

Introduction to Neutron Diffraction



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Outline

- 1. Characteristics of neutrons for diffraction**
- 2. Diffraction equations: Laue conditions**
- 3. Comparison neutrons – synchrotron X-rays**
- 4. Magnetic neutron diffraction**
- 5. Examples of neutron diffraction studies**


Neutrons for what?

Neutrons tell you
 “where the atoms
 are and what the
 atoms do”
 (Nobel Prize
 citation for
 Brockhouse and
 Shull 1994)

The Nobel Prize in Physics 1994



Ogilvie G. Shull, MIT, Cambridge, Massachusetts, USA, winner one half of the 1994 Nobel Prize in Physics for development of the neutron diffraction technique.



S Shull made use of elastic scattering, i.e. of neutrons which change direction without losing energy when they collide with atoms.

Because of the wave nature of neutrons, a diffraction pattern can be recorded which indicates where in the sample the atoms are situated. Even the placing of light electrons such as hydrogen in metallic hydrides, or hydrogen, carbon and oxygen in organic substances can be determined.

The pattern also shows how atoms behave are situated in magnetic materials, since neutrons are affected by magnetic forces. Shull also made use of this phenomenon in his neutron diffraction technique.





Photo: G. Shull, MIT, Cambridge, Massachusetts, USA


Neutrons see more than X-rays

X-rays are scattered by electron shells. For atoms with a high atomic number, the scattering is strong. For atoms with a low atomic number, the scattering is weak. Neutrons, on the other hand, are scattered by the nucleus. This means that they can see all atoms, whether they have a high or low atomic number.




Neutrons reveal laser stresses

It tells the bond length in an atomic crystal about how much it has changed under stress. This is important for understanding the mechanical properties of materials.




Neutrons show what atoms remember

They can see what happens to atoms in a material over time. This is useful for studying the dynamics of materials.



The Royal Swedish Academy of Sciences has awarded the 1994 Nobel Prize in Physics for pioneering contributions to the development of neutron scattering techniques for studies of condensed matter.

Brockhouse, Brockhouse University, Hamilton, Ontario, Canada, winner one half of the 1994 Nobel Prize in Physics for the development of neutron spectroscopy.



B Brockhouse made use of inelastic scattering, i.e. of neutrons, which change both direction and energy when they collide with atoms. They then start to vibrate, atomic oscillations in crystals and several movements in liquids and solids. Neutrons can also interact with spin waves in magnets.


With his 3-axis spectrometer Brockhouse measured energies of phonons (atomic vibrations) and magnons (magnetic waves). He also studied how atoms vibrate in liquids through with time.

Neutrons reveal structure and dynamics

Neutrons behave as particles and as waves


Neutrons show where atoms are

When the neutrons collide with atoms in the sample material, they change direction and sometimes - elastic scattering.



Calculations reveal the structure of the material and a diffraction pattern is obtained. The pattern gives the positions of the atoms relative to one another.

Neutrons bounce against atomic nuclei. They also react to the magnetism of the atoms.



Create the ions and for every thousand of a certain wavelength energy - more complicated reactions.





Photo: Brockhouse


Neutrons show what atoms do

Such an experiment with neutron systems and neutron beams.



When the neutrons penetrate the sample they start to vibrate. In the crystal, if the neutrons vibrate phonons or magnons they transfer energy to the sample. This energy then shows up as heat.


Changes in the energy of the neutrons are just another way of looking at it.



...and the neutrons have scattered in a detector.

Shull is thanked Brockhouse and Shull made their pioneering contributions to the first neutron reactor in the USA and Canada back in the 1950s and 1960s. It was then that the neutron became available for scientific research.

... See it ourselves There are over 30 neutron scattering facilities in the world. New and even advanced neutron scattering facilities have been built and are on the way in Europe, the USA and Asia. In these facilities, the neutrons are produced by the fission of low enriched uranium, natural or enriched uranium or by spallation of protons on a target. The neutrons are then moderated to the energy and flux required for the experiment.



Further reading:
 The Nobel Prize in Physics 1994
 The Nobel Prize in Physics 1994
 The Nobel Prize in Physics 1994

Particle-wave properties energy- velocity-wavelength ...

kinetic energy (E) velocity (v) temperature (T).

$$E = m_n v^2 / 2 = k_B T = p^2 / 2m_n = (\hbar k)^2 / 2m_n = (h/\lambda)^2 / 2m_n$$

momentum (p) $p = m_n v = \hbar k$

$$\hbar = h / 2\pi$$

wavevector (k) $k = 2\pi / \lambda = m_n v / \hbar$

wavelength (λ)

Neutrons, a powerful probe

Matter is made up atoms, aggregated together in organised structures

The properties of matter and materials are largely determined by their structure and dynamics (behaviour) on the **atomic scale**

distance between atoms $\sim 1 \text{ \AA} = 1/100\,000\,000 \text{ cm}$

Atoms are too small to be seen with ordinary light
(wavelength approx. 4000-8000 \AA)

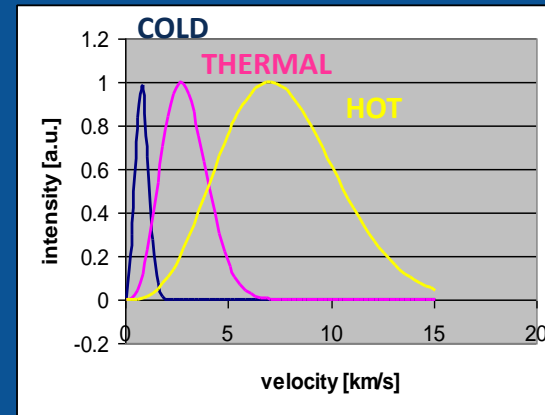
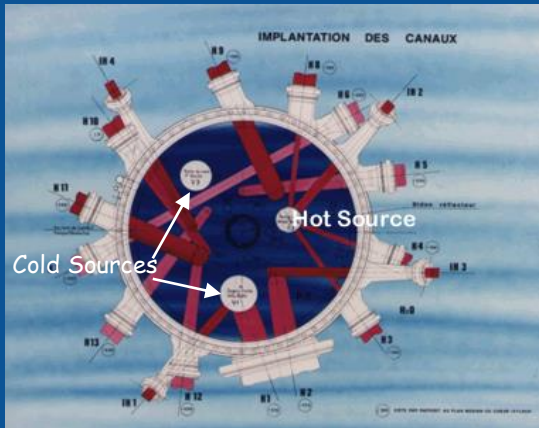
- **The wavelength** of the neutron is comparable to atomic sizes and the dimensions of atomic structures, which explains why neutrons can « see » atoms.
- **The energy** of thermal neutrons is similar to the thermal excitations in solids.
- Neutrons are **zero-charge particles** and have a **magnetic moment** that interacts with the magnetic dipoles in matter.

Techniques using neutrons can produce a picture of atomic and magnetic structures and their motion.

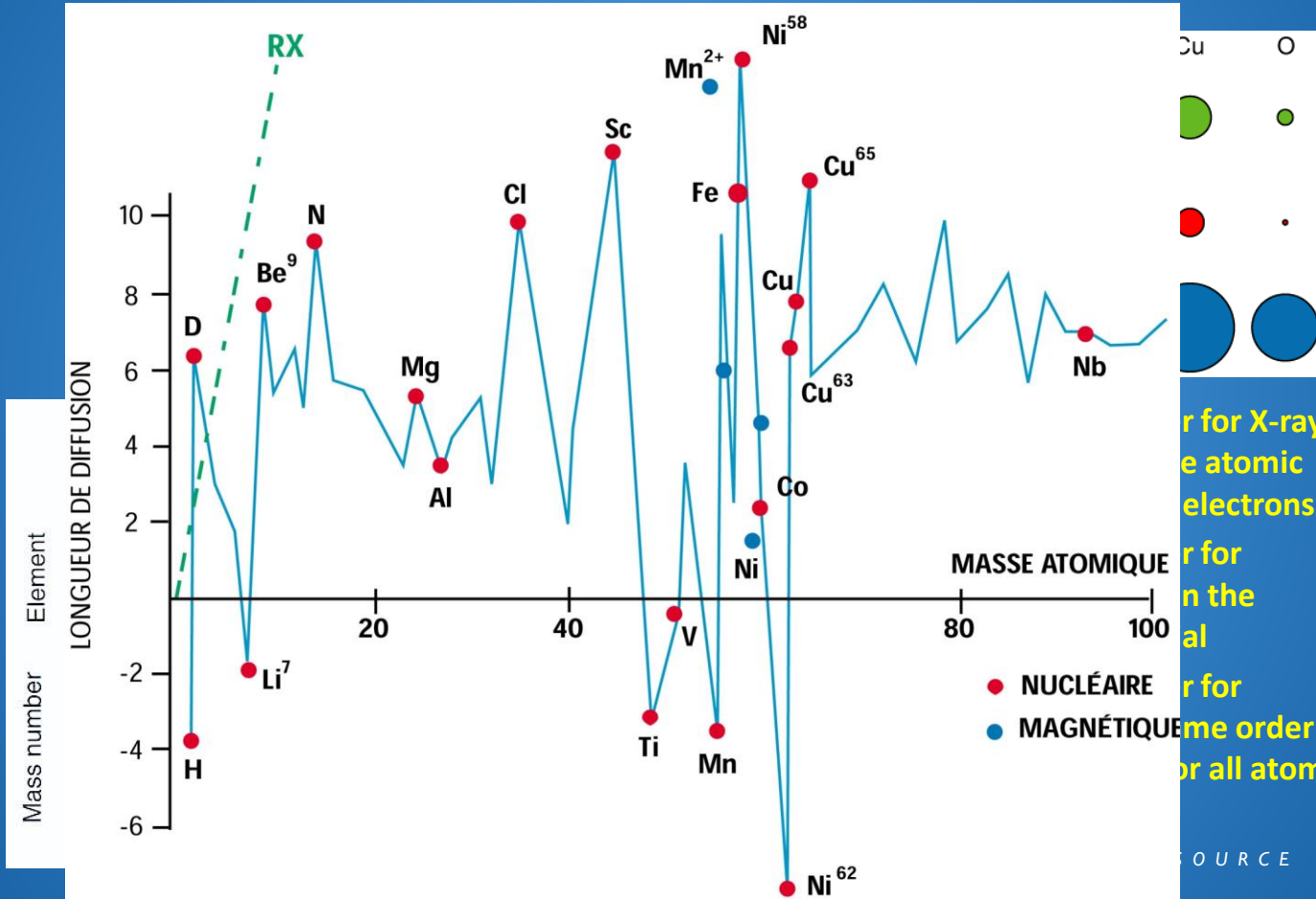
Particle-wave properties (Energy-Temperature-Wavelength)

$$E = m_n v^2 / 2 = k_B T = (\hbar k)^2 / 2m_n ; \quad k = 2\pi / \lambda = m_n v / \hbar$$

	<u>Energy (meV)</u>	<u>Temp (K)</u>	<u>Wavelength (Å)</u>
Cold	0.1 - 10	1 - 120	4 - 30
Thermal	5 - 100	60 - 1000	1 - 4
Hot	100 - 500	1000 - 6000	0.4 - 1



Scattering power of nuclei for neutrons



for X-rays
 e atomic
 electrons).
 for
 n the
 al
 for
 me order of
 or all atoms

SOURCE



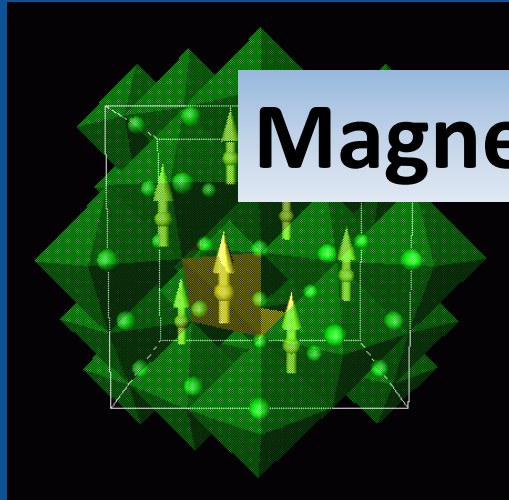
Neutrons for magnetism studies

Neutrons are strongly scattered by magnetic materials

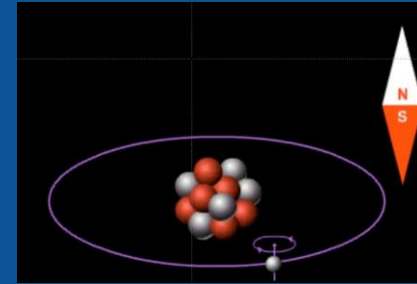
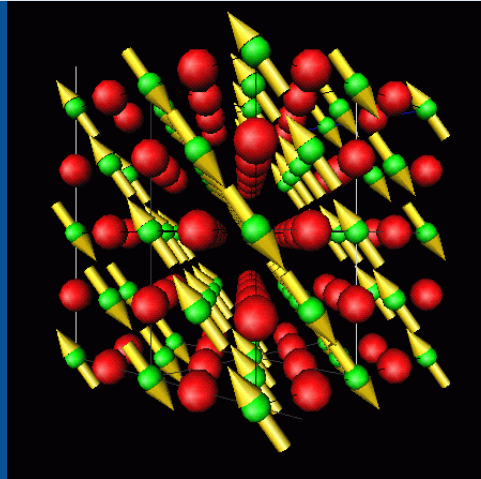
- Neutrons act as small magnets
- The dipolar magnetic moment of the neutron interacts strongly with the atomic magnetic moment
- Neutrons allow the determination of magnetic

Magnetic Crystallography

measure the
precision.



Ferromagnetic and antiferromagnetic oxides



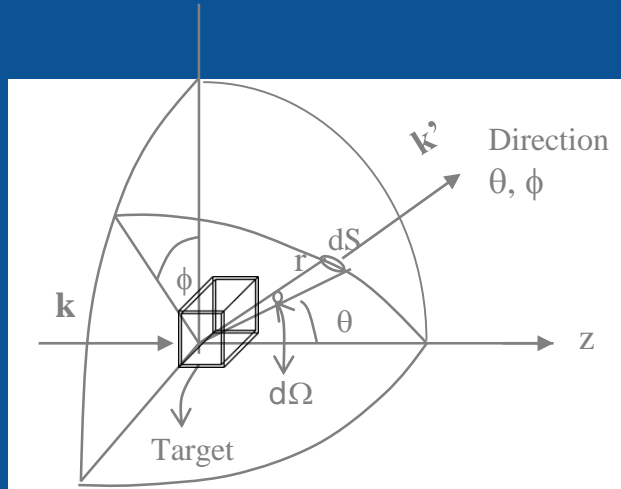
Outline

1. Characteristics of neutrons for diffraction
- 2. Diffraction equations: Laue conditions**
3. Comparison neutrons – synchrotron X-rays
4. Magnetic neutron diffraction
5. Examples of neutron diffraction studies

Interaction neutron-nucleus

Φ = number of incident neutrons /cm²/ second

σ = total number of neutrons scattered/second/ Φ



Fermi's golden rule gives the neutron-scattering Cross-section

→ number of neutrons of a given energy scattered per second in a given solid angle

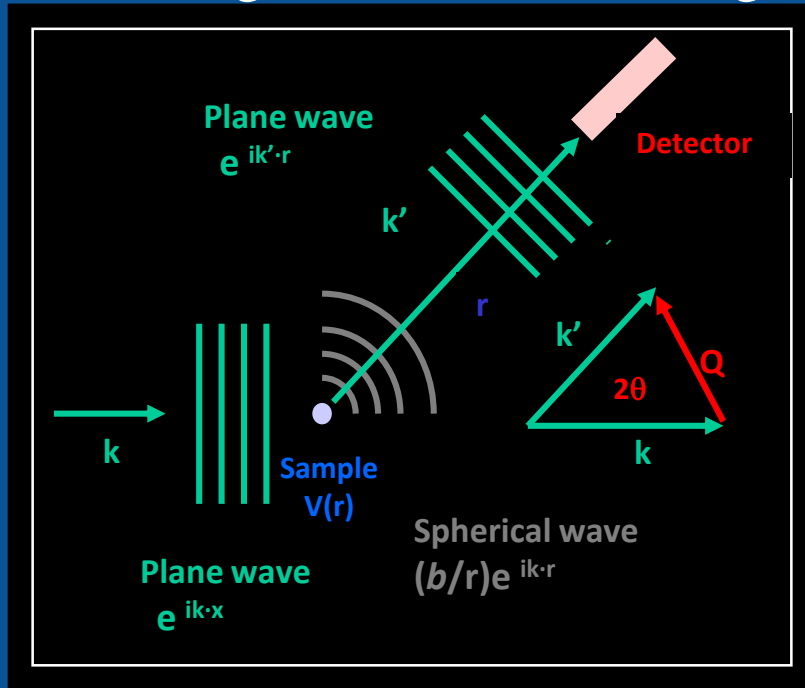
(the effective area presented by a nucleus to an incident neutron)

$$\frac{d^2\sigma}{d\Omega dE'} = \frac{k'}{k} \left(\frac{m}{2\pi\hbar^2} \right)^2 \sum_{\lambda,\sigma} p_{\lambda} p_{\sigma} \sum_{\lambda',\sigma'} \left| \langle \vec{k}' \sigma' \lambda' | V | \vec{k} \sigma \lambda \rangle \right|^2 \delta(\hbar\omega + E_{\lambda} - E_{\lambda'})$$

