

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




Clifford 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 tiny 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 atomic dipoles are oriented in magnetic materials, since neutrons are affected by magnetic forces. Shull also made use of this phenomenon in his neutron diffraction technique.




Neutrons see more than X-rays
 X-rays are scattered by electron shells in atoms and ions. With neutrons it is possible to study atoms that have no electrons. Hydrogen, for example, which has only one electron, is not so well seen with X-rays, but both of atoms are visible.



Neutrons show where atoms are

When the neutrons collide with atoms in the sample material, they change direction (see scattering - elastic scattering).



Detectors record the directions of the neutrons and a diffraction pattern is obtained. The pattern shows the positions of the atoms relative to one another.


Crystals that vary, just for heavy neutrons of a certain wavelength (energy) - strong unstimulated neutrons

Neutrons behave as particles and as waves

Neutrons reveal structure and dynamics

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

Research reactor




Beam splitter

Neutrons show what atoms do

3-axis spectrometer with sensitive systems and sensitive sample

Atoms in a crystalline sample




When the neutrons penetrate the sample they start to vibrate (oscillate) in the crystal. If the neutrons create phonons or magnons they transfer energy from the crystal - inelastic scattering

Changes in the energy of the neutrons are first analysed in an analyser crystal...

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

Brockhouse, McMaster 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 (oscillate) in crystals and record 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 atomic structures in liquids change with time.

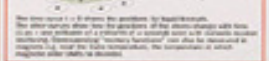
Neutrons reveal inner stresses

A hole has been punched in an aluminium metal sheet and the hole has been filled with a neutron diffraction beam. The hole has been filled with a neutron diffraction beam. The hole has been filled with a neutron diffraction beam.



Neutrons show what atoms remember

How the atoms remember their position after they have been displaced. The atoms remember their position after they have been displaced.




Where it started
 Brockhouse and Shull made their pioneering contributions at the first nuclear reactor in the USA and Canada back in the 1940s and 1950s. It was then that the neutron became available for peacetime research.

... Now it continues
 Thousands of neutrons have now working at the many neutron research centres throughout the world. New and more advanced neutron scattering installations have been built and more are planned in Europe, the USA and Asia. In these super-collaborations the researchers are studying the structure of new ceramic superconductors, material's interactions on surfaces of nanoscale for catalytic reactions, drug design, stress structures and the connections between the structure and the atomic properties of polymers.

Further reading:

- 1) The Nobel Prize in Physics 1994
- 2) The Nobel Prize in Physics 1994
- 3) The Nobel Prize in Physics 1994



3

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 Å)

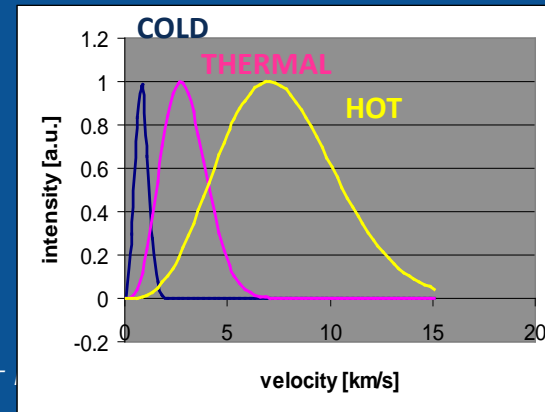
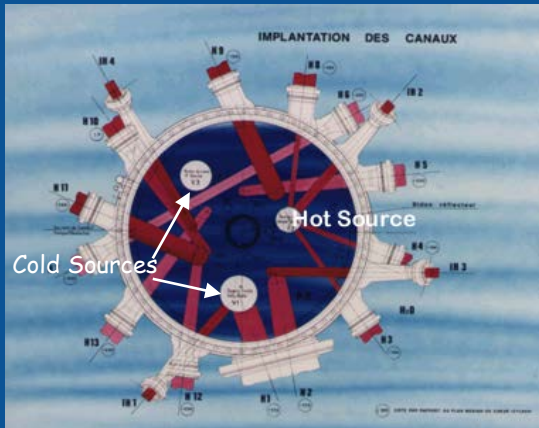
- **The wavelength** of the neutron is comparable to atomic sizes and the dimensions of atomic structures, which explains why neutrons can « see » atoms.
- **The energy** of thermal neutrons is similar to the thermal excitations in solids.
- Neutrons are **zero-charge particles** and have a **magnetic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

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

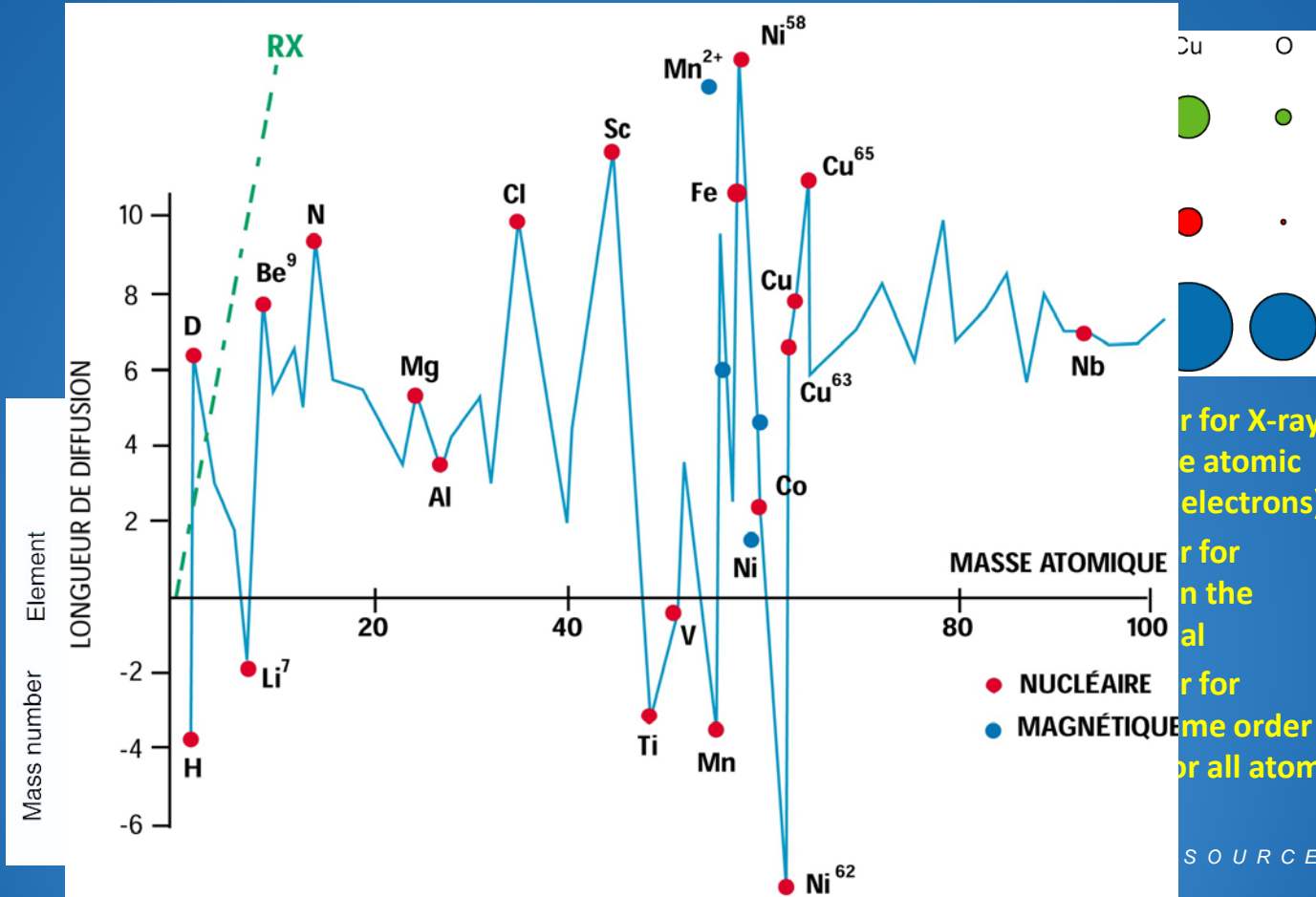
Particle-wave properties (Energy-Temperature-Wavelength)

