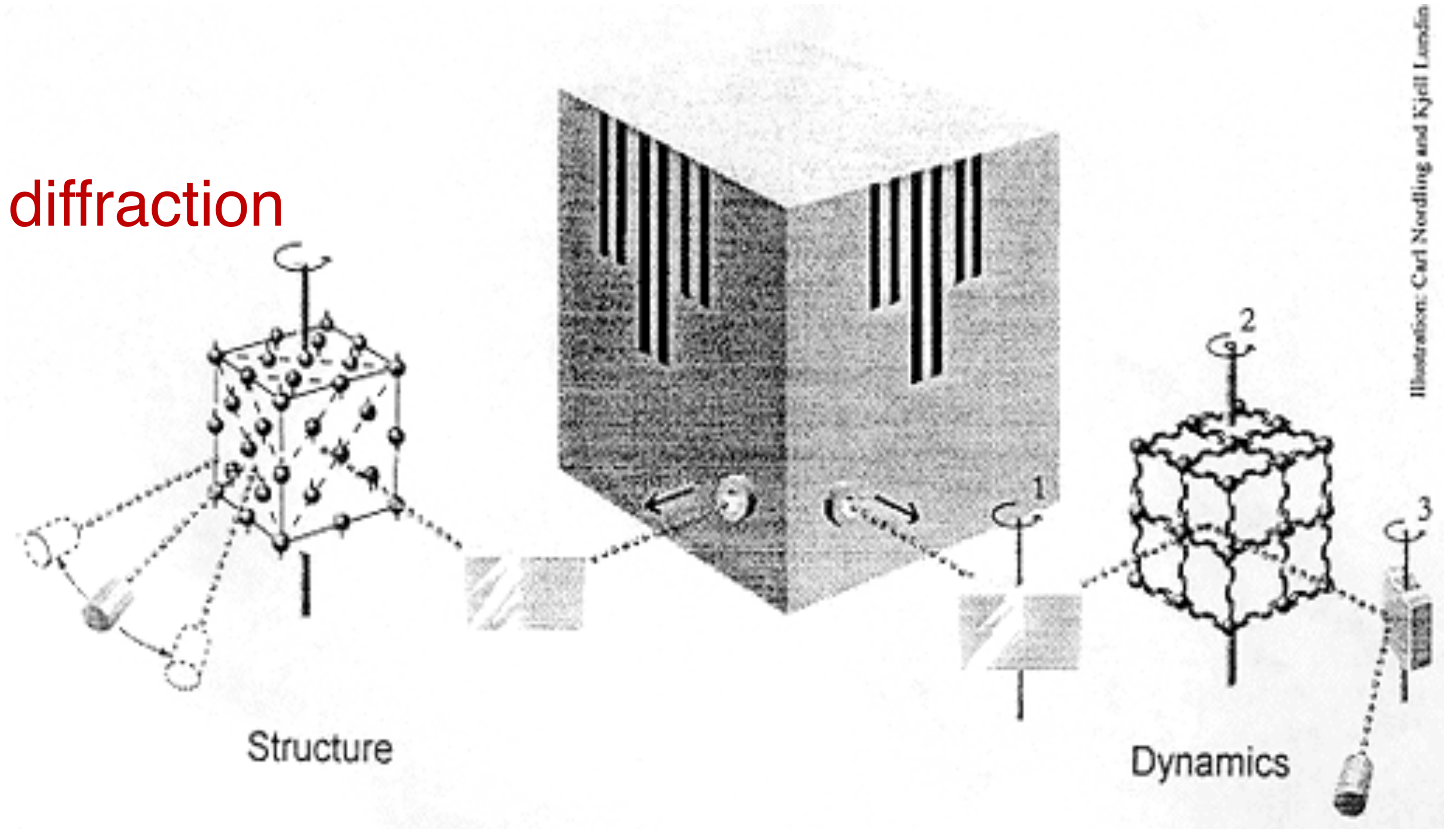


Father & son **Nobel Prize**, Physics, 1915

Where atoms are and what they do……

Instruments right on reactor faces to measure both structure and dynamics….

2-axis: diffraction



3-axis: dynamics

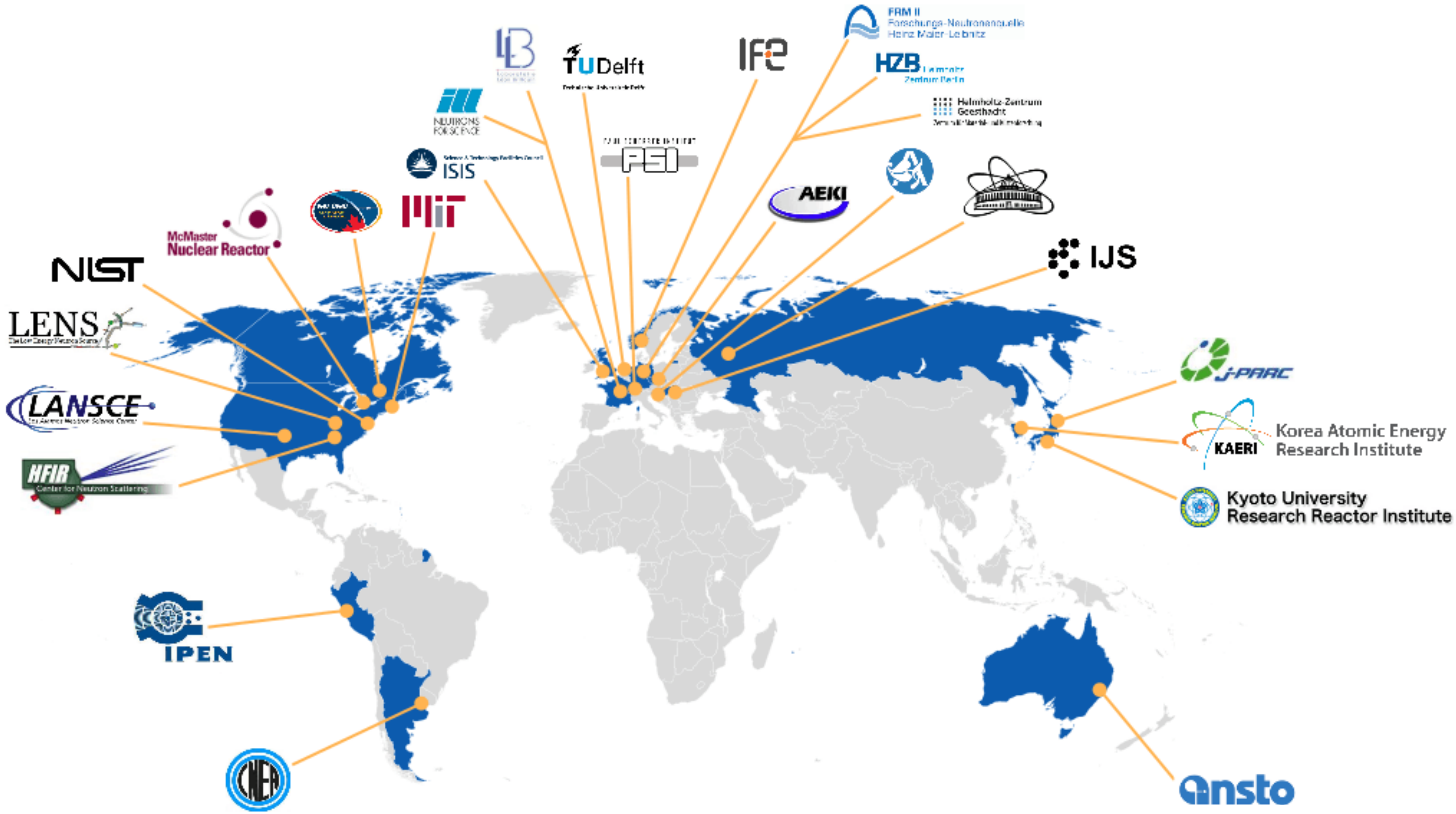
Field of neutron scattering emerges

- With reactors being built, more and more materials were being studied (and not just for reactor physics – or even non-peaceful means)

- By the early 1950s, a new field had emerged with pioneers of the field already in place…

….. much of this work, it has to be said, was largely facilitated by the emerging Cold War

Neutron sources around the world….



…a little out of date,

but it serves my purposes…

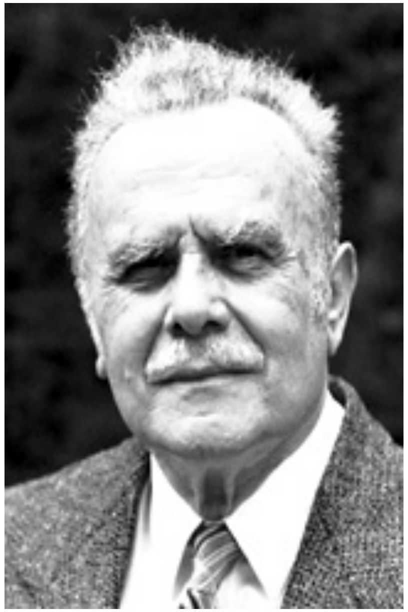
http://www.veqter.co.uk/assets/images/content-images/neutron-diffraction/map-of-world-neutron.png

Nobel Prize in Physics 1994

"for pioneering contributions to the development of neutron scattering techniques for studies of condensed matter"

Bertram N. Brockhouse (1918-2003)

McMaster University
Hamilton, Ontario, Canada



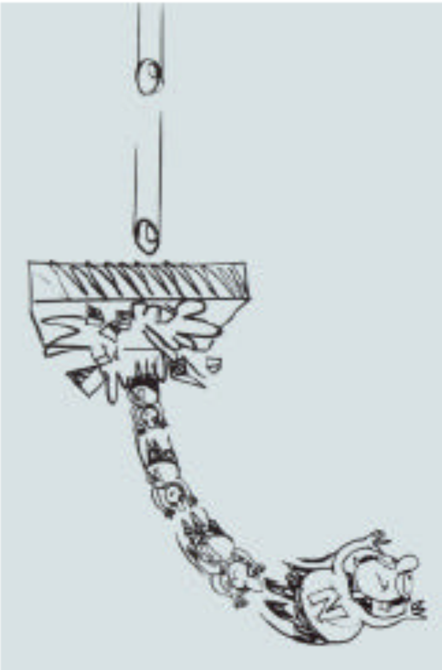
Clifford G. Shull (1915-2001)

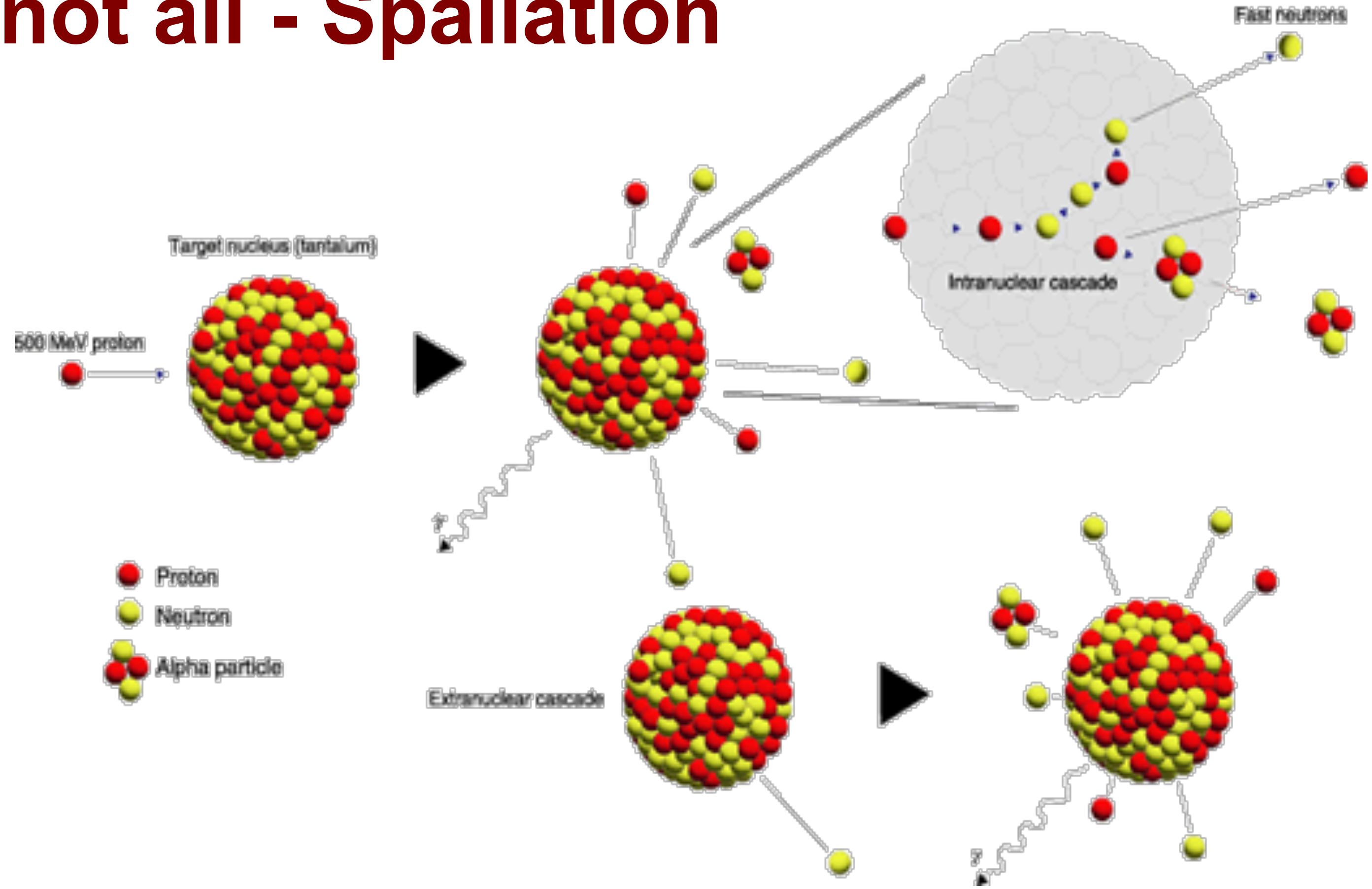
Massachusetts Institute of Technology (MIT)
Cambridge, MA, USA

"for the development of neutron spectroscopy"

"for the development of the neutron diffraction technique"

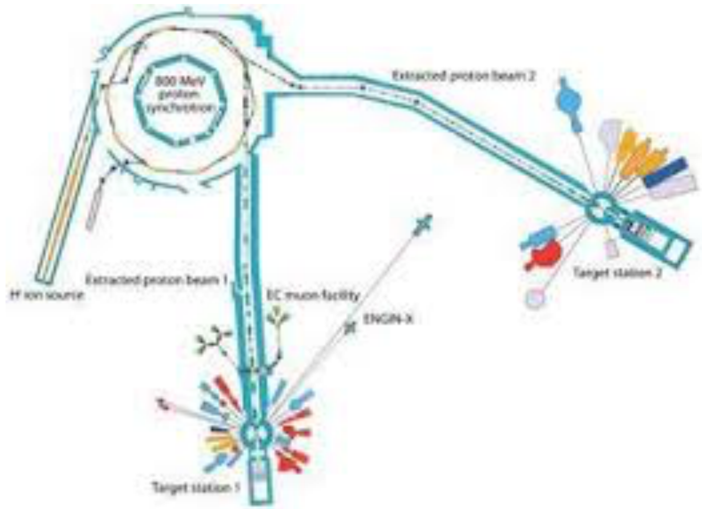
But that's not all - Spallation





- High energy incoming particle (typically protons)
- Heavy metal target (Ta, W, U)
- Neutron cascade; >10 neutrons per incident proton
- Low power load per outgoing neutron (~ 55 MeV)

World’s most intense spallation source was until recently, ISIS, UK….



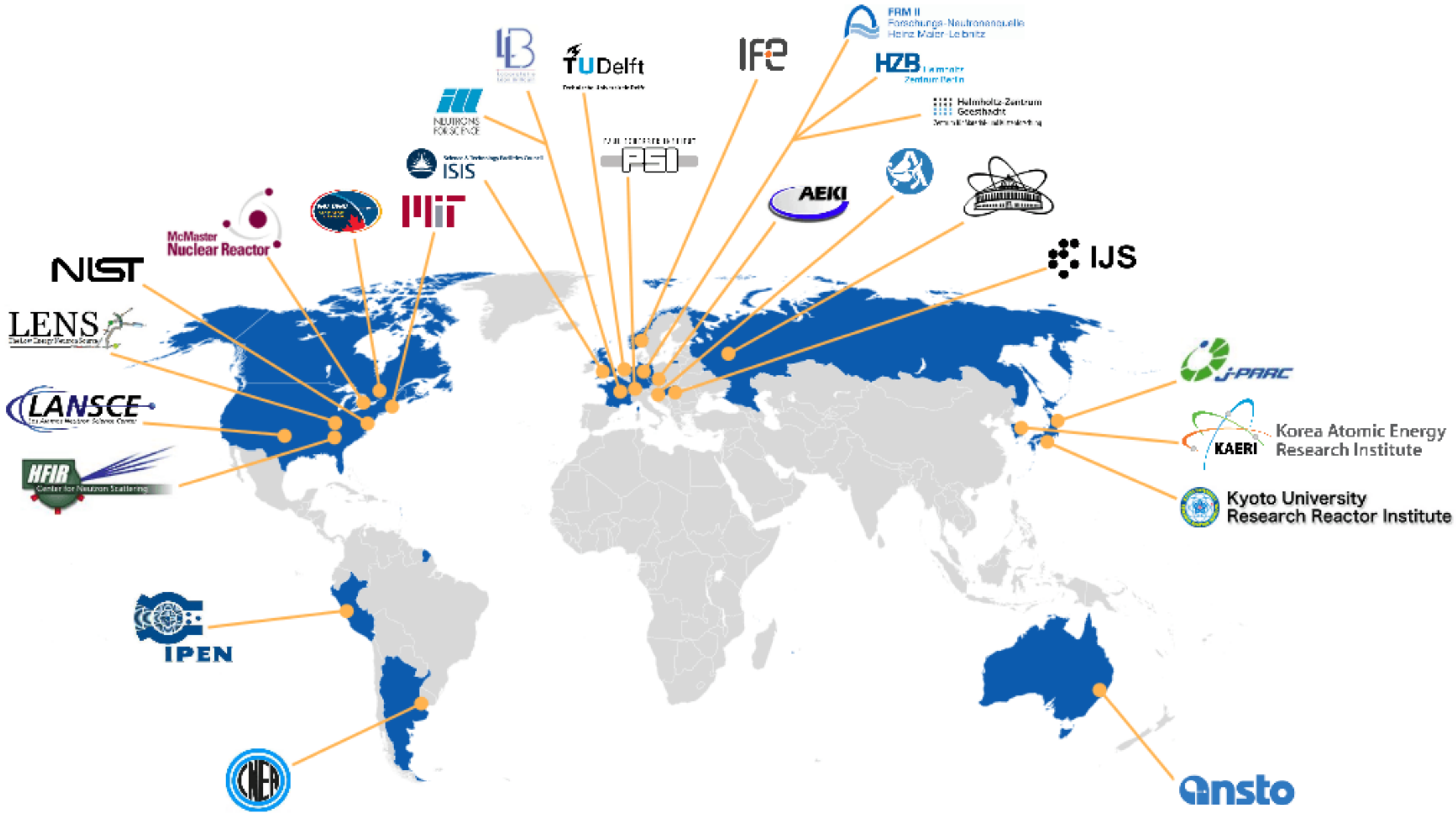
Born of an obsolete physics experiment on a disused airfield given over to nuclear science (Harwell), ISIS was Europe’s first high-intensity spallation source and has now ceded 1st place to the first of the 3rd gen. sources.