Interaction neutron-nucleus

Weak interaction with matter aids interpretation of scattering data

The range of nuclear force ($\sim 1\text{fm}$) is much less than neutron wavelength so that scattering is “point-like”



- Fermi Pseudo potential of a nucleus in \mathbf{r}_j

$$V_j = \frac{2\pi\hbar^2}{m} b_j \delta(\mathbf{r} - \mathbf{r}_j)$$

Potential with a single parameter

Diffraction Equations

For diffraction part of the scattering the Fermi's golden rule resumes to the statement: **the diffracted intensity is the square of the Fourier transform of the interaction potential**

$$|\mathbf{k}'| = |\mathbf{k}| = 2\pi / \lambda$$

$$A(\mathbf{Q}) = \int V(\mathbf{r}) \exp(i\mathbf{Q}\cdot\mathbf{r}) d^3\mathbf{r} \rightarrow I(\mathbf{Q}) = A(\mathbf{Q})A^*(\mathbf{Q}) = |A(\mathbf{Q})|^2$$

$$\mathbf{Q} = \mathbf{k}' - \mathbf{k} = 2\pi(\mathbf{s} - \mathbf{s}_0) / \lambda = 2\pi\mathbf{s} = 2\pi\mathbf{h}$$

There are different conventions and notations for designing the scattering vector (we use here crystallographic conventions).

$$A_X(\mathbf{s}) = \int \sum \rho_{ej}(\mathbf{r}) \delta(\mathbf{r} - \mathbf{R}_j) \exp(2\pi i \mathbf{s} \cdot \mathbf{r}) d^3\mathbf{r} = \sum f_j(\mathbf{s}) \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_j)$$

$$f_j(\mathbf{s}) = \int \rho_{ej}(\mathbf{r}) \exp(2\pi i \mathbf{s} \cdot \mathbf{r}) d^3\mathbf{r} \quad \leftarrow \text{Atomic form factor.}$$

Scattering length

$$A_N(\mathbf{s}) = \frac{2\pi\hbar^2}{m} \int \sum b_j \delta(\mathbf{r} - \mathbf{R}_j) \exp(2\pi i \mathbf{s} \cdot \mathbf{r}) d^3\mathbf{r} \sim \sum b_j \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_j)$$

Diffraction Equations for crystals

In a crystal the atoms positions can be decomposed as the vector position of the origin of a unit cell plus the vector position with respect to the unit cell

$$\mathbf{R}_{lj} = \mathbf{R}_l + \mathbf{r}_j$$

$$A_N(\mathbf{s}) = \sum_{lj} b_j \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_{lj}) = \sum_l \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_l) \sum_{j=1,n} b_j \exp(2\pi i \mathbf{s} \cdot \mathbf{r}_j)$$

$$\sum_l \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_l) = 0 \quad \text{for general } \mathbf{s}$$

$$\sum_l \exp(2\pi i \mathbf{s} \cdot \mathbf{R}_l) = N \quad \text{for } \mathbf{s} = \mathbf{H} \rightarrow \mathbf{H}\mathbf{R}_l = L_H \text{ integer}$$

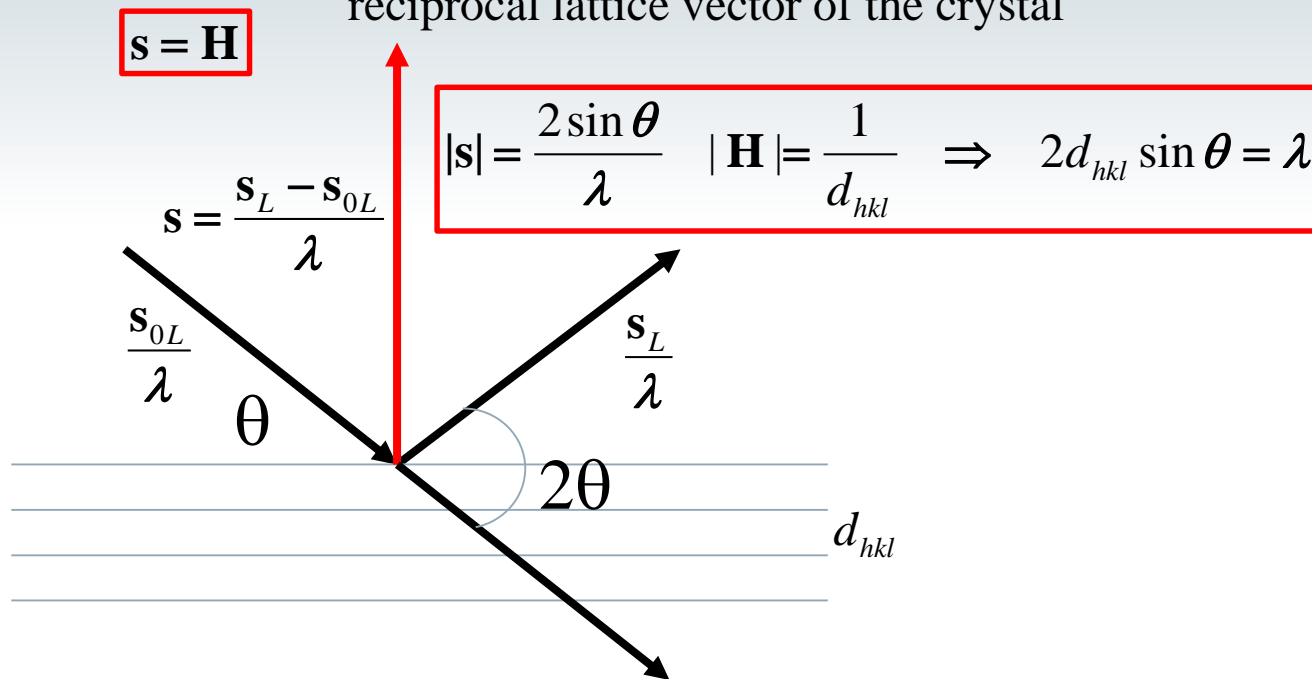
$\mathbf{s} = \mathbf{H}$ Laue conditions: the scattering vector is a reciprocal lattice vector of the crystal

$$I_N(\mathbf{H}) \sim \left| \sum_{j=1,n} b_j \exp(2\pi i \mathbf{H} \cdot \mathbf{r}_j) \right|^2 = |F(\mathbf{H})|^2$$

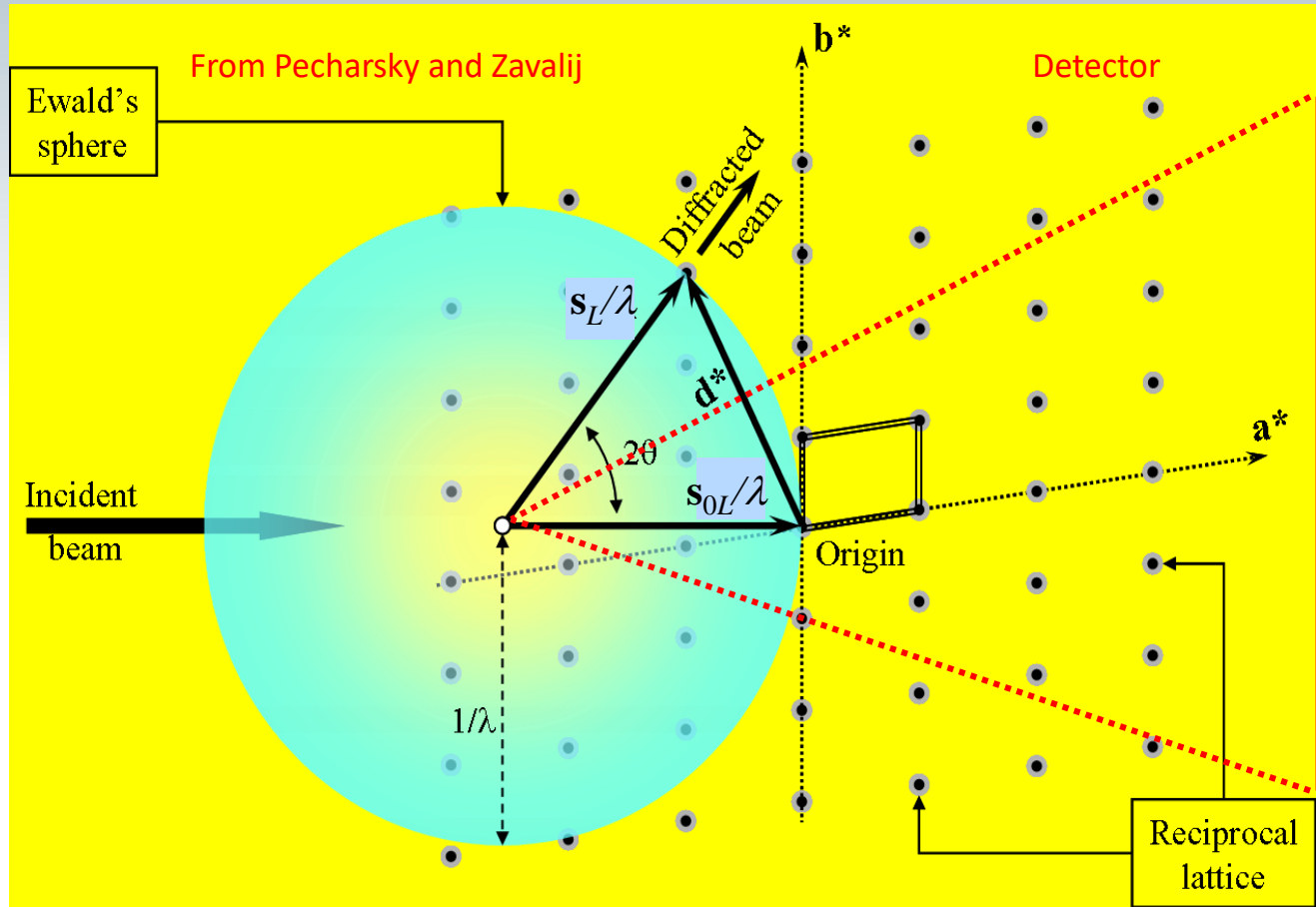
Diffraction Equations for crystals

The Laue conditions have as a consequence the Bragg Law

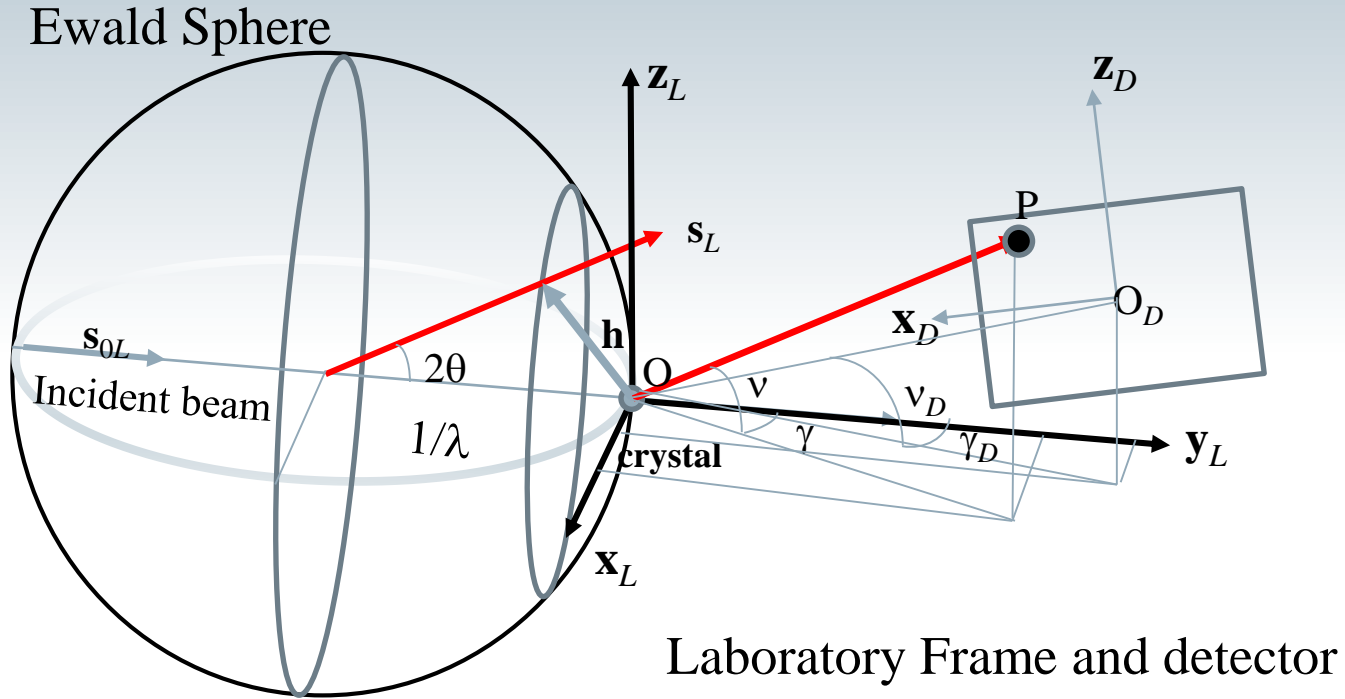
Laue conditions: the scattering vector is a reciprocal lattice vector of the crystal



Ewald construction



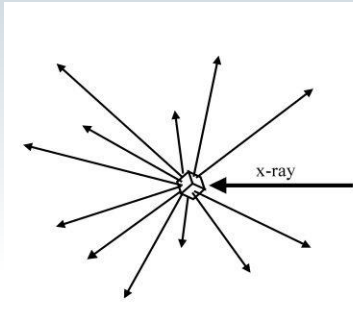
Ewald construction



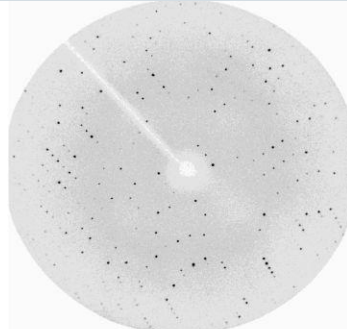
Diffraction patterns

Single Xtal - 2D image + scan \rightarrow 3D Int vs 2θ

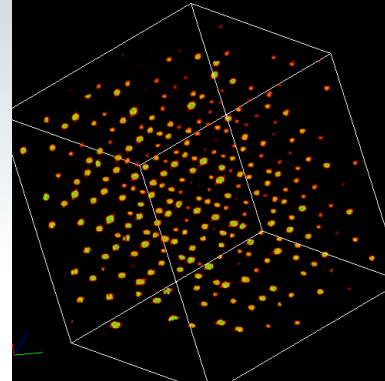
Powder - 2D image \rightarrow 1D Int vs 2θ



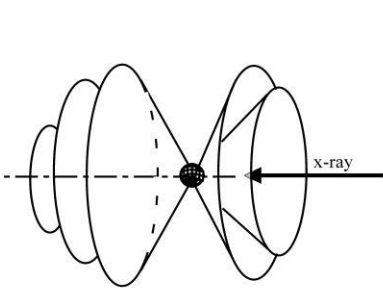
(a)



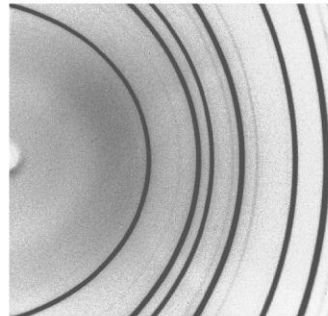
(b)