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

	<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



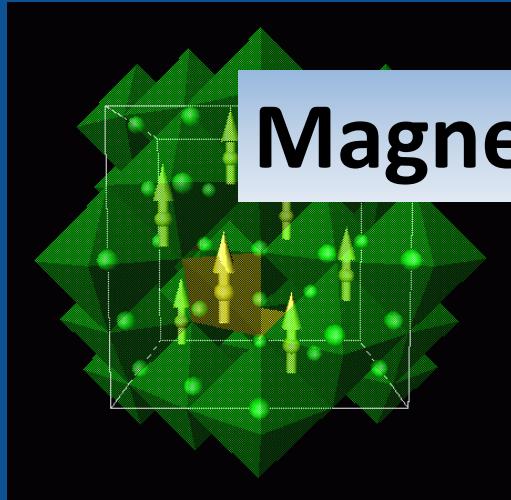
Neutrons for magnetism studies

Neutrons are strongly scattered by magnetic materials

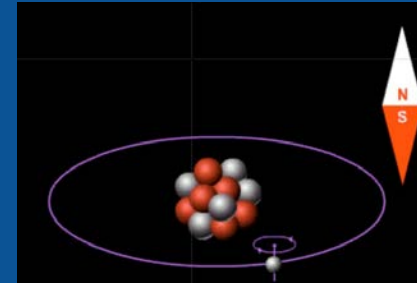
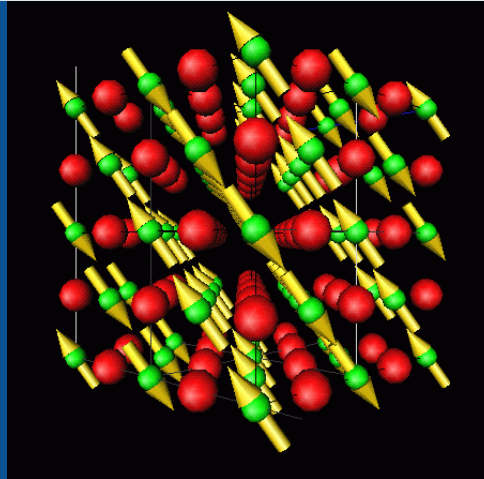
- Neutrons act as small magnets
- The dipolar magnetic moment of the neutron interacts strongly with the atomic magnetic moment
- Neutrons allow the determination of magnetic

Magnetic Crystallography

measure the
precision.



Ferromagnetic and antiferromagnetic oxides



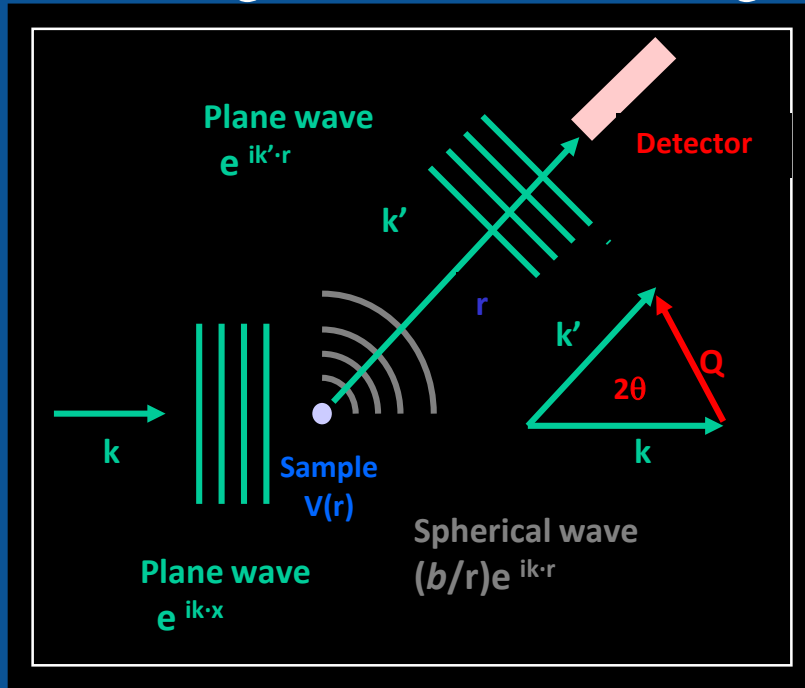
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- 2. Diffraction equations: Laue conditions**
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Interaction neutron-nucleus