Meanwhile, the world's most intense neutron source is still the 1960s-designed ILL, France

An ageing piece of infrastructure (consortium of European countries) that has served the field well for almost 50 years!

On the second reactor vessel, ILL is currently slated for retirement around 2030….

Neutron sources around the world....

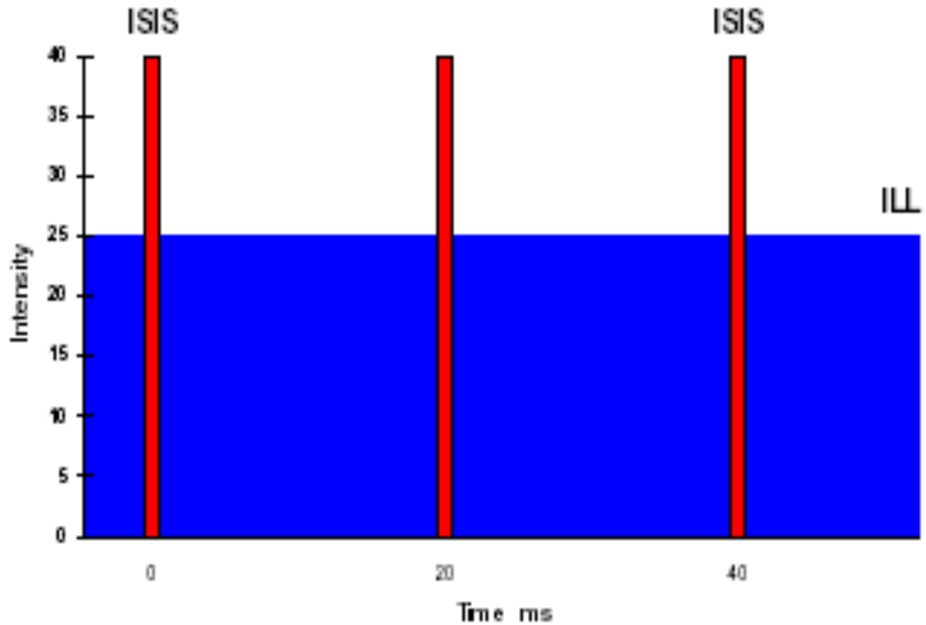


…a little out of date,

but it serves my purposes…

http://www.veqter.co.uk/assets/images/content-images/neutron-diffraction/map-of-world-neutron.png

Advantages & disadvantages of spallation sources..



- large epithermal neutron flux
- intrinsically sharp pulses (hi-res).
- ability to work with restricted angular coverage (complex sample environments)
- pulsed operation mode gives low background - source is OFF when measuring

- low time-averaged flux
- fixed duty cycle (often want >50 Hz)
- reliability less than reactor
- unsuited to some measurement types

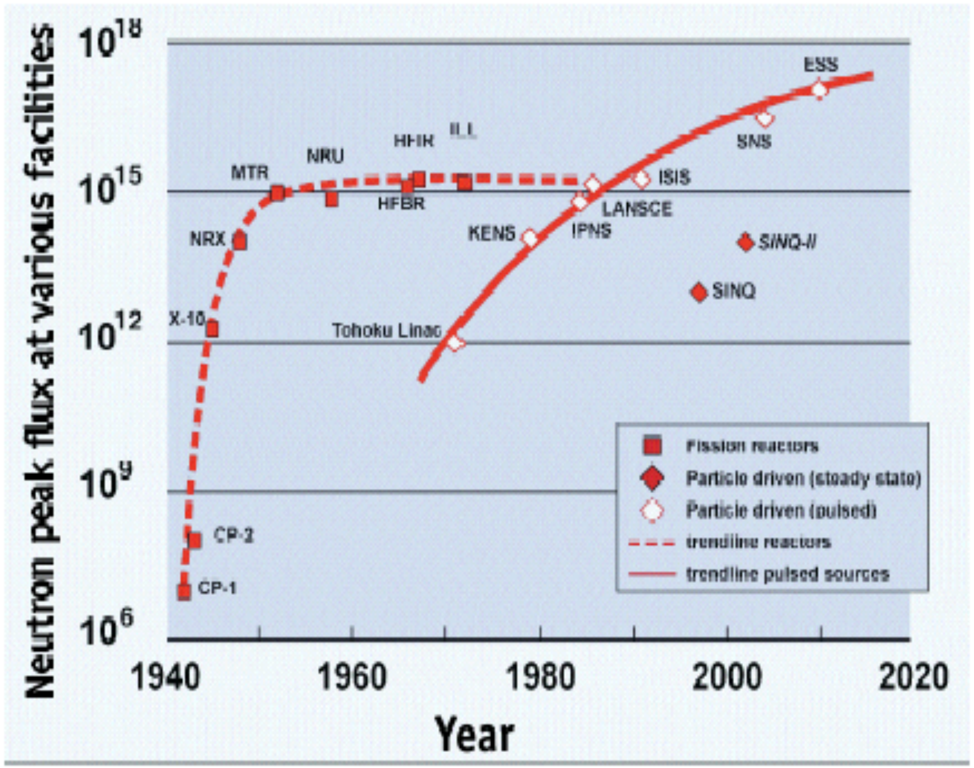
ILL is close to the limits of reactor design

- In 1990's US killed ANS project due to marginal gains & cost
- political component (!)

In order to continue to higher fluxes, pulsed spallation sources offer the best hope..

.. higher fluxes needed for more demanding experiments, smaller samples

.. 3rd generation sources will have time integral flux ~ILL and novel modes of operation (LPSS)

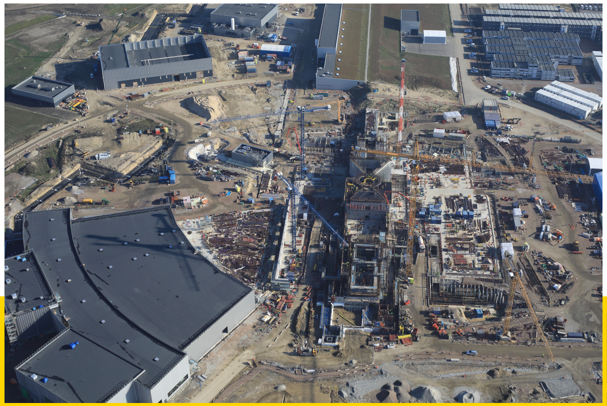


3rd generation neutron sources

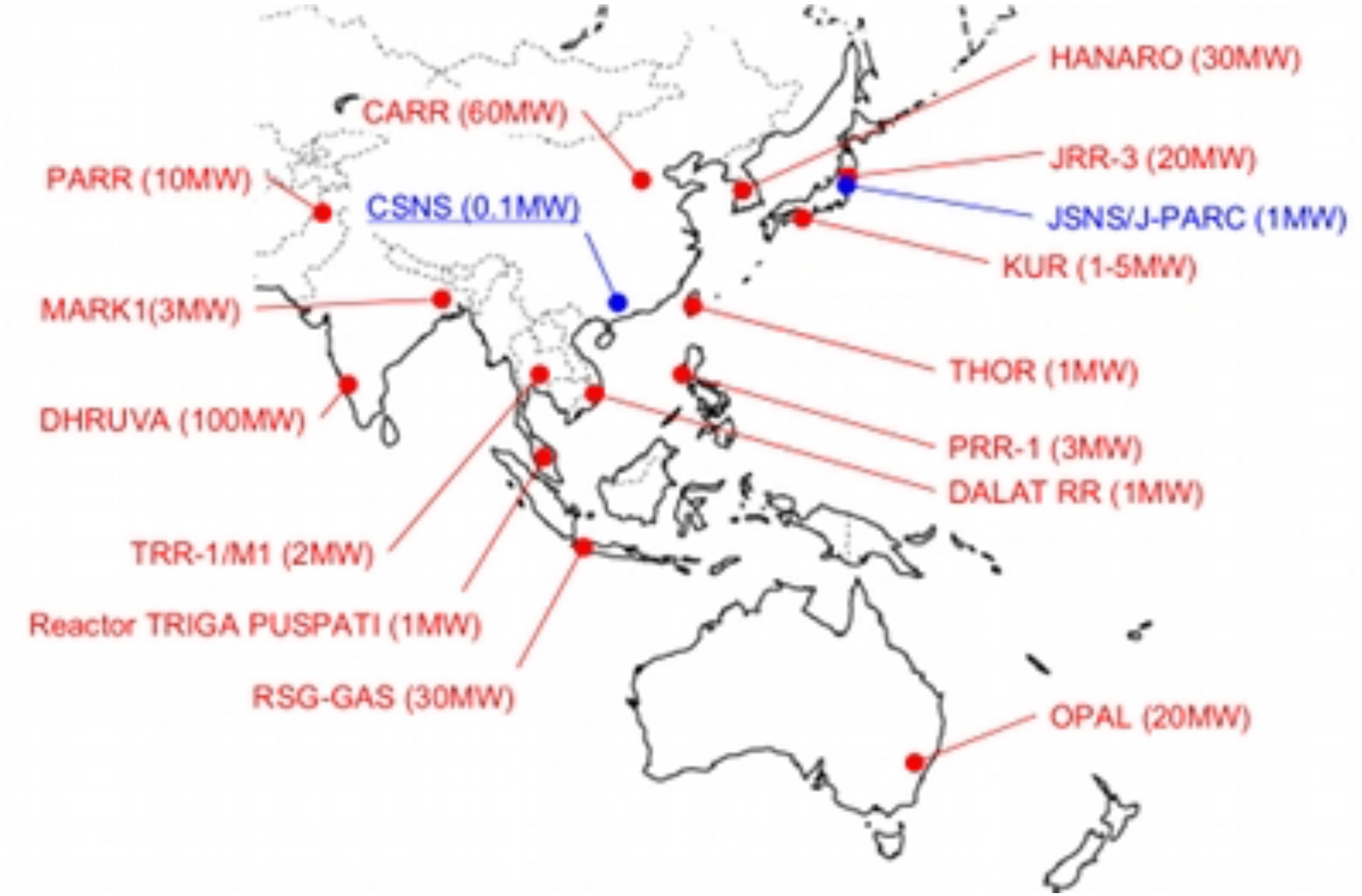
SNS - Oak Ridge, USA

JPARC - Tokai, Japan

ESS - Lund, Sweden

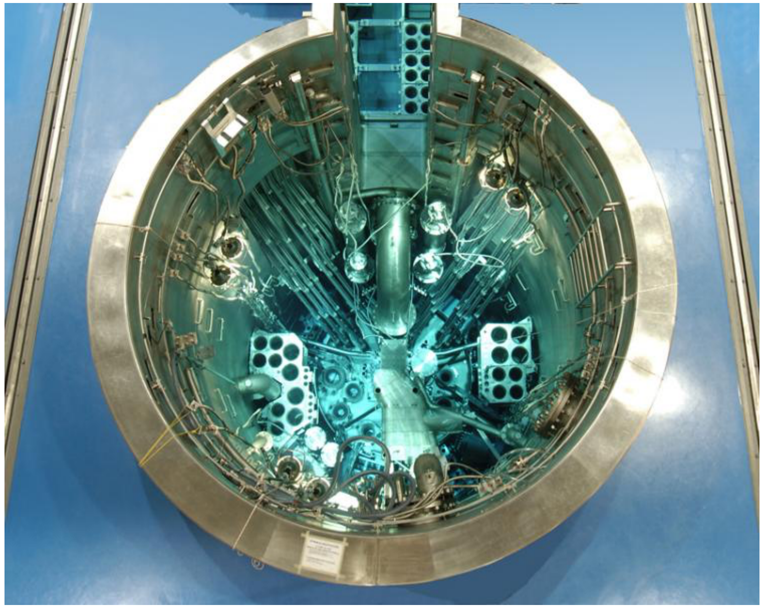


Closer to home.... AONSA:



http://www.epsnews.eu/2014/04/neutron-facilities-asia-oceania/

https://youtube/GooWJywwfgo



Here at ANSTO

OPAL reactor (20 MW) feeds 14 instruments:

WOMBAT	high-int. PND
ECHIDNA	high-res. PND
KOALA	SXD
PLATYPUS	reflectometer (i)
SPATZ	reflectometer (ii)
QUOKKA	SANS (i)
BILBY	SANS (ii)
KOOKABURRA	USANS
JOEY	Laue camera
TAIPAN	thermal-TAS
PELICAN	cold-TOF
SIKA	cold-TAS
EMU	backscattering (high-res.)
DINGO	imaging

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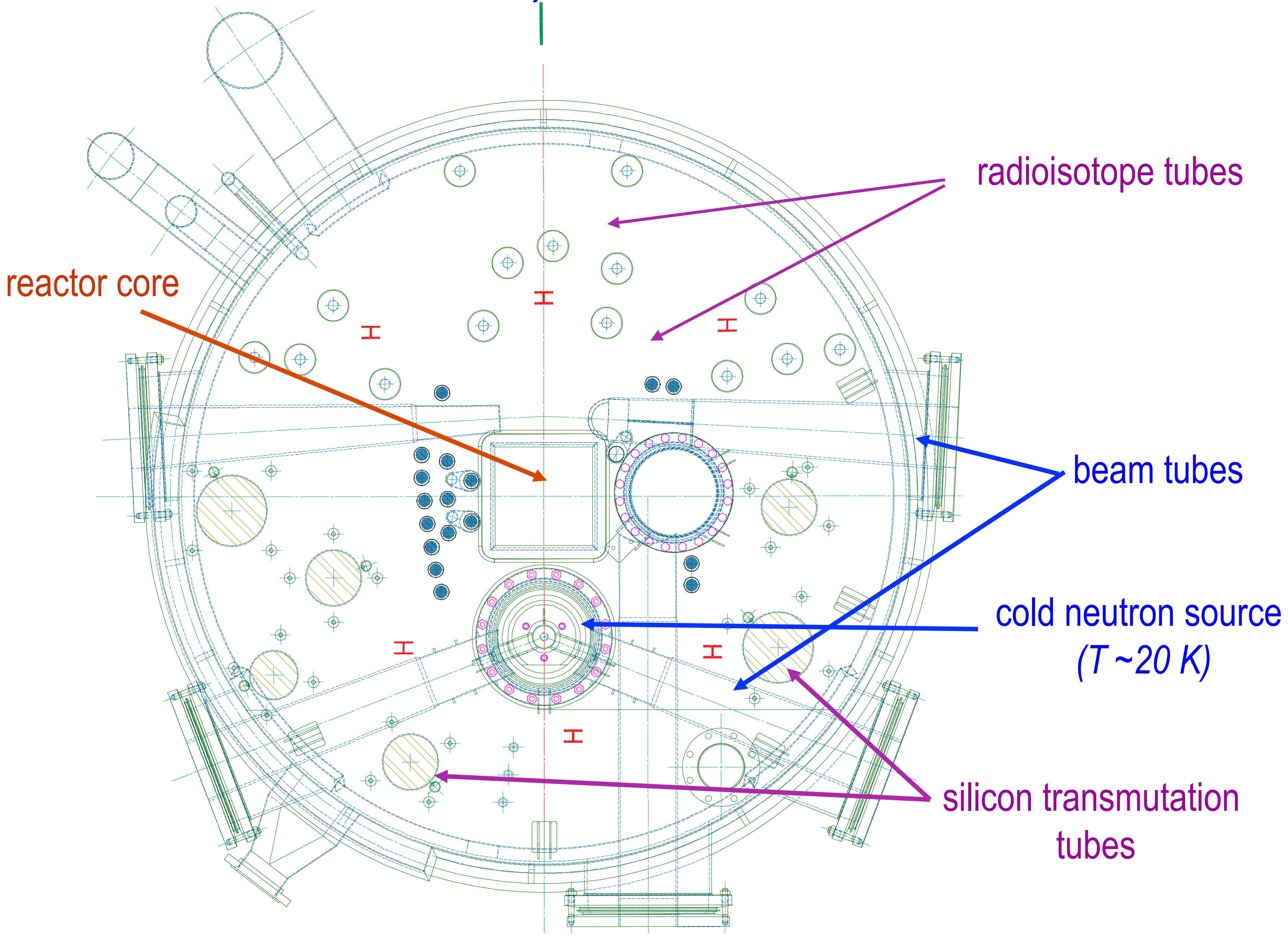
OPAL reactor core, moderator and beam tubes



Applications, examples & techniques.....

The following slides are not mine.

Dr. Rob Robinson put them together and has kindly allowed me to use them today....

I cannot fail to agree with the examples that Rob has chosen to highlight the continued impact that neutron scattering is making to condensed matter science...

Some simple questions from 1979

How many forms of carbon are there?
Can we make perfect (practical) electrical conductors?
Can 5-fold symmetry exist in a crystal?
What is the world’s best permanent magnet?
Can we improve on lead-acid batteries?
    what about an electric sports car?
What is the strongest engineering material?
Can one use solid-state technology to produce white light?
Can recording technology keep up with Moore’s Law?
    what about Kryder’s Law and Hatz’s Law?

