New ILL Partners, Krakow Sept 2004

Structure and Materials – A Bridge for European Science

Alan Hewat, Diffraction Group Leader, ILL Grenoble, FRANCE

Grunwaldzki Bridge, Krakow (photo: P.Prokop)

European Neutron & Synchrotron Sources
ILL & ESRF Grenoble

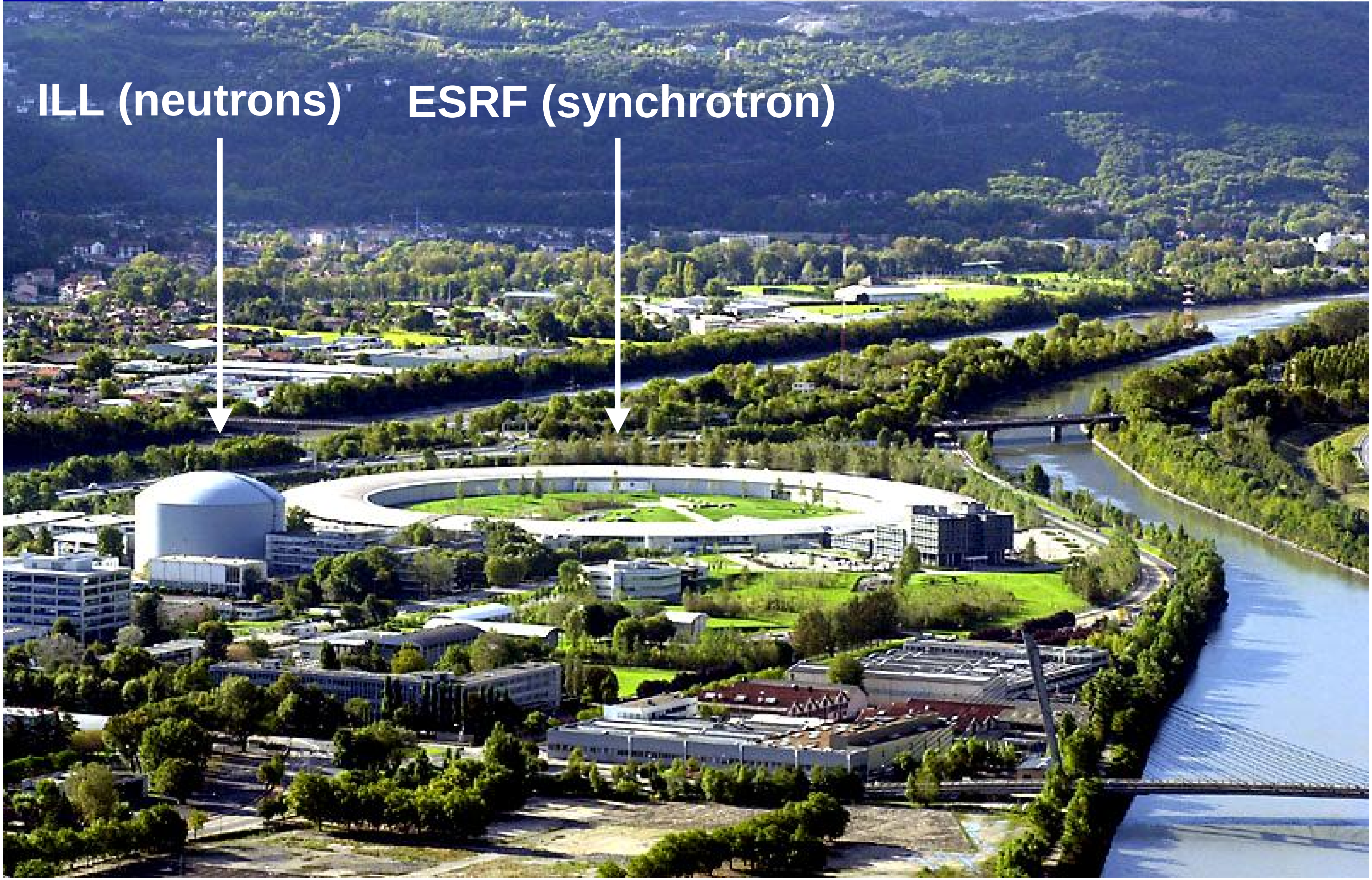
- World's most intense neutron source
- 1280 visiting scientists each year
- 300+ scientific papers each year
- physics, chemistry, biology, materials

- Proposals for experiments welcome
  Next deadline 2 weeks from now
  see: www.ill.fr (visitors club)

ILL member countries are shown in green

European Neutron & Synchrotron Sources

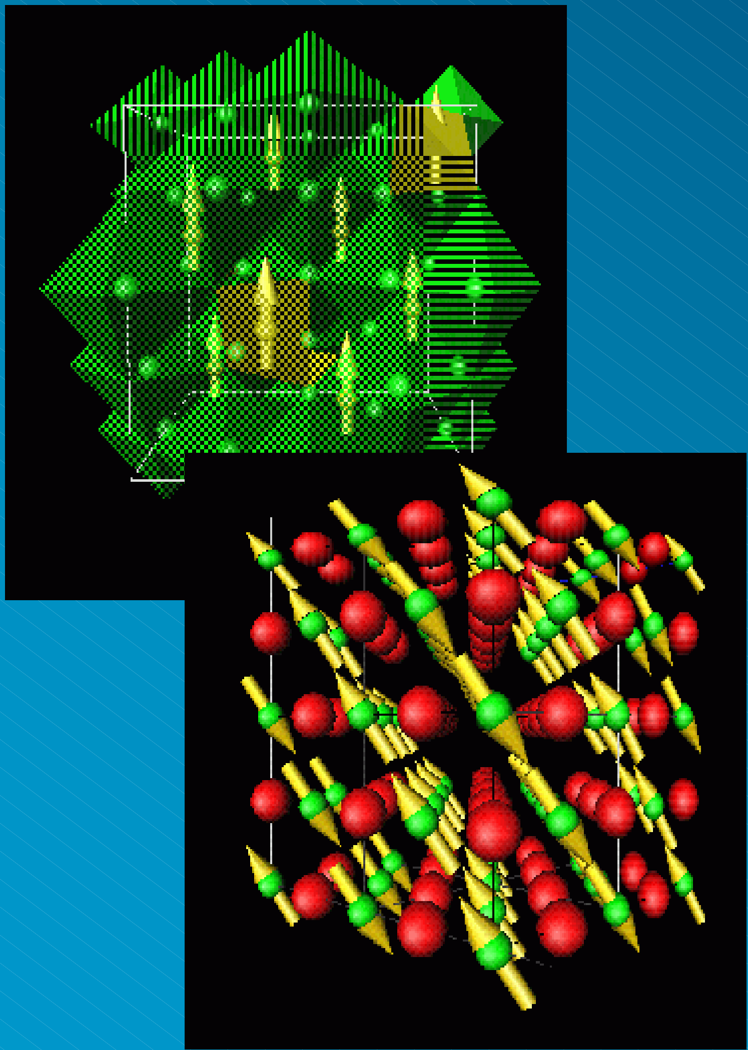
ILL & ESRF Grenoble



Maria Sklodowska-Curie
1867-1934

Advantages of Neutrons

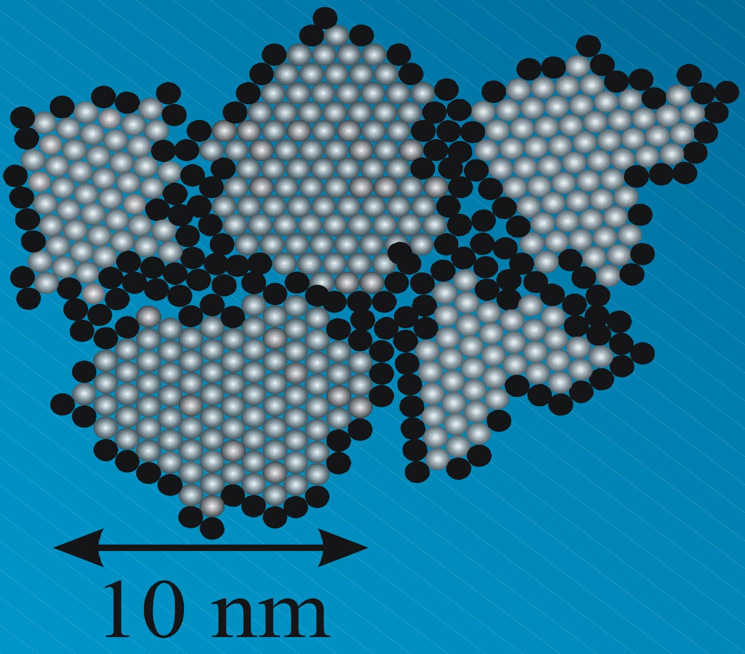
Neutrons scatter from magnetic atoms



- Neutrons act like tiny magnets
- Interact with atomic magnetic moments
- Neutrons determine magnetic structures
- Ferromagnetic magnetite Fe_3O_4 (top)
- Antiferromagnetic manganese oxide MnO

Magnetic ordering in electro-deposited nano-crystalline Chromium

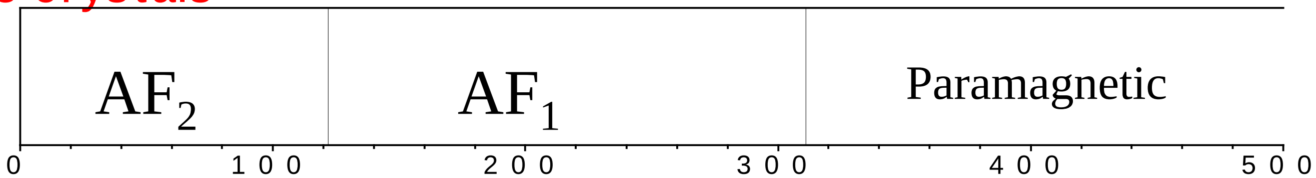
Przenioslo R., Sosnowska I, Rousse G, Hempelmann R (2002) Phys.Rev. B 66, 014404.



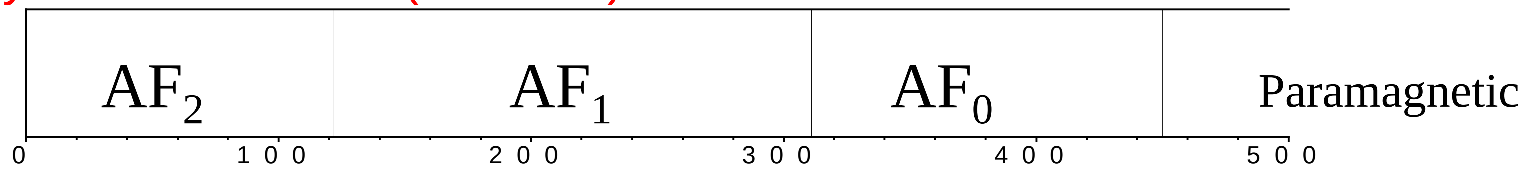
How does the magnetic order change with crystalline size ?

Magnetic order observed in different kinds of Chromium

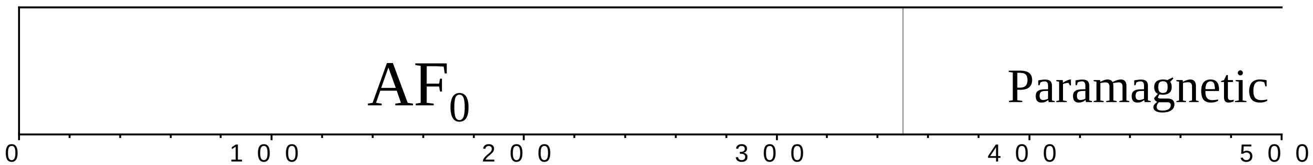
Single crystals



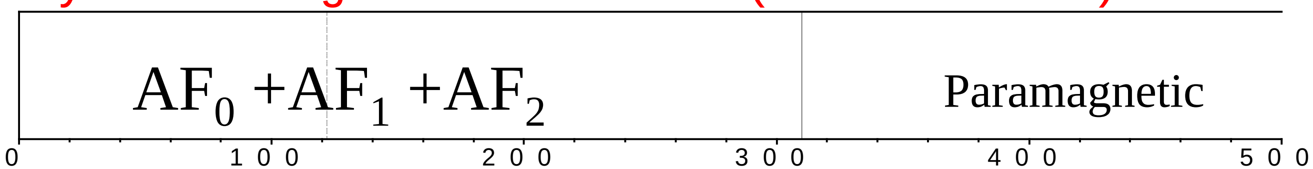
Polycrystalline material (strained)



Nanocrystalline small size D = 13 nm (Tsunoda 1993)



Nanocrystalline large size D = 100 nm (Ishibashi 1993)