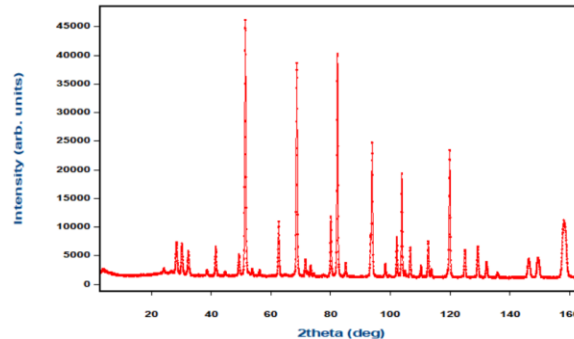
Single
Crystal



(c)



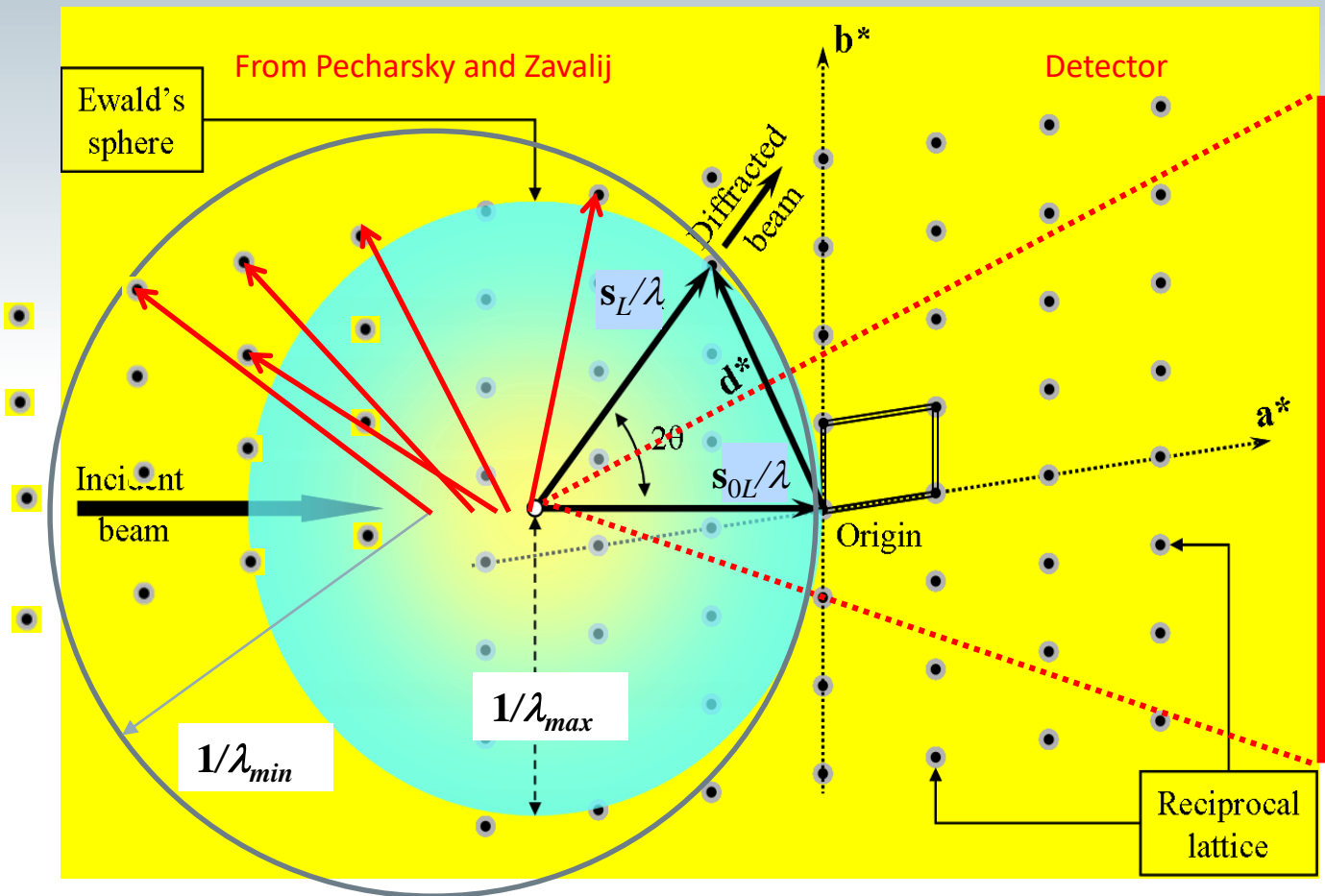
(d)



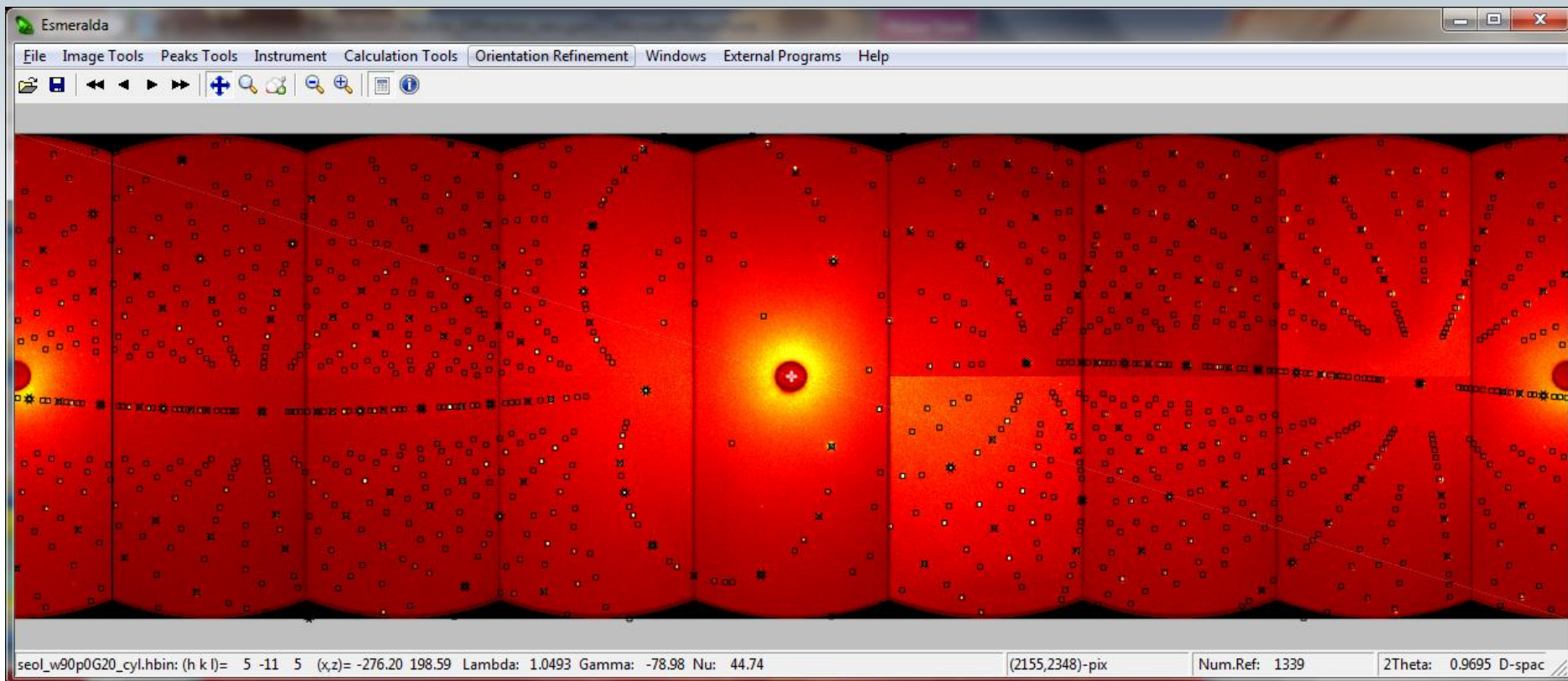
Powder or
polycrystalline
solid

Courtesy of Jim

Ewald construction Laue



Laue image obtained in Cyclops



Single Crystal and Powder Diffraction

Single Crystal diffraction allows to get with high precision subtle structural details: thermal parameters, anharmonic vibrations.

Drawbacks: big crystals for neutrons, extinction, twinning

Data reduction: Needs only the indexing and integration of Bragg reflection and obtain structure factors. List: $h k l$ F^2 $\sigma(F^2)$

Data Treatment: SHELX, FullProf, JANA, GSAS, ...

Powder diffraction no problem with extinction or twinning.

Data reduction: minimalistic, needs only the profile intensities and their standard deviations

Data Treatment: FullProf, JANA, GSAS, TOPAS, ...

$$y_{ci} = \sum_{\{h\}} I_h \Omega (T_i - T_h) + b_i$$

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Why neutrons?

NEUTRON DIFFRACTION FOR FUNDAMENTAL AND APPLIED RESEARCH IN CONDENSED MATTER AND MATERIALS SCIENCE

Location of light elements and distinction between adjacent elements in the periodic table.

Examples are:

Oxygen positions in High- T_C superconductors and manganites

Structural determination of fullerenes and their derivatives,

Hydrogen in metals and hydrides

Lithium in battery materials

Determination of atomic site distributions in solid solutions

Systematic studies of hydrogen bonding

Host-guest interactions in framework silicates

Role of water in crystals

Magnetic structures, magnetic phase diagrams and magnetisation densities

Relation between static structure and dynamics (clathrates, plastic crystals).

Aperiodic structures: incommensurate structures and quasicrystals

Why neutrons?

The complementary use of X-ray Synchrotron radiation and neutrons (1)

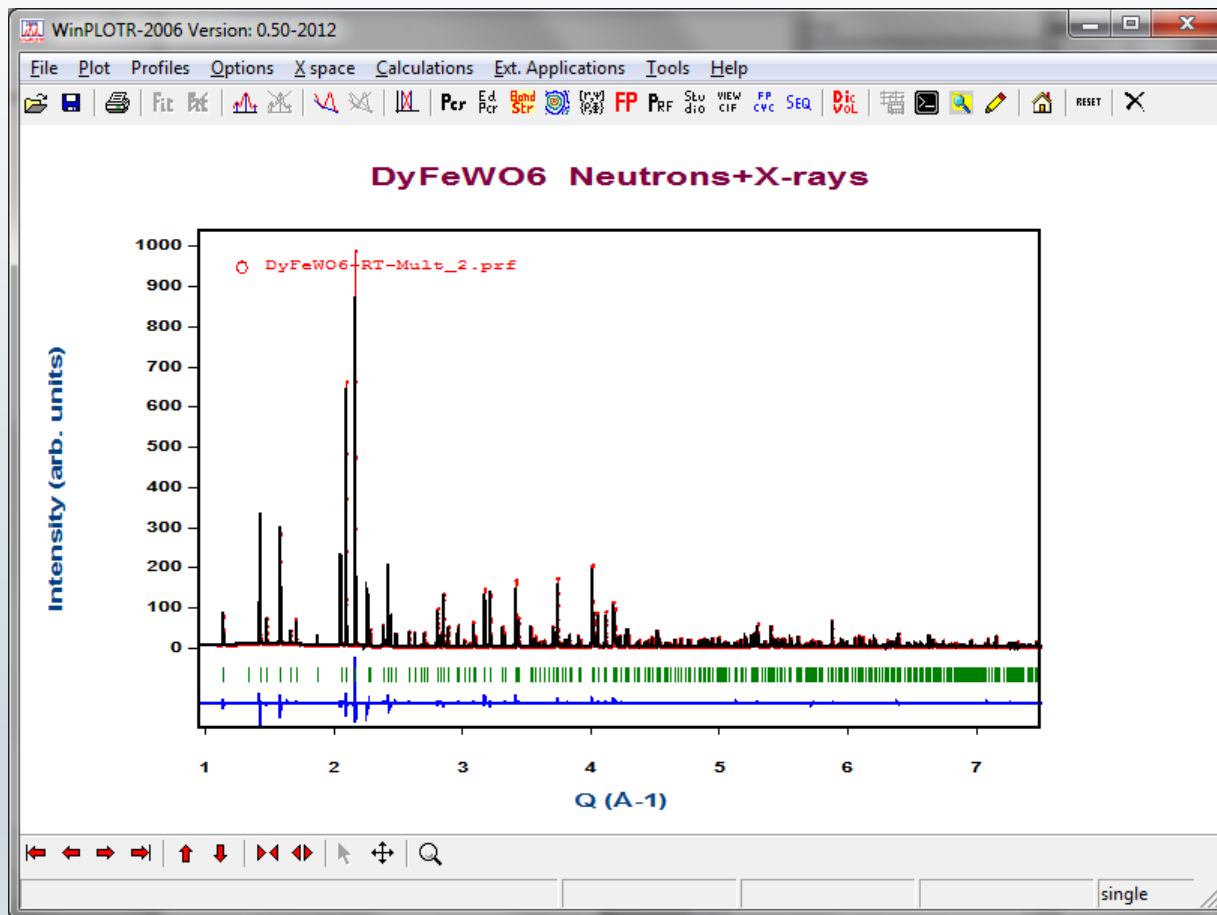
The **advantages of thermal neutrons** with respect to X-rays as far as diffraction is concerned are based on the following properties of thermal neutrons:

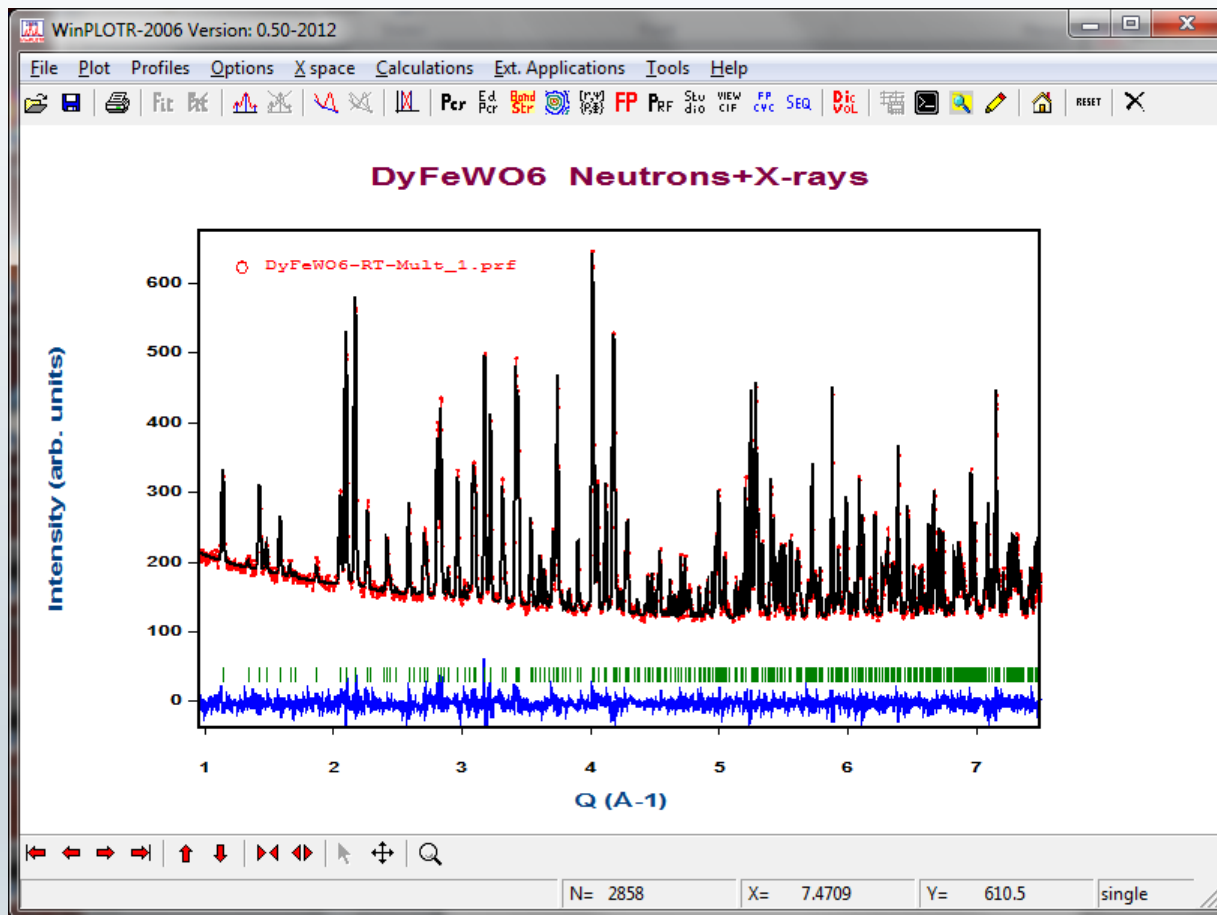
- **constant scattering power** (b is Q -independent) having a non-monotonous dependence on the atomic number
- **weak interaction** (the first Born approximation holds) that implies simple theory can be used to interpret the experimental data
- **the magnetic interaction is of the same order of magnitude as the nuclear interaction**
- **low absorption**, making it possible to use complicated sample environments

Why neutrons?