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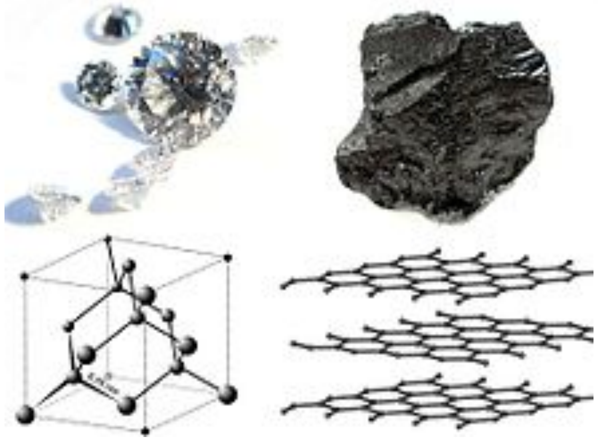
Example 1a – 1985
Nobel Prize in Chemistry 1996

The most important Element – What we were taught in school about Carbon

Two allotropes:

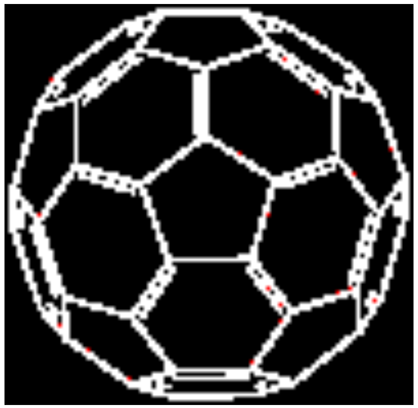
Diamond

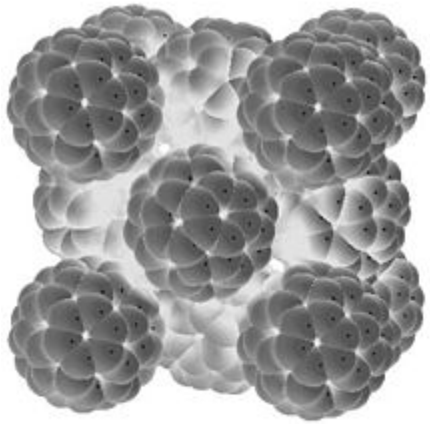
Graphite



But we also knew about *Carbon Black, Soot, Coke*,….

1985 – C_{60} discovered (a.k.a. *Buckminsterfullerene, buckballs*)





C_{60} – what's it useful for?

No application of C_{60} has been commercialized

But:

- Non-radioactive tracing (e.g. with helium)
- Inhibits HIV
- Potential photovoltaic applications
  - (absorption matches solar spectrum well)
- Electron acceptors in solar cells

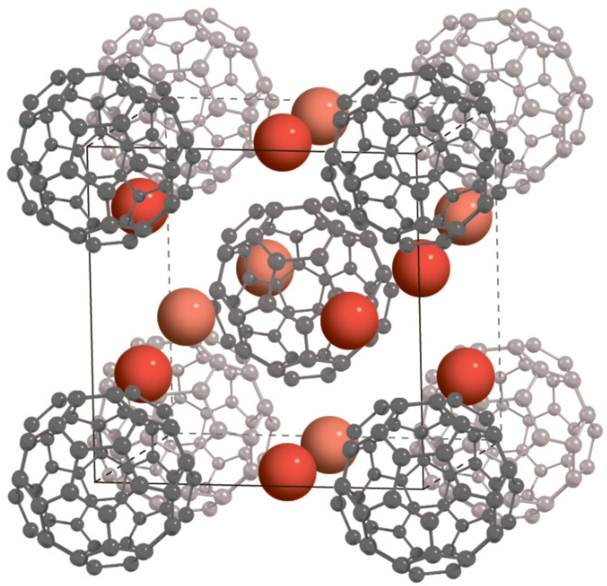
See https://en.wikipedia.org/wiki/Buckminsterfullerene

C_{60} – why is it interesting?

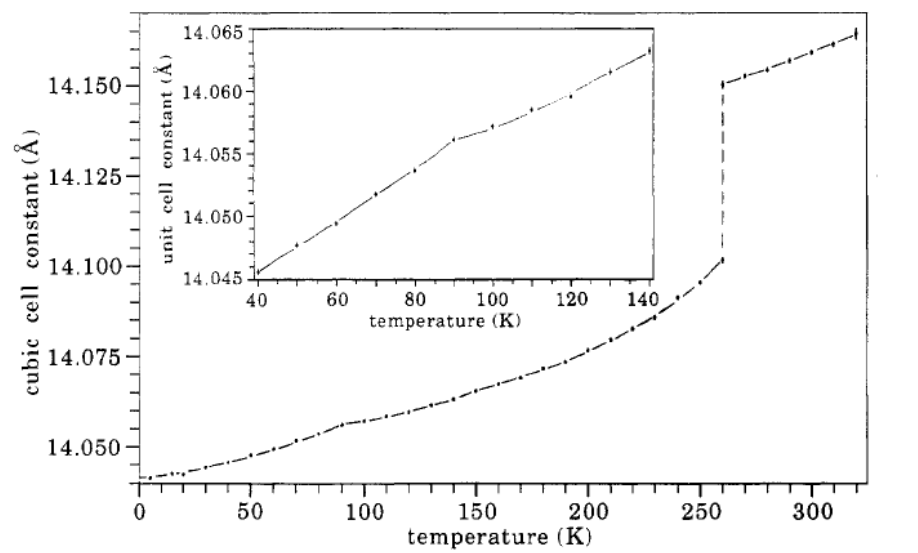
C_{60} does occur in soot

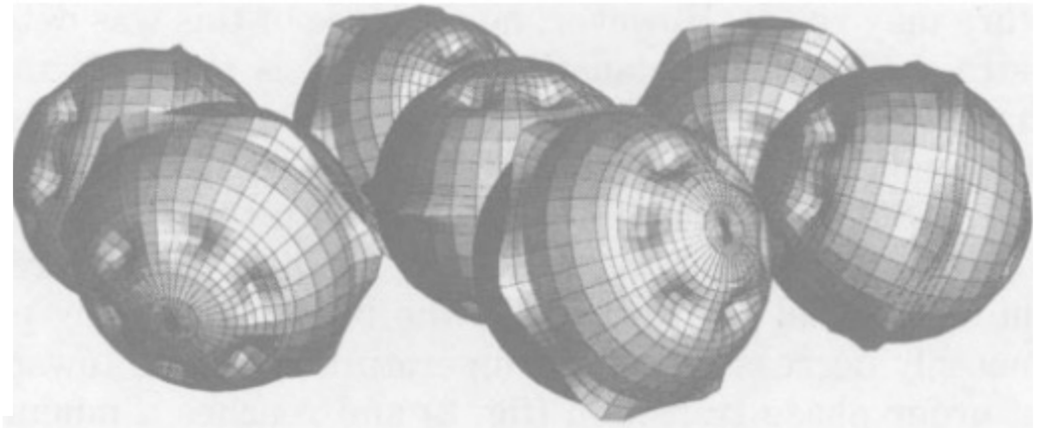
Other fullerenes: C_{20}, C_{70}, C_{72}, C_{76}, C_{84}, etc.

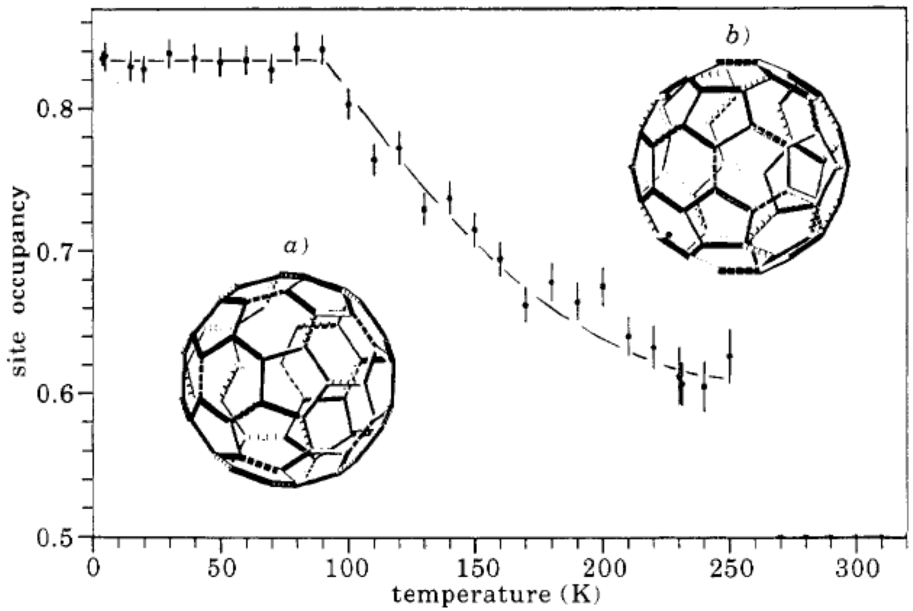
Alkali metal + C_{60} is a superconductor



C_{60} – what did neutrons contribute?







Bill David *et al.*

Nature **353**, 147 (1991).

Europhys. Letters **18**, 219 (1992).

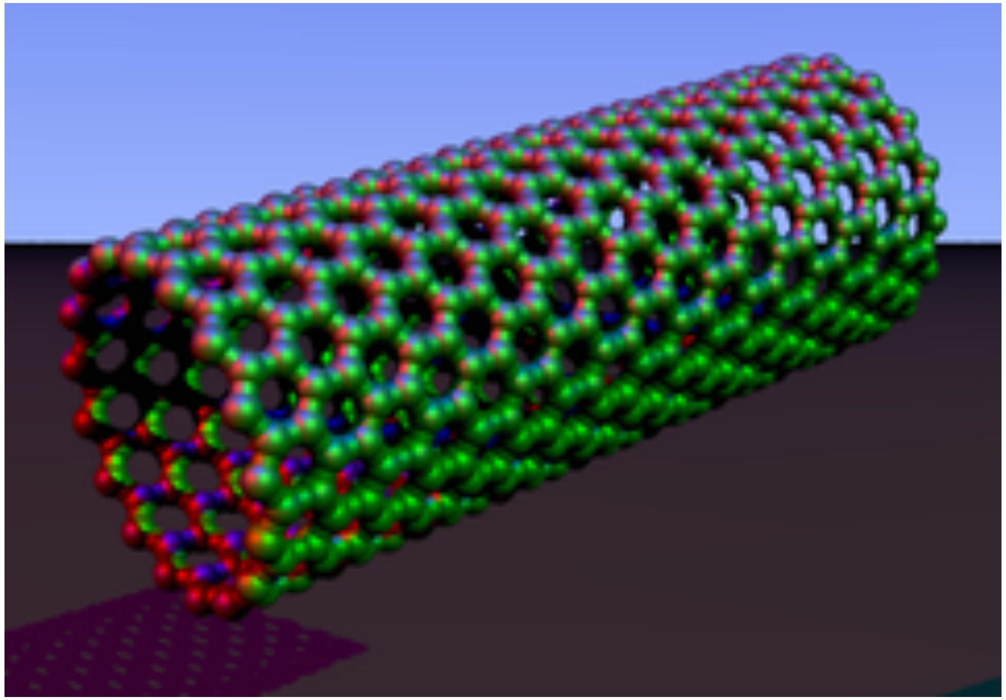
Nanotubes

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

Discovered 1991 (or earlier)

Single-walled (SWNT)

Multi-walled (MWNT)



Nanotubes – what are they useful for?

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

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

Strength – highest tensile strength of any material
- reinforcement in polymers
- bicycles
- wind turbines, marine paints, sports equipment

Hardness

Wettability

Kinetic properties – v. low friction

Electrical properties
- AFM tips
- transistors
- in Li-ion batteries

Optical properties

Thermal properties

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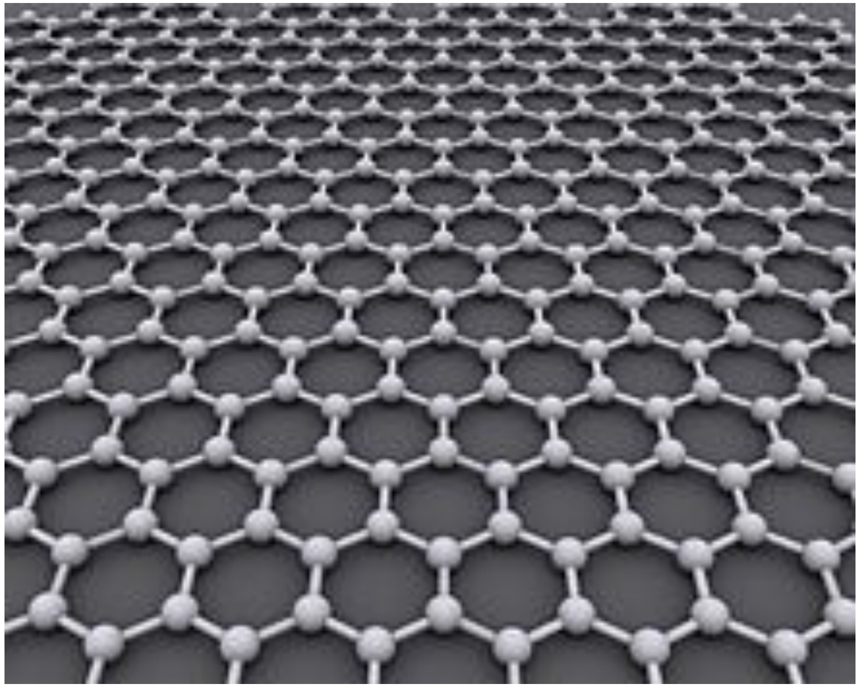
Example 1b – 2004
Nobel Prize in Physics 2010

Graphene

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

First patents 2002 & 2006
First bulk production – 2004 (Geim and Novoselov)
Lots of claims for applications (like nanotubes)
v. Interesting electronic properties



C_{60} is 0-D carbon
Nanotubes are 1-D carbon
Graphene is 2-D carbon
Graphite is 2.5D carbon
Diamond is 3-D carbon

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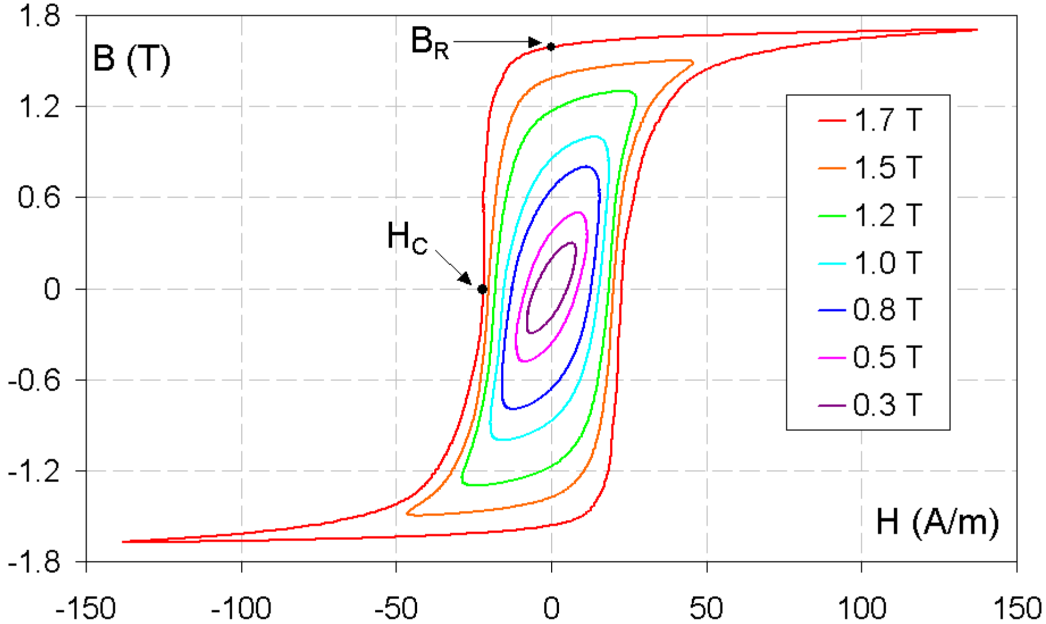
Example 2 – 1982

Hard magnets – permanent magnets

1982 – General Motors and Sumitomo rediscovered $Nd_2Fe_{14}B$



Largest "energy product" – area of hysteresis curve

$SmCo_5$ has larger coercivity, but is expensive

A factor in interest over supply of rare earths

Hard magnets – permanent magnets $Nd_2Fe_{14}B$

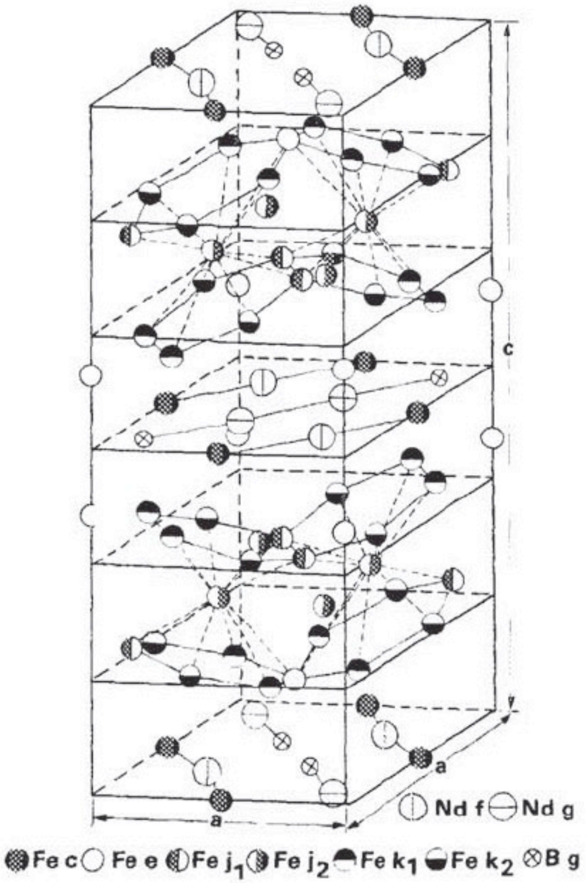
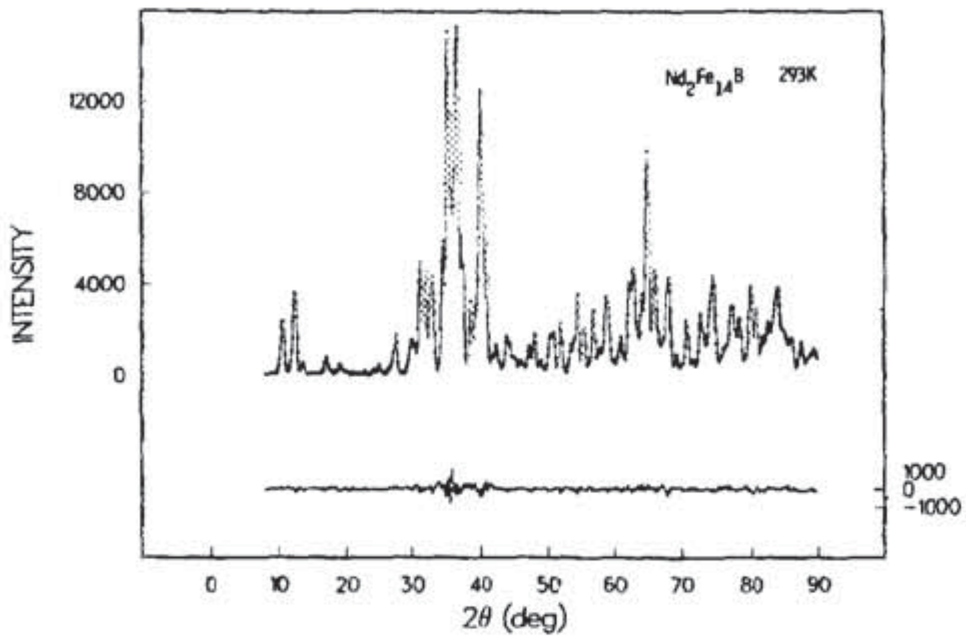


FIG. 1. Tetragonal unit cell of the $Nd_2Fe_{14}B$ structure.