Magnetic ordering in electro-deposited nano-crystalline Chromium

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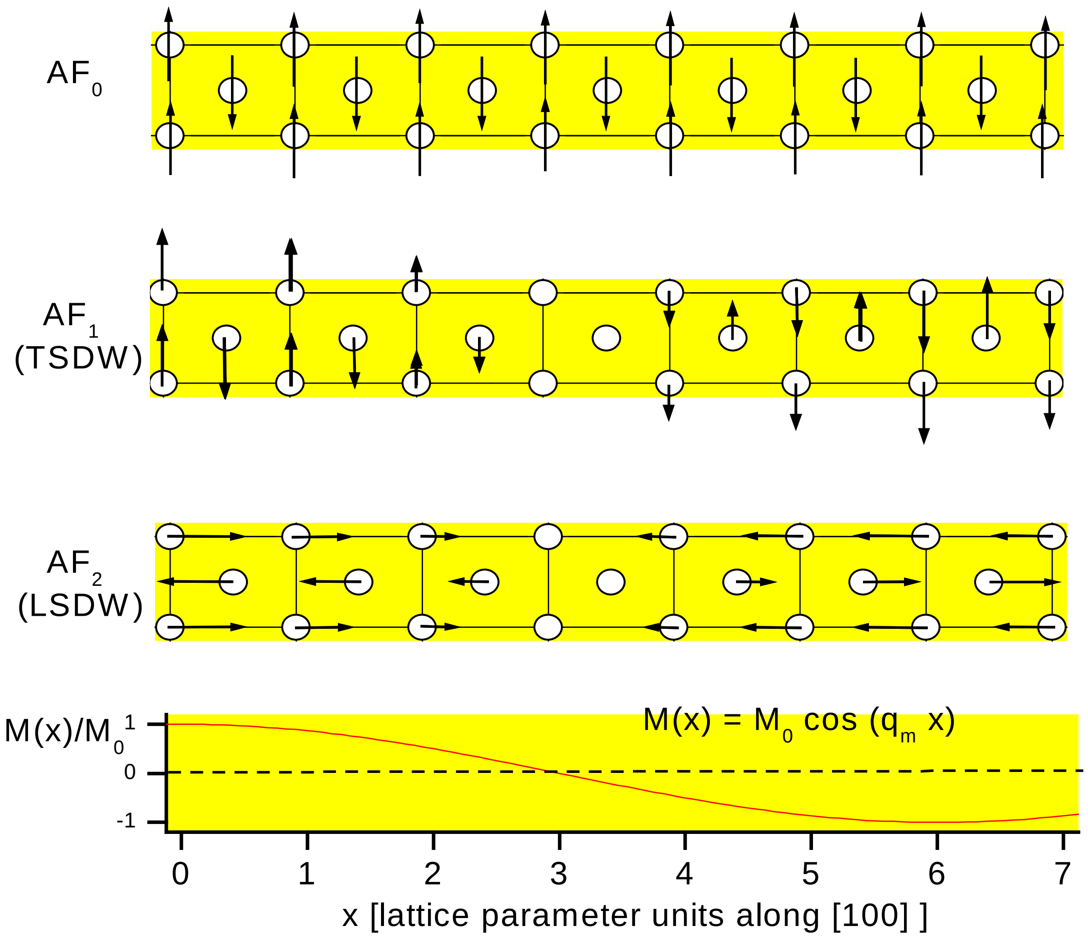
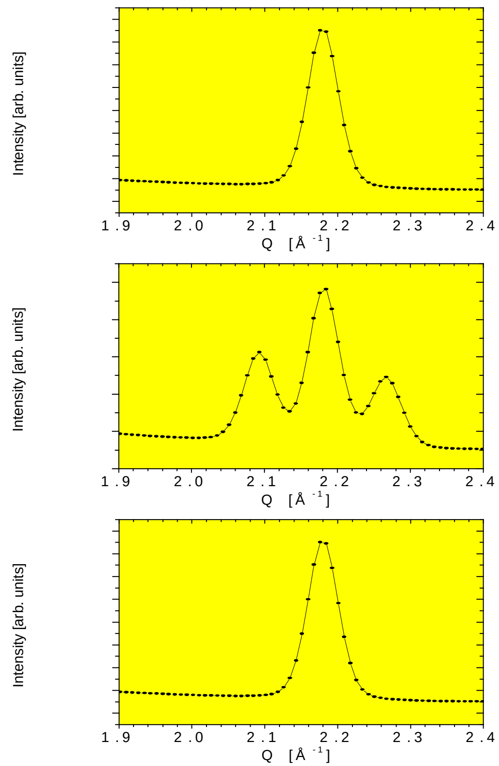
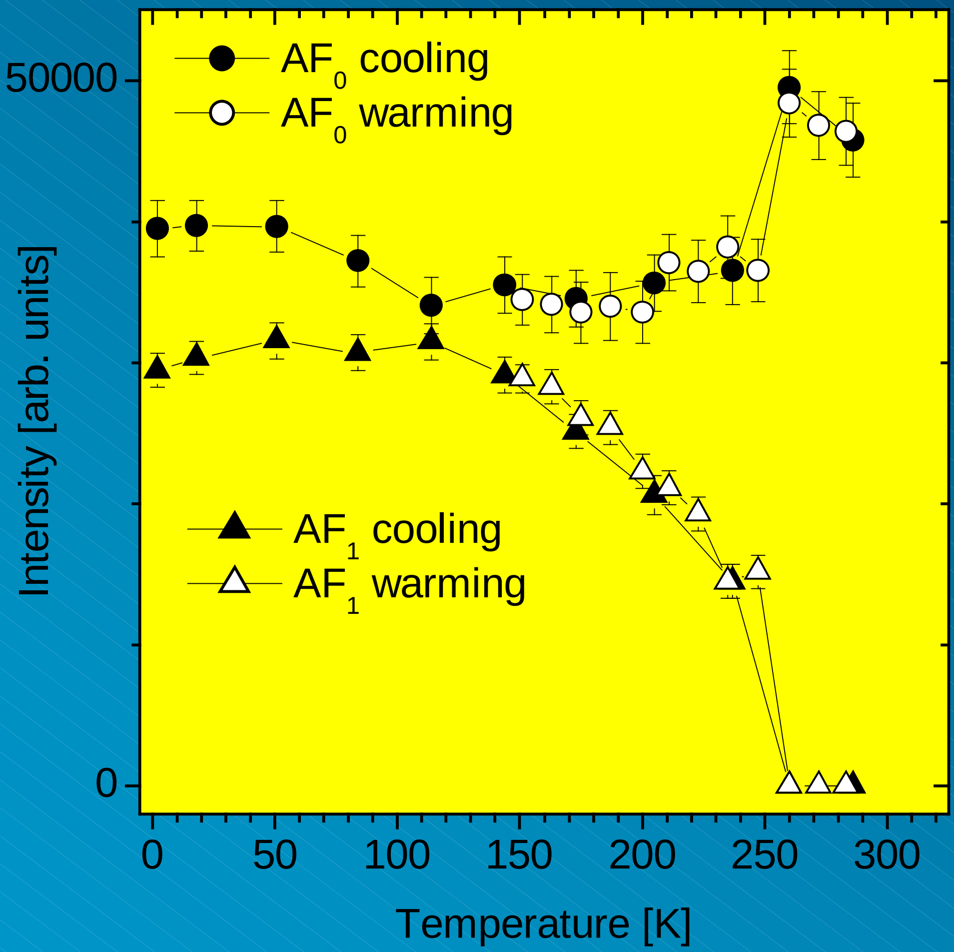




Temperature changes of the AF0 and AF1 magnetic orderings

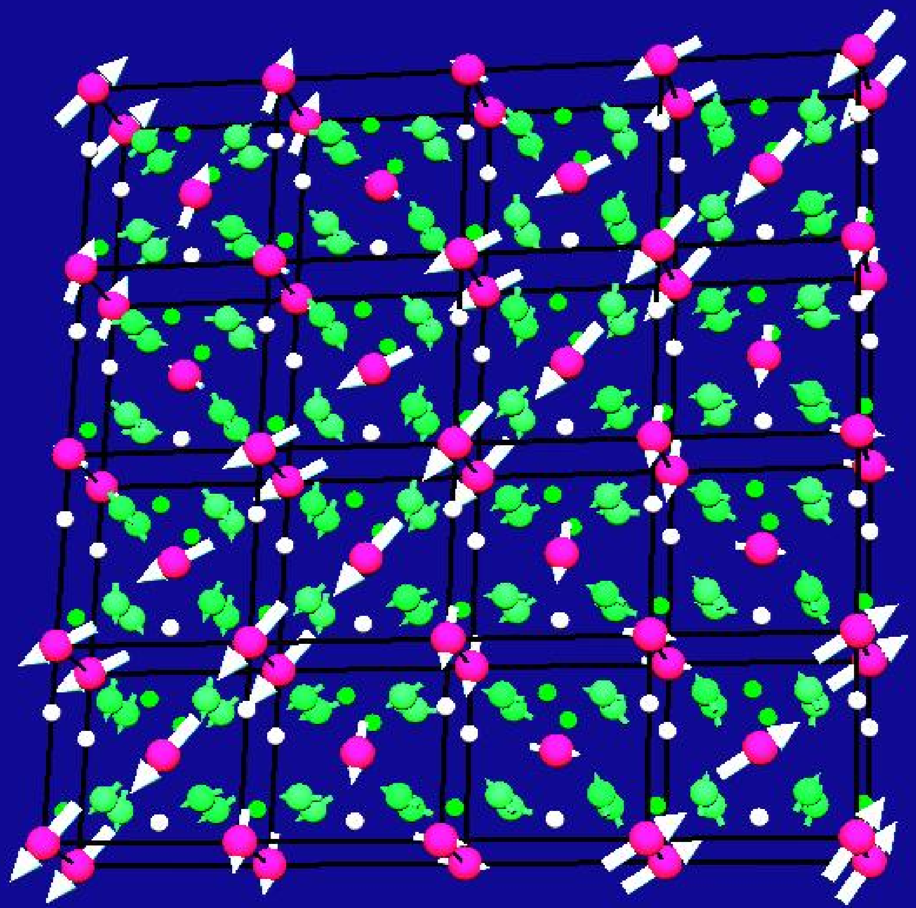
Przenioslo R., Sosnowska I, Rousse G, Hempelmann R (2002) Phys.Rev. B 66, 014404.



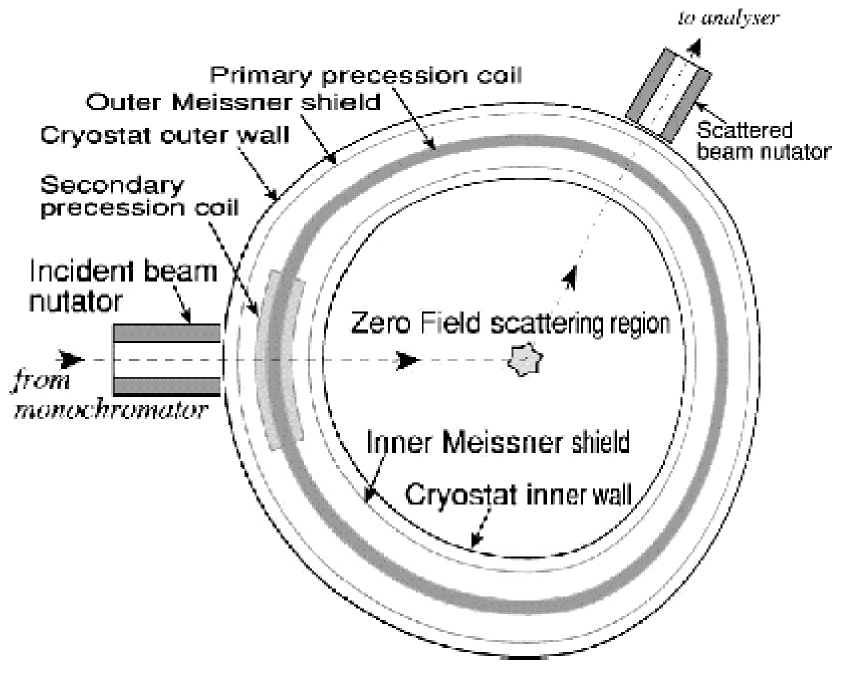
Neutrons - Unique for Magnetic Structures

Recent Portugese work at ILL

Neutron-Scattering study of the magnetic structure of $DyFe_4Al_8$ and $HoFe_4Al_8$.
Paixao ,J. A., Ramos ,Silva ,M, ... Brown ,P J et al. (2000). Phys.Rev. B61, 6176.



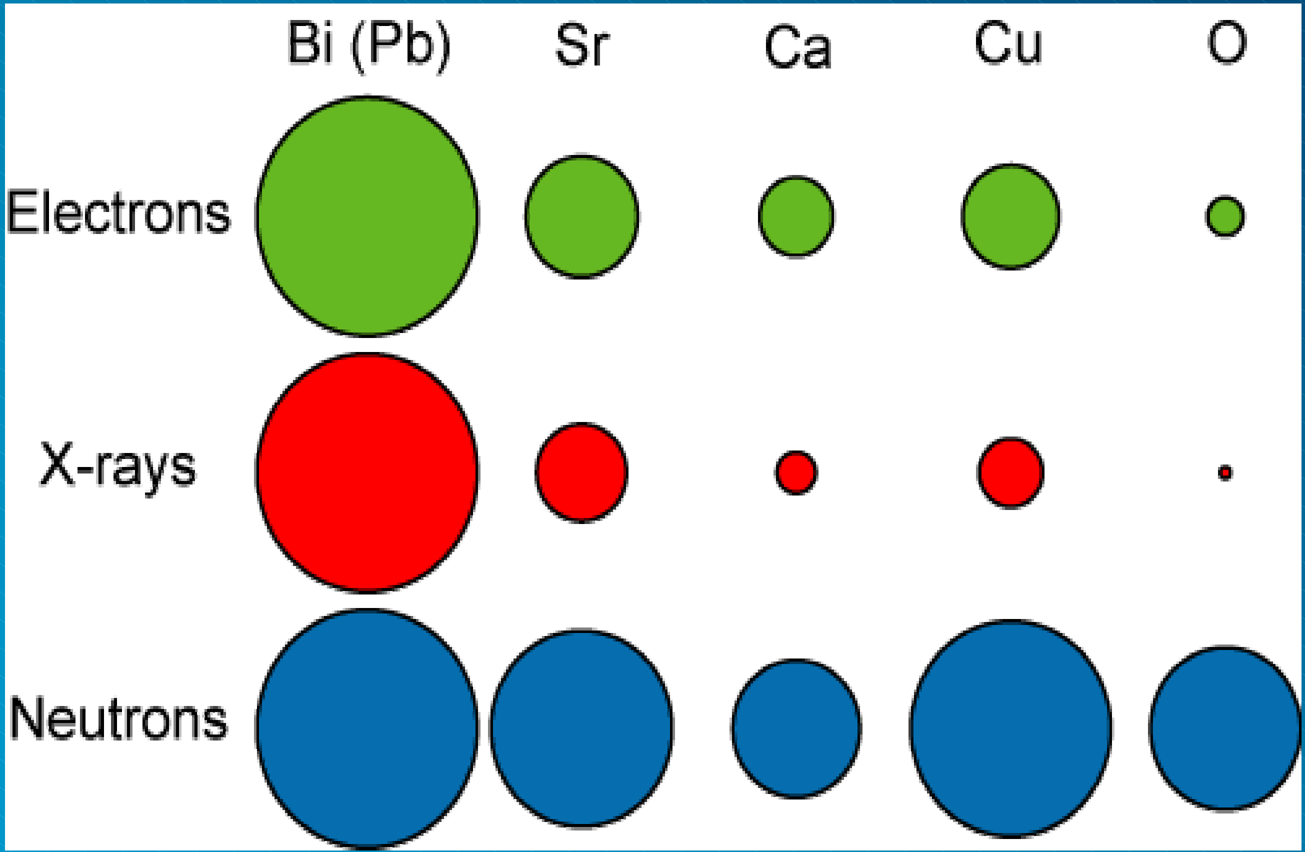
- Complex cycloidal magnetic structure of $HoFe_4Al_8$ using polarised neutrons from CryoPad on D3 at ILL



Advantages of Neutrons

Neutrons scatter strongly from light atoms

- Neutron scattering is of similar magnitude for all atoms
- X-ray scattering is proportional to the number of electrons
- Electron scattering depends on the electrical potential