The complementary use of X-ray Synchrotron radiation and neutrons (2)

- Powder diffraction with SR can be used for *ab initio* structure **determination** and microstructural analysis due to the current extremely high Q-resolution.
- **Structure refinement is better done with neutrons** (or using simultaneously both techniques) because systematic errors in intensities (texture effects) are less important and because scattering lengths are Q-independent in the neutron case.





Why neutrons?

The complementary use of X-ray Synchrotron radiation and neutrons (3)

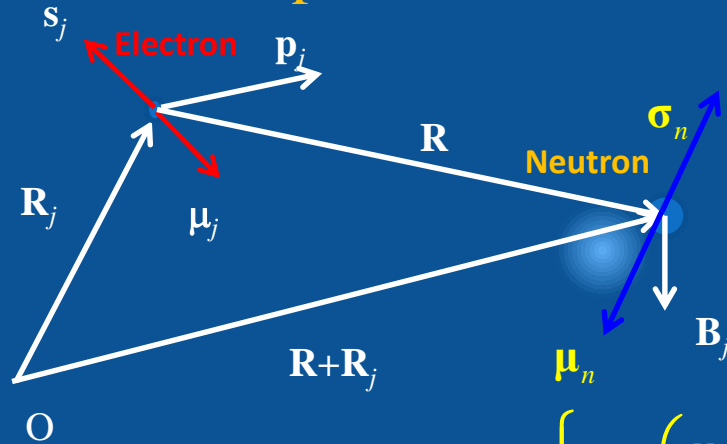
- Magnetic X-ray scattering allows in principle the separation of orbital and spin components. However, **SR cannot compete with neutrons in the field of *magnetic structure determination*** from powders.
- The contribution of SR to that field is on details of magnetic structures (already known from neutrons) for selective elements using resonant magnetic scattering (rare earths, U, ...)

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Magnetic scattering: magnetic fields

The interaction potential to be evaluated in the FGR is: $V_m^j = \boldsymbol{\mu}_j \mathbf{B}_j$
 Magnetic field due to spin and orbital moments of an electron:



Magnetic vector potential of a dipolar field due to electron spin moment

$$\mathbf{B}_j = \mathbf{B}_{jS} + \mathbf{B}_{jL} = \frac{\mu_0}{4\pi} \left\{ \nabla \times \left(\frac{\boldsymbol{\mu}_j \times \hat{\mathbf{R}}}{R^2} \right) - \frac{2\mu_B}{\hbar} \frac{\mathbf{p}_j \times \hat{\mathbf{R}}}{R^2} \right\}$$

Biot-Savart law for a single electron with linear momentum \mathbf{p}

Magnetic scattering: magnetic fields

Evaluating the spatial part of the transition matrix element for electron j :

$$\langle \mathbf{k}' | V_m^j | \mathbf{k} \rangle = \exp(i\mathbf{Q}\mathbf{R}_j) \left\{ \mathbf{e} \times (\mathbf{s}_j \times \mathbf{e}) + \frac{i}{\hbar Q} (\mathbf{p}_j \times \mathbf{e}) \right\} \quad \mathbf{e} = \frac{\mathbf{Q}}{Q}$$

Where $\hbar\mathbf{Q} = \hbar(\mathbf{k} - \mathbf{k}')$ is the momentum transfer

Summing for all unpaired electrons we obtain:

$$\sum_j \langle \mathbf{k}' | V_m^j | \mathbf{k} \rangle = \mathbf{e} \times (\mathbf{M}(\mathbf{Q}) \times \mathbf{e}) = \mathbf{M}(\mathbf{Q}) - (\mathbf{M}(\mathbf{Q}) \cdot \mathbf{e}) \cdot \mathbf{e} = \mathbf{M}_\perp(\mathbf{Q})$$

$\mathbf{M}_\perp(\mathbf{Q})$ is the perpendicular component of the Fourier transform of the magnetisation in the scattering object to the scattering vector. It includes the orbital and spin contributions.

Scattering by a collection of magnetic atoms

We will consider in the following only elastic scattering.

We suppose the magnetic matter made of atoms with unpaired electrons that remain close to the nuclei.

Vector position of electron e : $\mathbf{R}_e = \mathbf{R}_{lj} + \mathbf{r}_{je}$

The Fourier transform of the magnetization can be written in discrete form as

$$\mathbf{M}(\mathbf{Q}) = \sum_e \mathbf{s}_e \exp(i\mathbf{Q} \cdot \mathbf{R}_e) = \sum_{lj} \exp(i\mathbf{Q} \cdot \mathbf{R}_{lj}) \sum_{e_j} \exp(i\mathbf{Q} \cdot \mathbf{r}_{je}) \mathbf{s}_{je}$$

$$\mathbf{F}_j(\mathbf{Q}) = \sum_e \mathbf{s}_{je} \exp(i\mathbf{Q} \cdot \mathbf{r}_{je}) = \int \boldsymbol{\rho}_j(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r}$$

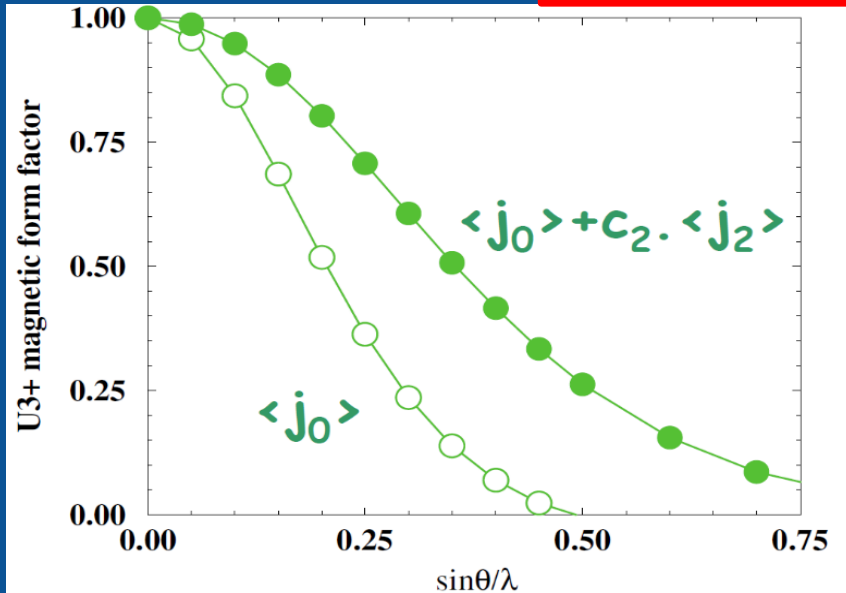
$$\mathbf{F}_j(\mathbf{Q}) = \mathbf{m}_j \int \rho_j(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r} = \mathbf{m}_j f_j(Q)$$

$$\mathbf{M}(\mathbf{Q}) = \sum_{lj} \mathbf{m}_{lj} f_{lj}(Q) \exp(i\mathbf{Q} \cdot \mathbf{R}_{lj})$$

Scattering by a collection of magnetic atoms

$$\mathbf{F}_j(\mathbf{Q}) = \sum_e \mathbf{s}_{je} \exp(i\mathbf{Q} \cdot \mathbf{r}_{je}) = \int \rho_j(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r}$$

$$\mathbf{F}_j(\mathbf{Q}) = \mathbf{m}_j \int \rho_j(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r} = \mathbf{m}_j f_j(Q)$$



If we use the common variable $s = \sin\theta/\lambda$, then the expression of the form factor is the following:

$$f(s) = \sum_{l=0,2,4,6} W_l \langle j_l(s) \rangle$$

$$\langle j_l(s) \rangle = \int_0^\infty U^2(r) j_l(4\pi sr) 4\pi r^2 dr$$

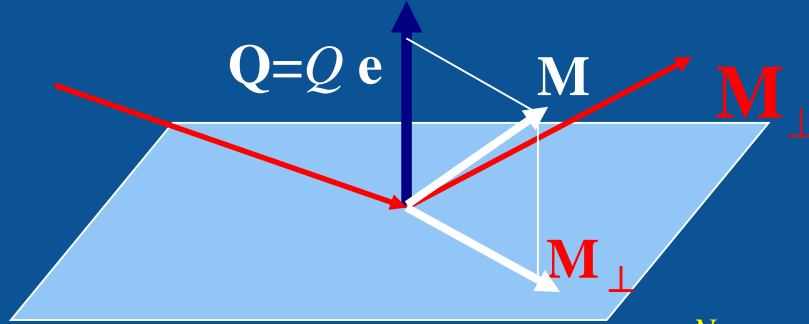
$$\langle j_l(s) \rangle = s^2 \left(A_l \exp\{-a_l s^2\} + B_l \exp\{-b_l s^2\} + C_l \exp\{-c_l s^2\} + D_l \right) \quad \text{for } l = 2, 4, 6$$

$$\langle j_0(s) \rangle = A_0 \exp\{-a_0 s^2\} + B_0 \exp\{-b_0 s^2\} + C_0 \exp\{-c_0 s^2\} + D_0$$

Magnetic scattering

$\mathbf{M}_\perp(\mathbf{Q})$ is the perpendicular component of the Fourier transform of the magnetisation in the sample to the scattering vector.

$$\mathbf{M}(\mathbf{Q}) = \int \mathbf{M}(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r}$$



Magnetic interaction vector
 $= \mathbf{e} \times \mathbf{M} \times \mathbf{e} = \mathbf{M} - \mathbf{e} (\mathbf{e} \cdot \mathbf{M})$

$$\left(\frac{d\sigma}{d\Omega} \right) = (\gamma r_0)^2 \mathbf{M}_\perp^* \mathbf{M}_\perp$$

Magnetic structure factor: $\mathbf{M}(\mathbf{H}) = p \sum_{m=1}^{N_{mag}} \mathbf{m}_m f_m(H) \exp(2\pi i \mathbf{H} \cdot \mathbf{r}_m)$

Neutrons only see the components of the magnetisation that are perpendicular to the scattering vector

Elastic Magnetic Scattering by a crystal

For a general magnetic structure that can be described as a Fourier series:

$$\mathbf{m}_{lj} = \sum_{\{\mathbf{k}\}} \mathbf{S}_{\mathbf{k}j} \exp\{-2\pi i \mathbf{k} \cdot \mathbf{R}_l\}$$

$$\mathbf{M}(\mathbf{h}) = \sum_{lj} \sum_{\mathbf{k}} \mathbf{S}_{\mathbf{k}j} \exp(-2\pi i \mathbf{k} \cdot \mathbf{R}_l) f_{lj}(h) \exp(2\pi i \mathbf{h} \cdot \mathbf{R}_{lj})$$

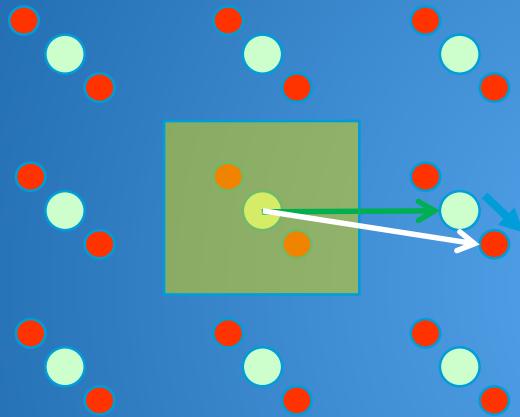
$$\mathbf{M}(\mathbf{h}) = \sum_j f_j(h) \exp(2\pi i \mathbf{h} \cdot \mathbf{r}_j) \sum_{\mathbf{k}} \mathbf{S}_{\mathbf{k}j} \sum_l \exp(2\pi i (\mathbf{h} - \mathbf{k}) \cdot \mathbf{R}_l)$$

$$\mathbf{M}(\mathbf{h}) = \sum_j \mathbf{S}_{\mathbf{k}j} f_j(Q) \exp(2\pi i (\mathbf{H} + \mathbf{k}) \cdot \mathbf{r}_j)$$

The lattice sum is only different from zero when $\mathbf{h} - \mathbf{k}$ is a reciprocal lattice vector \mathbf{H} of the crystallographic lattice. The vector \mathbf{M} is then proportional to the **magnetic structure factor of the unit cell** that now contains the Fourier coefficients $\mathbf{S}_{\mathbf{k}j}$ instead of the magnetic moments \mathbf{m}_j .

Diffraction Patterns of magnetic structures

Portion of reciprocal space



● Magnetic reflections

● Nuclear reflections

$$\mathbf{h} = \mathbf{H} + \mathbf{k}$$

Magnetic reflections: indexed by a set of propagation vectors $\{\mathbf{k}\}$

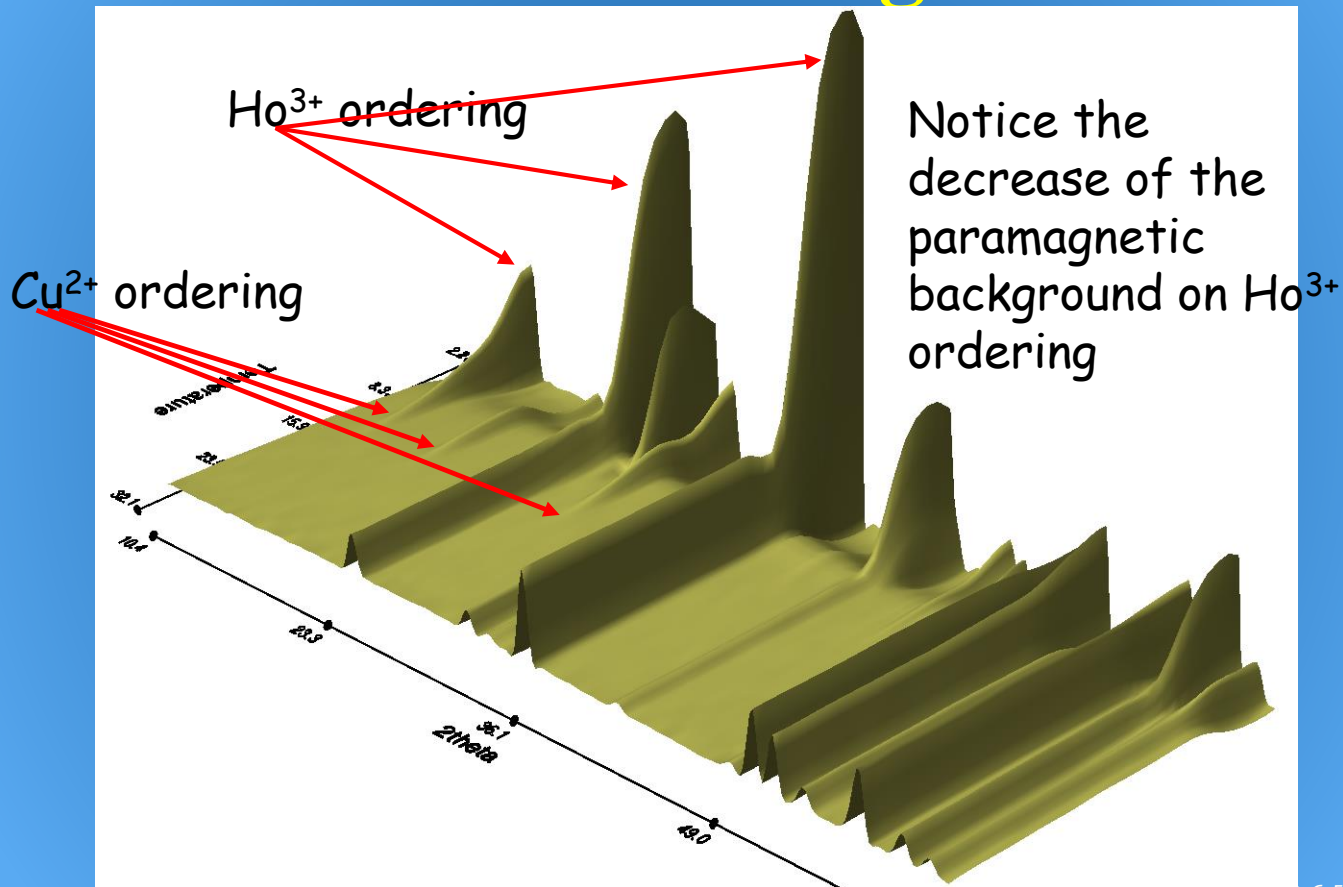
\mathbf{h} is the scattering vector indexing a magnetic reflection