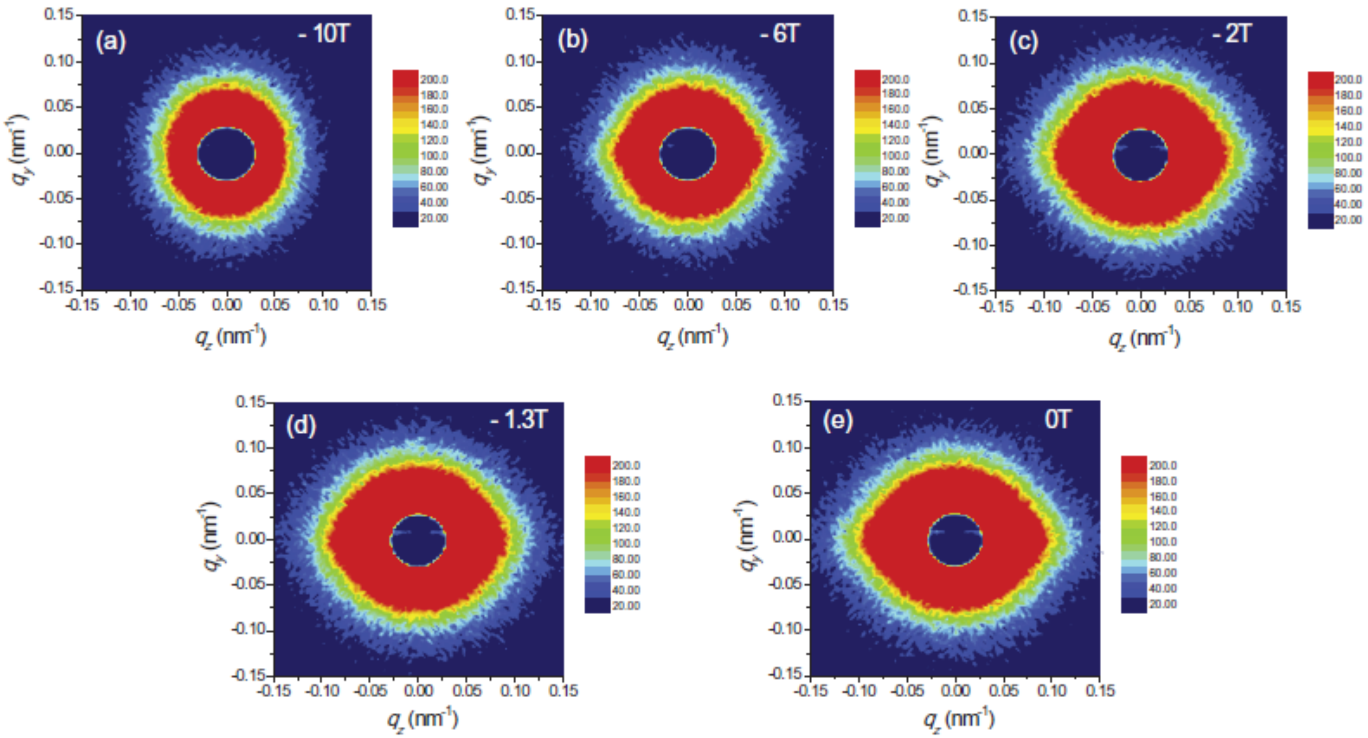
Neutron powder diffraction done at University of Missouri Research Reactor

J. F. Herbst, J. J. Croat, W. B. Yelon, *J. Appl. Phys*. **57**, 4086 (1985)

Hard magnets – permanent magnets $Nd_2Fe_{14}B$

Current work at OPAL – with U. of Luxemburg + Toyota and KEK + interest from elsewhere)

Small-angle neutron scattering, to understand synthesis and defects:



E.A. Perigo, E.P. Gilbert and A. Michels, *Acta Materialia* **87** (2015) 142–149

Hard Magnets – what are they useful for?

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

50,000 tons per year produced in China

Head actuators for computer hard disks
Erase heads for cheap cassette recorders
Magnetic resonance imaging (MRI)
Magnetic guitar pickups
Mechanical e-cigarette firing switches
Locks for doors
Loudspeakers and headphones
Magnetic bearings and couplings
Benchtop NMR spectrometers
Electric motors:
- Cordless tools
- Servomotors
- Lifting and compressor motors
- Synchronous motors
- Spindle and stepper motors
- Electrical power steering
- Drive motors for hybrid and electric vehicles. The electric motors of each Toyota Prius require 1 kilogram of neodymium.[13]
- Actuators

Electric generators for wind turbines (only those with permanent magnet excitation)
- direct-drive wind turbines require c. 600 kg of PM material per megawatt[18]
- turbines using gears require less PM material per megawatt

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Example 3 – 1991

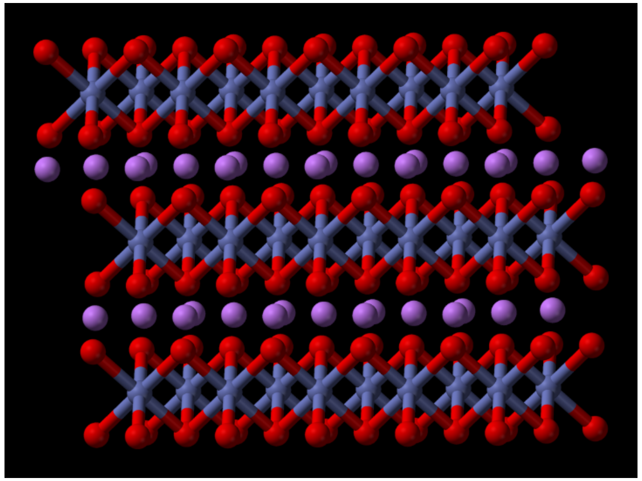
Batteries – an old problem

Batteries – an old problem

Lithium-ion batteries

https://en.wikipedia.org/wiki/Lithium-ion_battery

1991 – SONY and produced 1st commercial Li-ion battery

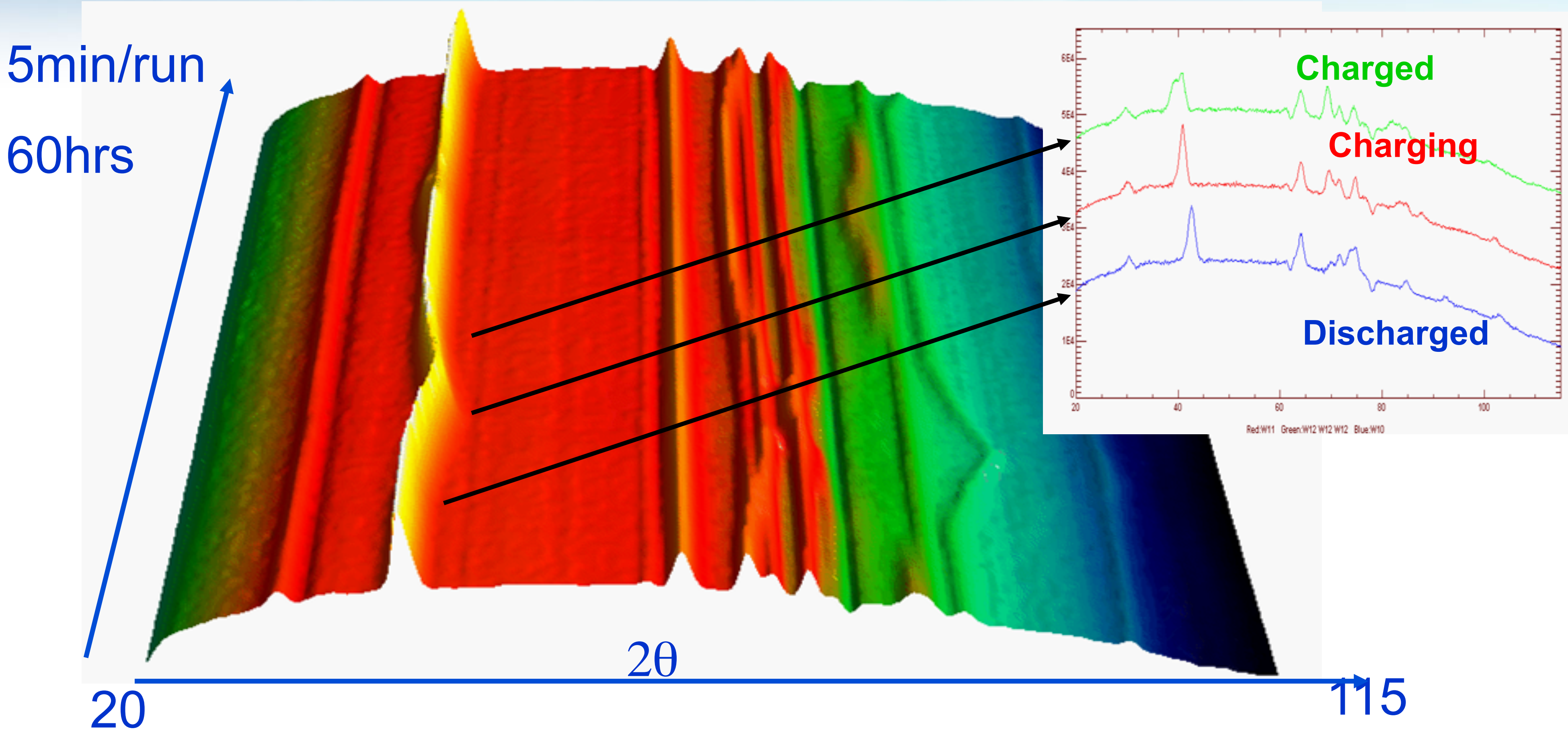


$LiCoO_2$

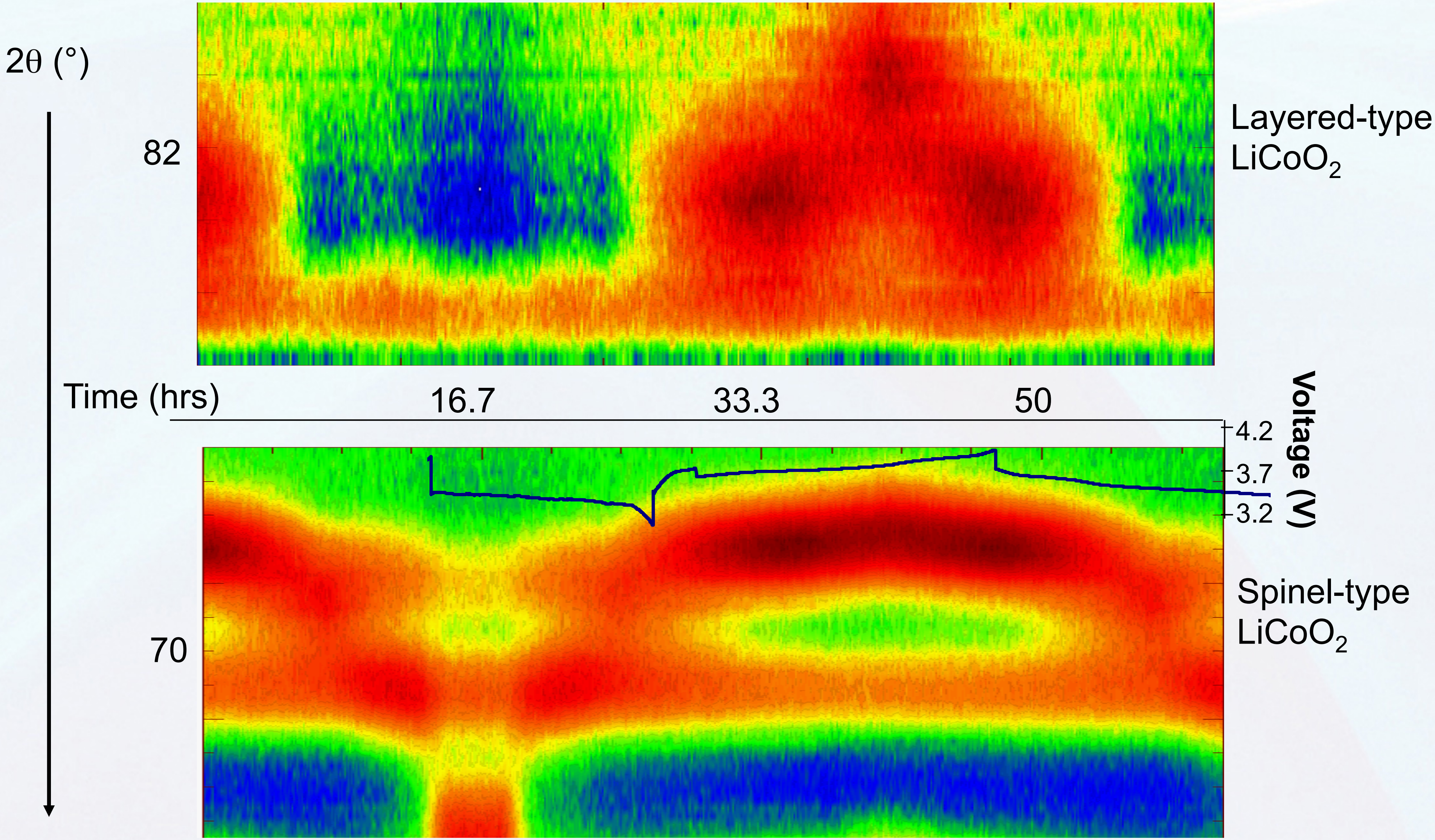
$LiFePO_4$

and other Li-compounds

Li-ion batteries on WOMBAT at OPAL

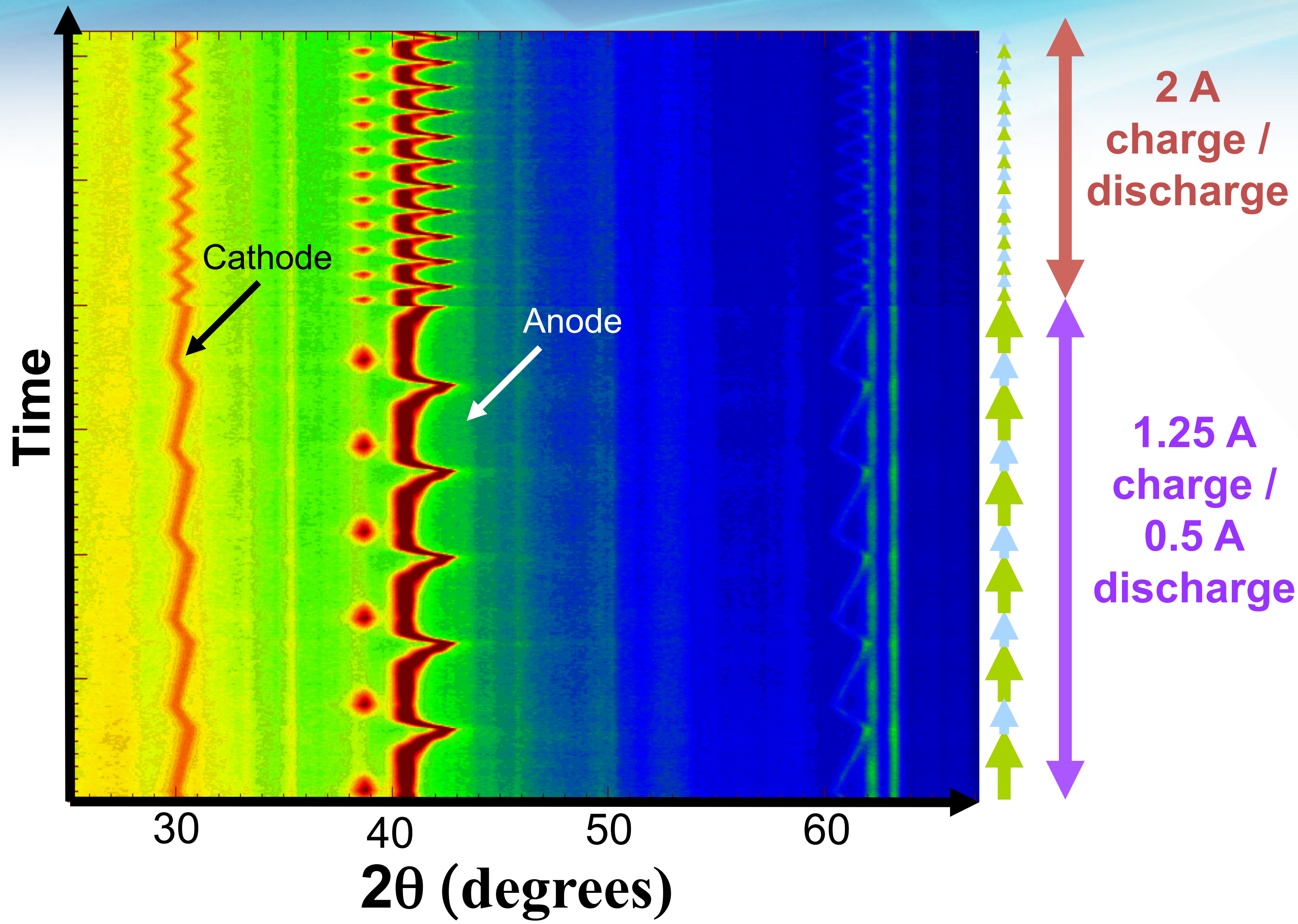


- *In-situ* cycling on Wombat
- Background reduction is essential to progressing this research: New cell designs

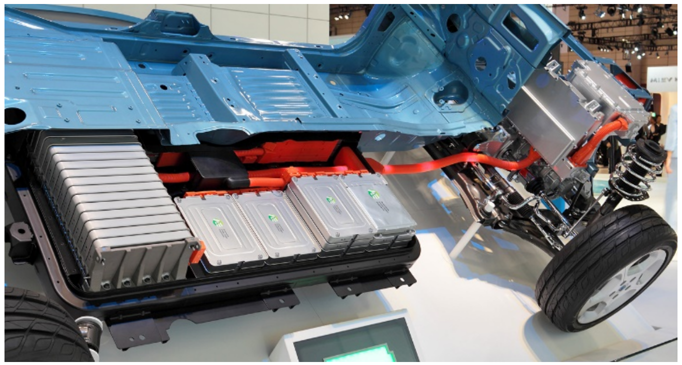


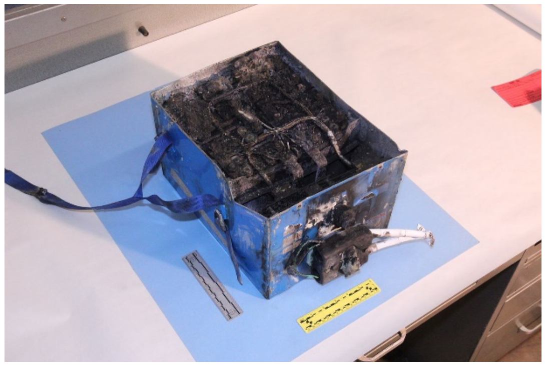
N. Sharma *at al.*.