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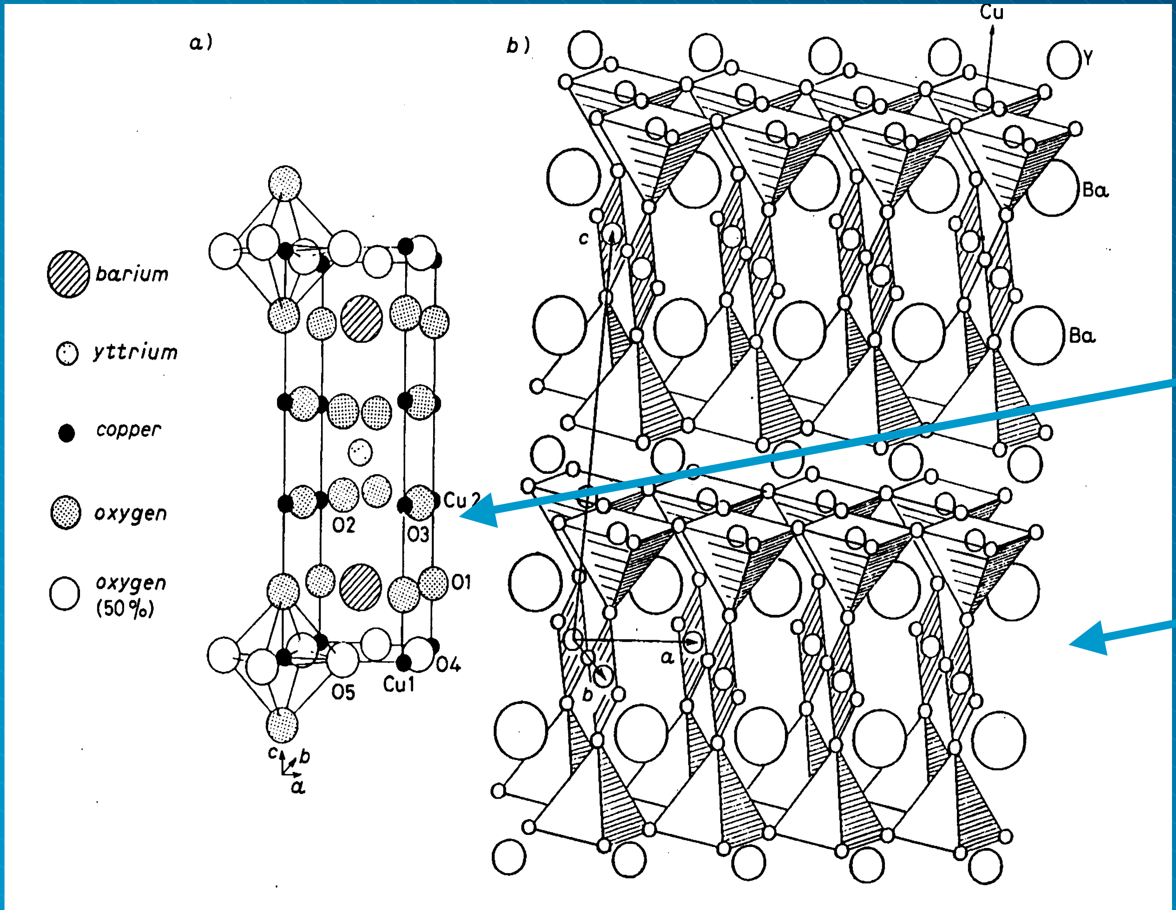


The 90K high Tc Superconductor $Y_1Ba_2Cu_3O_7$

- Left - by X-rays (Bell labs. & others)
- Right -by Neutrons (ILL & others)

Neutrons gave new insight, important in searching for similar materials.

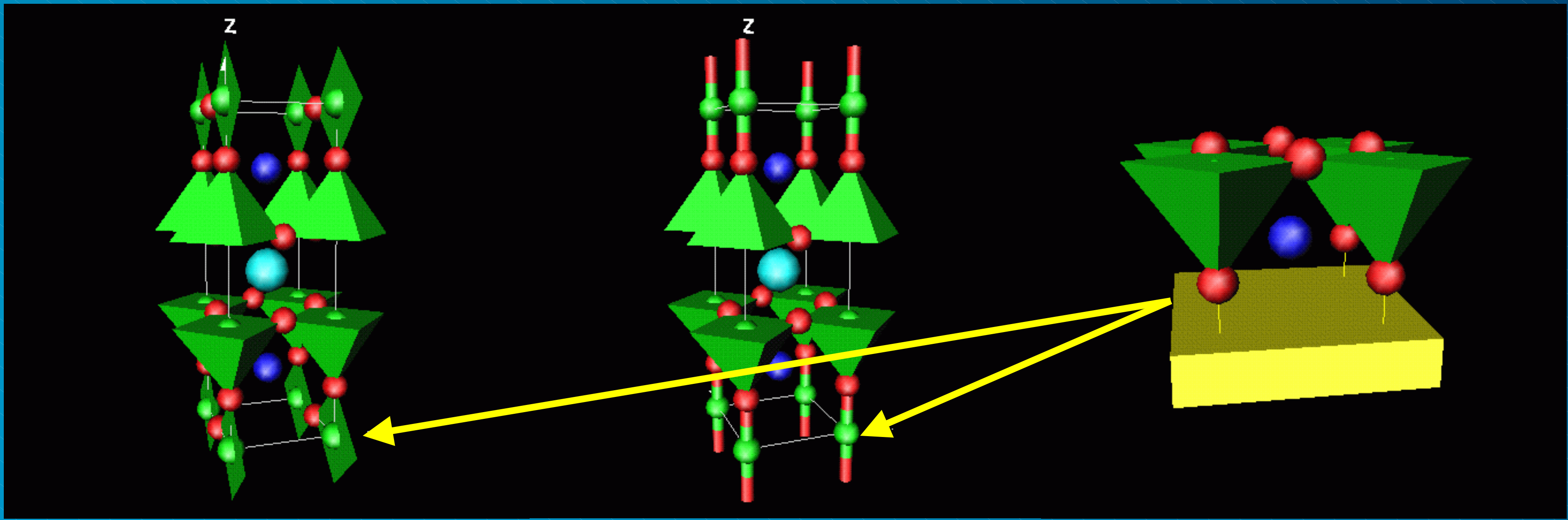
M. Marezio, J-J. Capponi, A. Hewat... (1987)

Measure precise metal-oxygen distances

The importance of oxygen for high-Tc superconductors

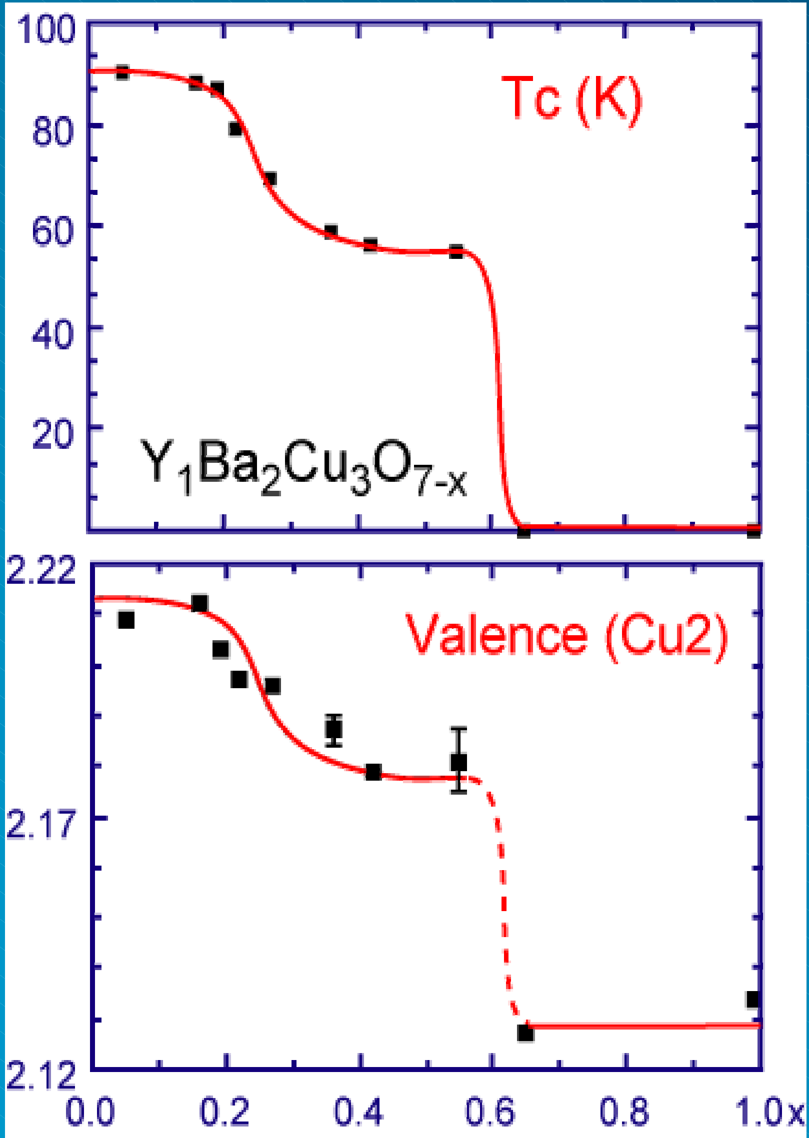
Neutrons are sensitive to oxygen – “charge reservoir” concept

Superconducting $YBa_2Cu_3O_7$ Non-supercond. $YBa_2Cu_3O_6$ Charge reservoir layer



R. Cava, A. Hewat, E. Hewat, M. Marezio (Bell labs & ILL)

Measure precise metal-oxygen distances



Oxidation state of Copper

Charge Transfer in High-Tc Superconductors

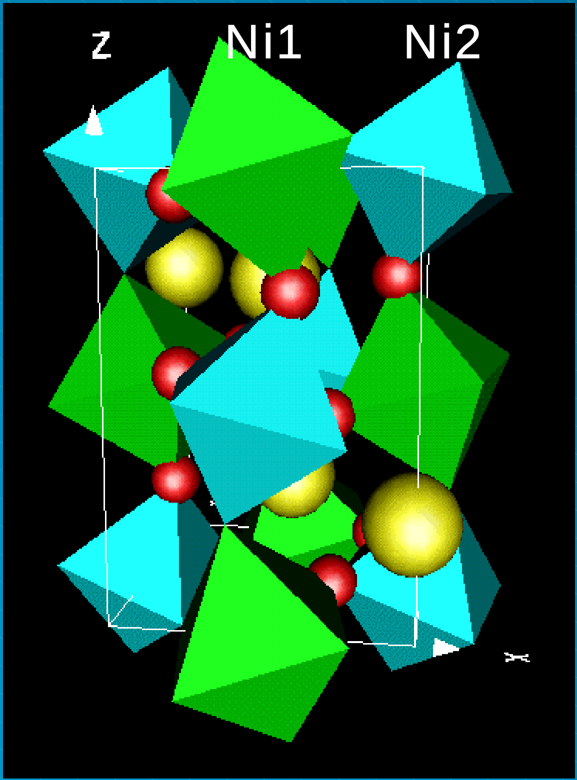
- Charge reservoir concept
- Tc depends on oxidation
- Imagine new charge reservoirs
- Discovery of new materials

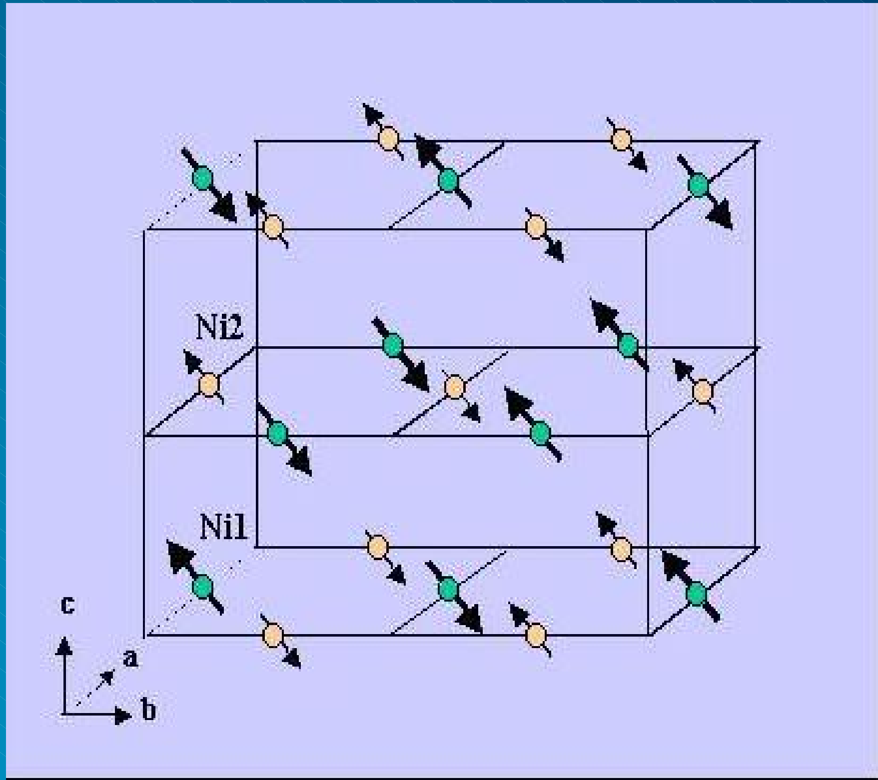
ILL and Bell labs. (1990)

Neutrons - precise metal-oxygen distances
Charge Transfer in $YNiO_3$

Combined ESRF, D1B and D2B data - Alonso J.A. et al (1999) PRL 82, 3873

Metallic Ortho. $YNiO_3$ -> Insulating Mono. $YNiO_3$ T < 582K   Ni valence 3-d, 3+ d





V(Ni1) = 2.62   V(Ni2) = 3.17

M(Ni1) = -1.4 m_B M(Ni2) = 0.7 m_B

Neutrons - precise metal-oxygen distances
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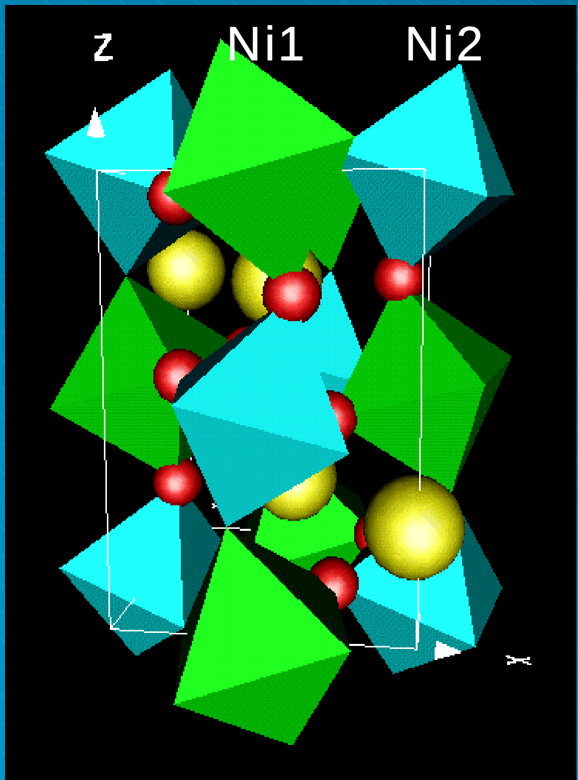