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

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

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

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

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

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

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

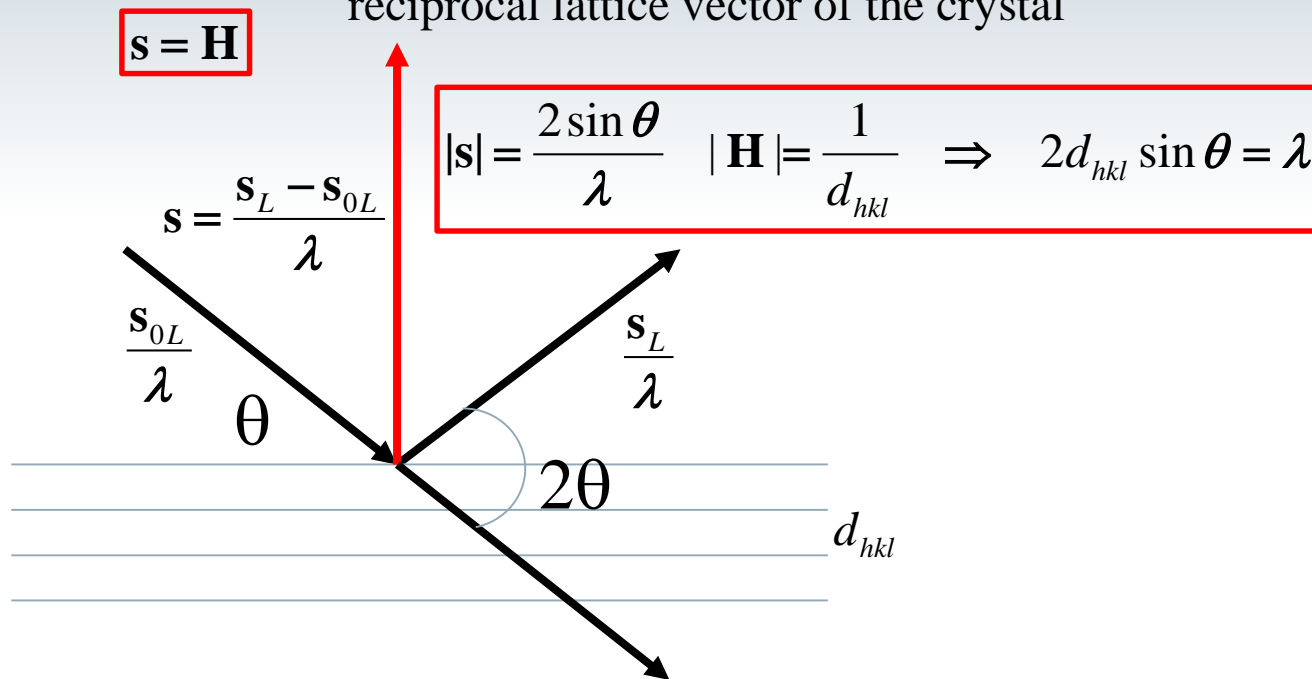
$\mathbf{s} = \mathbf{H}$ Laue conditions: the scattering vector is a reciprocal lattice vector of the crystal