Battery Research Scientific Highlight



Lithium-ion batteries
– what are they useful for?





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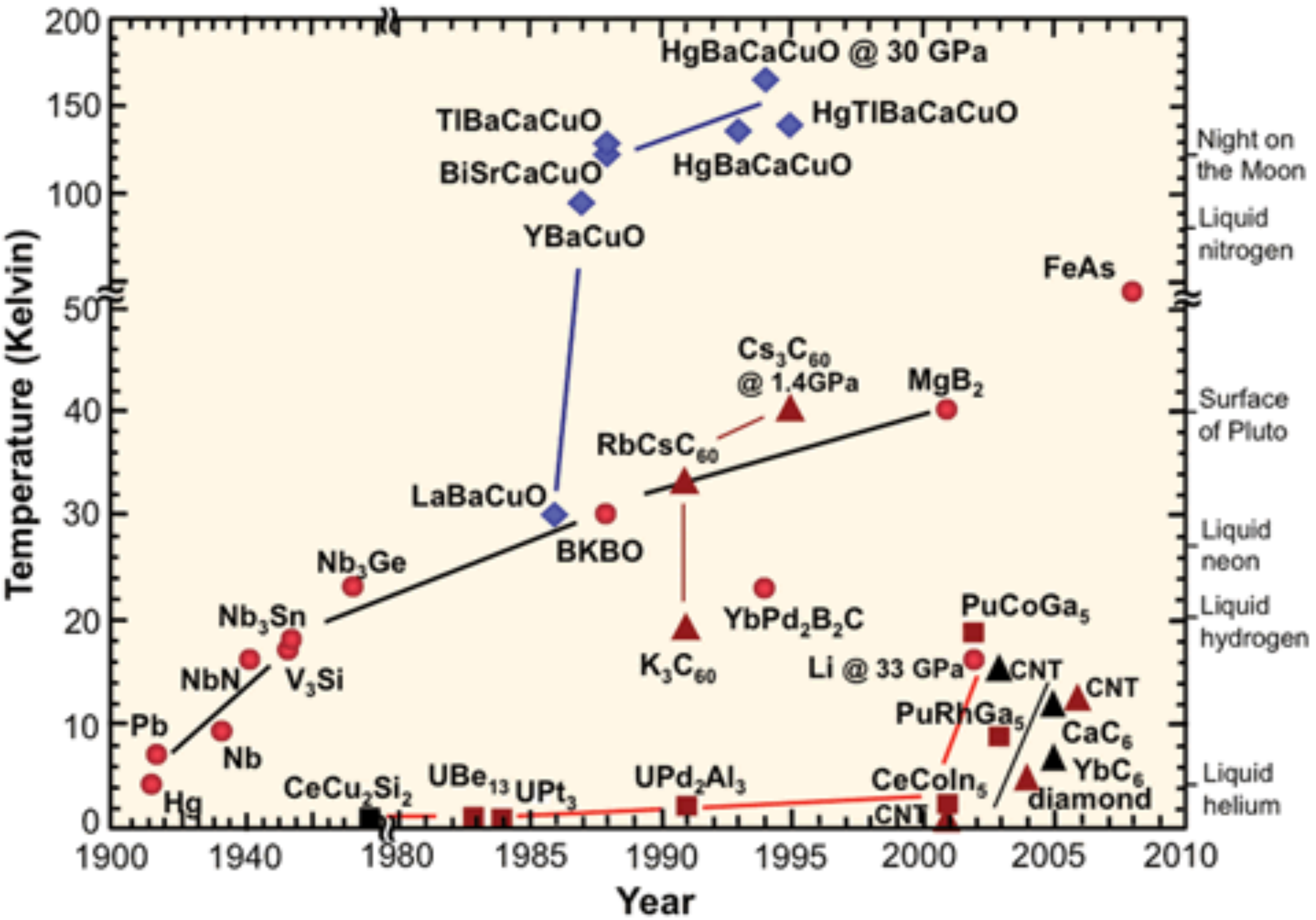
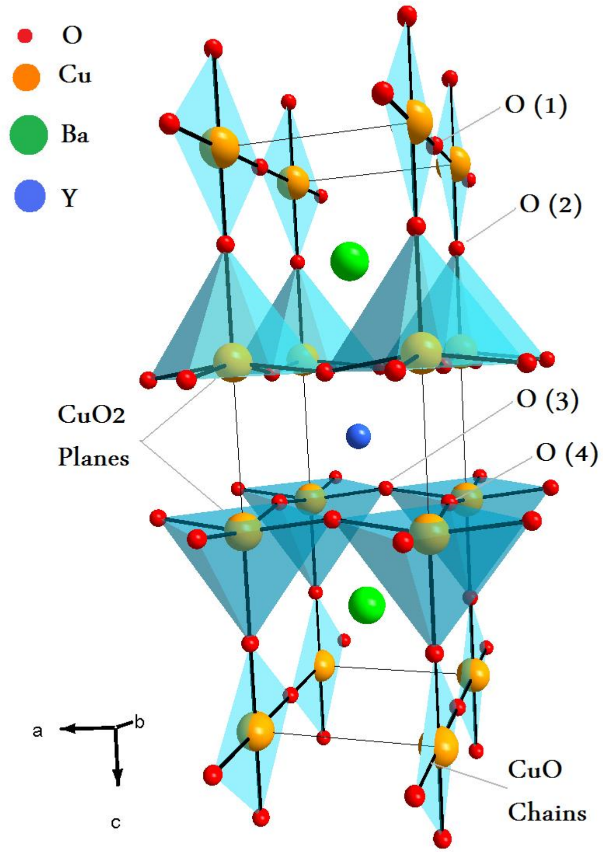
Example 4a – 1986
Nobel Prize in Physics 1987

High-Tc Superconductors https://en.wikipedia.org/wiki/High-temperature_Superconductivity

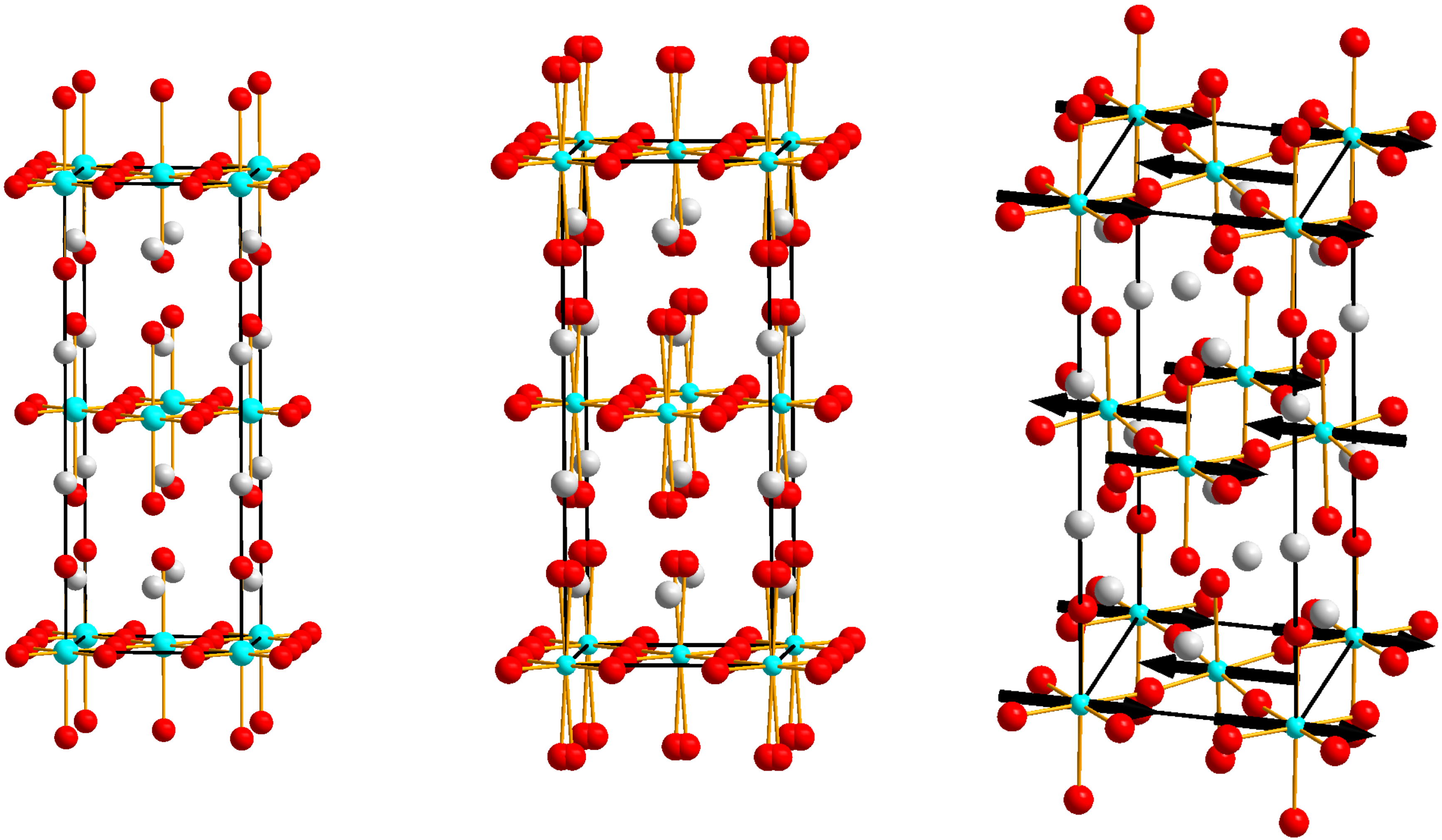
1986 – discovered by IBM in Zurich





Neutron Powder Diffraction & High-Tc Superconductors

Basic structure | oxygen positions | magnetism



What did neutrons contribute?

Structural properties of oxygen-deficient $YBa_2Cu_3O_{7-\delta}$

J. D. Jorgensen, B. W. Veal, A. P. Paulikas, L. J. Nowicki, G. W. Crabtree,
H. Claus,* and W. K. Kwok[†]
Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439
(Received 17 July 1989; revised manuscript received 25 September 1989)

The structural properties of oxygen-deficient $YBa_2Cu_3O_{7-\delta}$ have been determined by neutron powder diffraction for $0.07 < \delta < 0.91$. The samples were produced by quenching into liquid nitrogen from controlled oxygen partial pressures at 520 °C, and they exhibit a clearly defined "plateau" behavior of T_c versus δ. Superconductivity disappears at the orthorhombic-to-tetragonal transition that occurs near $\delta = 0.65$. Structural parameters, including the copper-oxygen bond lengths, vary smoothly with δ within each phase but exhibit different behavior in the superconducting and nonsuperconducting phases. These observations are consistent with a model in which superconducting behavior is controlled by charge transfer between the conducting two-dimensional CuO_2 planes and the CuO_x chains, which act as reservoirs of charge.

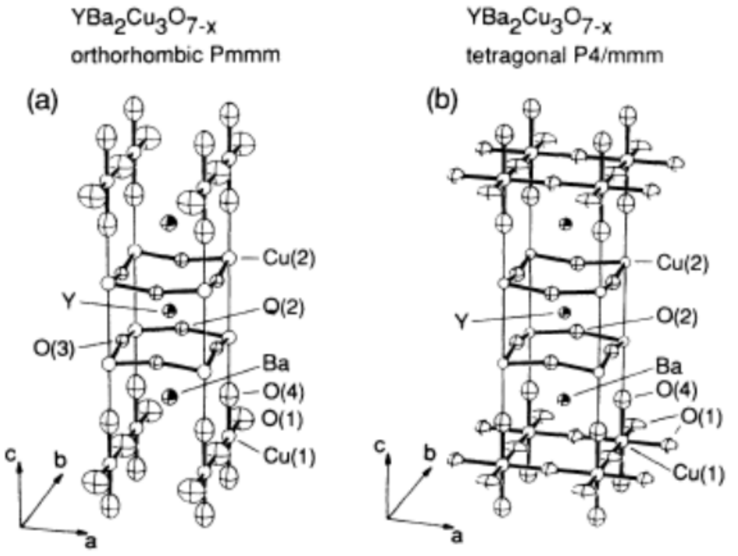
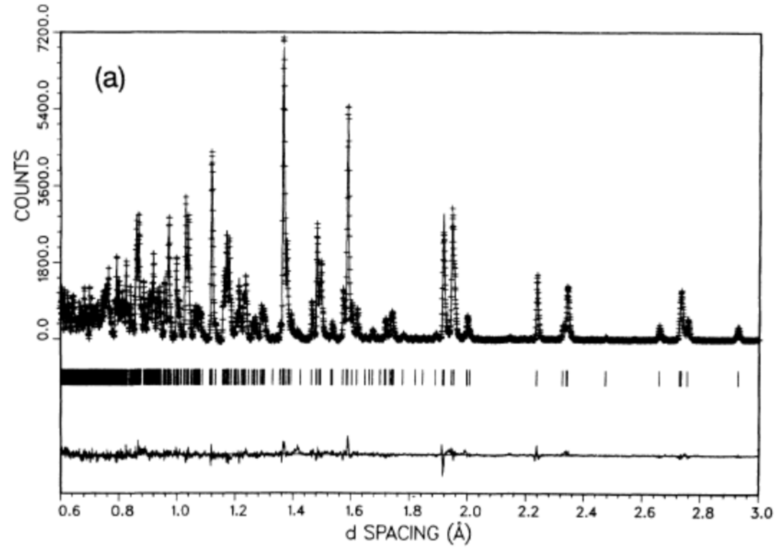
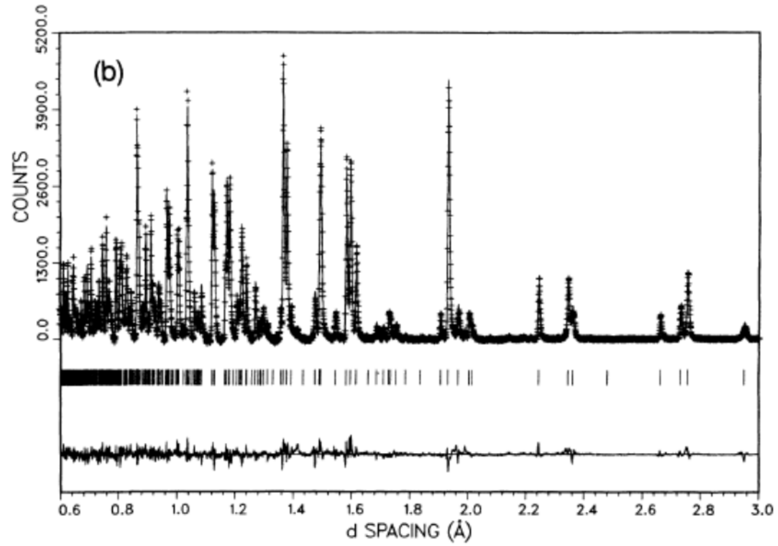


FIG. 1. (a) Orthorhombic and (b) tetragonal structures of $YBa_2Cu_3O_{7-\delta}$. In the tetragonal structure (b) the different atom symbol for the O(1) site is used to indicate that this site is not fully occupied.





What did neutrons contribute?

Antiferromagnetism in La_2CuO_{4-y}

D. Vaknin,[a] S. K. Sinha, D. E. Moncton, D. C. Johnston, J. M. Newsam,
C. R. Safinya, and H. E. King, Jr.
Corporate Research Laboratories, Exxon Research and Engineering Company, Annandale, New Jersey 0880l
(Received 4 May 1987)

Powder neutron diffraction studies of undoped La_2CuO_{4-y} have revealed new superlattice peaks below $\simeq$220 K. The absence of corresponding x-ray superlattice lines and an observed susceptibility anomaly near 220 K suggest the occurrence of antiferromagnetism. From the magnetic peak intensities we deduce a structure consisting of ferromagnetic sheets of Cu spins alternating along the [100] orthorhombic axis, with the spins aligned along the [001] orthorhombic axis. The low-temperature magnetic moment is approximately $0.5\mu_B$/Cu-atom. The tetragonal-orthorhombic transition at 505 K has also been studied.

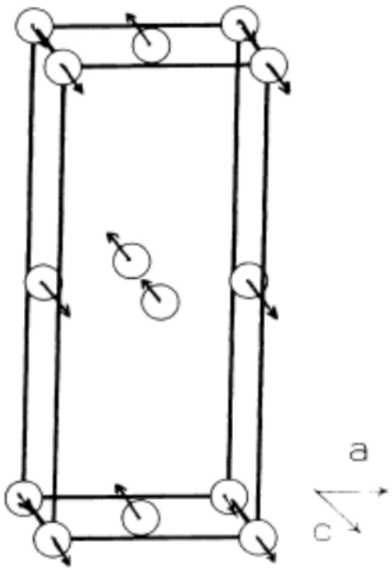


FIG. 3. Proposed spin structure of antiferromagnetic La_2CuO_{4-y}. Only copper sites in the orthorhombic unit cell are shown for clarity.