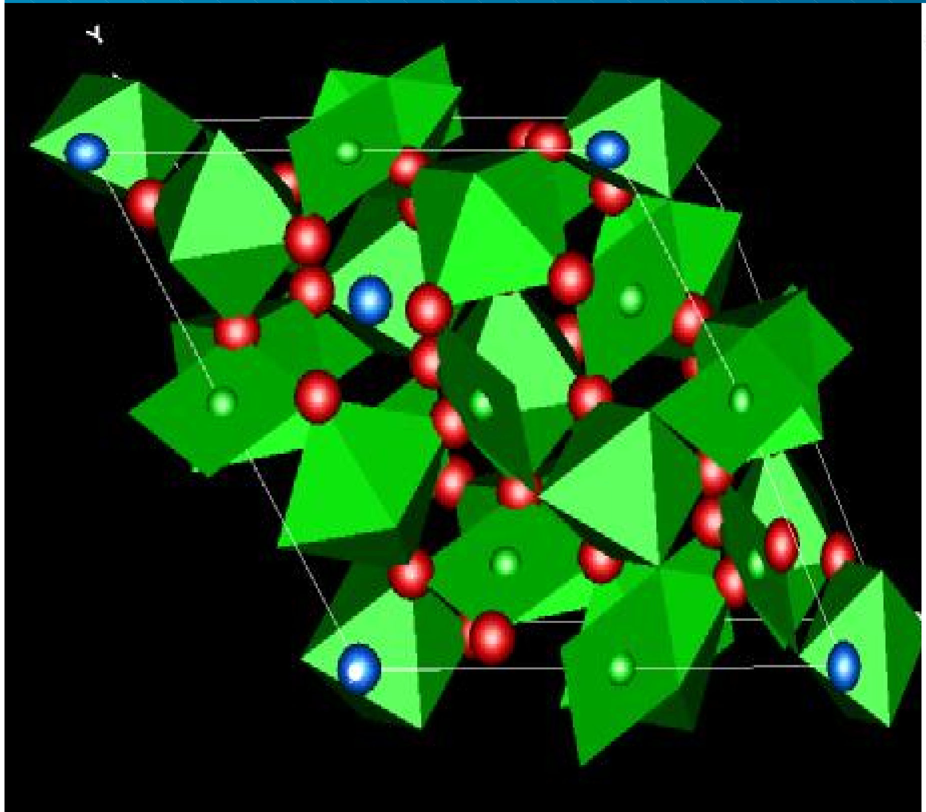
V(Ni1) = 2.62   V(Ni2) = 3.17

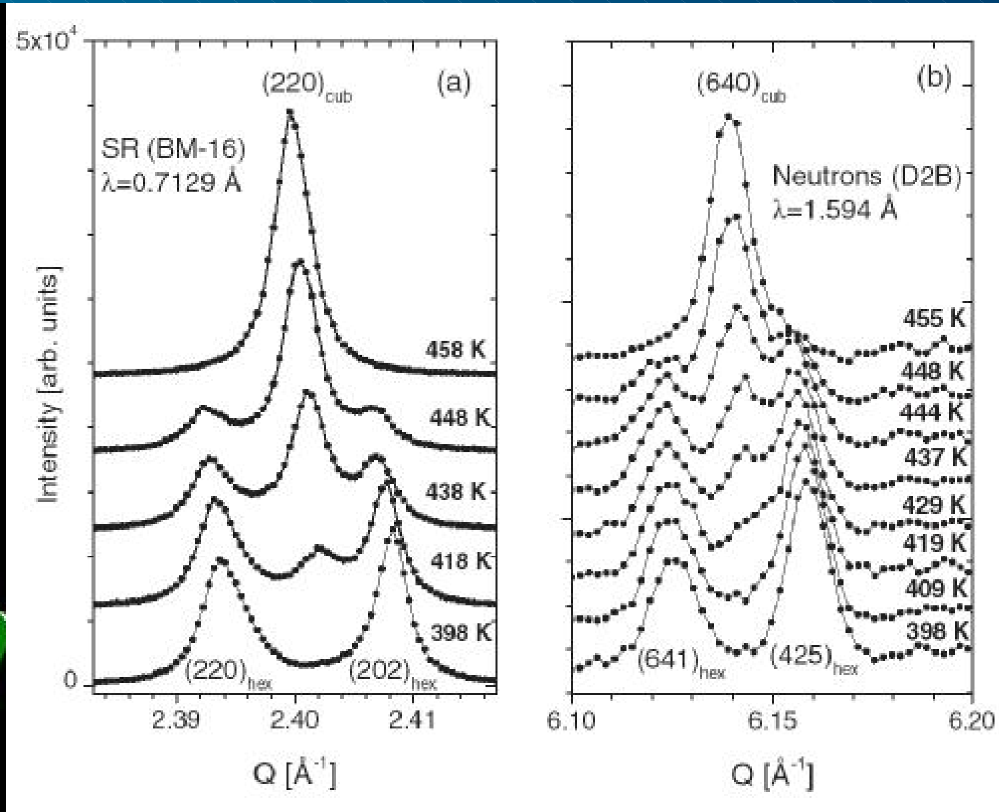
- Double evidence for charge transfer
  - Magnetic superstructure and different moments on Ni-sites
  - Different Ni-O distances around Ni1 and Ni2 sites mean 'charge transfer'
- Neutrons provide both. But need:
  - High resolution to resolve symmetry
  - High flux to see superstructure

Phase co-existence in the charge ordering transition in $CaMn_7O_{12}$

R Przeniosło, I Sosnowska, E Suard, A Hewat & A N Fitch (2002)

J. Phys. Condens. Matter 14, 5747–5753





Recent Papers from Sosnowska, Przenioslo (Warsaw Uni.) et al. at ILL

Phase separation in CaCuxMn7-xO12 (x=0.38). Przenioslo , R., Sosnowska ,I, Van-Beek ,W, Suard ,E, Hewat ,A (2004). **Journal of Alloys and Compounds 362**, 218.

Charge ordering and anisotropic thermal expansion of the manganese perovskite CaMn7O12. Przenioslo , R., Sosnowska ,I, Suard ,E, Hewat ,A, Fitch ,A N (2004). **Physica B 344**, 358.

Phase coexistence in annealed CaMn7O12. Przenioslo , R., Van-Beek ,W, Sosnowska ,I (2003). **Solid State Communications 126**, 485.

Magnetic ordering in electrodeposited nanocrystalline chromium particles. Przenioslo , R., Sosnowska ,I, Rousse ,G, Hempelmann ,R (2002). **Physical Review B 66**, 014404.

Magnetic order parameter in the perovskite system CaMn7O12. Przenioslo , R., Sosnowska ,I, Suard ,E, Hansen ,T (2002). **Applied Physics A 74**, S1731.

Phase coexistance in the charge ordering transition in CaMn7O12. Przenioslo , R., Sosnowska I, Suard ,E, Hewat ,A W, Fitch ,A N (2002). **Journal of Physics Condensed Matter 14**, 5747.

A Sample of other Polish Work at ILL

Uranium form factors in selected Utx ,compounds. Javorsky , P., Schweizer ,J, Givord ,F, Boucherle ,J X, Sechovsky ,V, Andreev ,A V, Lelievre-Berna ,E, Bourdarot ,F (2003). **Acta Physica Polonica B 34**, 1425-1428.

Magnetic structures of the R2Ni3Si5 compounds (R=Tb, Dy, Ho). Szytula , A., Kolenda ,M, Ressouche ,E (1997). **Physica B 234-236**, 663-664.

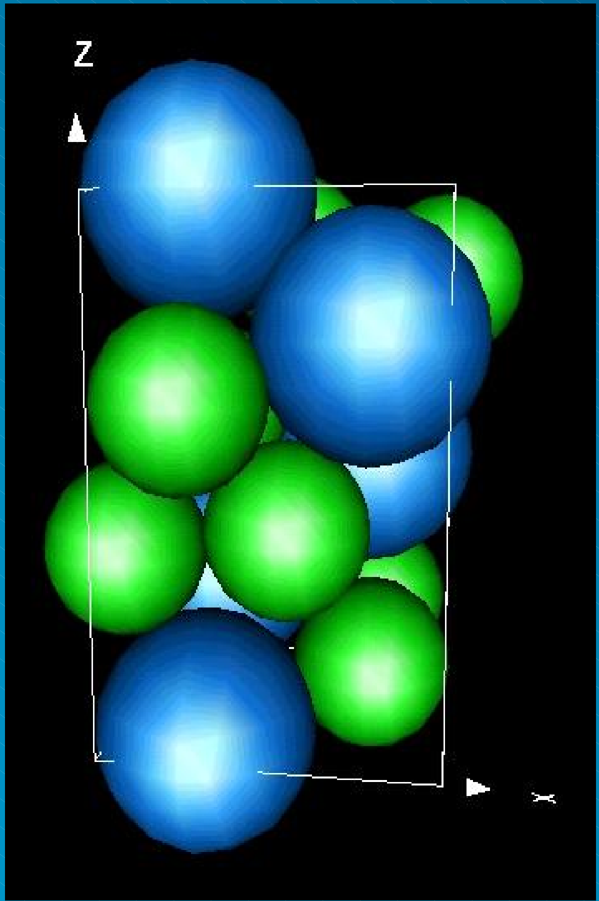
Neutron diffraction studies of magnetic structures of R2Ni3Si5 (R=Tb, Dy, Ho) compounds. Szytula , A., Kolenda ,M, Ressouche ,E, Sikora ,W (1997). **Journal of Physics Condensed Matter 9**, 6651-6663.

Magnetic properties of RPdGe (R=Ce, Pr and Tb) compounds. Szytula , A., Kolenda ,M, Ressouche ,E, Zygmunt ,A (1997). **Journal of Alloys and Compounds 259**, 36-41.

Peculiar ferromagnetic insulator state in the low-Hole-doped manganites. Algarabel , P. A., De-Teresa ,J M, Blasco ,J, Ibarra ,M R, Kapusta ,C, Sikora ,M, Zajac ,D, Riedi ,P C, Ritter ,C (2003). **Physical Review B 67**, 134402-1-134402-6.

Etc…Etc…

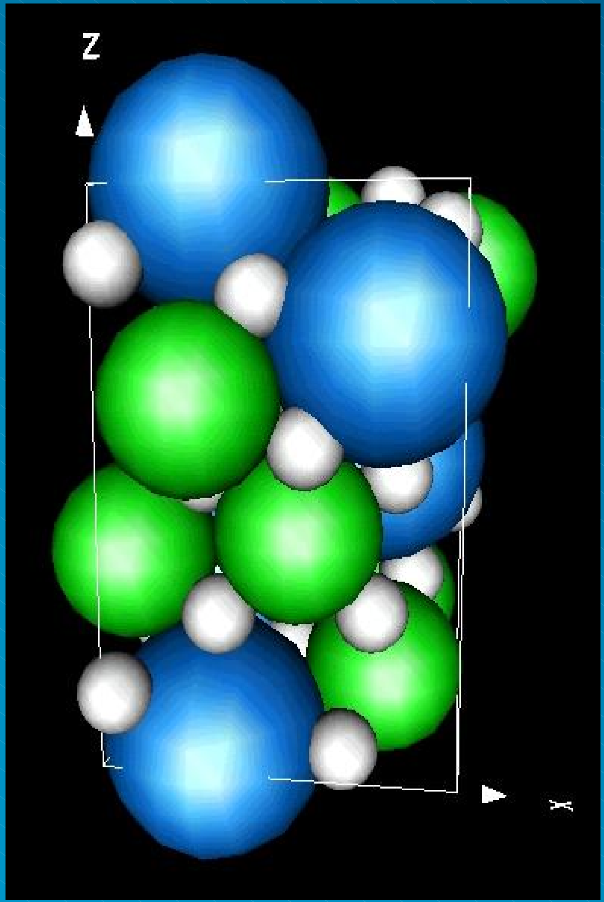
Hydrogen storage in metals



- Hydrogen storage in metals
  - Location of H among heavy atoms
  - No single crystals
- Laves phases eg $LnMg_2H_7$ (La,Ce)
- Can even find H in Eu on D20 !

Gingl, Yvon et al. (1997) J. Alloys Compounds 253, 313.
Kohlmann, Gingl, Hansen, Yvon (1999) Angew. Chemie 38, 2029. etc..

Neutrons scatter strongly from light atoms
Hydrogen storage in metals



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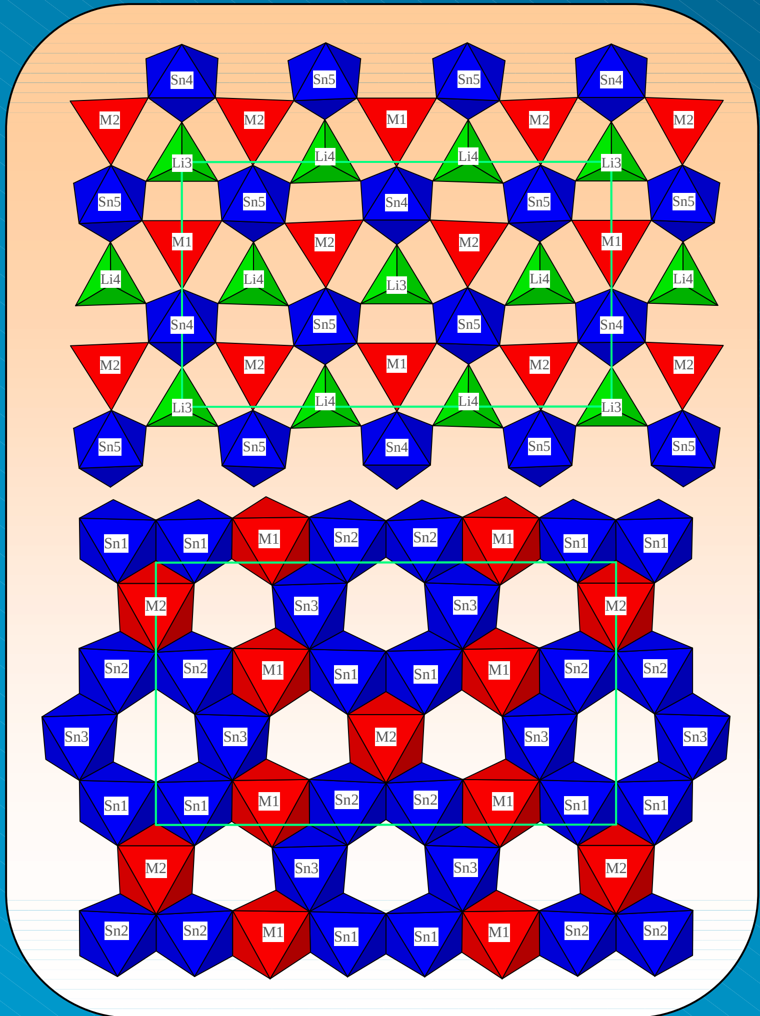
Kohlmann, Gingl, Hansen, Yvon (1999) Angew. Chemie 38, 2029. etc..