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

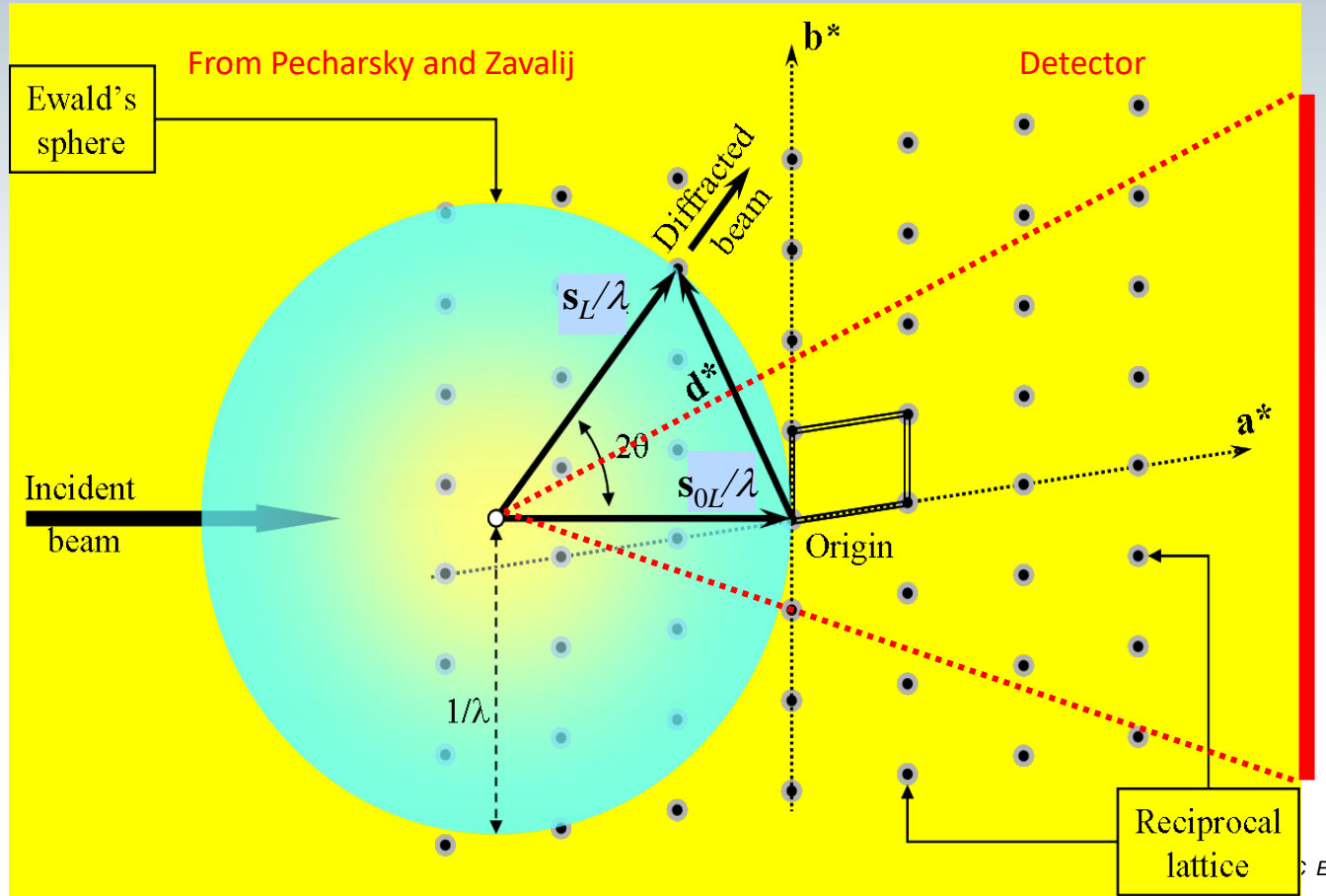
Diffraction Equations for crystals

The Laue conditions have as a consequence the Bragg Law

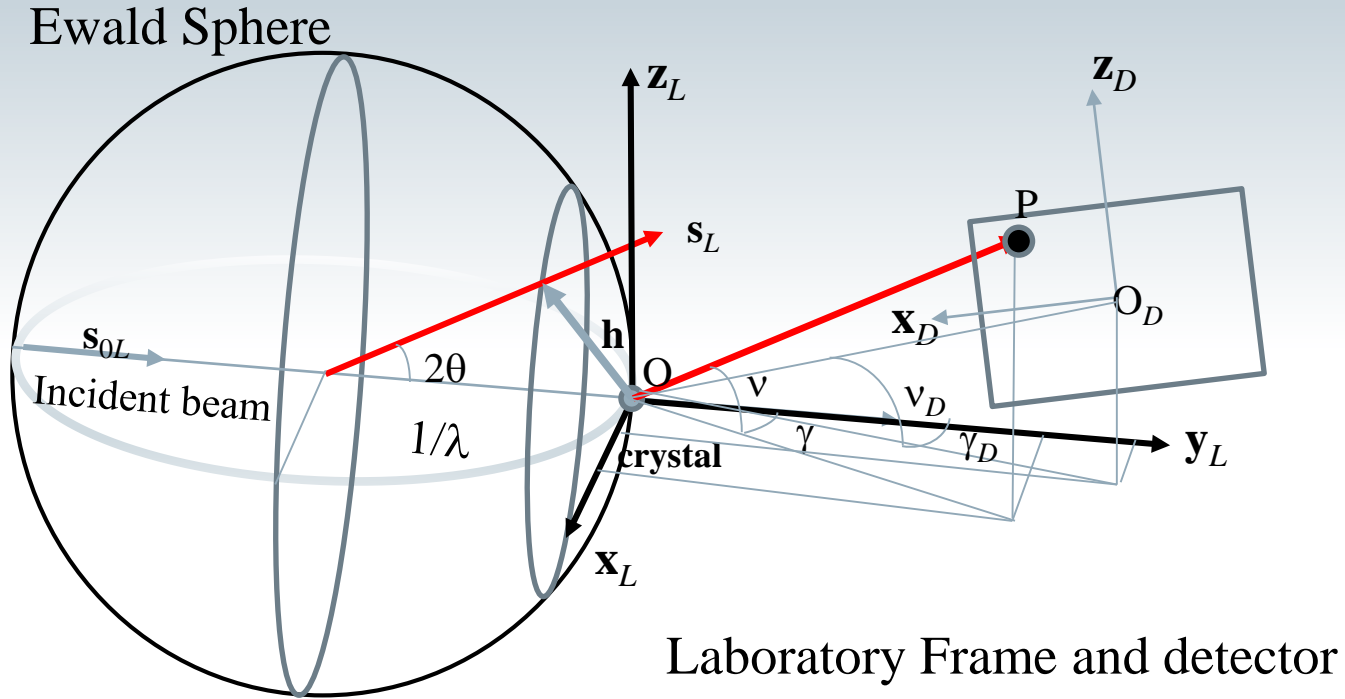
Laue conditions: the scattering vector is a reciprocal lattice vector of the crystal



Ewald construction



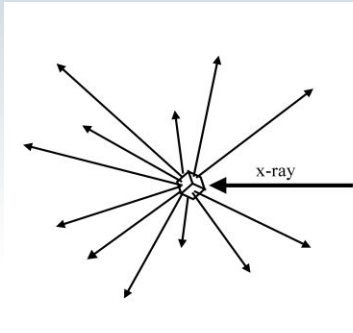
Ewald construction



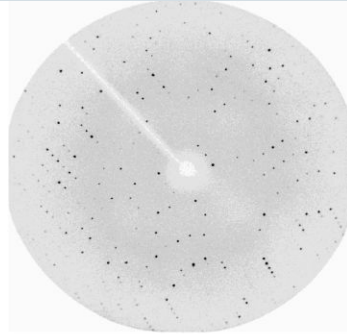
Diffraction patterns

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

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



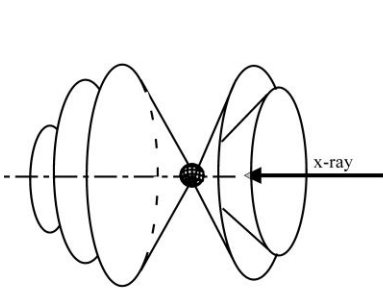
(a)



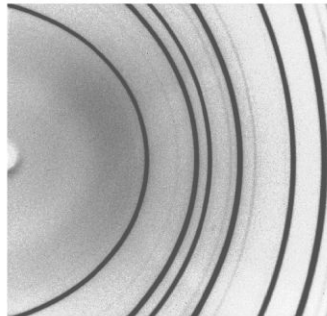
(b)



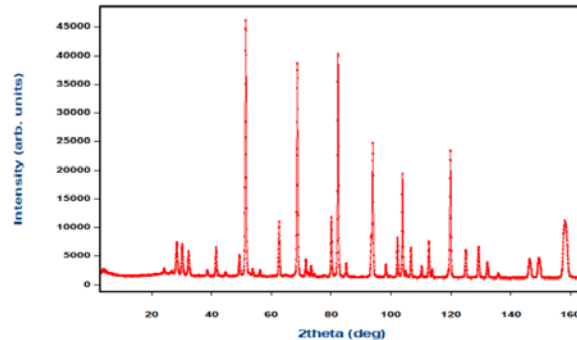
Single
Crystal



(c)