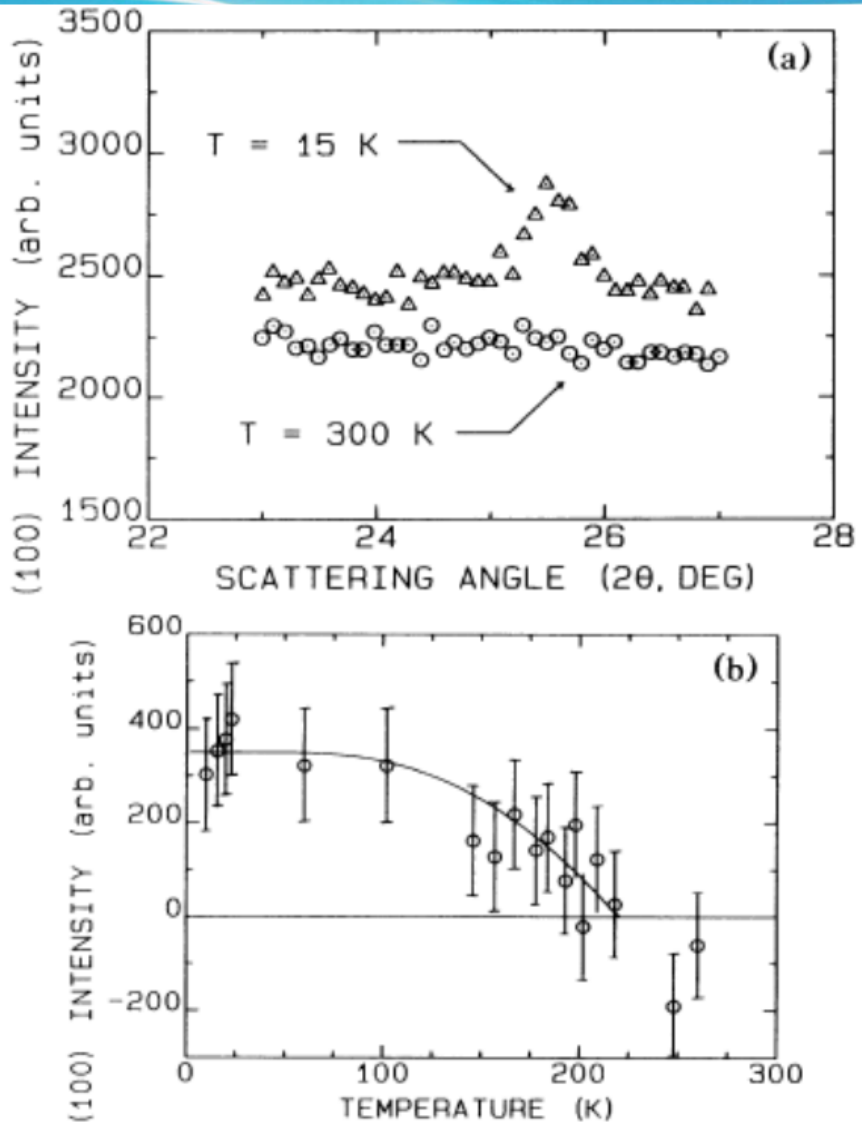
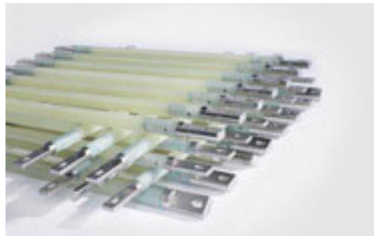


FIG. 2. (a) Intensity vs scattering angle 2θ for neutron powder scans of the (100) peak region at 15 K and at room temperature. (b) (100) peak intensity vs temperature. The line is a spin-$\frac{1}{2}$ magnetization curve for $T_N = 220$ K, calculated from molecular-field theory.

What are superconductors good for?

Magnets
- MRI machines
- Magnets and RF cavities in accelerators
- Insertion devices in synchrotrons

Conductors (transmission lines) and current leads



Fault-current interruptors

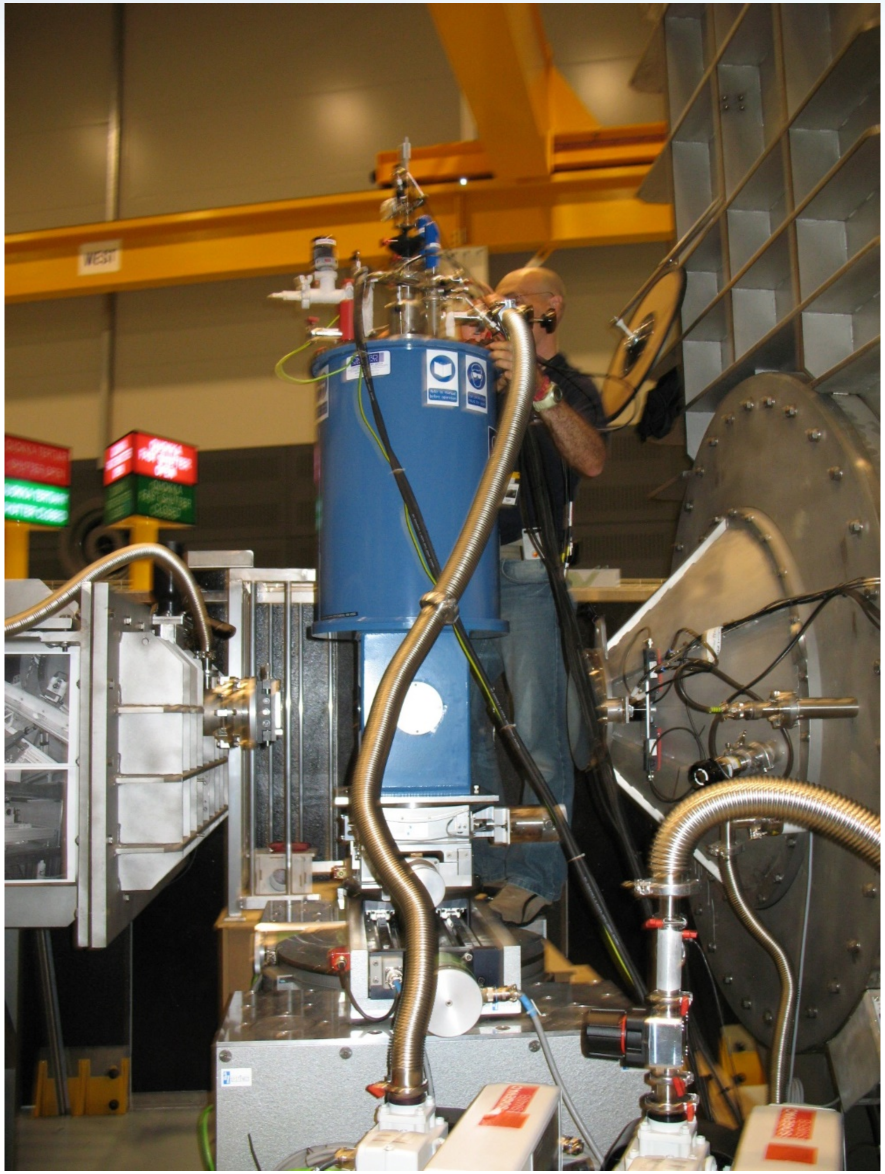
Sensors

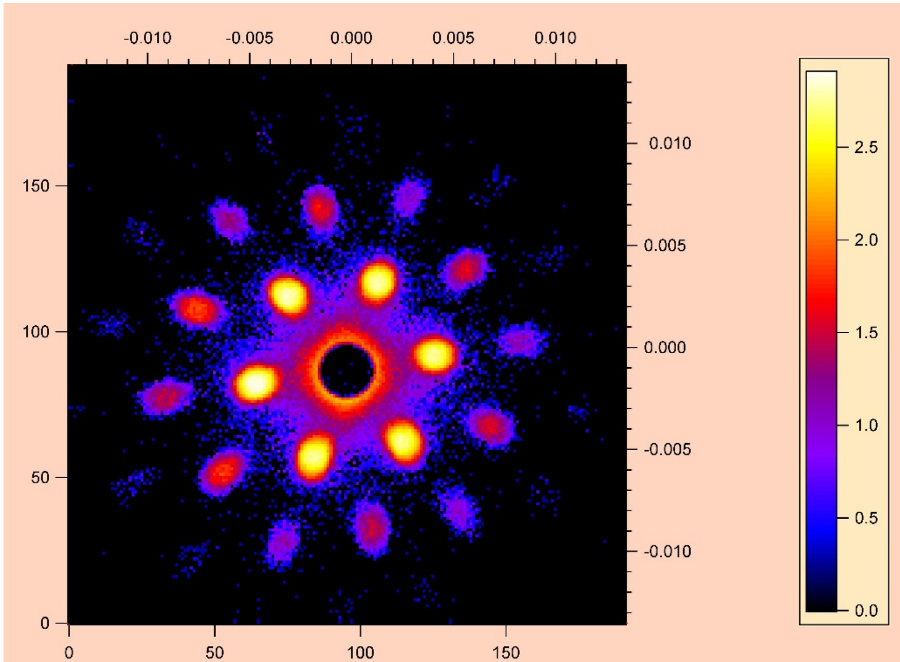
Logic elements in computers (Josephson junctions)

To date, High-Tc has mostly been used in research applications

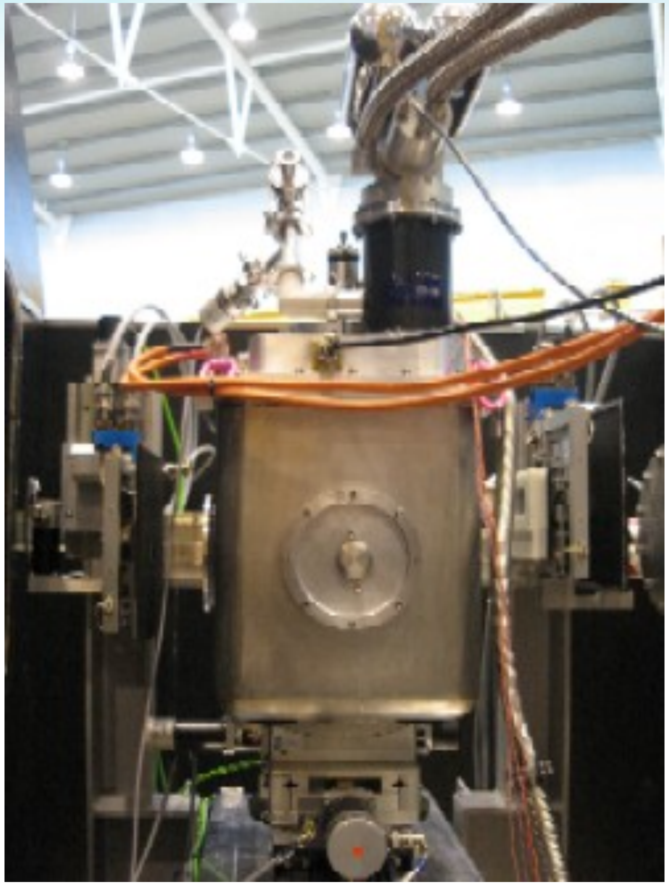
Other superconducting devices are typically Nb-Ti or Nb_3Sn

Some superconducting magnets at OPAL





More superconducting magnets at OPAL



All high-Tc magnet from HTS-110 in New Zealand

HTS-110

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Example 4b – 2006

Fe-based superconductors

https://en.wikipedia.org/wiki/Iron-based_superconductor

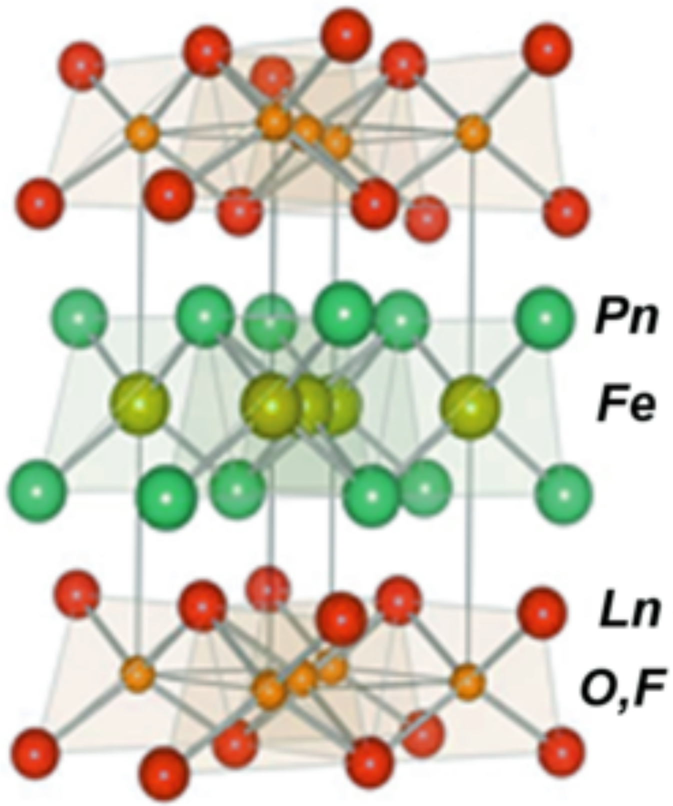
Discovered 2006

Also known as *ferropnictides*

Race to discover new materials:

- synthesise
- Grow crystals
- Bulk measurements
- Structures, Phase Diagrams, Magnetism

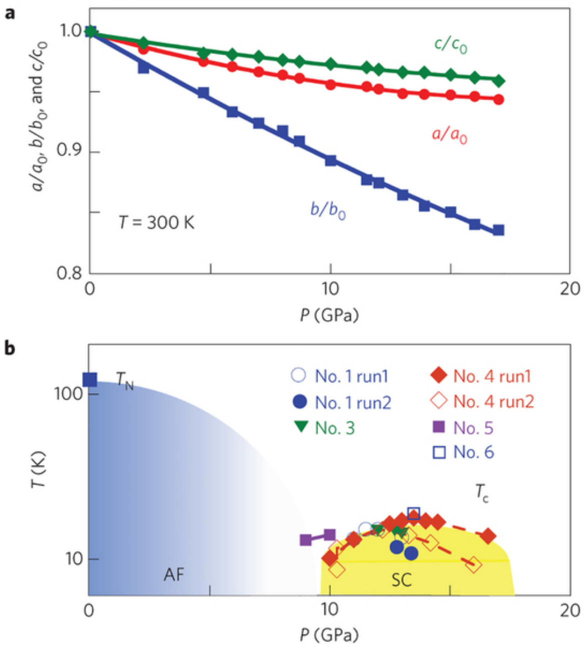
Lots of neutron characterisation done at the NIST Reactor (USA)

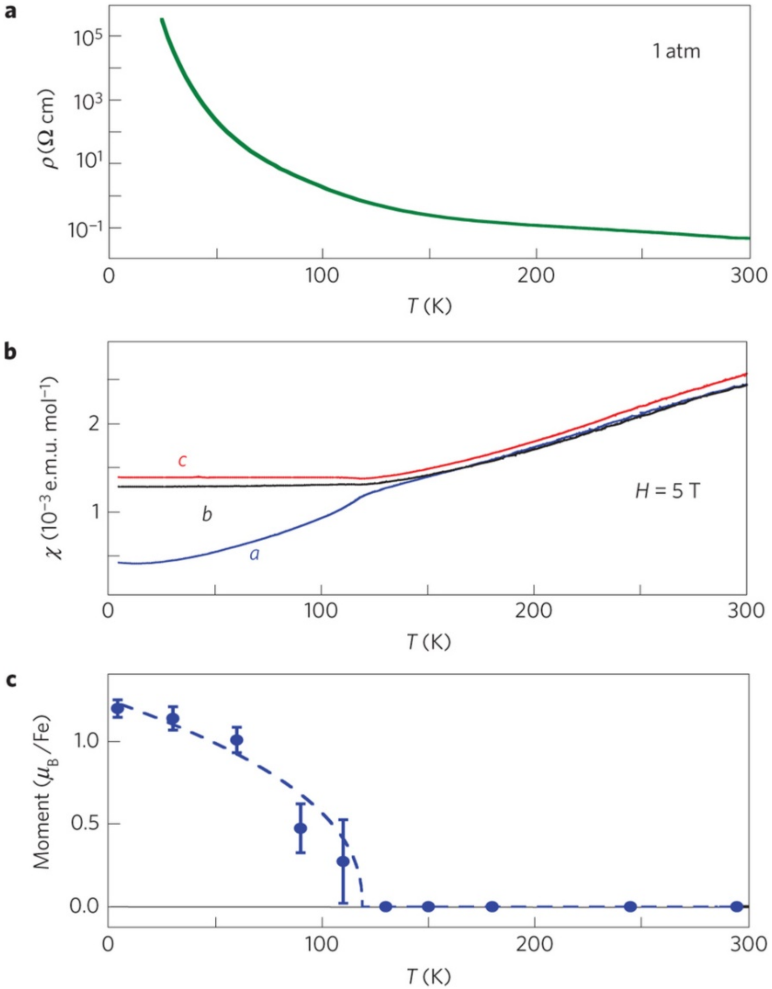


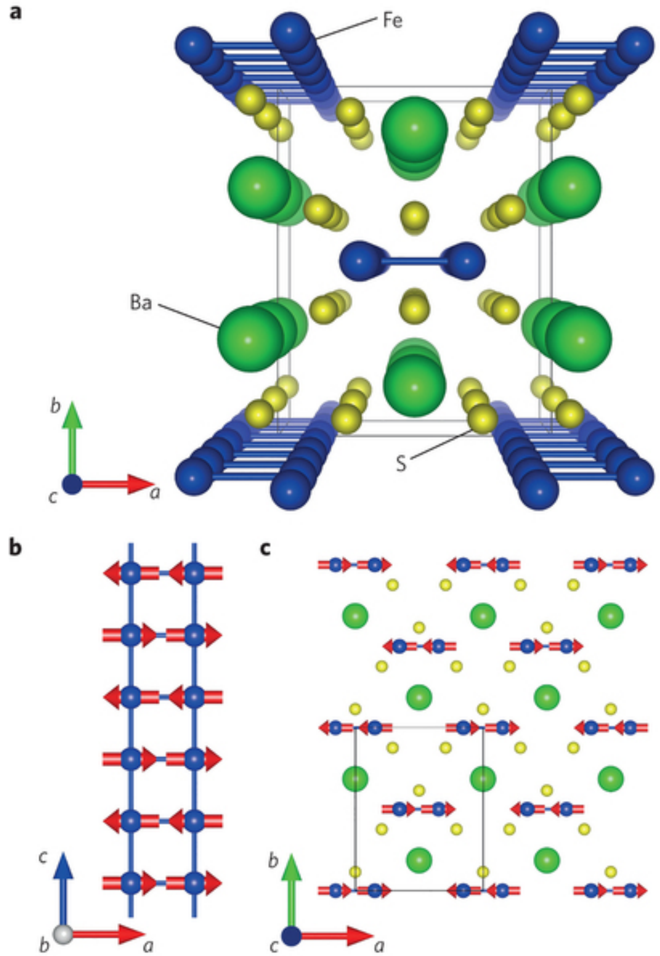
RFeAsOF

Something recent from OPAL –

Superconducting ladders in Fe superconductors







H. Takahashi *et al*. *Nature Materials* (2015)

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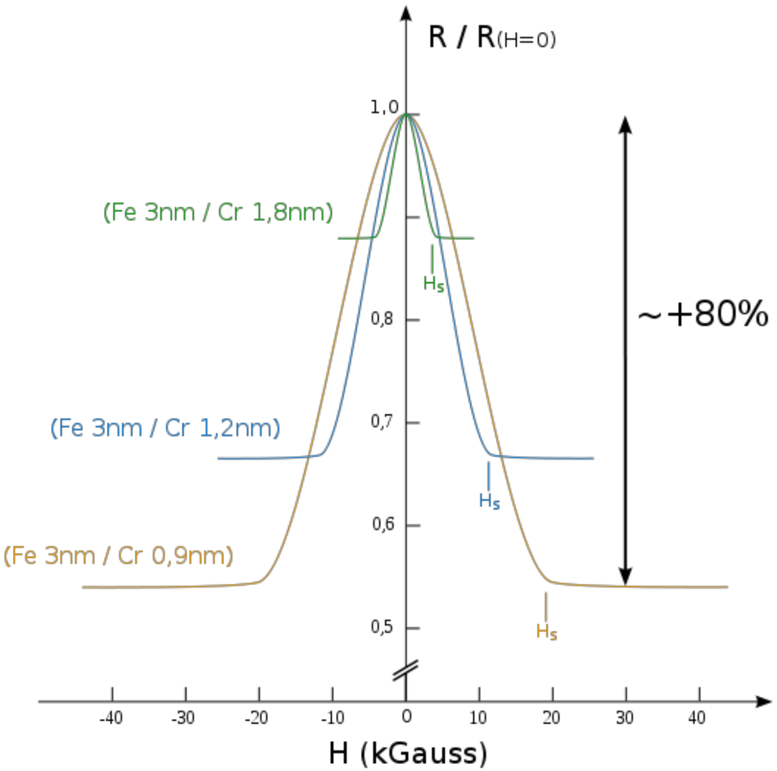
Example 5 – 1988
Nobel Prize in Physics 2007