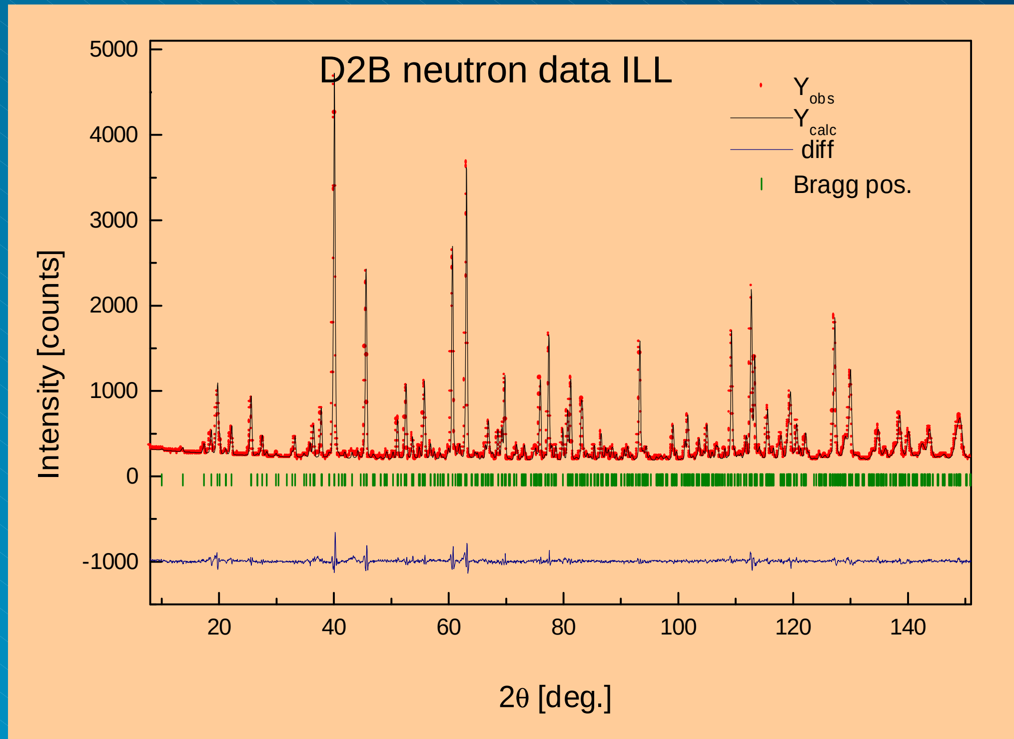
Neutrons - Unique Locating Light Atoms
Bulgarian Academy of Science work at ILL

Cation distribution in $Li_2M(II)Sn_3O_8$, M(II)=Mg, Co, Fe.

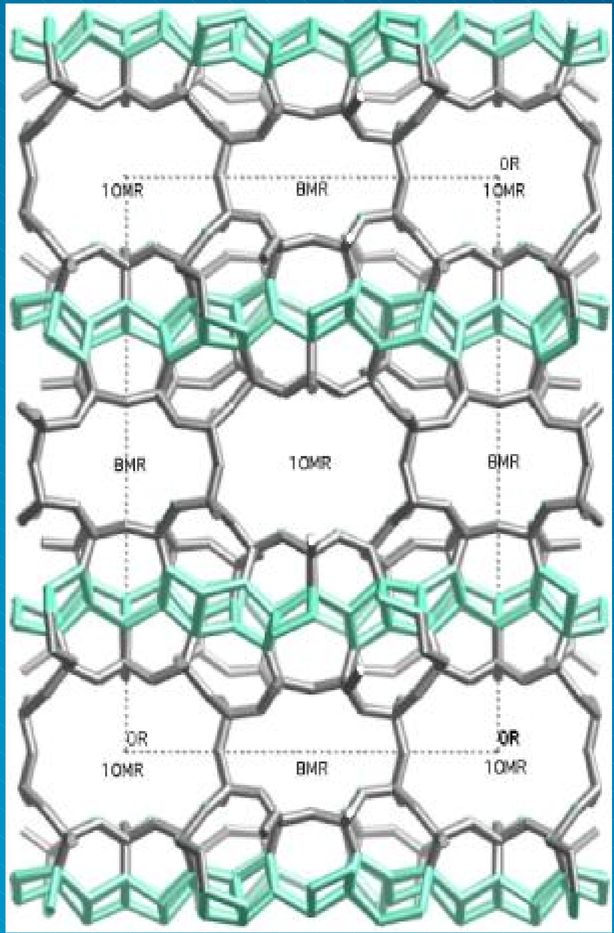
T. Trendafilova, D. Kovacheva, K. Petrov & A. Hewat (2004) EPDIC-IX, Prague.



- Complex oxide structures containing lithium were solved using both ILL-ESRF neutron and synchrotron data in Grenoble



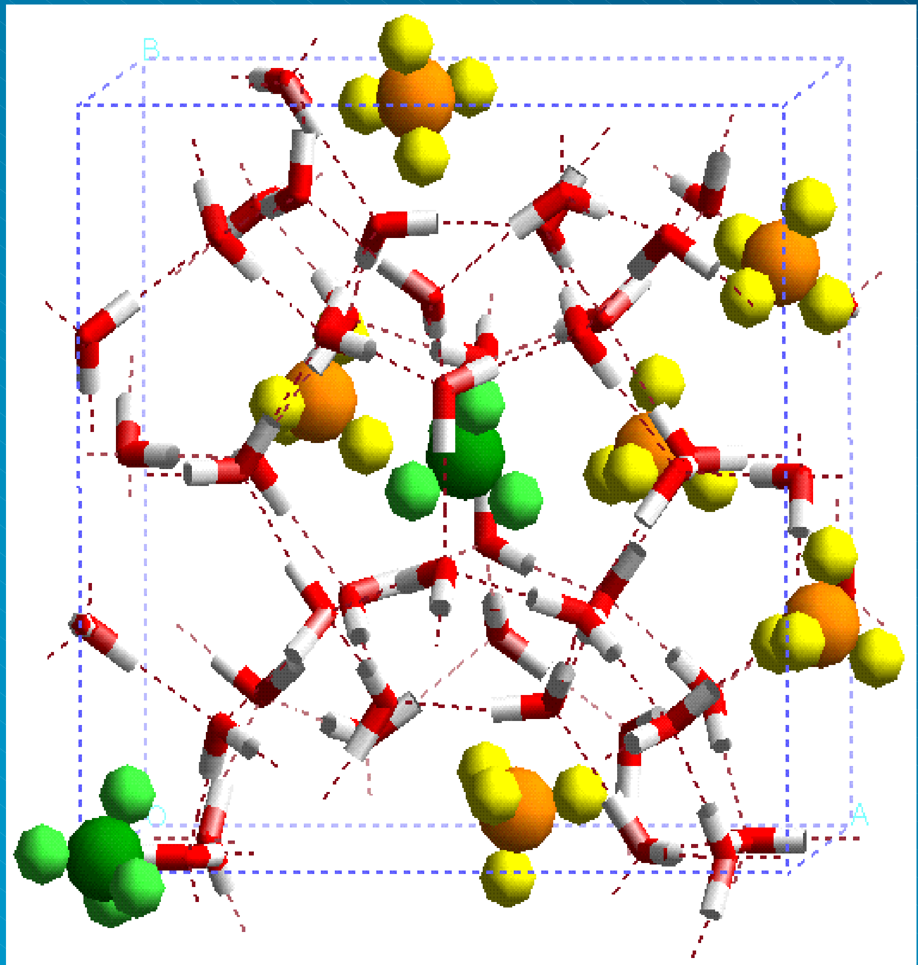
Neutrons scatter strongly from light atoms
Molecular sieves and ion exchangers



- Ion exchangers can remove toxic metals from the environment
- New types of zeolite ion-exchangers are needed to trap specific elements
- Neutron and synchrotron radiation are used to understand ion exchange
- RUB29, a new lithium zeolite for cleaning up radioactive caesium

J.B.Parise, S-H.Park, A.Tripathi, T.Nenoff, M.Nymann (SUNY & SANDIA)

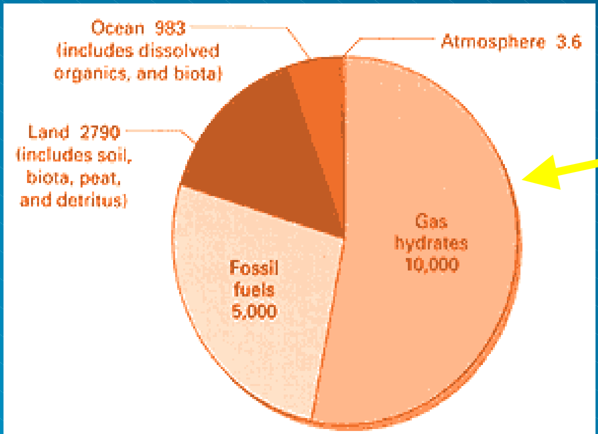
Neutrons scatter strongly from light atoms
Clathrates - hydrate fuel from the ocean

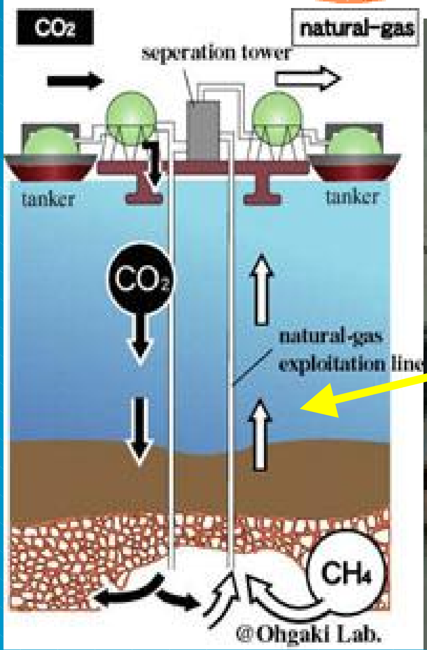


- Clathrates consist of molecular cages that can trap methane (spheres)
- Neutrons are important – they scatter strongly from the light methane atoms
- High pressure compressibility was studied, to help with seismic searches for clathrates

B.Chazallon, A.Klaproth, D.Staykova, W.Kuhs (Göttingen)

Neutrons scatter strongly from light atoms
Clathrates - hydrate fuel from the ocean



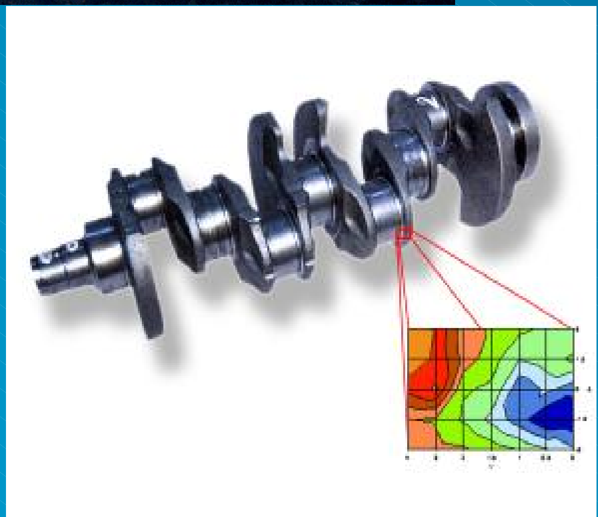


- Most hydrocarbons are locked in water cages at the bottom of the oceans
- These gas hydrates can be used as fuel
- A closed fuel cycle – extraction of methane and storage of CO2 in the deep ocean
- Neutrons are needed to learn more about these strange “clathrates” eg density at HP for seismic searches

Advantages of Neutrons

Neutrons are more penetrating than X-rays

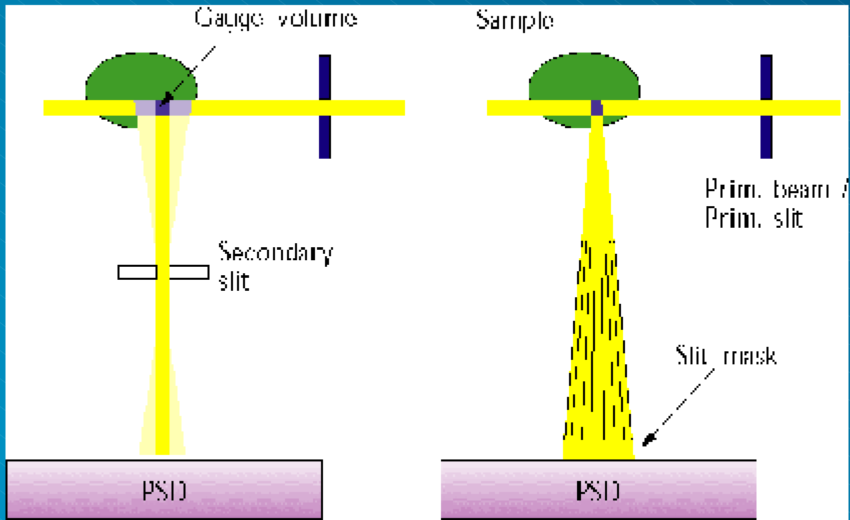
Measuring stresses deep inside engineering components



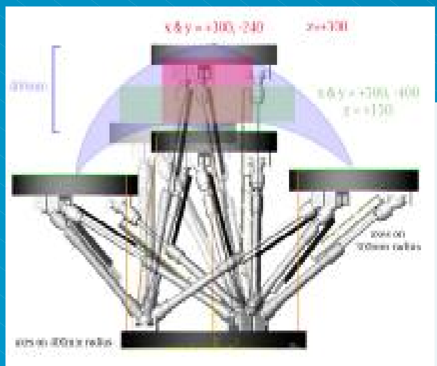
- Tensile stress can produce cracks
- Compressive stress toughens materials
- Neutrons can penetrate deep inside materials (~10cm) and measure stress by changes in atom spacings
- The compressive stress (blue) deep inside a VW crankshaft
- Design of stronger, lighter engines