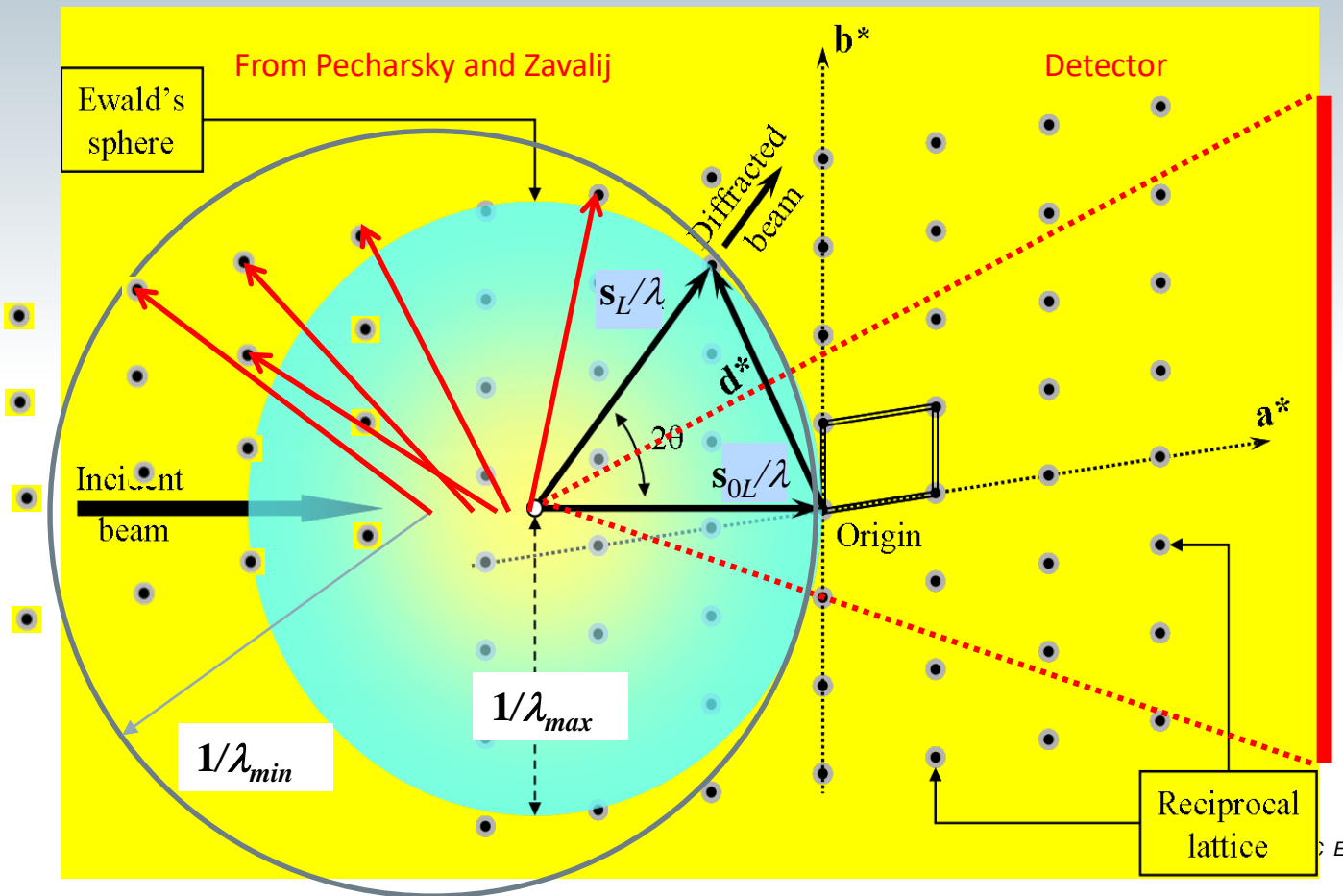
(d)



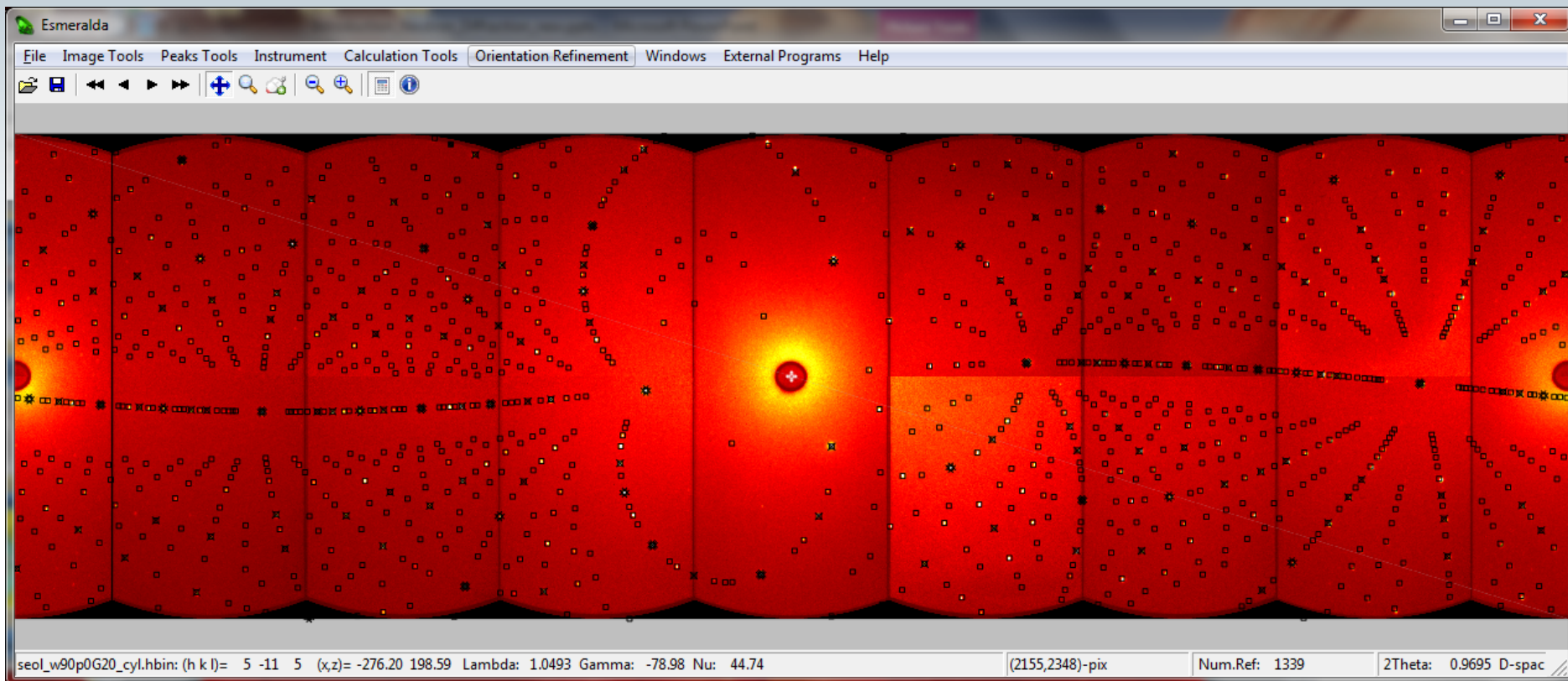
Powder or
polycrystalline
solid

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)