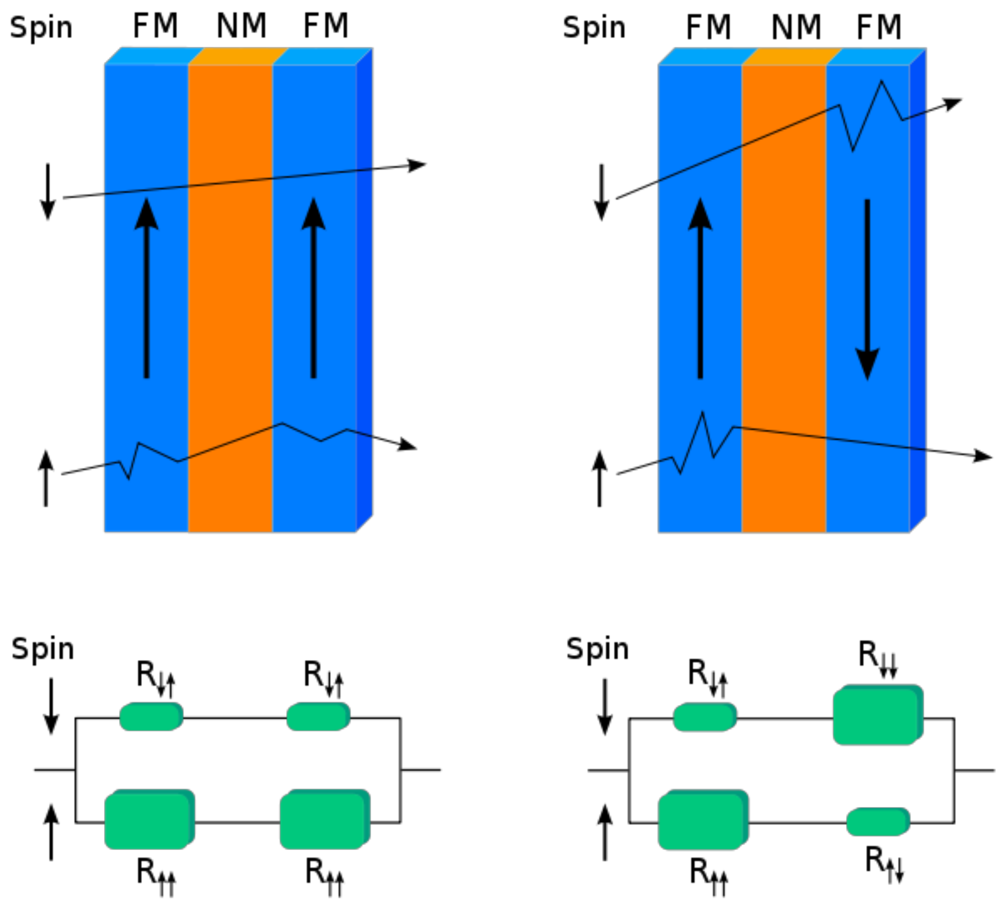
Giant Magnetoresistance

https://en.wikipedia.org/w/index.php?title=Giant_magnetoresistance&redirect=no

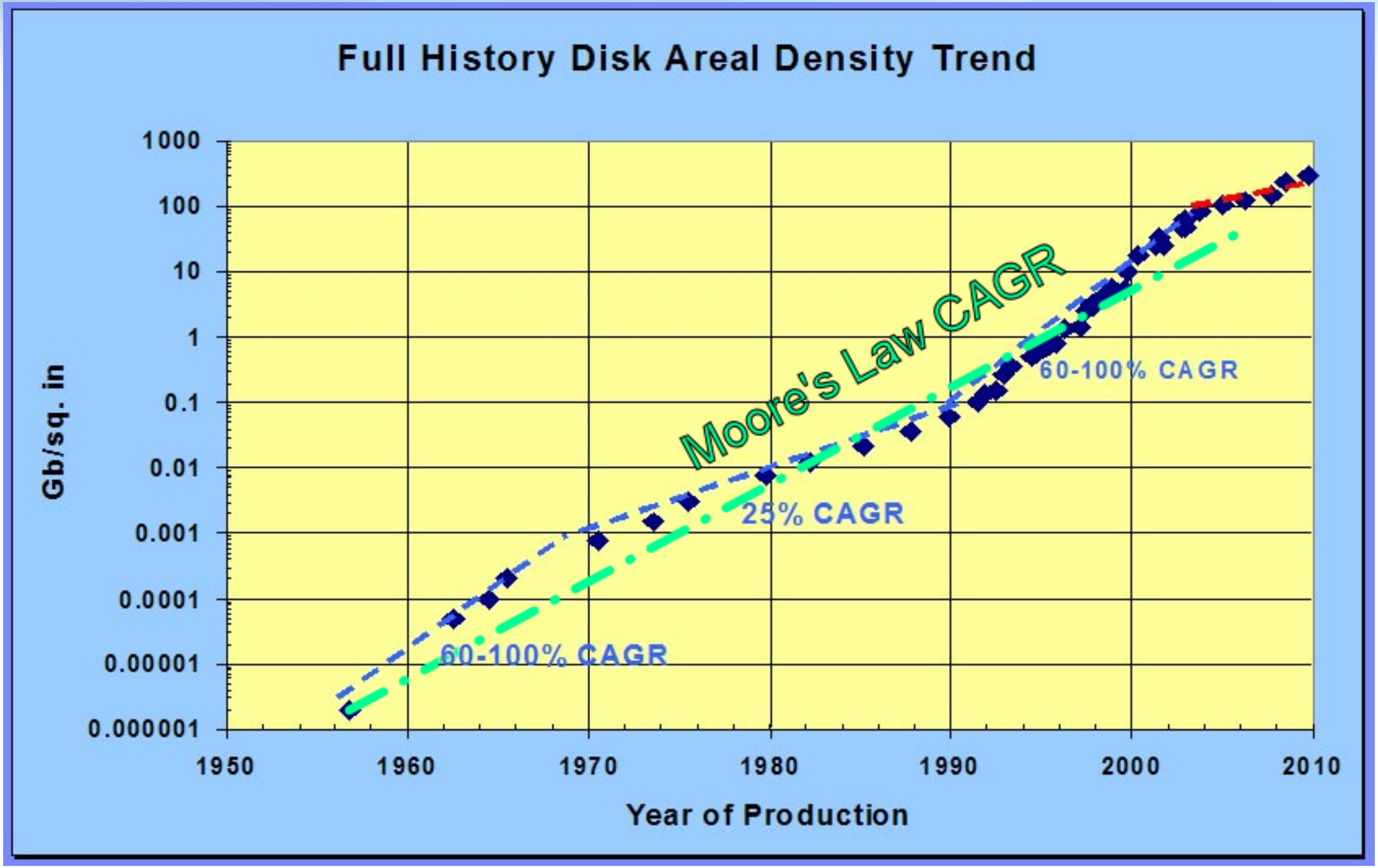
Discovered 1988 by Fert & Grunberg

In Fe/Cr multilayers





Kryder's Law



Full History Disk Areal Density Trend
1000
100
10
1
0.1
0.01
0.001
0.0001
0.00001
0.000001
Gb/sq. in
1950
1960
1970
1980
1990
2000
2010
Year of Production
Moore's Law CAGR
60-100% CAGR
25% CAGR
60-100% CAGR

What is GMR useful for?

In 2013 - $32B in sales of hard disks

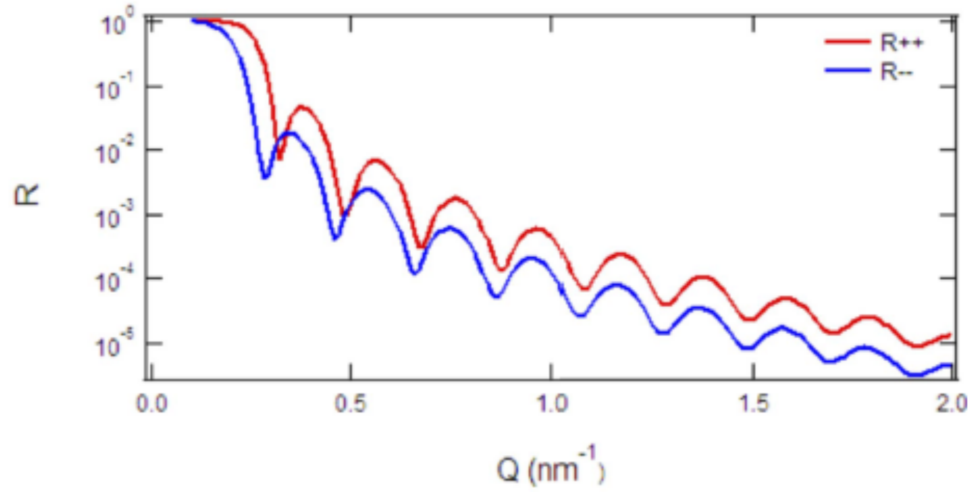
10 years from materials discovery to $1B business

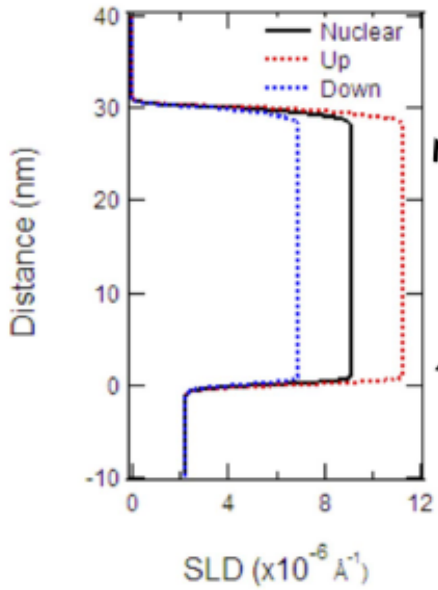
Stuart Parkin (IBM) realised that GMR was useful to IBM for hard-disk read-heads

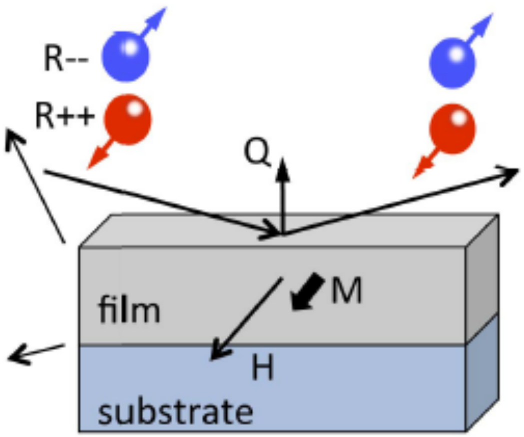
Tunneling magnetoresistance (TMR) has now superceded GMR

What can neutrons offer to this field?

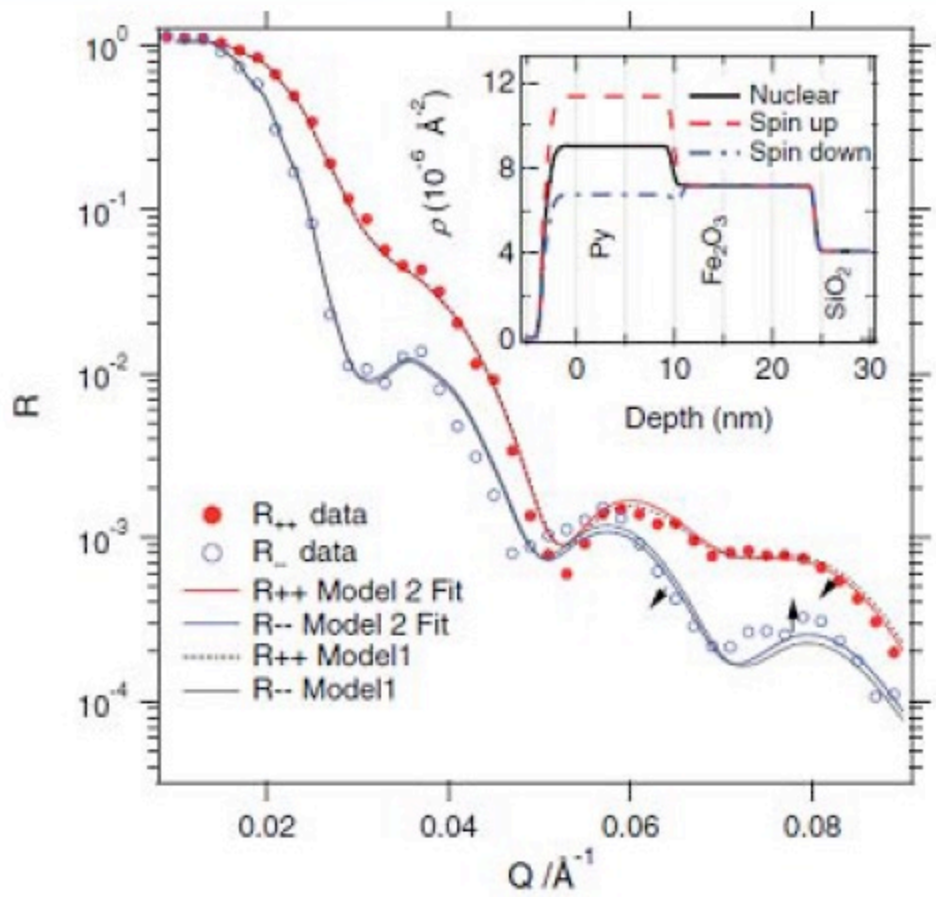
S. J. Callori and F. Klose, *IEEE Trans Magnetics* **50**, 6400107(2014)





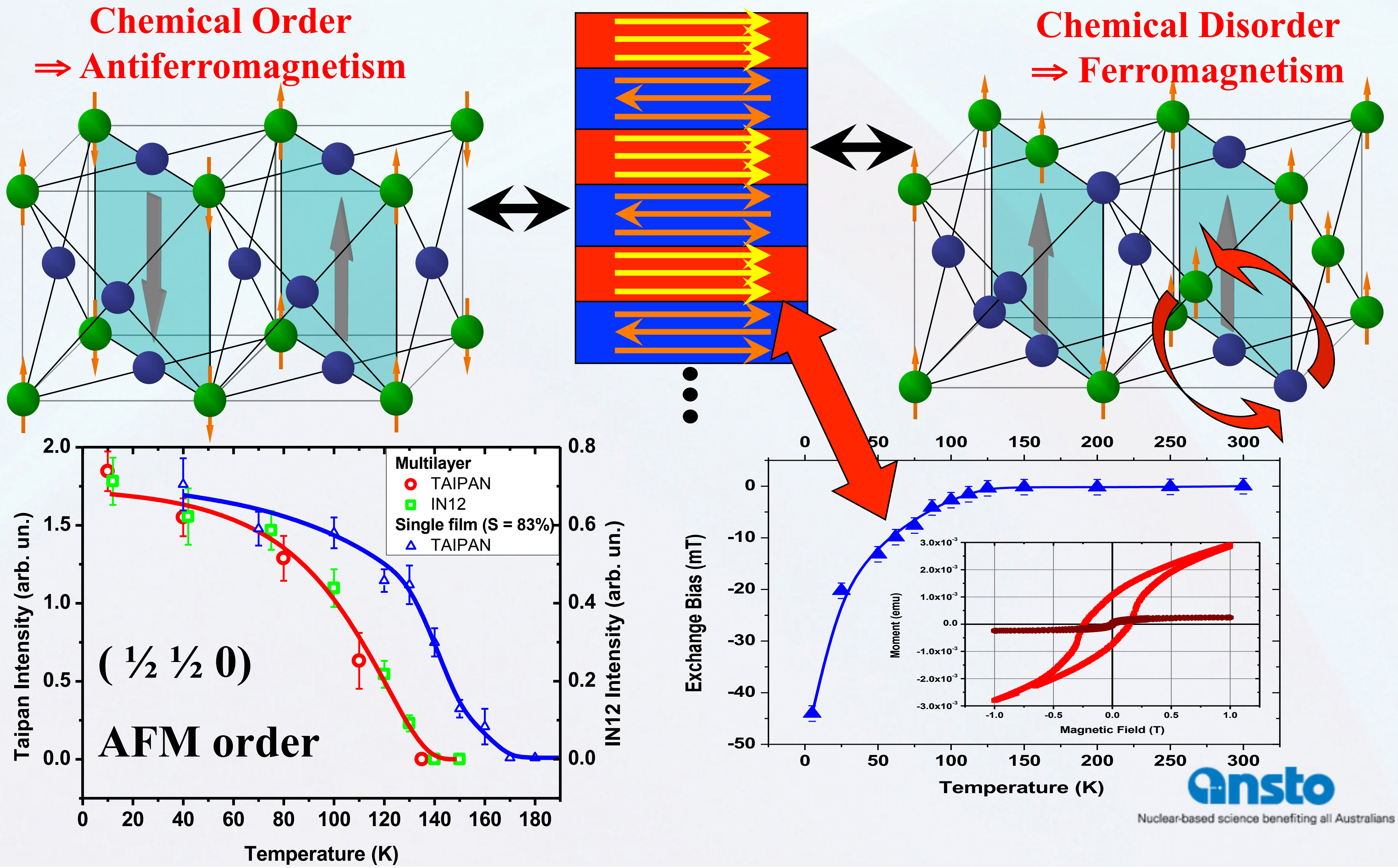


30-nm magnetic film



Permalloy on hematite bi-layer

$FePt_3$ multilayer: AFM/FM interfaces from chemical order modulation



Nuclear-based science benefiting all Australians

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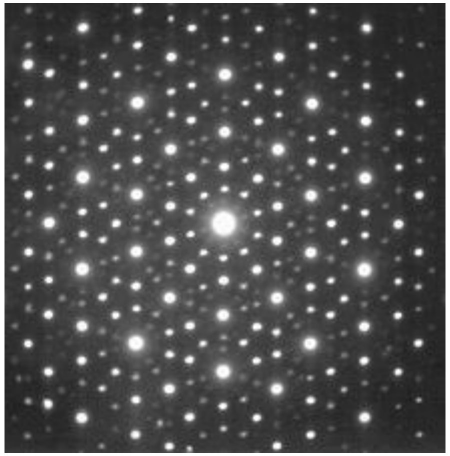
Example 6 – 1982
Nobel Prize in Chemistry 2011

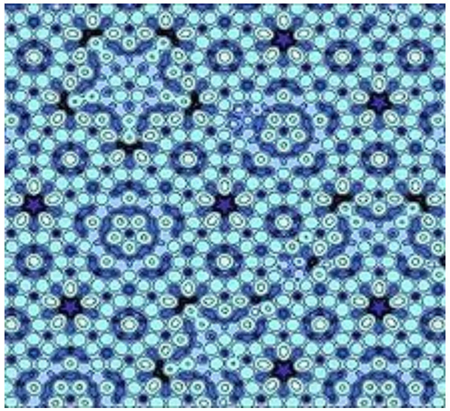
Can one have tiles made of pentagons?