T. Pirling, G. Bruno (ILL & Manchester)

Measuring stresses deep inside engineering components



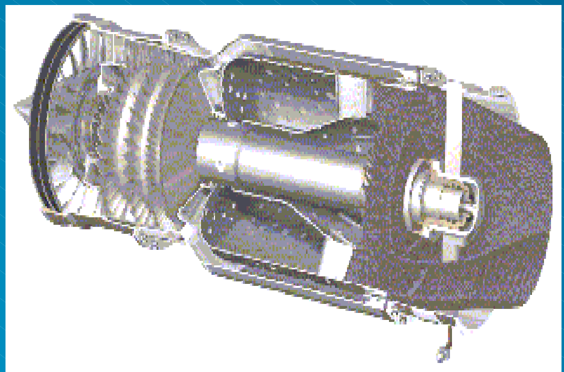
- The neutron beam is collimated to a 1mm^3 “gauge volume” of measurement
- The scattered peak is measured on a position-sensitive detector (PSD)
- Small shifts in peak positions map the strain as the sample is scanned
- Very large engineering components (1 tonne) can be scanned using a “hexapod” platform (similar to the platform of an aircraft flight simulator)

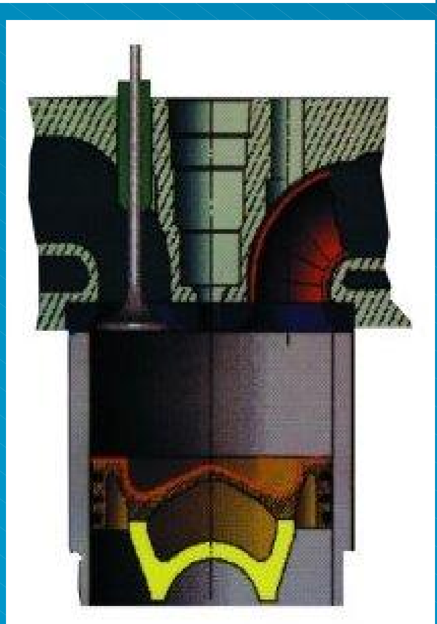


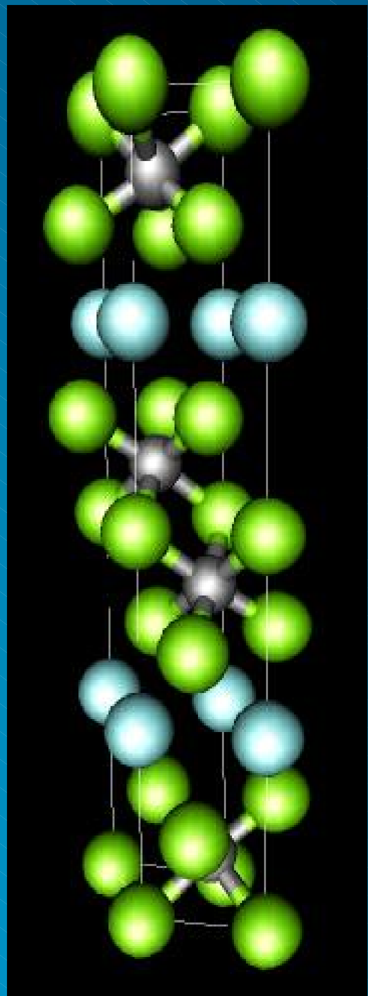
T. Pirling, G. Bruno (ILL & Manchester)

Neutrons are more penetrating than X-rays

New ceramics to replace metals in engineering components







- Titanium silicon carbide Ti_3SiC_2 conducts heat and electricity
- It is tough, easily machinable
- Potential engineering applications as a light replacement for metals
- BUT, difficult to prepare pure

D.Riley, E.Kisi, T.Hansen, A.Hewat

Advantages of Neutrons

Neutrons are more penetrating than X-rays

New ceramics to replace metals in engineering components

- The explosive SHS reaction was studied in real time with neutrons
- The reaction is exothermic, & heats the sample to 2200ºC in <1 sec
- The complete diffraction pattern (left) is collected at 300 ms intervals – A World Record
- Knowledge of the SHS process allows us to prepare a pure Ti_3SiC_2 product

D.Riley, E.Kisi, T.Hansen, A.Hewat

Disadvantage of Neutrons

Neutron Flux is low – Need Big Detectors

Construction of a microstrip position-sensitive detector (printed circuit)

A.Oed,
P.Convert,
T. Hansen,
et al...
(ILL)

Disadvantage of Neutrons

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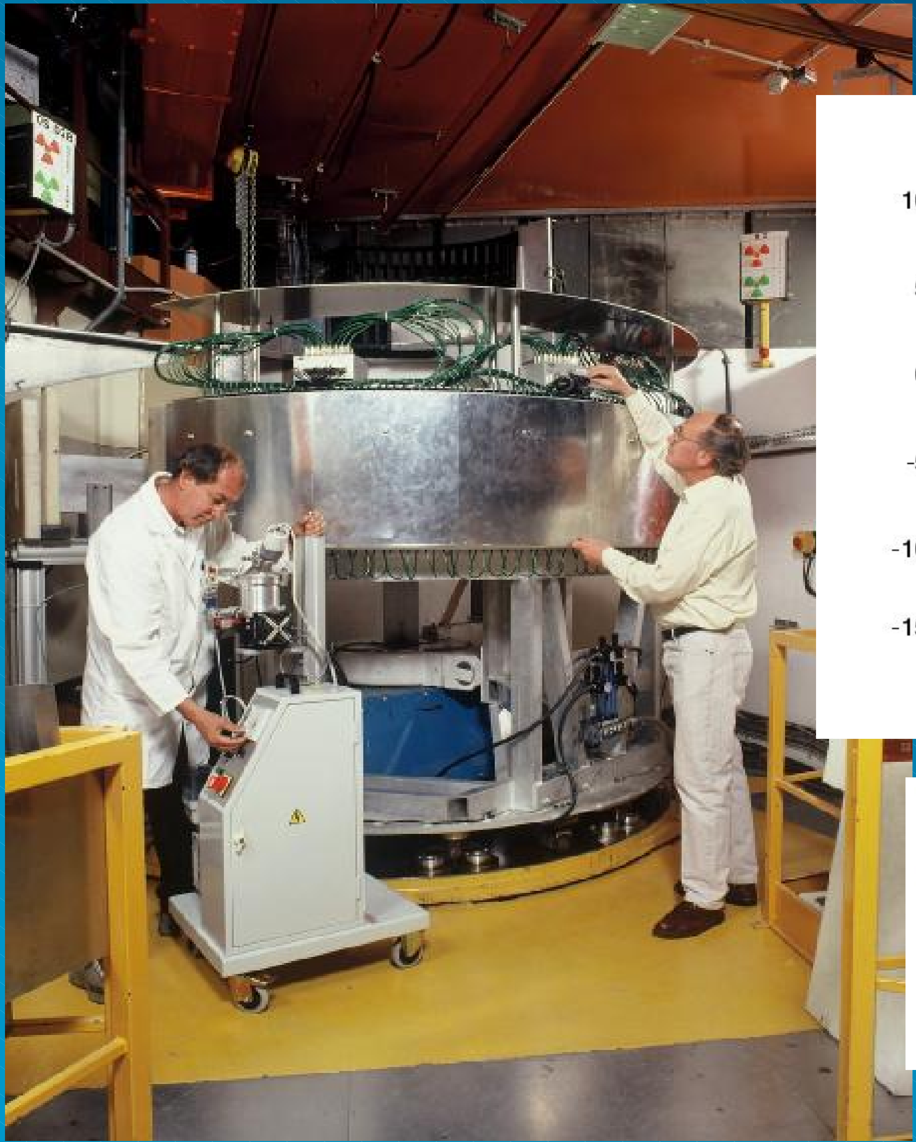
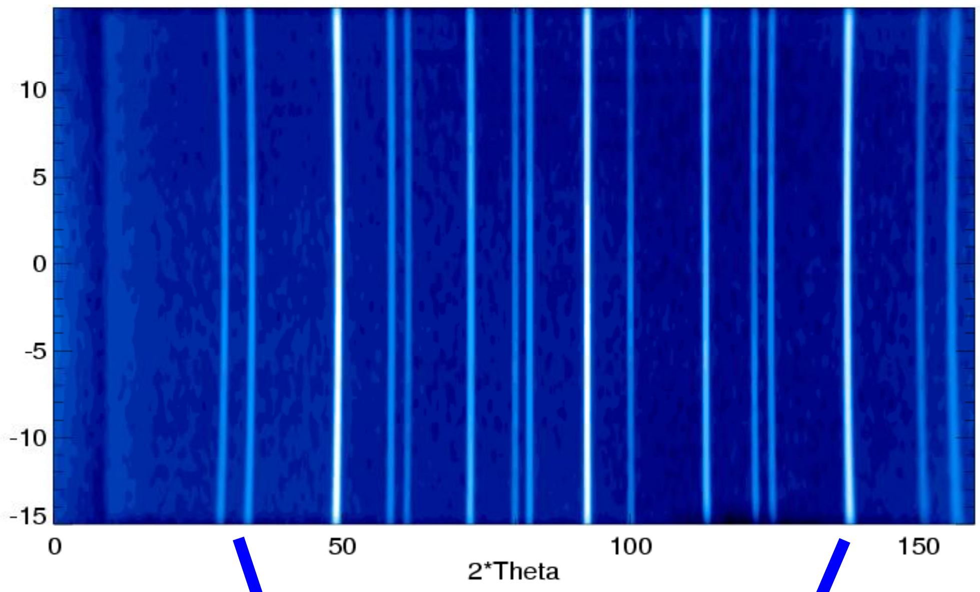
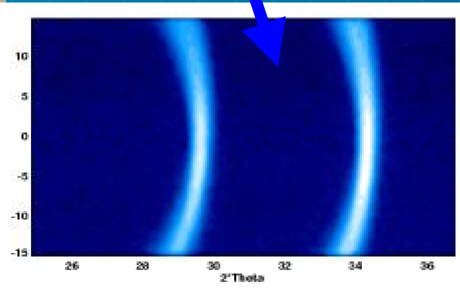
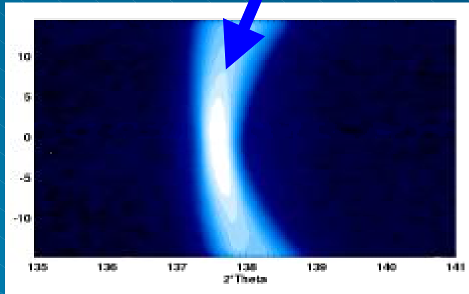


UK-EPSRC project Super-D2B







E.Suard, C.Ritter, A.Hewat, P.Attfield... (Edin.)

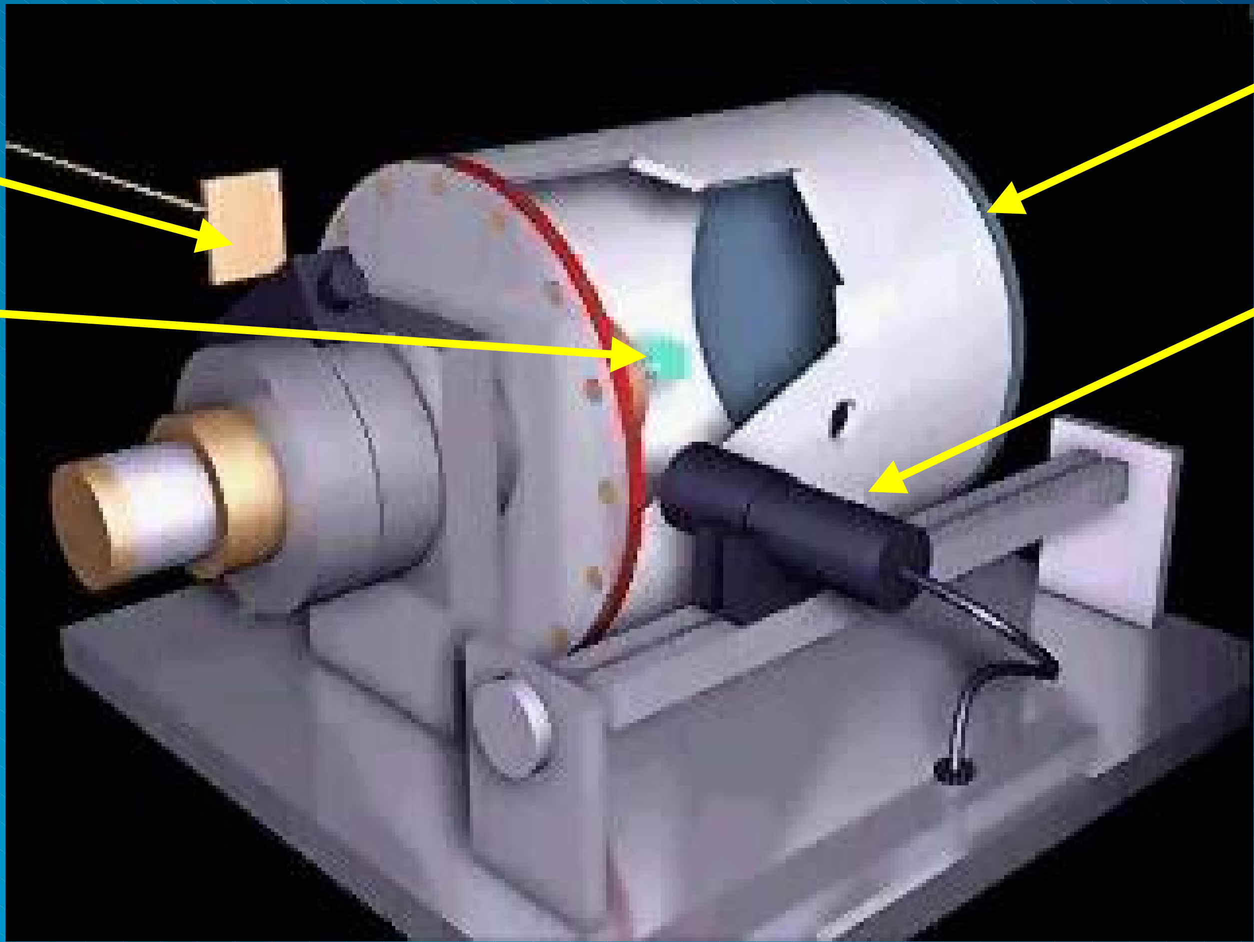
DRACULA – Ultra fast, very small samples