\mathbf{H} is a reciprocal vector of the crystallographic structure

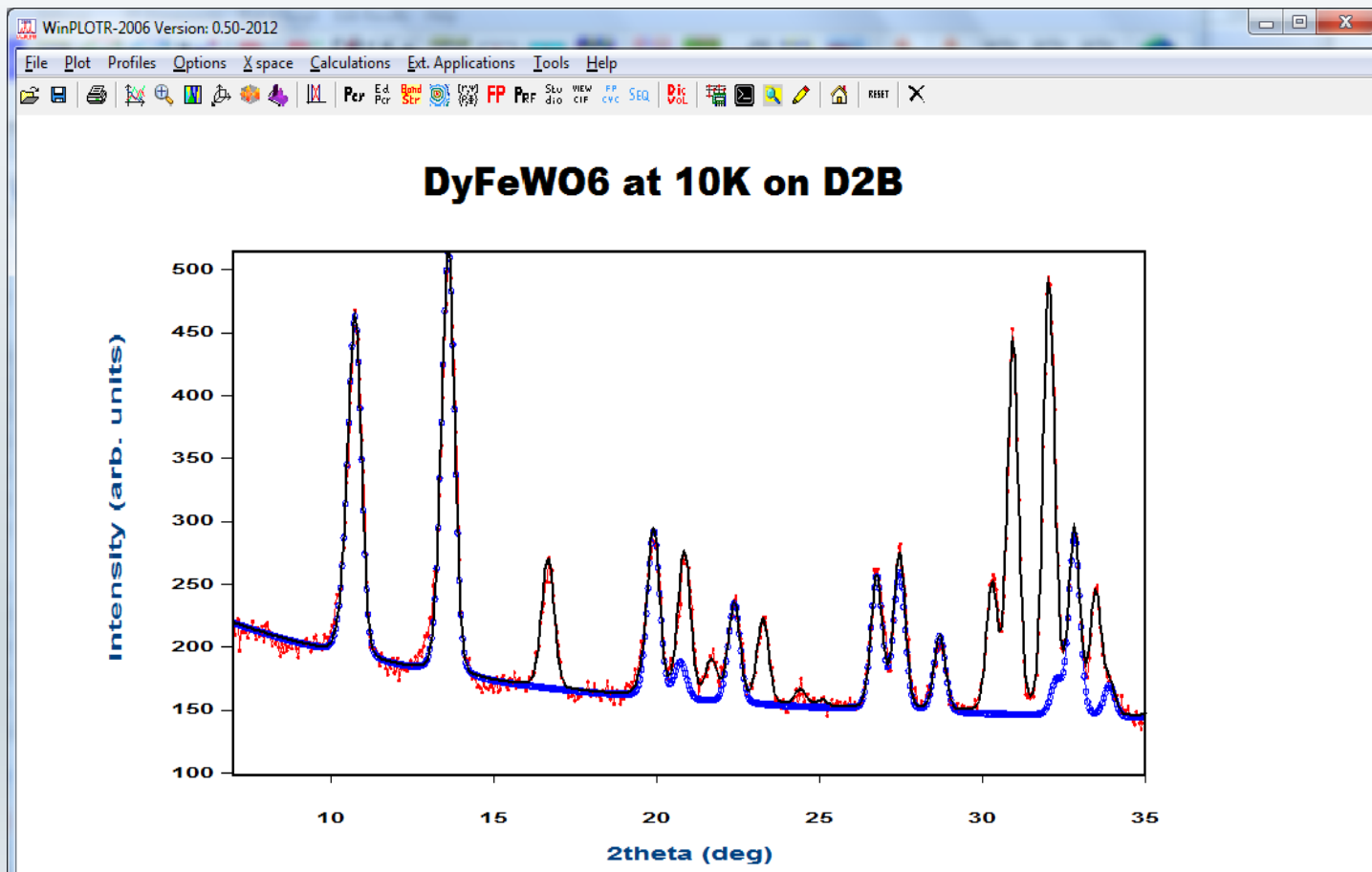
\mathbf{k} is one of the propagation vectors of the magnetic structure

(\mathbf{k} is reduced to the Brillouin zone)

Diffraction Patterns of magnetic structures



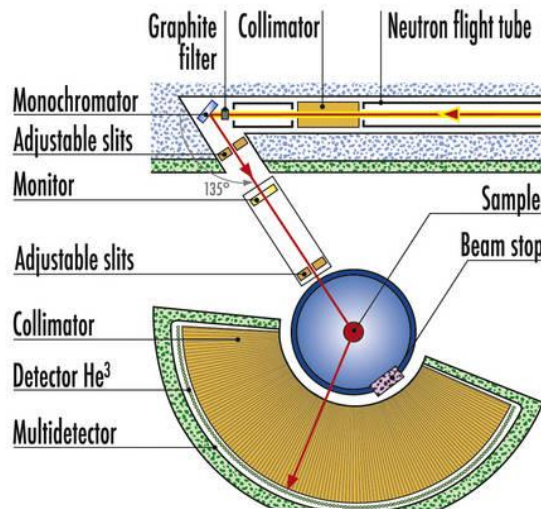
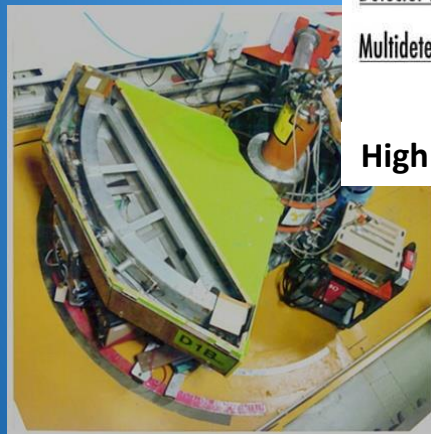
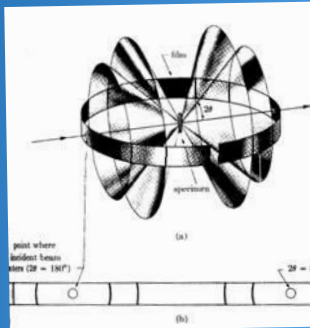
Magnetic refinement on D2B



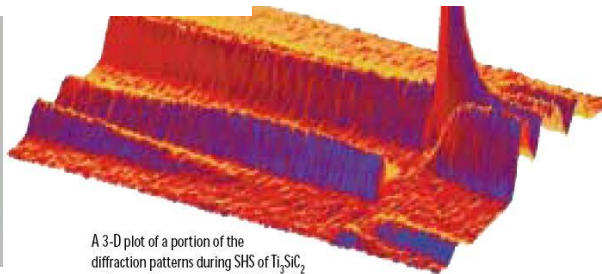
Outline

1. Characteristics of neutrons for diffraction
2. Diffraction equations: Laue conditions
3. Comparison neutrons – synchrotron X-rays
4. Magnetic neutron diffraction
- 5. Examples of neutron diffraction studies**

Two Axes Diffractometers: Powders and Liquids

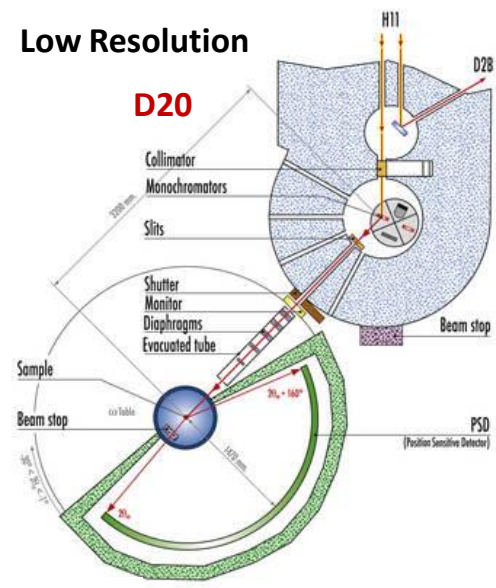


High Resolution D2B



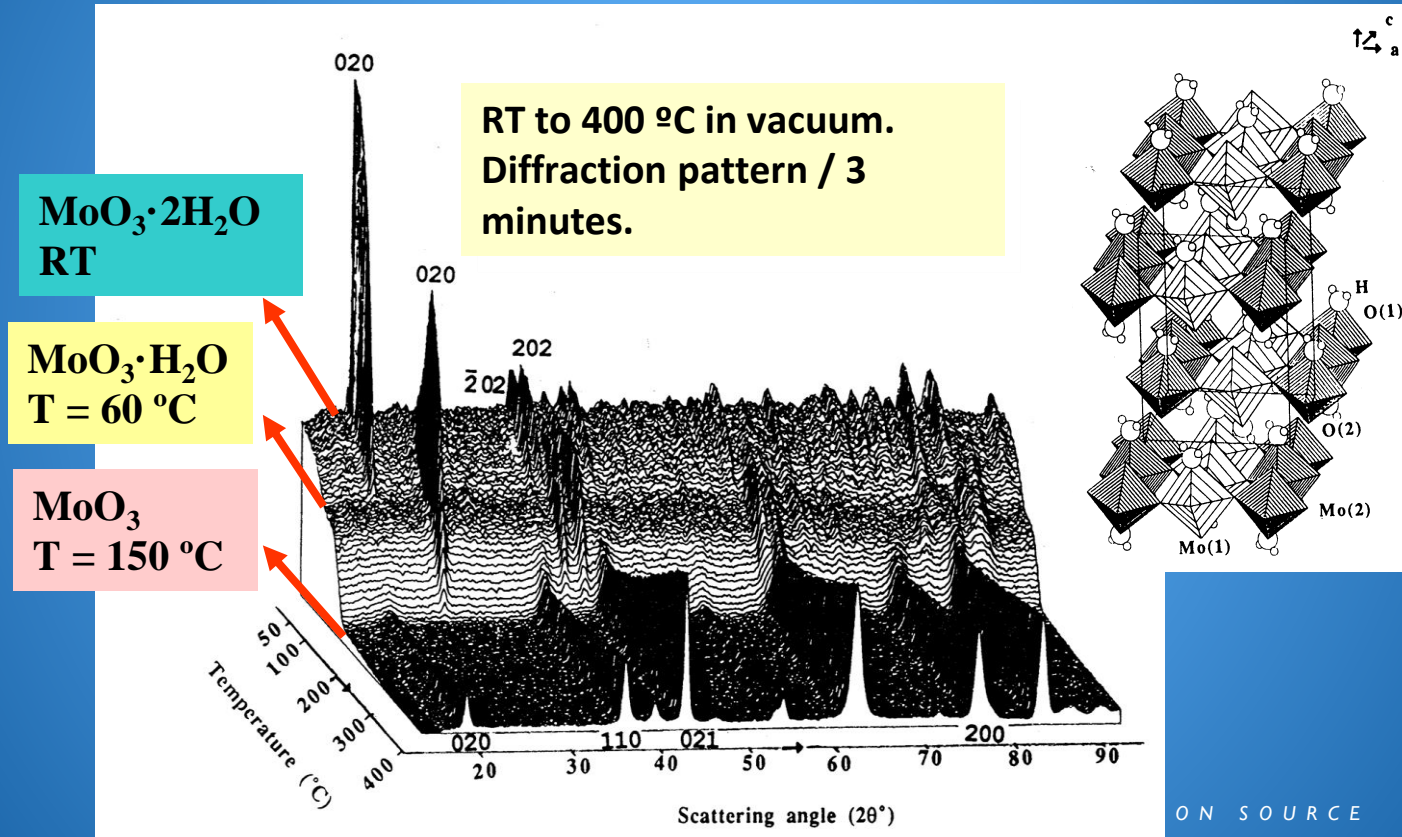
A 3-D plot of a portion of the diffraction patterns during SHS of Ti_5SiC_2 as the reaction progresses (left to right)

Low Resolution

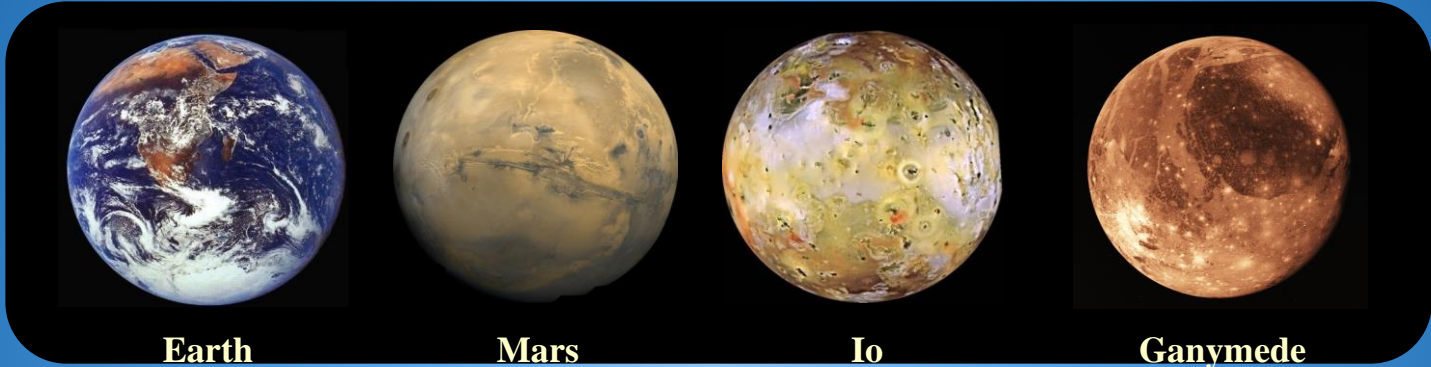


Real time powder diffraction on D1B

Dehydration of $\text{MoO}_3 \cdot 2\text{H}_2\text{O}$ [N. Boudjada et al.; *J. Solid State Chem.* **105**, 211 (1993)]



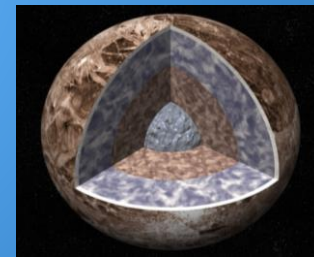
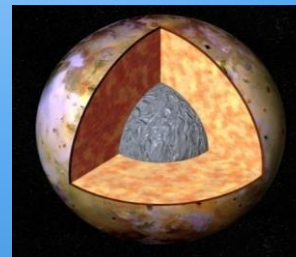
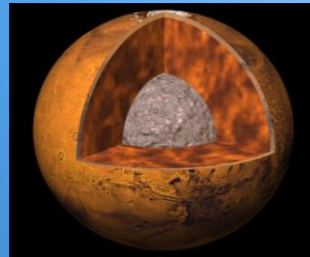
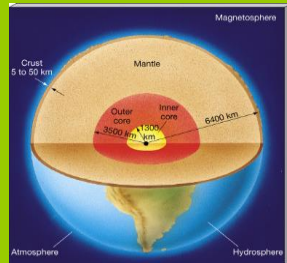
Some Applications: Liquid state → D4



What they have in common ...?