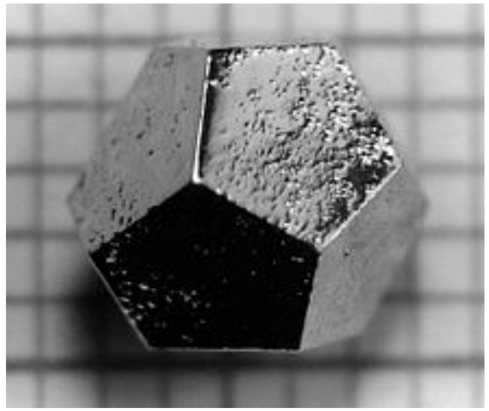
No – according to normal crystallography

But – in 1982 electron-diffraction patterns with 5-fold symmetry were recorded in Al-Mn alloys



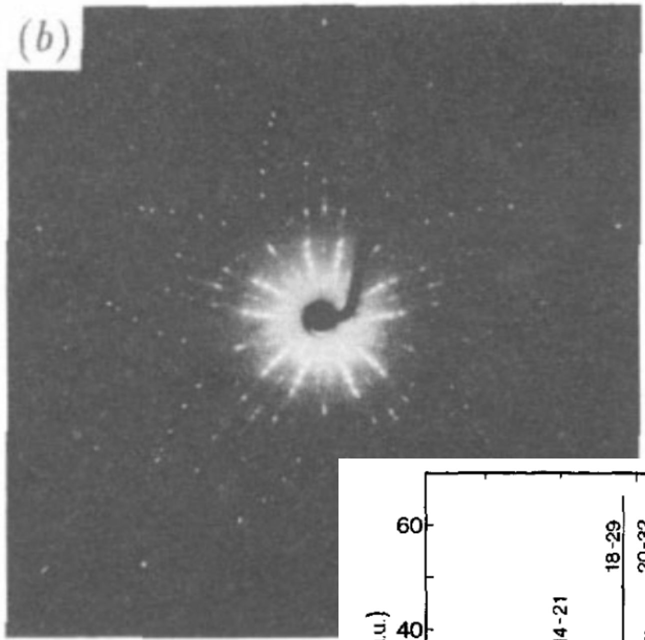
Dan Schechtman





Neutron & X-ray Diffraction from Quasicrystals

J. Phys.: Condens. Matter 4, 10149 (1992).



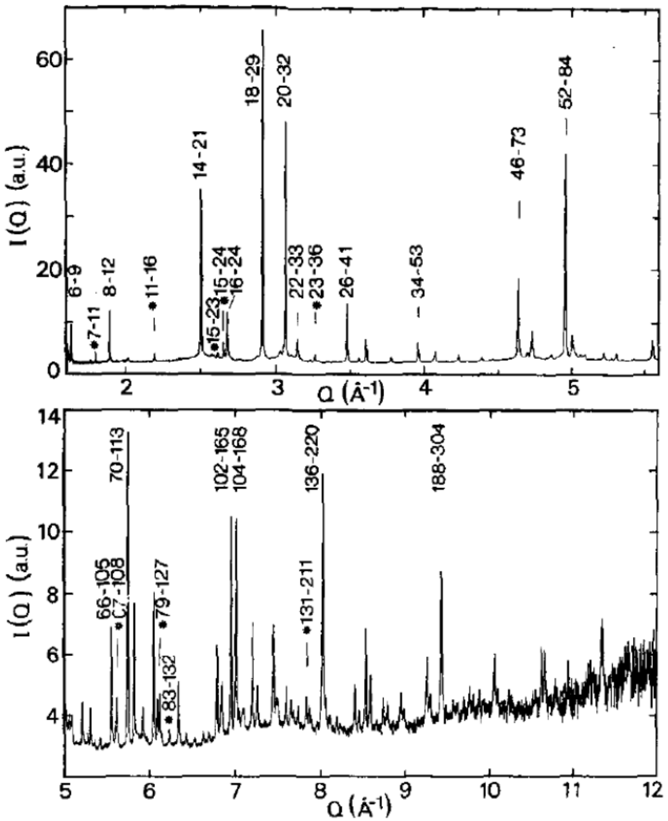
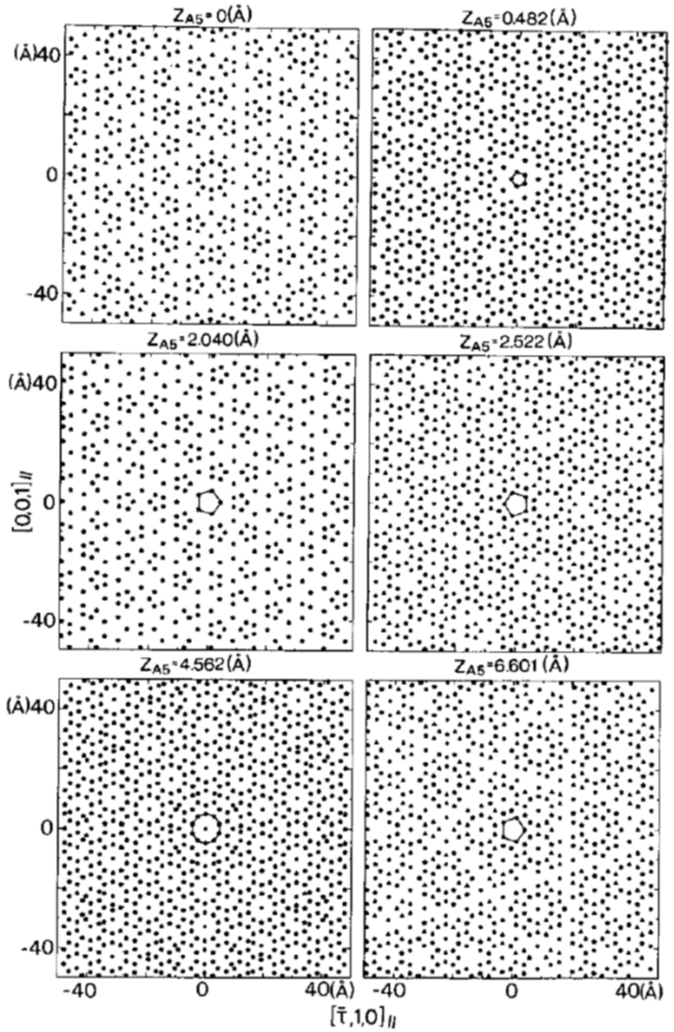


Figure 3. High-resolution powder diffraction pattern. Main Bragg reflections are indexed following the indexing scheme proposed by Cahn *et al* [33].



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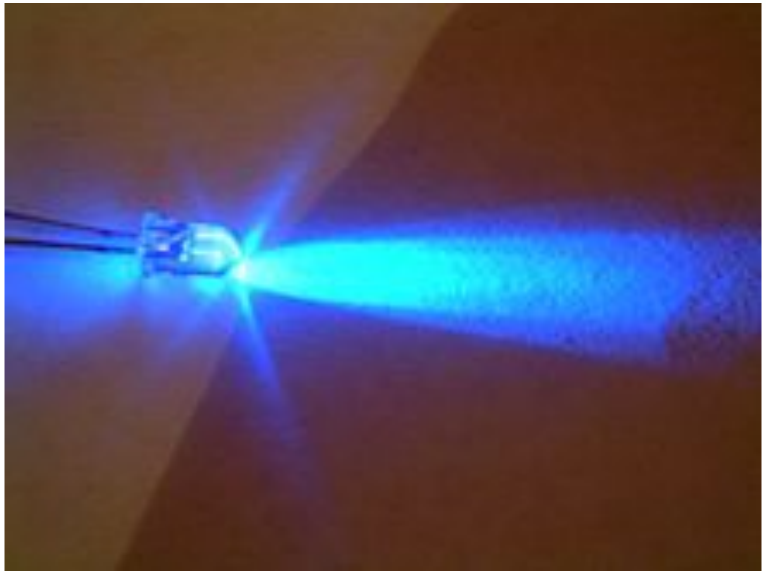
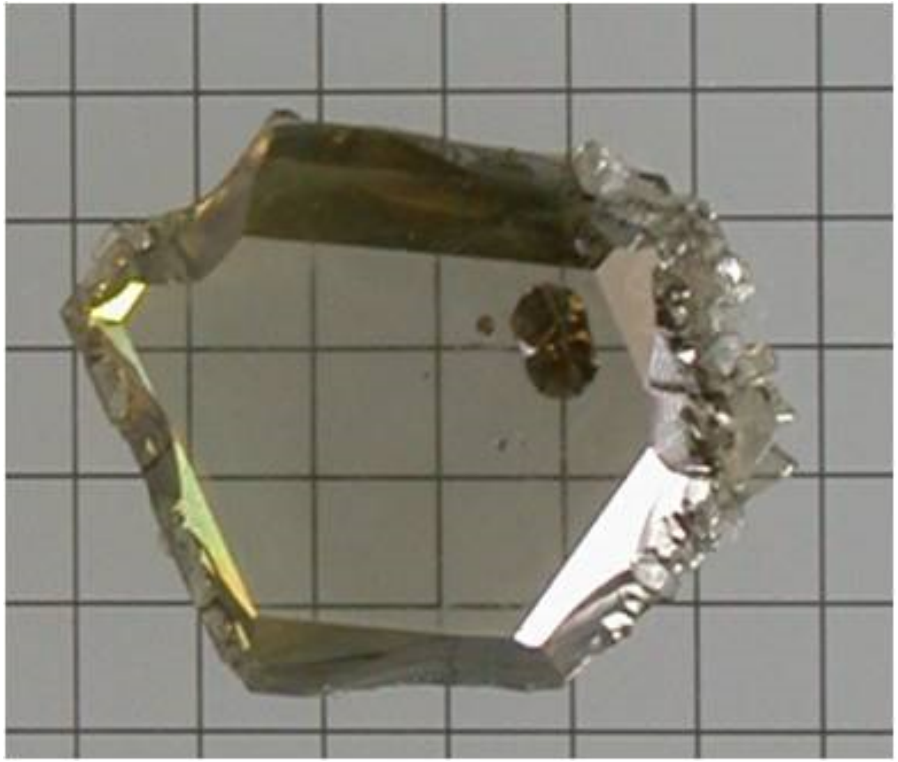
Example 7 – 1994 Nobel Prize in Physics 2014

Blue LEDs (1994)

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

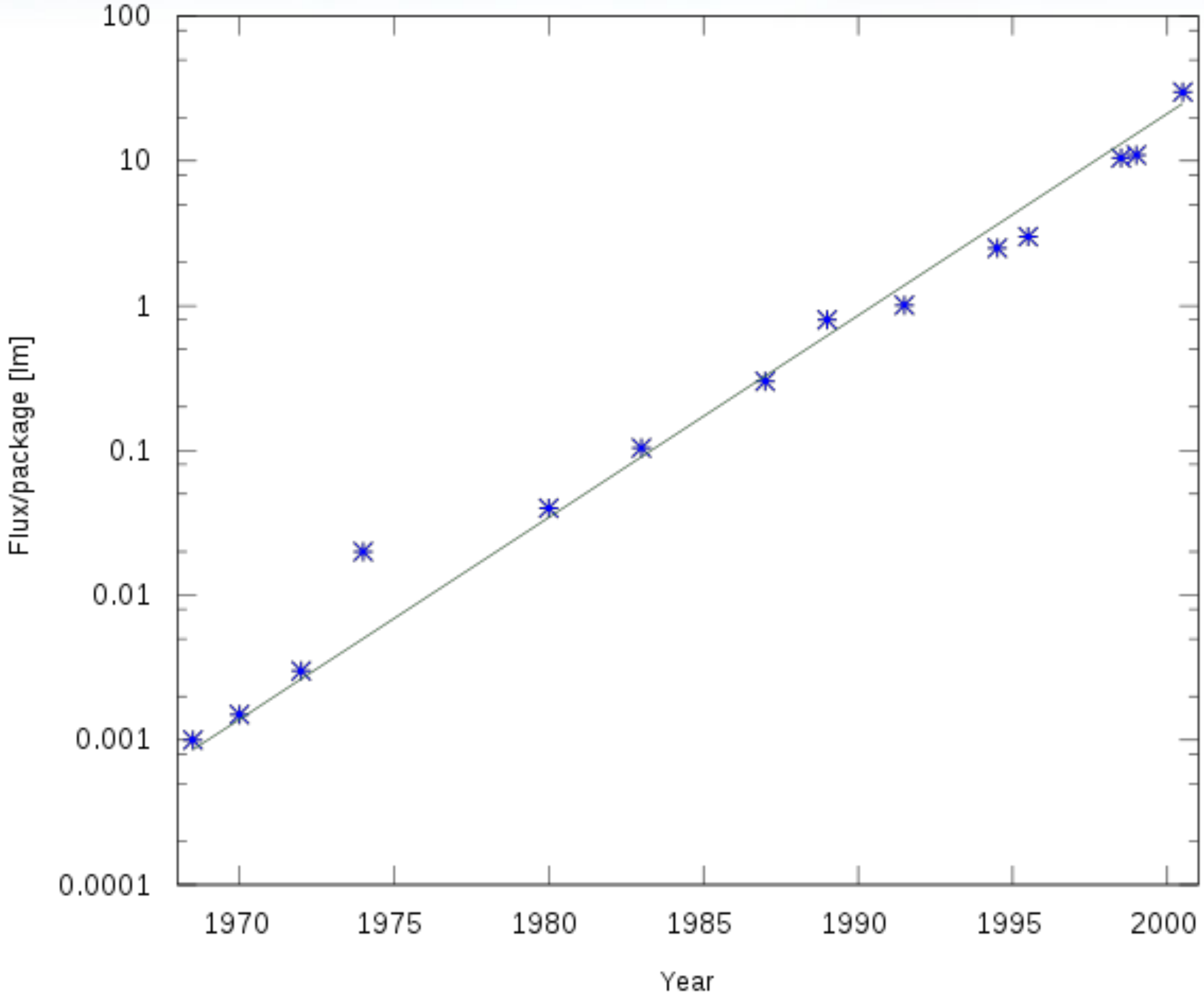




Displacing Edison incandescent bulbs and fluorescent lamps

Huge energy savings

Haitz's Law:
Light Output per LED



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Summary

Nobel Prizes

2014 – Physics: Blue LEDs

2011 – Chemistry: Quasicrystals

2010 – Physics: Graphene

2007 – Physics: Giant Magnetoresistance

2000 – Chemistry: Conducting Polymers

1996 – Chemistry: Fullerenes (C_{60})

1994 – Physics: *Neutron Scattering*

1987 – Physics: High-T_c Superconductors

Other exciting new materials

Bulk Metallic Glasses (1990s)

Metal-Organic Frameworks (for gas storage)

Carbon nanotubes (1991)

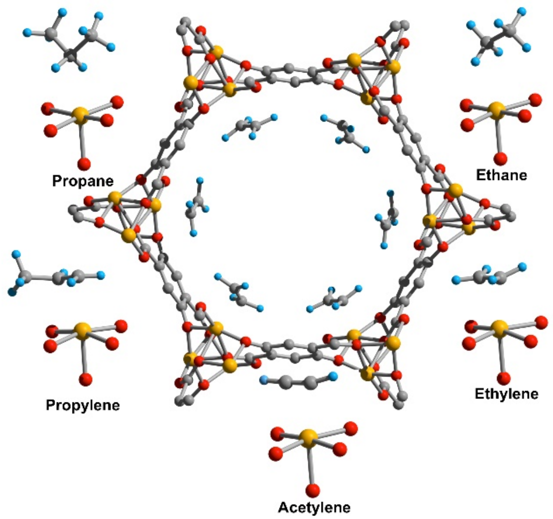
Magneto-caloric materials

Diamond-like carbon

Other unconventional superconductors

MCM-41 Molecular Sieve (1992)

Colossal Magnetoresistance (revived 1990s)



The exciting thing - there will be more!

A natural sequence

1. Materials discovery (anywhere in the world)
2. Characterisation (using powder diffraction)
3. Understanding Mechanisms (using spectroscopy, single-crystal diffraction, polarised neutrons)
4. Improving production (using SANS, powder diffraction, *in-situ* experiments)
5. Into devices/products (with or without (3))

Closing thoughts

Who would have thought that 3 new forms of carbon would be discovered?

GMR, Li-ion batteries, blue LEDs and hard magnets have changed our lives

We can expect more big surprises every few years

We need to be in early in the materials-discovery cycle – as in business, there is “first-mover advantage” thank you