Disadvantage of Neutrons

Neutron Flux is low – Need Big Detectors

Neutron Image Plate Detectors – like photographic film

All of the scattered neutron peaks are recorded simultaneously



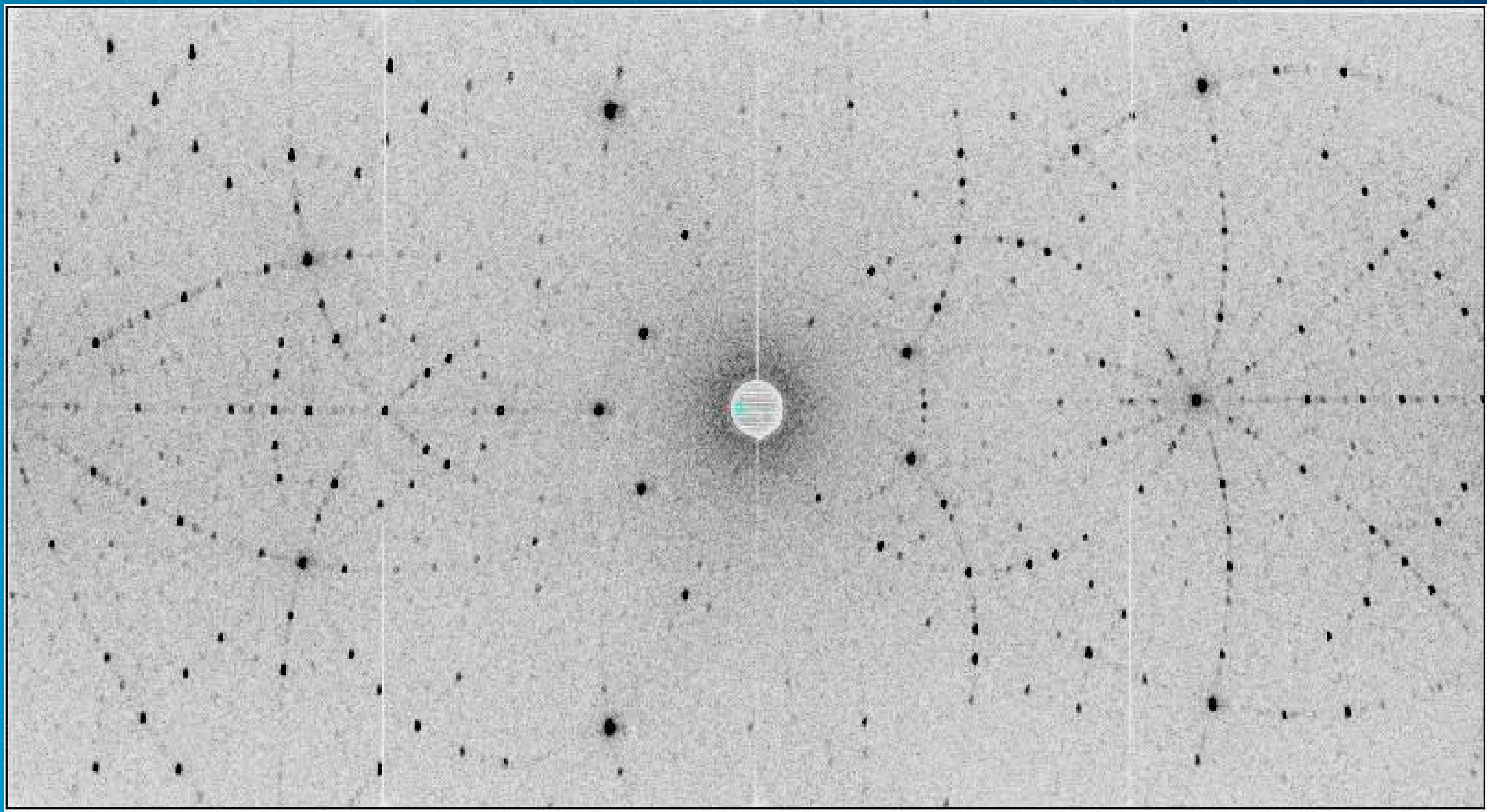
C.Wilkinson
M.Lehmann
D.Myles
F.Cipriani
G.McIntyre
(EMBL & ILL)

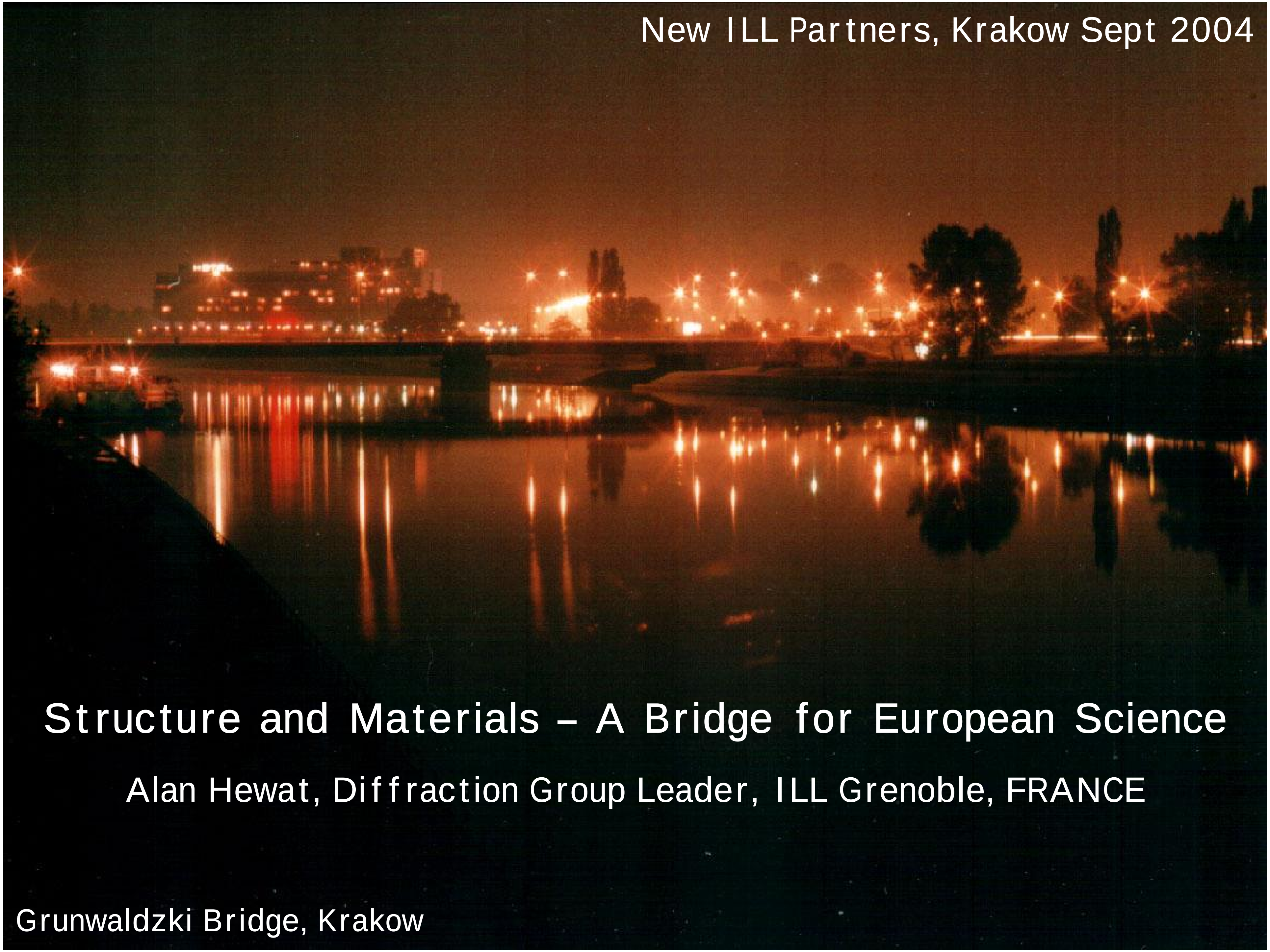
Disadvantage of Neutrons

Neutron Flux is low – Need Big Detectors

Neutron Image Plate & 5-fold symmetry of a quasi-crystal

All of the scattered neutron peaks are recorded simultaneously




New ILL Partners, Krakow Sept 2004
Structure and Materials – A Bridge for European Science
Alan Hewat, Diffraction Group Leader, ILL Grenoble, FRANCE
Grunwaldzki Bridge, Krakow

A Bridge for European Science

Science & Technology in the New Europe

Number of cited Spanish papers with ILL

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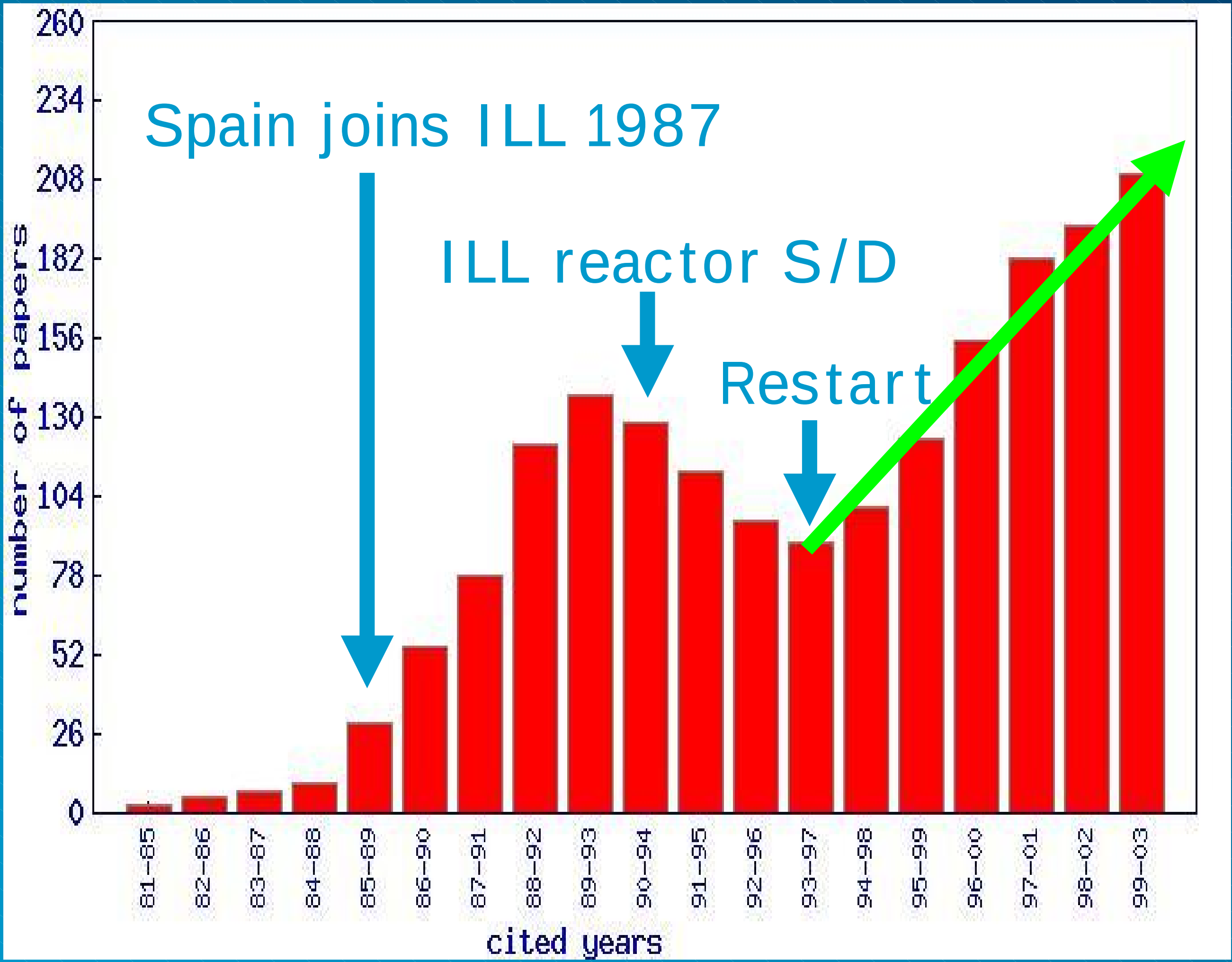