Metallic cores

Proximity in Solar System



Fluid Outer Core: from 3000 to 5200 km

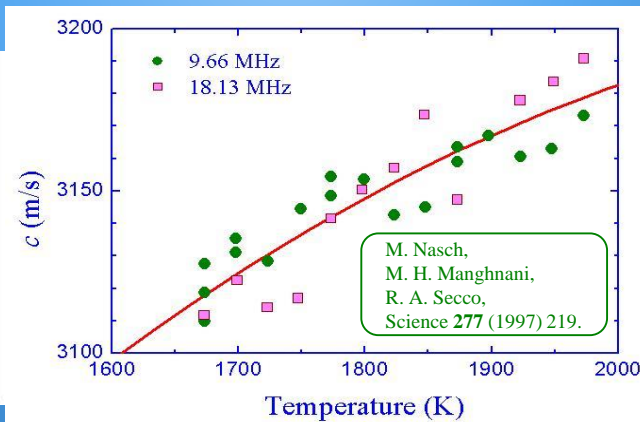
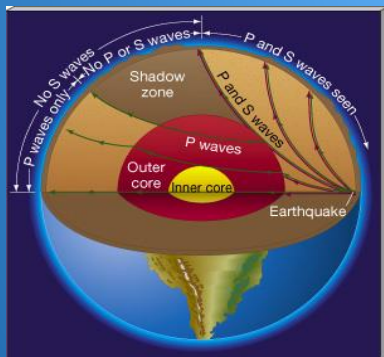
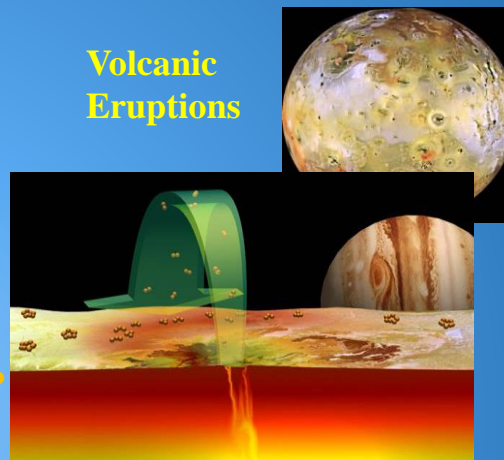
Primarily Fe with some Ni

Some Applications: Liquid state

But ρ is 5-10% less than pure Fe+Ni ...



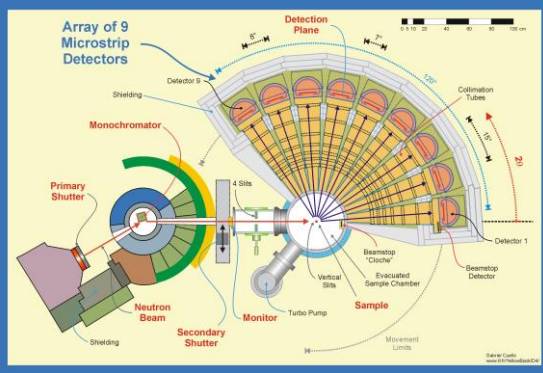
Volcanic Eruptions



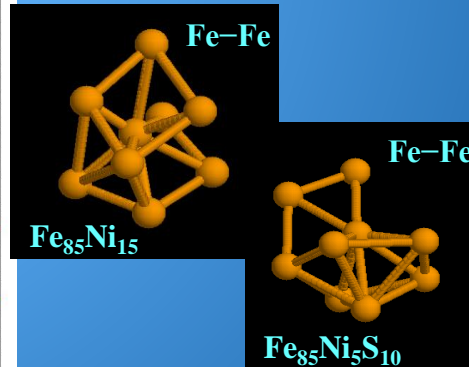
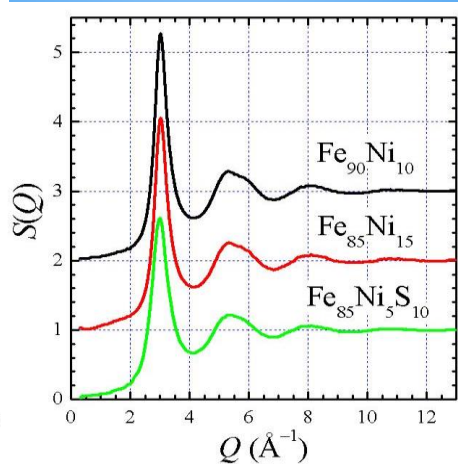
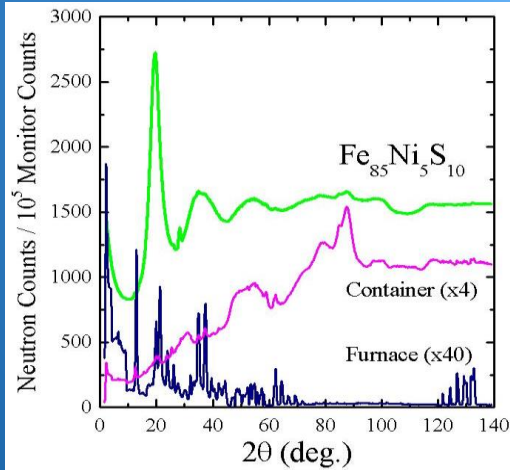
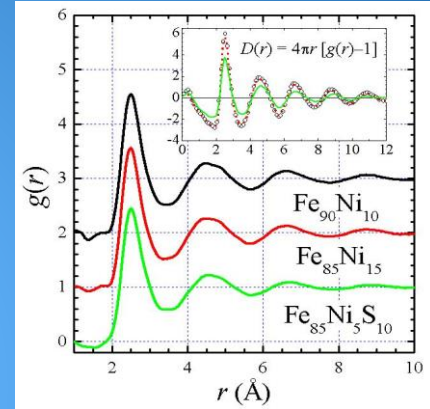
Hypothesis:

The light element helps aggregating clusters, which in turn are disaggregated by heating the system.

Some Applications: Liquid state

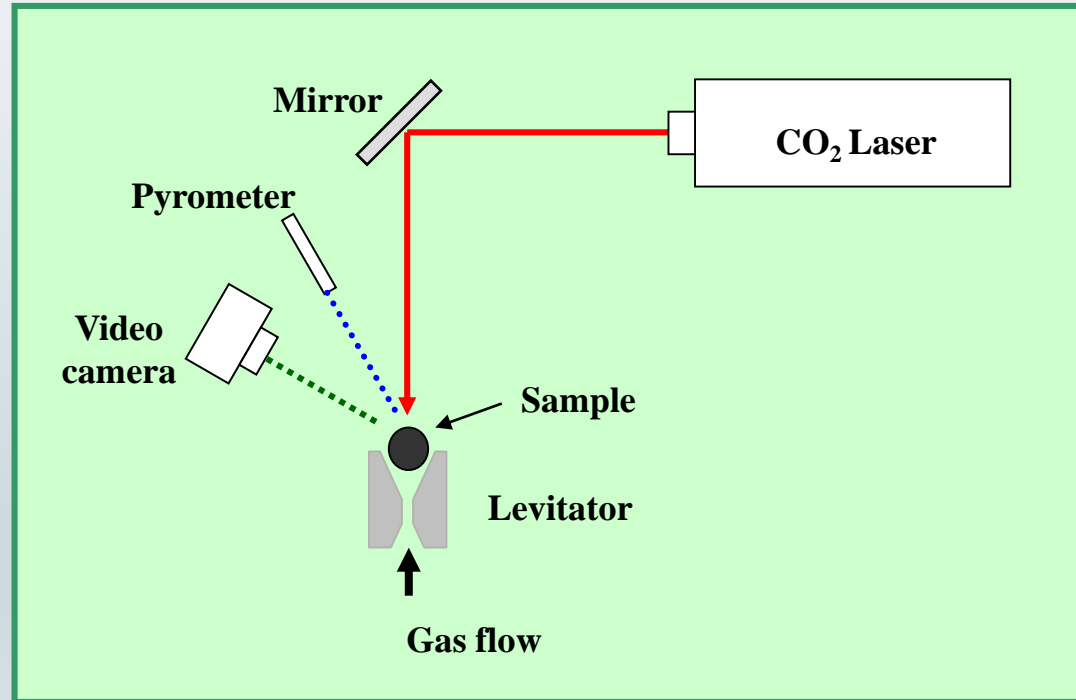


FeNi and FeNiS alloys
 Liquid state
 High temperature
 Special furnace
 Two-axis diffractometer

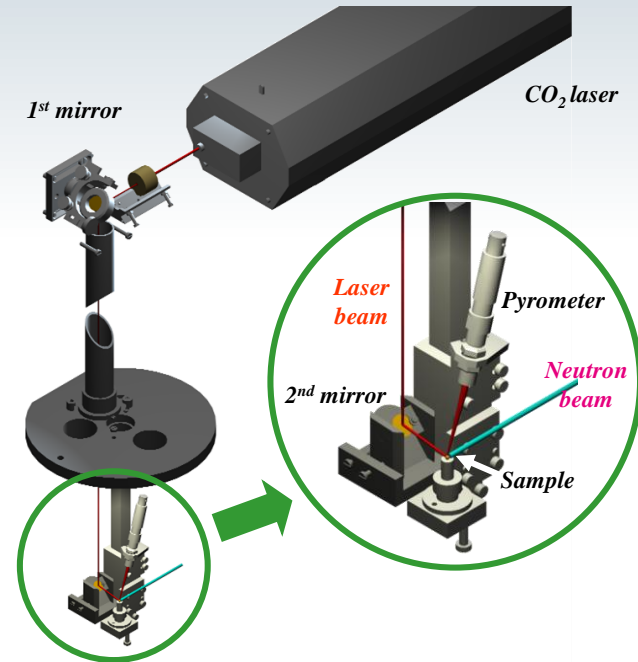
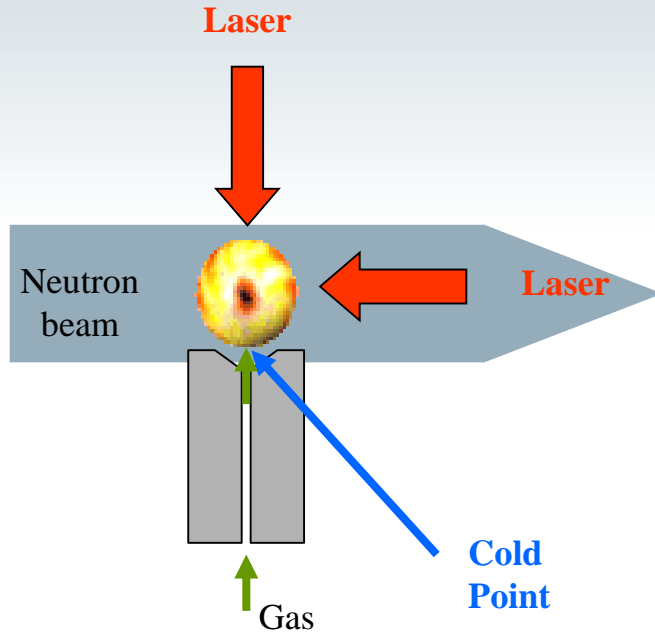


Levitation of Liquids

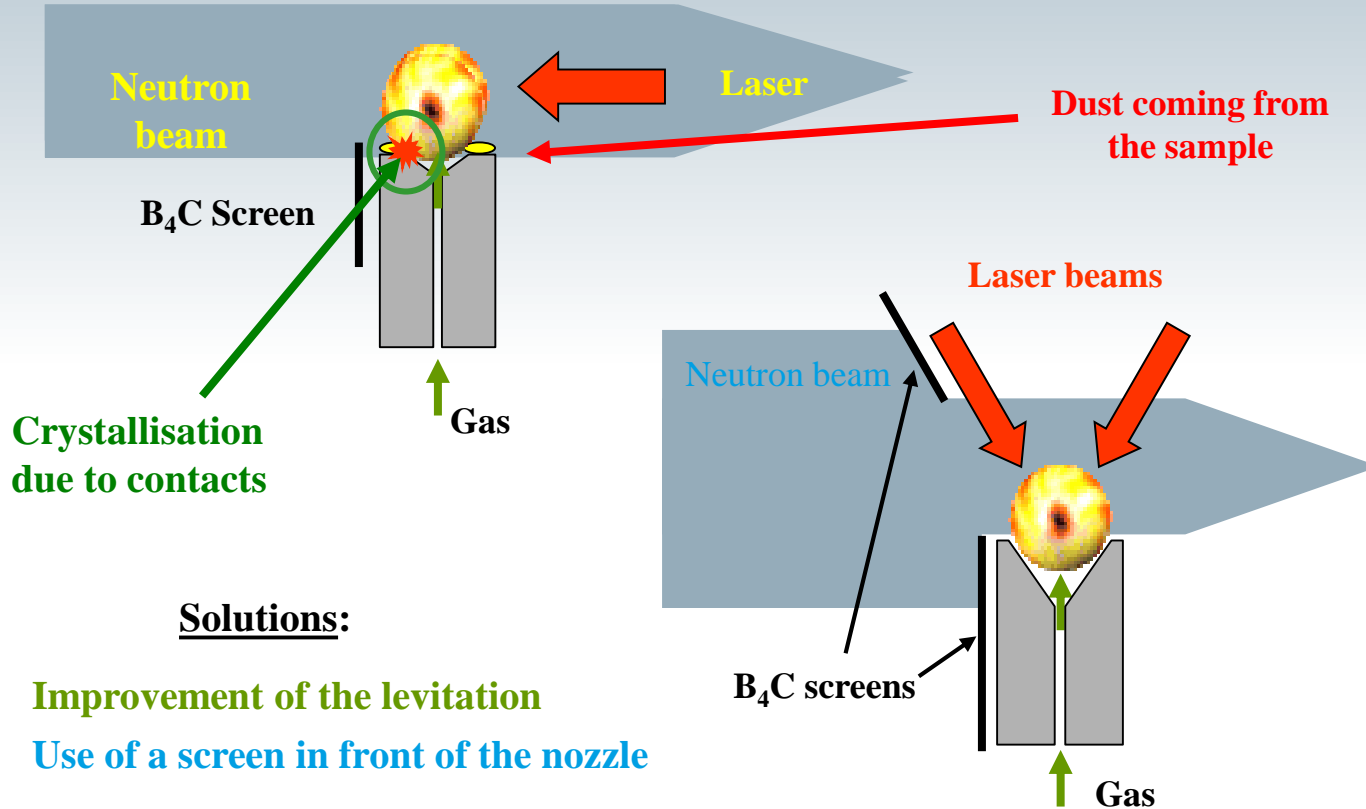
Principle of the aerodynamic levitation and laser heating