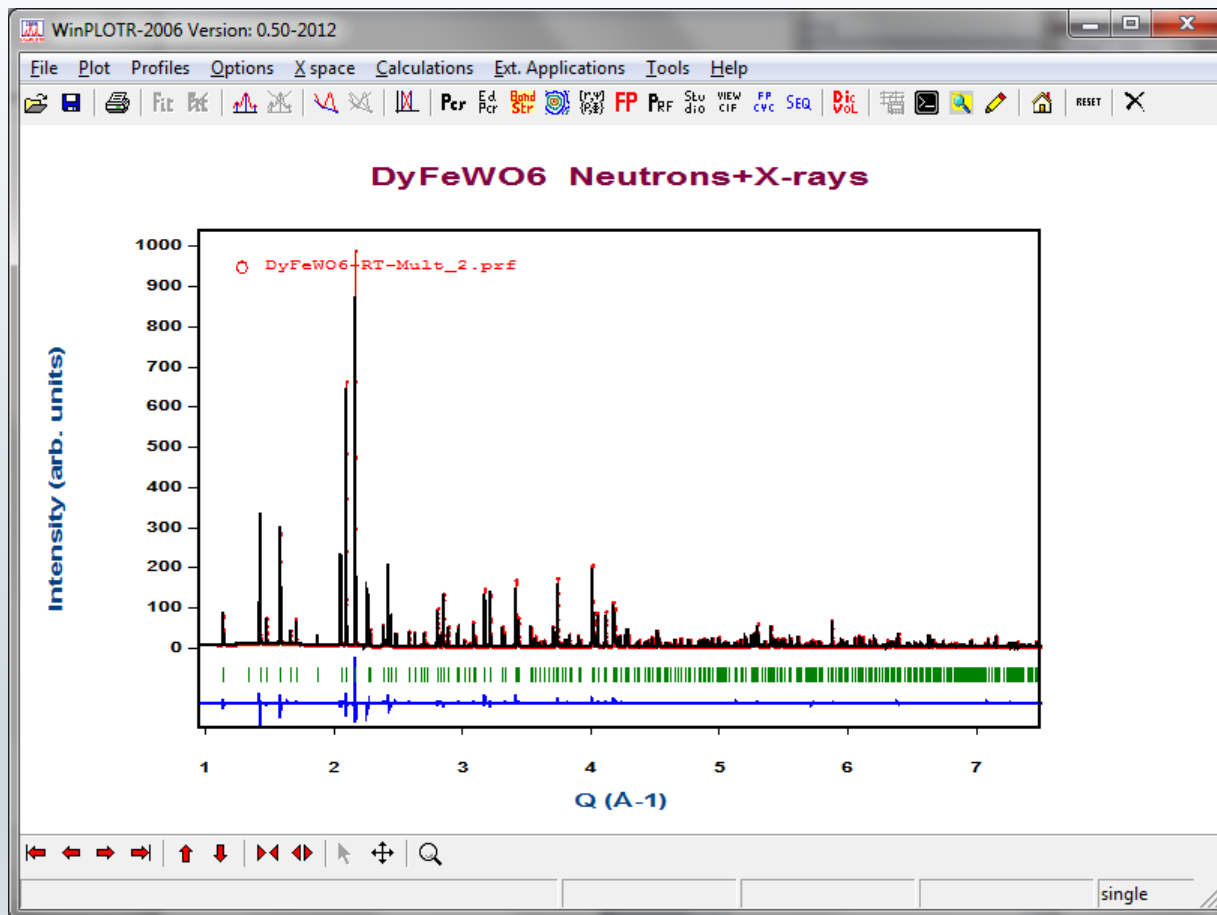
The **advantages of thermal neutrons** with respect to X-rays as far as diffraction is concerned are based on the following properties of thermal neutrons:

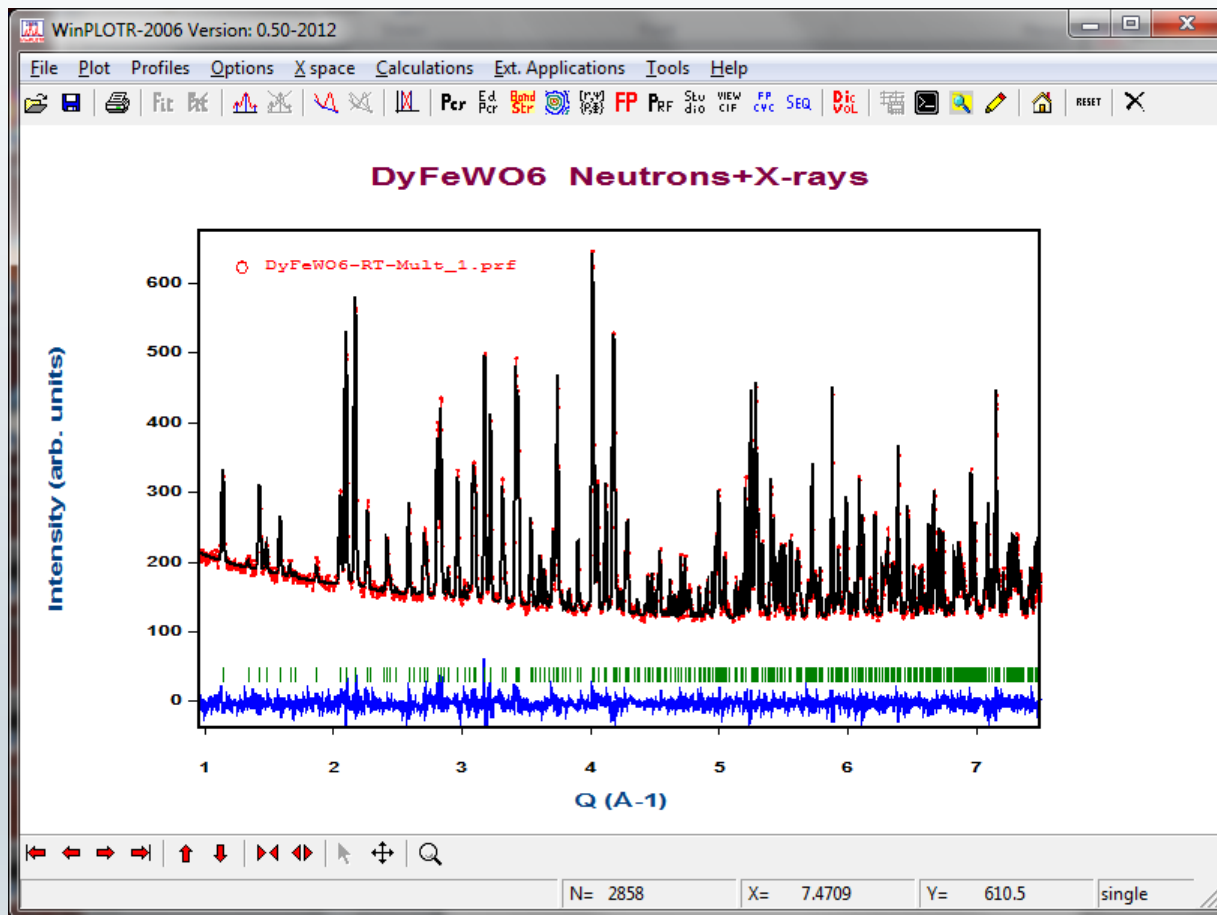
- **constant scattering power** (b is Q -independent) having a non-monotonous dependence on the atomic number
- **weak interaction** (the first Born approximation holds) that implies simple theory can be used to interpret the experimental data
- **the magnetic interaction is of the same order of magnitude 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)