Number of cited Spanish papers with ILL

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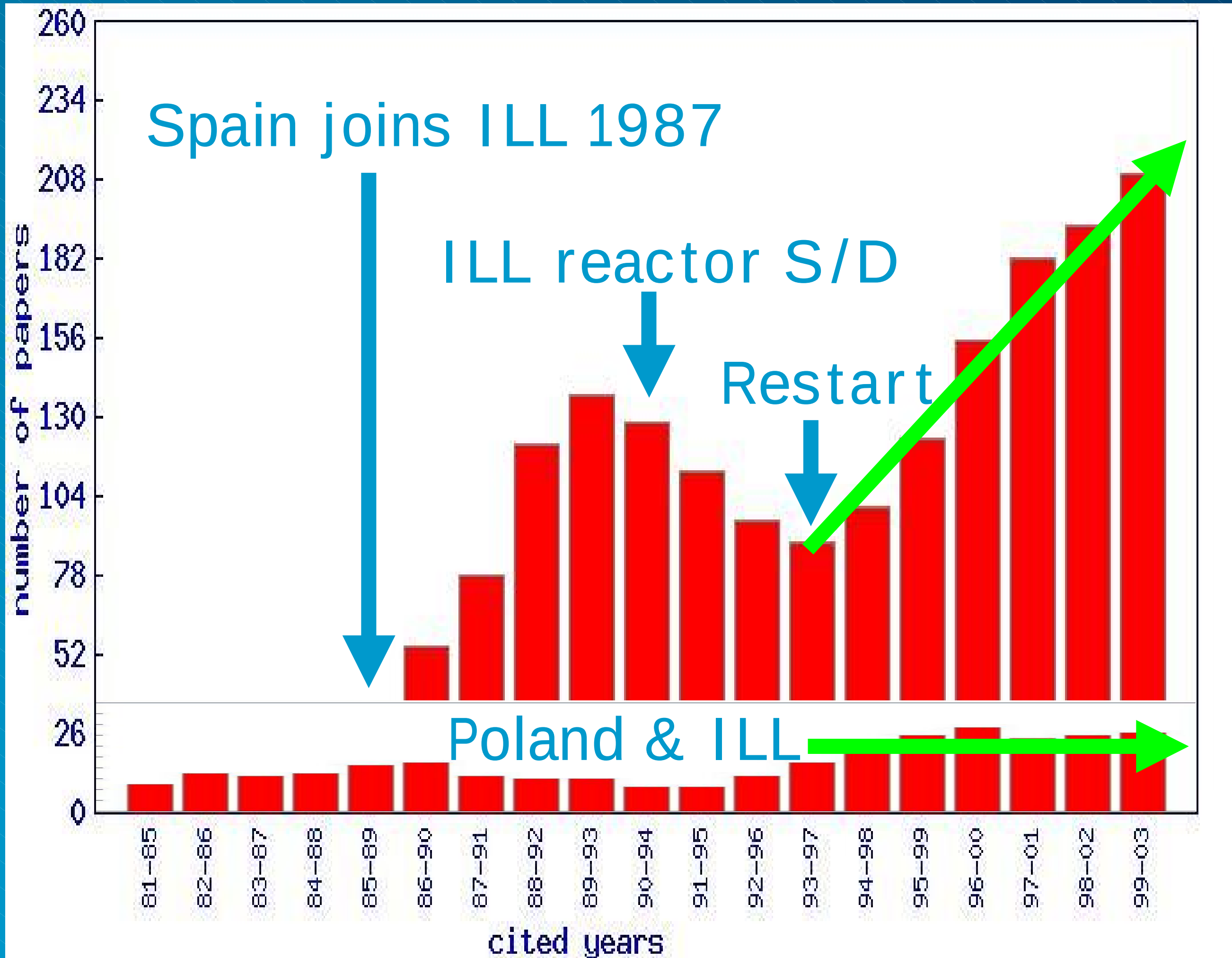
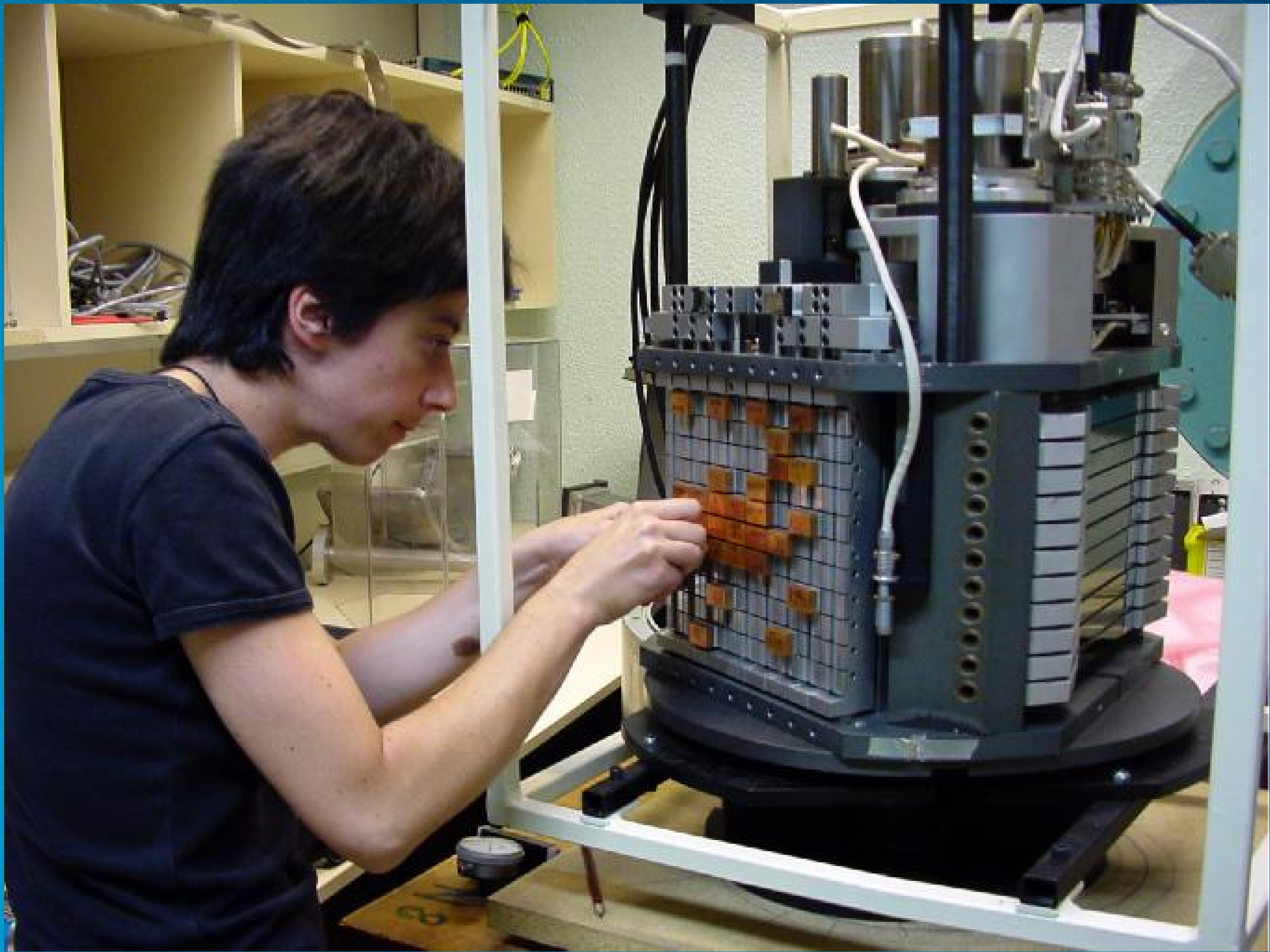


97 Participants – 55 contributions

Dr Monica Jiminez aligns the new IN8 Spanish Monochromator



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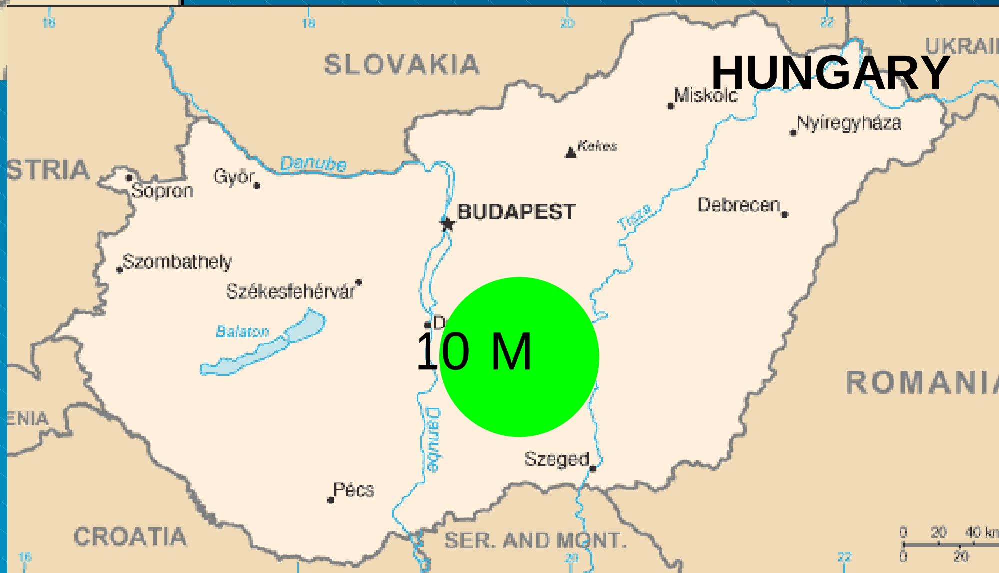

Number of cited Czech papers with ILL

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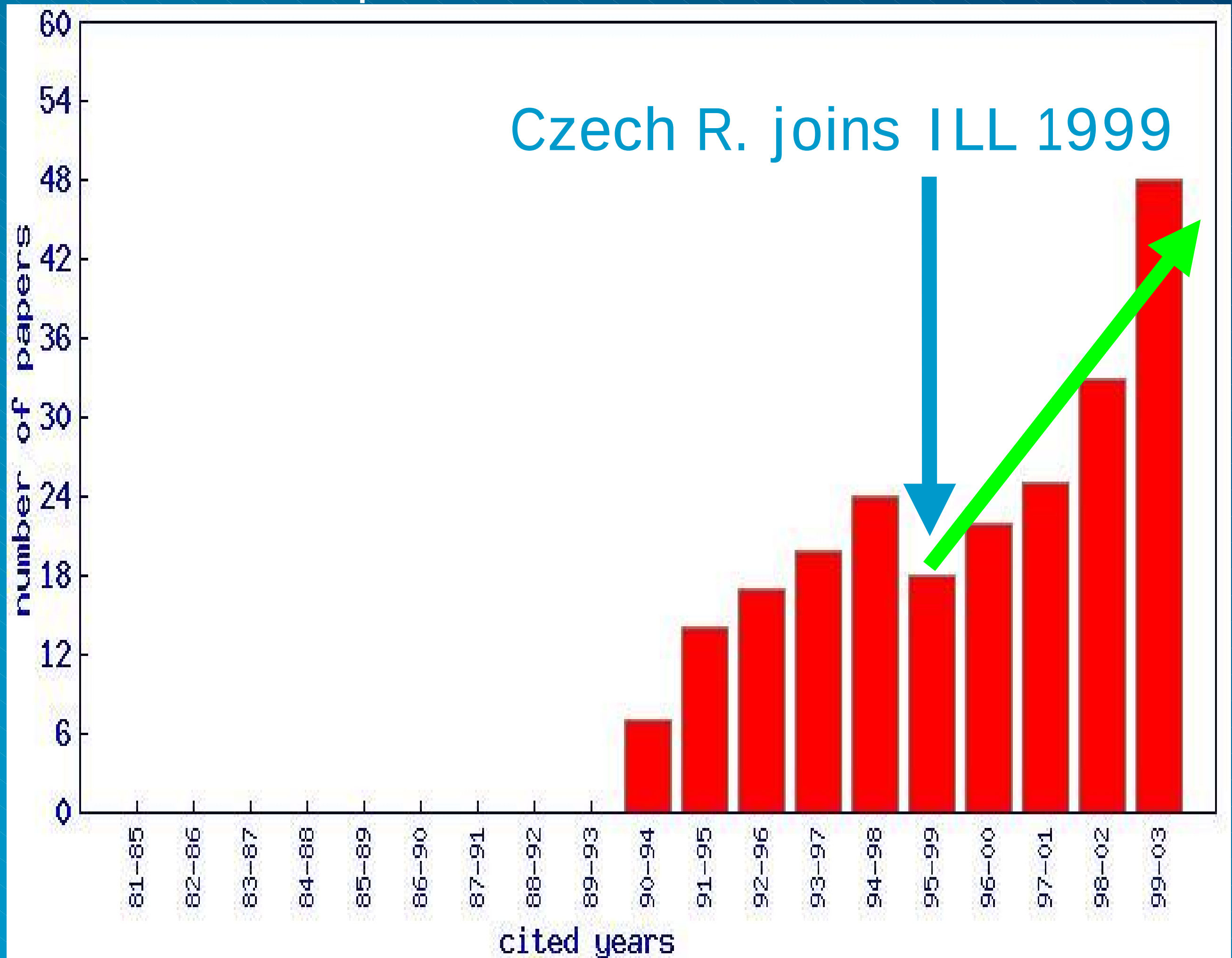

Number of cited Czech & Hungarian papers with ILL

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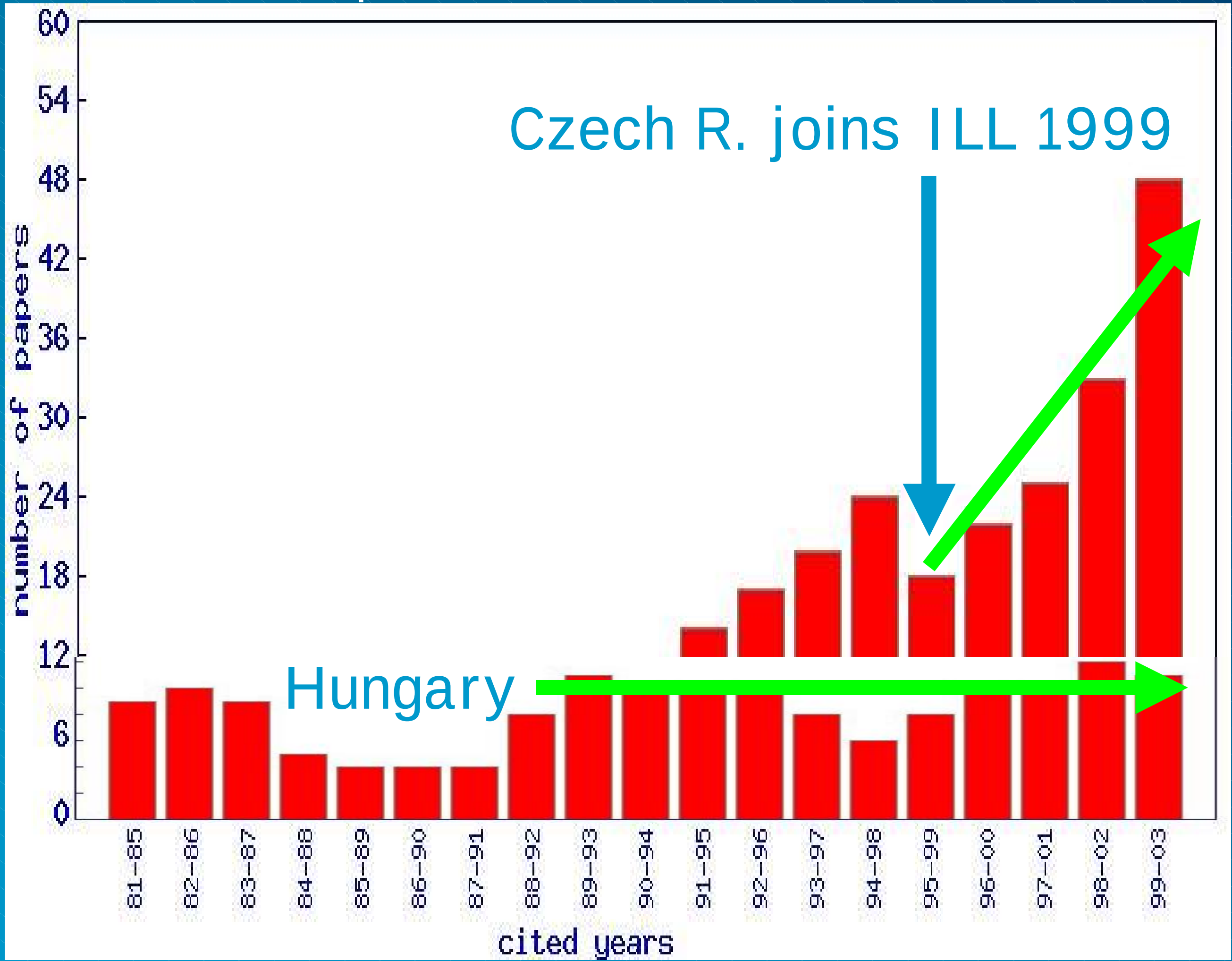

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Grunwaldzki Bridge, Krakow (photo: P.Prokop)