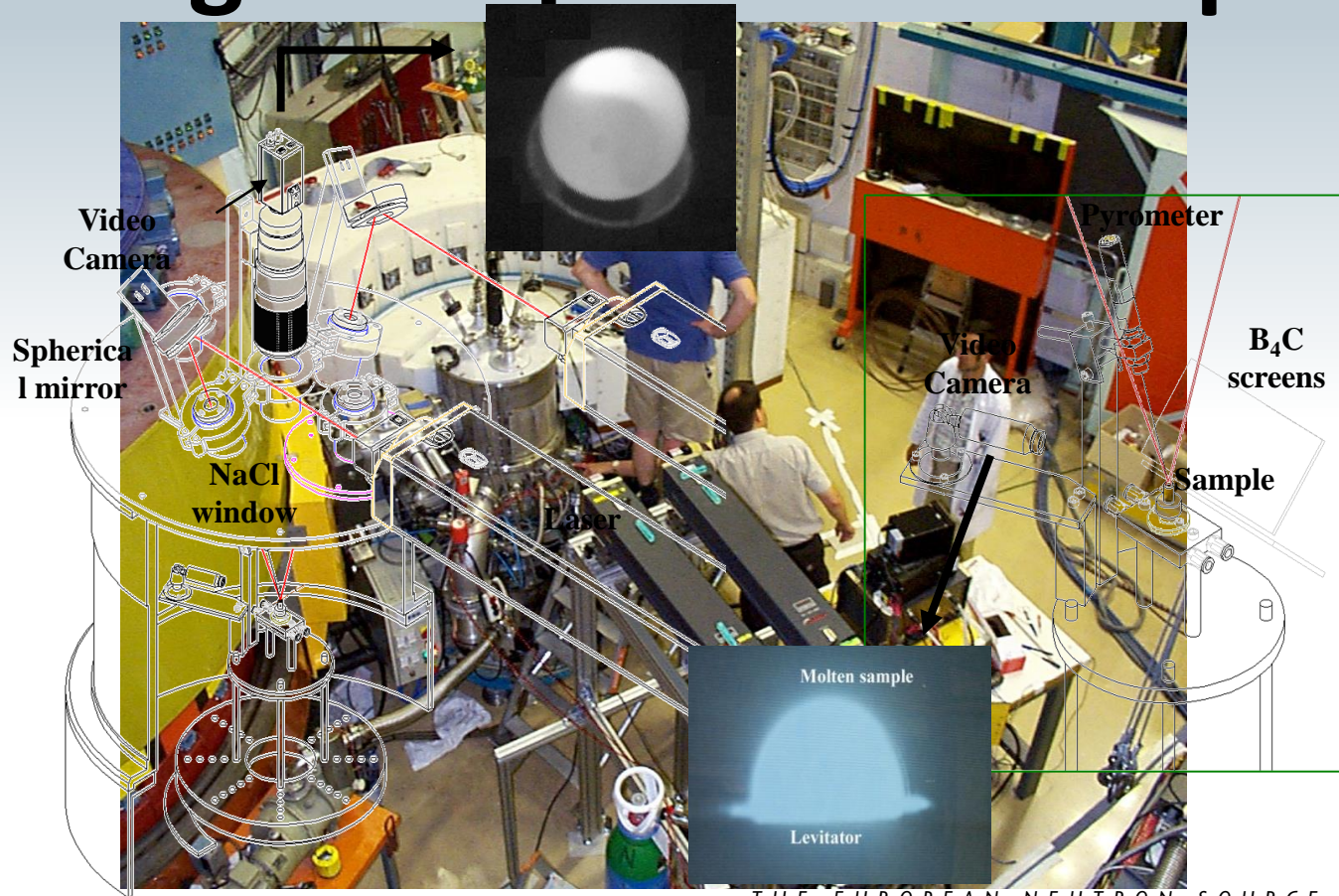
Crystallisation



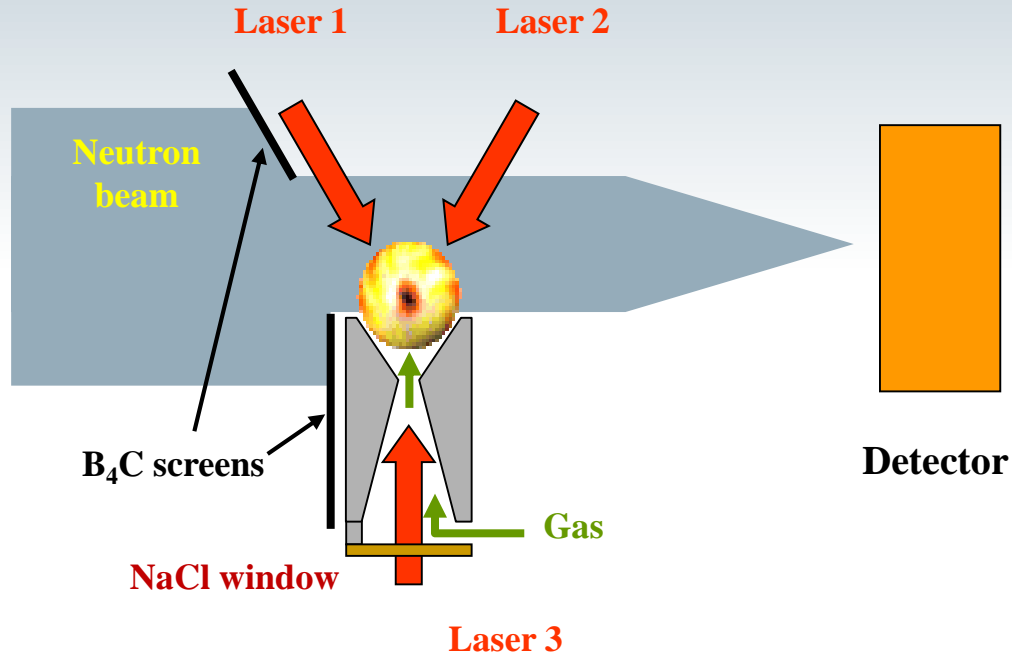
Again Crystallisation



High Temperature Setup

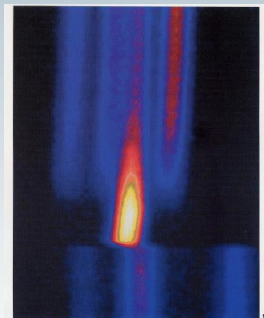


Three Lasers Setup

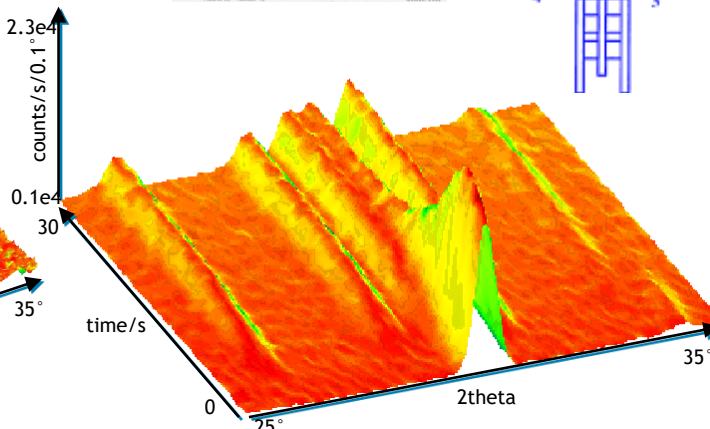
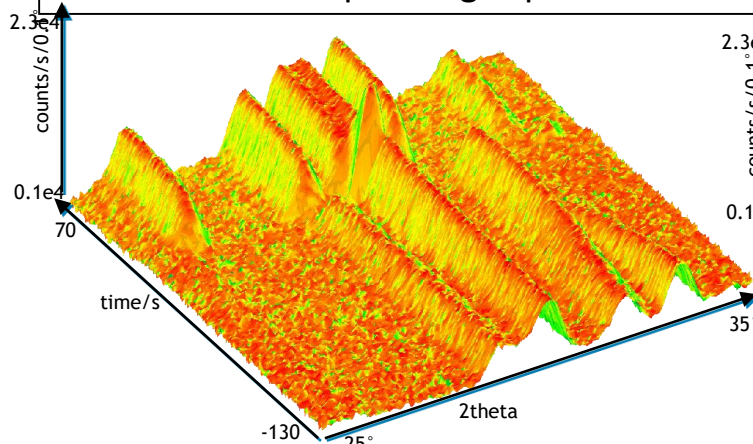
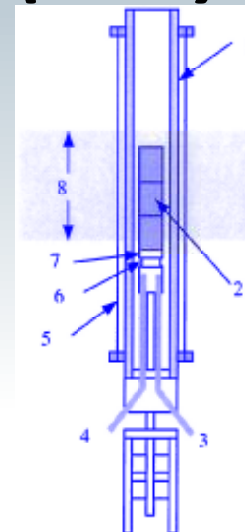


Self-propagating High-T Synthesis (SHS) D20

- Titanium silicon carbide Ti_3SiC_2
- Self-propagating High-temperature Synthesis (SHS)
 - Riley, Kisi et al.: 3 Ti : 1 Si : 2 C, 20 g pellet in furnace
 - Heating from 850 C to 1050 C at 100 K/min
 - Acquisition time 500 ms (300 ms)
- Hot isostatic pressing expensive



Journal
of the American Ceramic Society
Incorporating Advanced Ceramic Materials and Communications
Volume 85 Number 10 October 2002

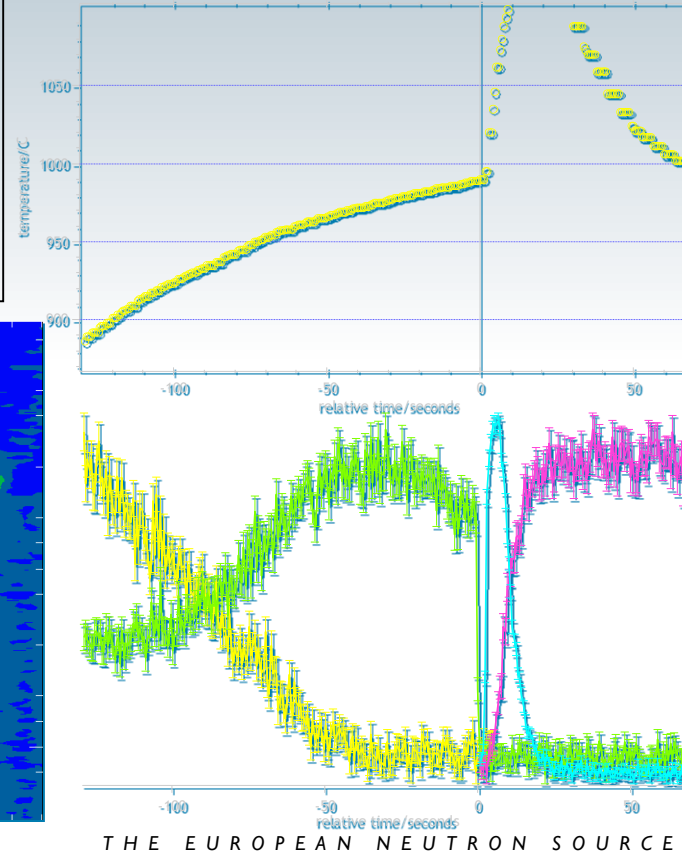
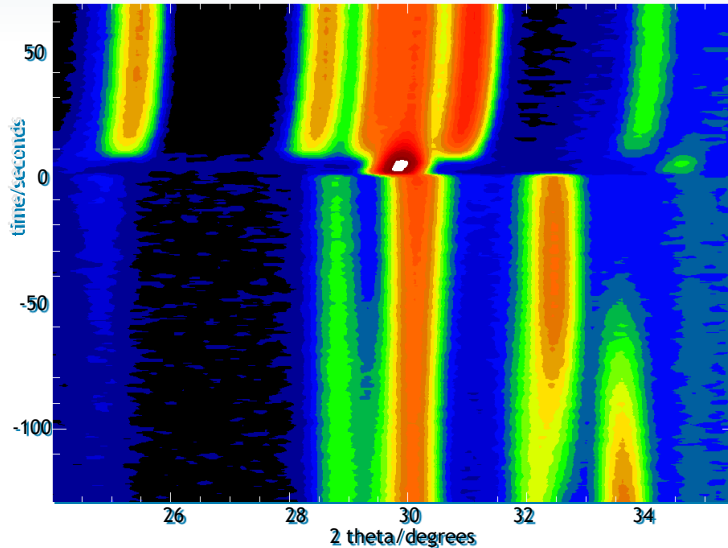


Thanks to
Thomas Hansen

D.P. Riley, E.H. Kisi, T.C. Hansen, A. Hewat, *J. Am. Ceramic Soc.* **85** (2002) 2417-2424.

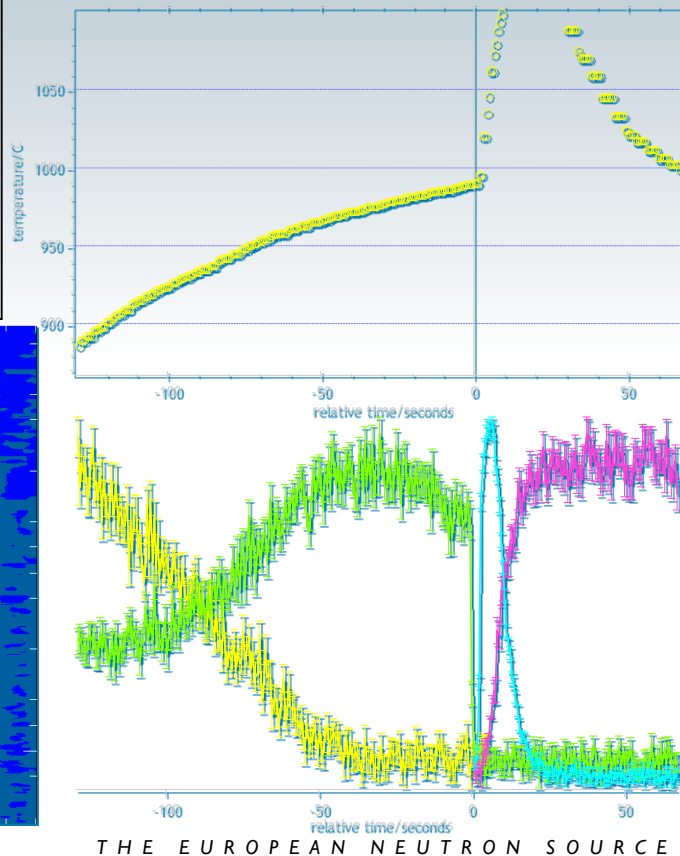
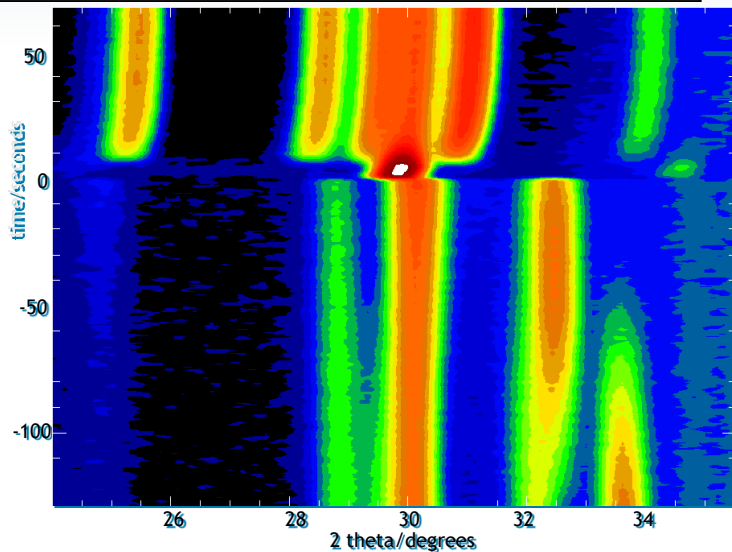
SHS: pre-ignition

- Ti α - β transition
 - starting at 870 C
- Pre-ignition:
 - TiC_x growth during 1 min
- Melting (?) in 0.5 s



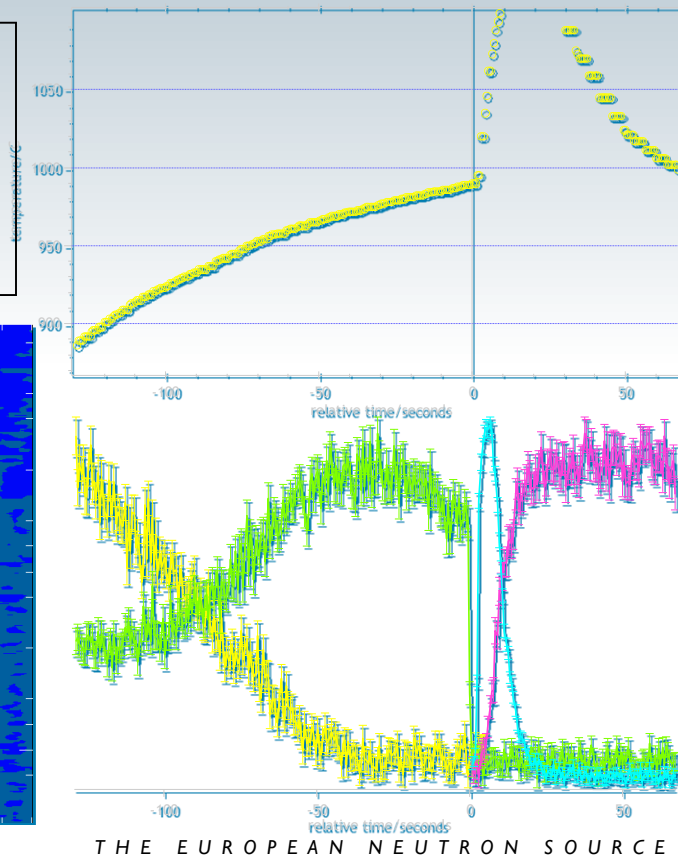
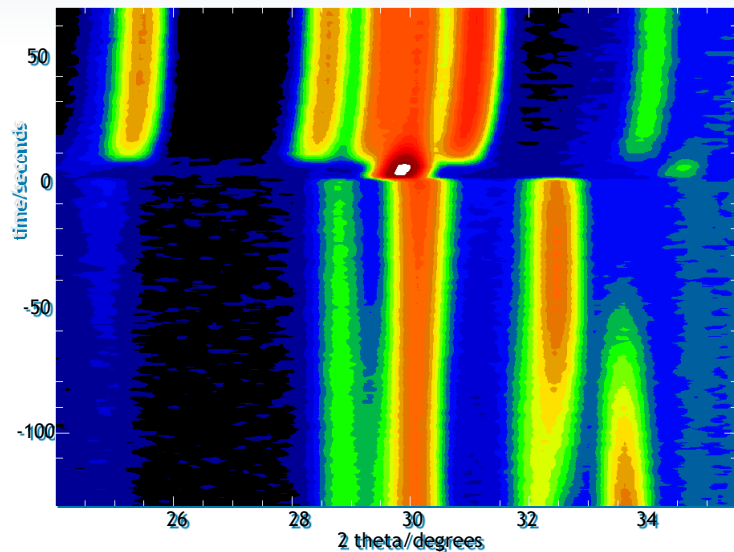
SHS: intermediate product