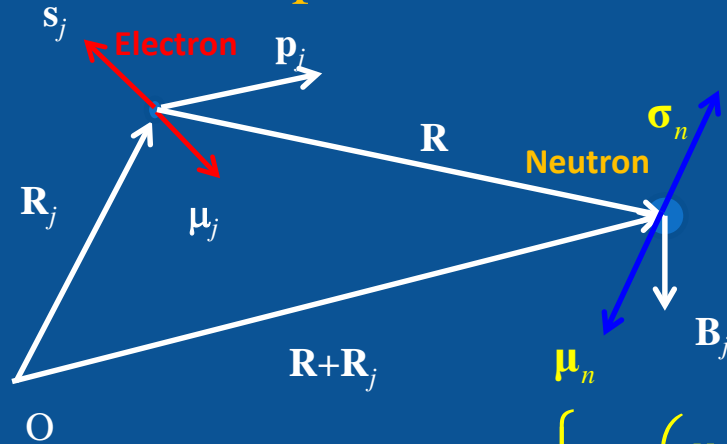
- Magnetic X-ray scattering allows in principle the separation of orbital and spin components. However, **SR cannot compete with neutrons in the field of *magnetic structure determination*** from powders.
- The contribution of SR to that field is on details of magnetic structures (already known from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

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

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

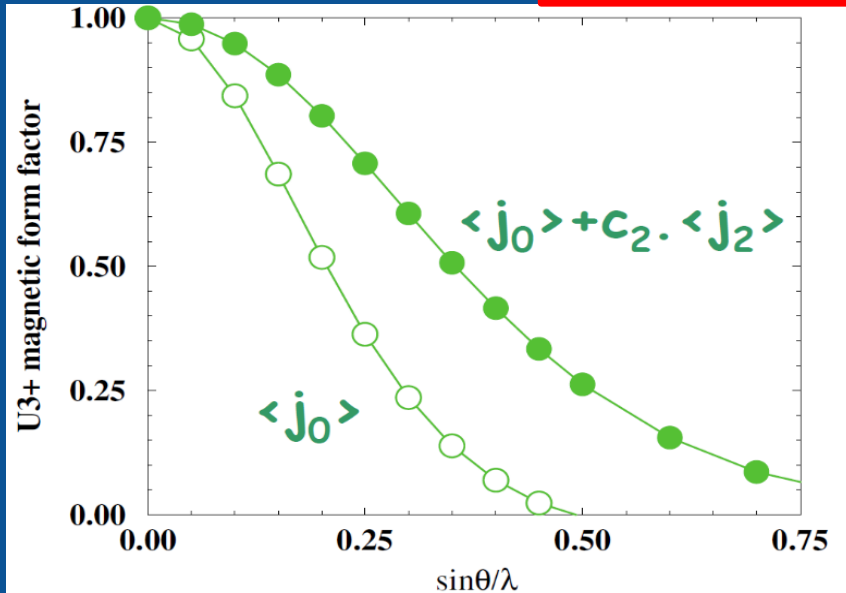
$$\mathbf{F}_j(\mathbf{Q}) = \mathbf{m}_j \int \rho_j(\mathbf{r}) \exp(i\mathbf{Q} \cdot \mathbf{r}) d^3\mathbf{r} = \mathbf{m}_j f_j(Q)$$

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

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:

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

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

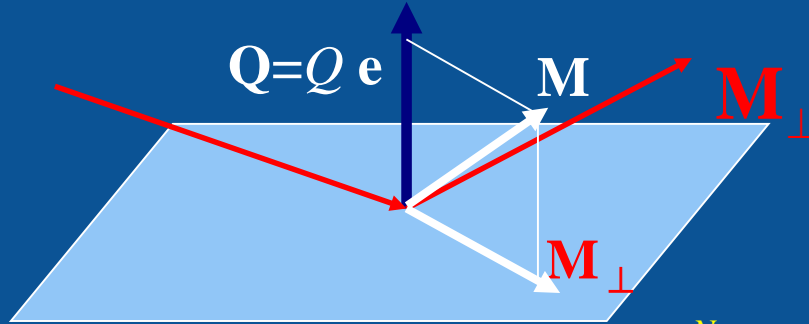
$$\langle j_l(s) \rangle = s^2 \left(A_l \exp\{-a_l s^2\} + B_l \exp\{-b_l s^2\} + C_l \exp\{-c_l s^2\} + D_l \right) \quad \text{for } l = 2, 4, 6$$

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

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:

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

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

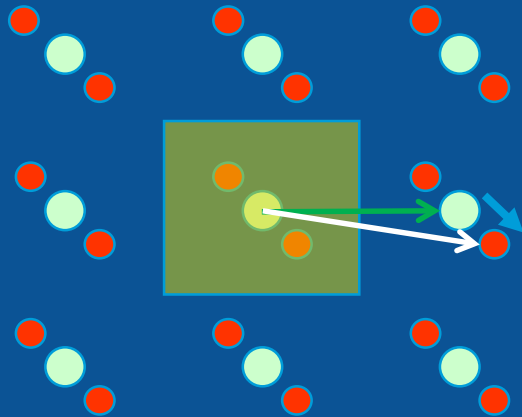
$$\mathbf{M}(\mathbf{h}) = \sum_j f_j(h) \exp(2\pi i \mathbf{h} \cdot \mathbf{r}_j) \sum_{\mathbf{k}} \mathbf{S}_{\mathbf{k}j} \sum_l \exp(2\pi i (\mathbf{h} - \mathbf{k}) \cdot \mathbf{R}_l)$$