- Intermediate phase
 - TiC, Si substituted
 - formed in 0.5 s, 2s delay
 - Heating up to 2500 K
 - afterwards decay in 5 s

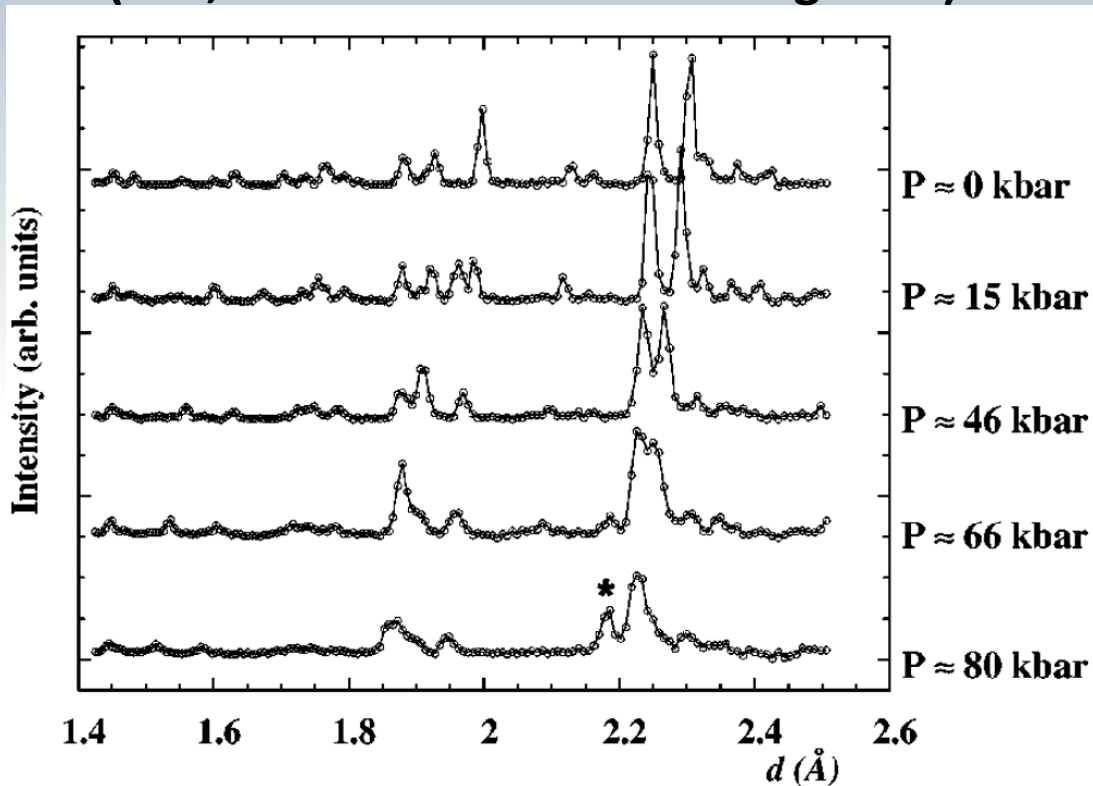


SHS: final product

- Product Ti_3SiC_2
 - starts after 5 s incubation
 - time constant about 5 s



Diffraction patterns of LaMnO_3 vs Pressure (ISIS, POLARIS + Paris-Edinburgh cell)

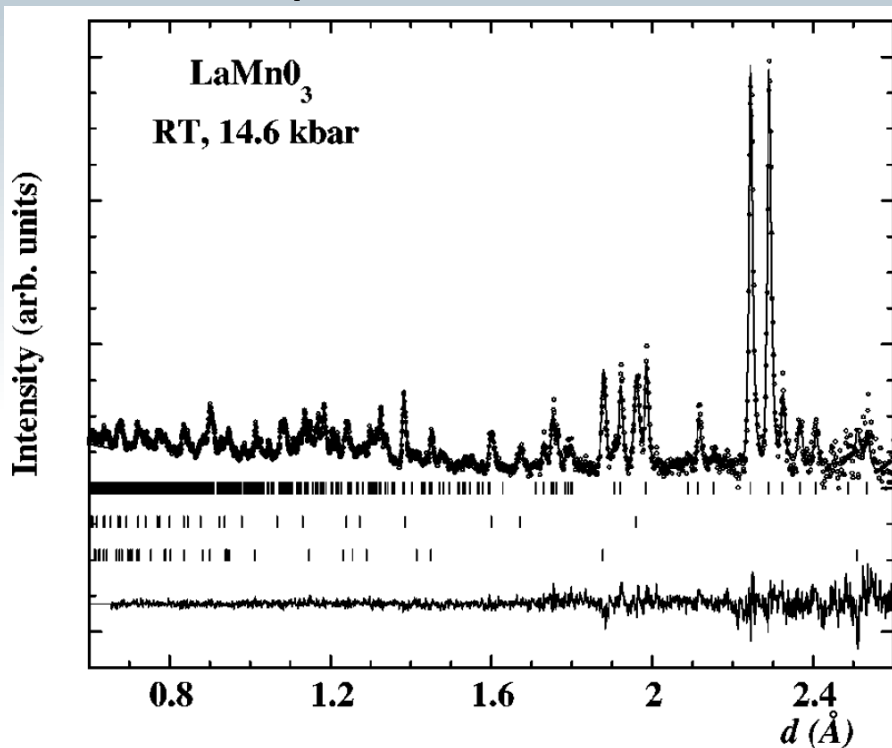


Stability of the Jahn-Teller effect and magnetic study of LaMnO_3 under pressure

L. Pinsard-Gaudart, J.Rodriguez-Carvajal, A. Daoud-Aladine, I.N.Goncharenko, M. Medarde,
R.I. Smith and A. Revcolevschi. PRB 64, 064426 (2001)

THE EUROPEAN NEUTRON SOURCE

Diffraction patterns of LaMnO_3 vs Pressure (ISIS, POLARIS + Paris-Edinburgh cell)



**Rietveld refinement
of LaMnO_3 at RT and
14.6 kbar**

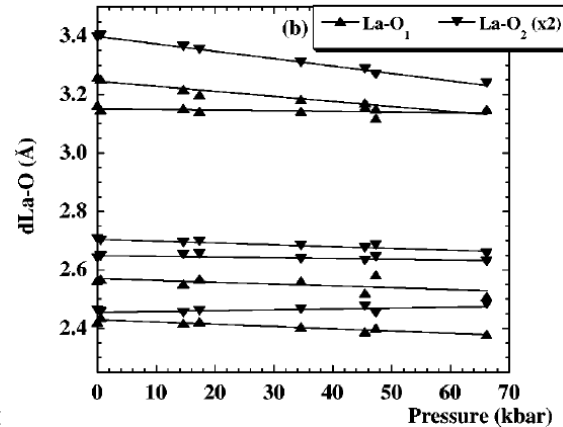
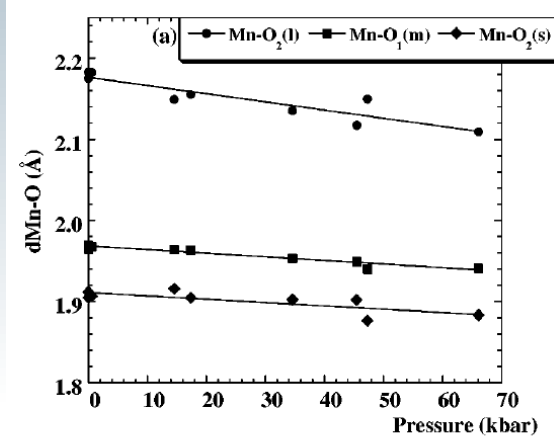
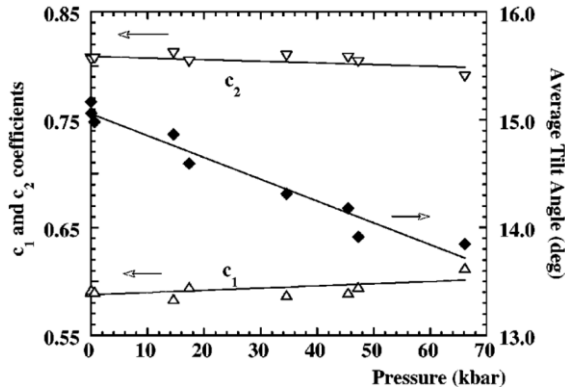
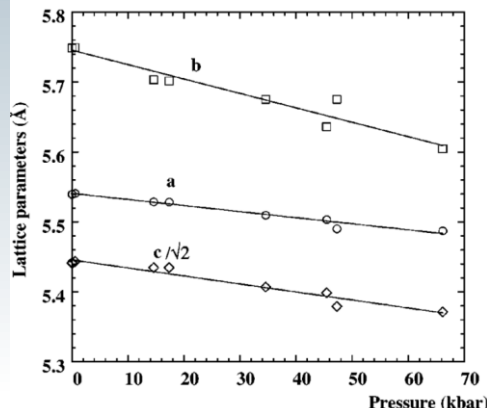
**In this range of
pressure the
reflections in the
diffraction pattern are
still sharp enough for
performing a proper
refinement**

Stability of the Jahn-Teller effect and magnetic study of LaMnO_3 under pressure

L. Pinsard-Gaudart, J.Rodriguez-Carvajal, A. Daoud-Aladine, I.N.Goncharenko, M. Medarde,
R.I. Smith and A. Revcolevschi. PRB 64, 064426 (2001)

LaMnO₃ vs Pressure (ISIS, POLARIS + Paris-Edinburgh cell)

Behaviour of
different
structural
parameters of
LaMnO₃ up to
70kbar



Magnetic scattering of LaMnO₃ vs Pressure (LLB, G61 + Goncharenko cell)

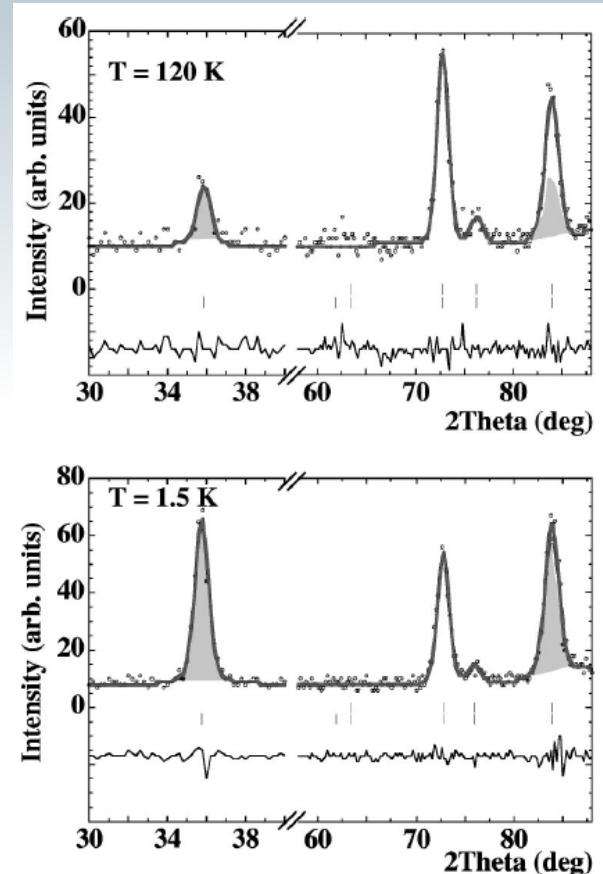
Rietveld refinement of the nuclear (fixed) and magnetic scattering of LaMnO₃

The analysis of the magnetic contribution to the diffraction patterns indicates that the mode A continues to be dominant for all pressure range studied. However the mode Ay is unable to explain the diffraction pattern at 67 kbar.

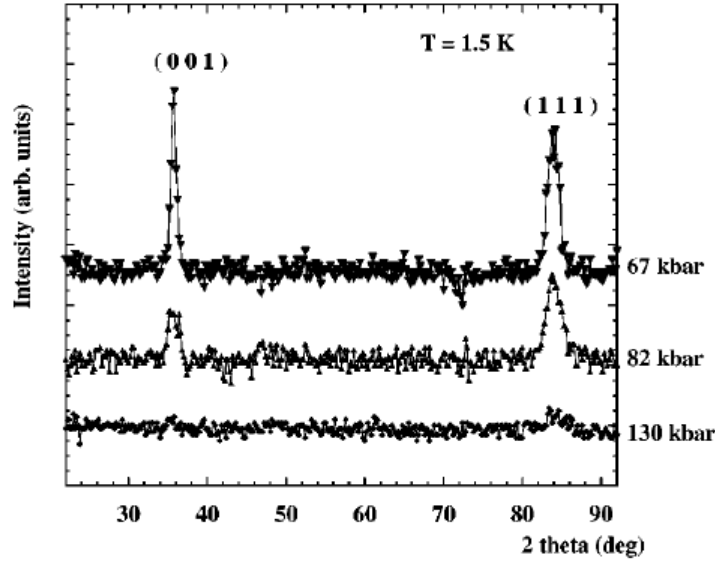
The magnetic structure corresponds to the mixture of two representations

$\Gamma_{3g}(+-)$ and $\Gamma_{4g}(--)$ of Pbnm for $k=0$
 $[G_x, A_y, F_z] \sim [0, A_y, 0]$ and $[C_x, F_y, A_z] \sim [0, 0, A_z]$

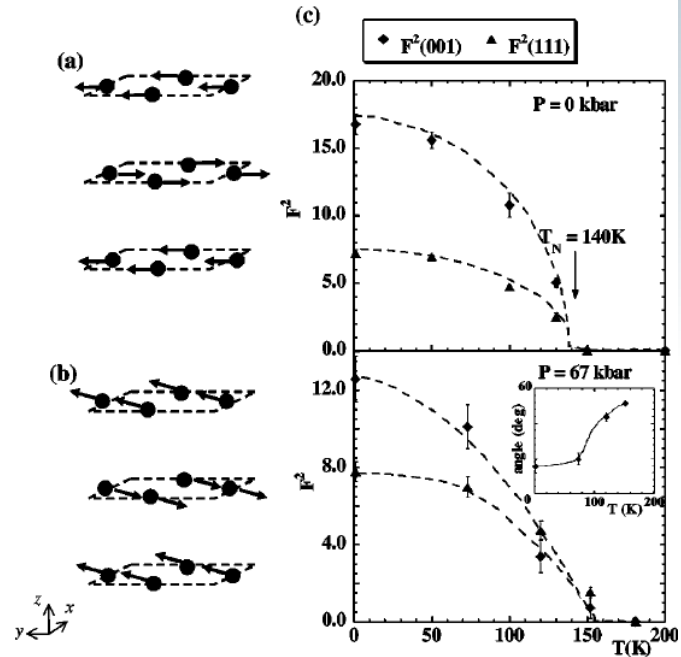
Magnetic structure at 67 kbar: $[0, A_y, A_z]$



Magnetic scattering of LaMnO_3 vs Pressure (LLB, G61 + Goncharenko cell)



Magnetic structure vs pressure



Conclusions LaMnO_3

Stability of the Jahn-Teller effect and magnetic study of LaMnO_3 under pressure

L. Pinsard-Gaudart, J.Rodriguez-Carvajal, A. Daoud-Aladine, I.N.Goncharenko, M. Medarde, R.I. Smith and A. Revcolevschi. [PRB 64, 064426 \(2001\)](#)

- 1:** The main effect of pressure is to decrease the orthorhombic distortion by diminishing the tilt angle MnO_6 octahedra (the interatomic distances diminish near isotropically)
- 2:** The Jahn-Teller effect is stable up to 70 kbar
- 3:** The magnetic structure conserve the A-type of ordering but with a deviation of the magnetic moment from the b-axis towards the c-axis

Thank you for your attention



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Facilities

Reference

Software

Conferences

Announcements

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Scattering
Web**

www.neutron.anl.gov

Neutron Scattering Mailing Lists