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

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 $\{\mathbf{k}\}$

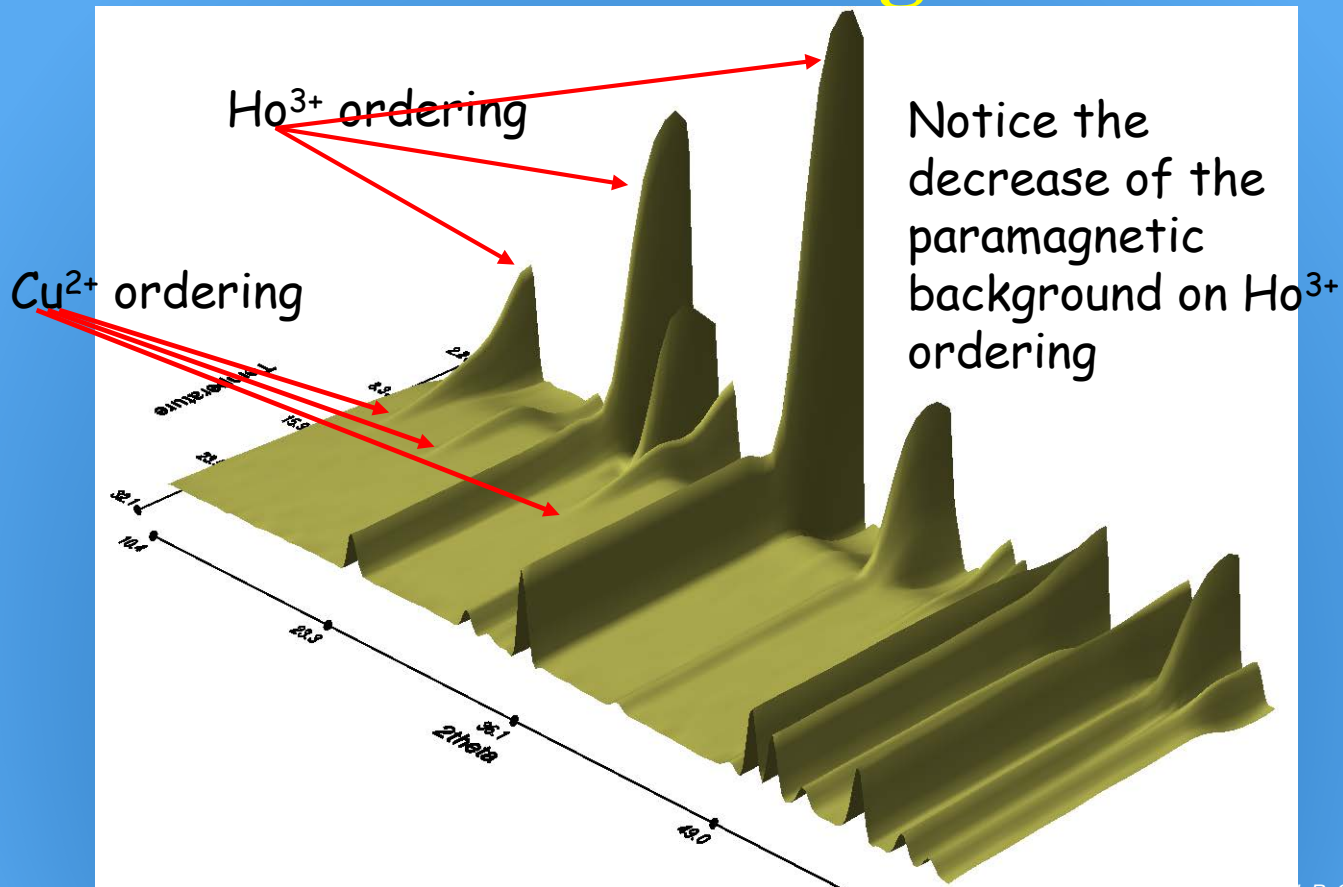
\mathbf{h} is the scattering vector indexing a magnetic reflection

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

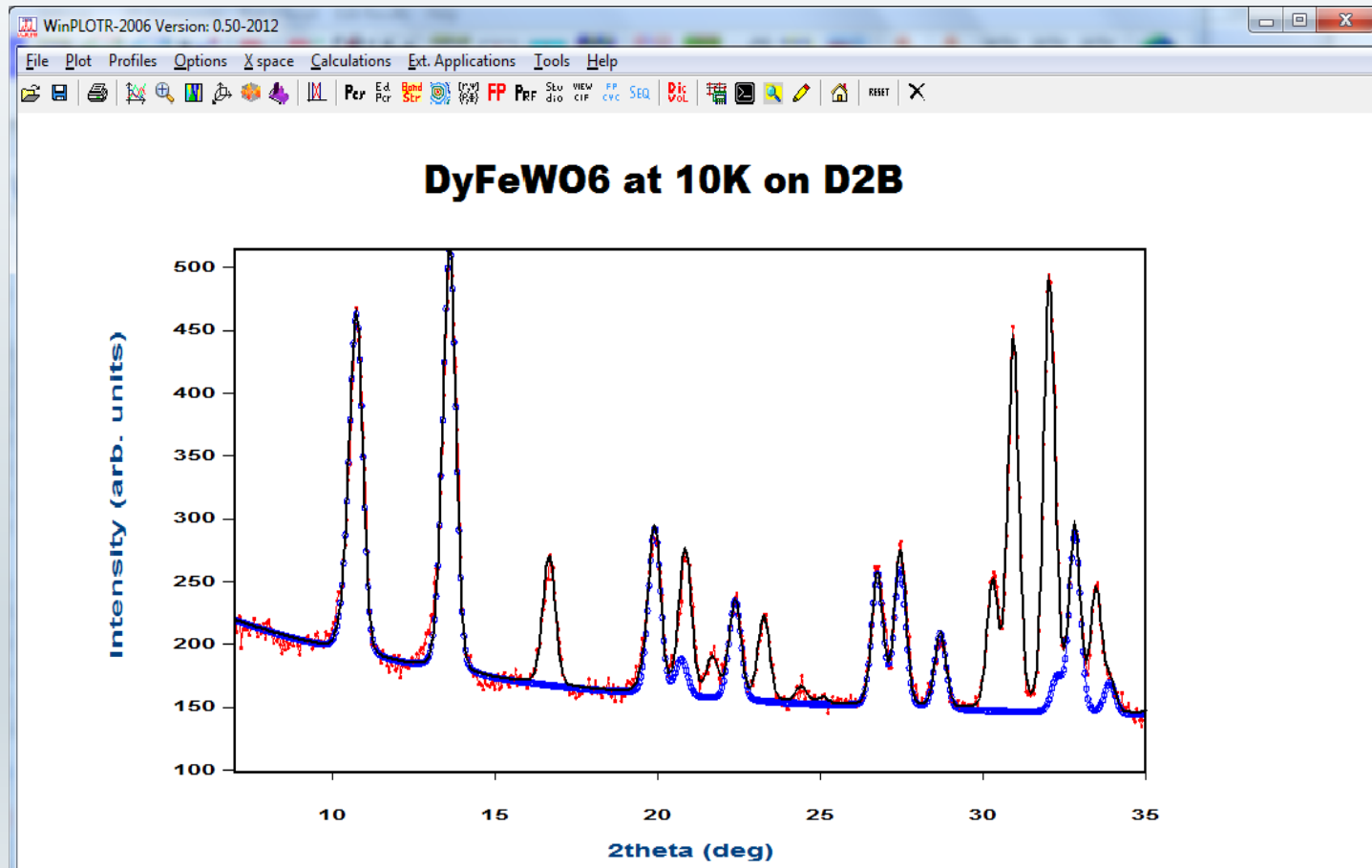
\mathbf{k} is one of the propagation vectors of the magnetic structure

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

Diffraction Patterns of magnetic structures



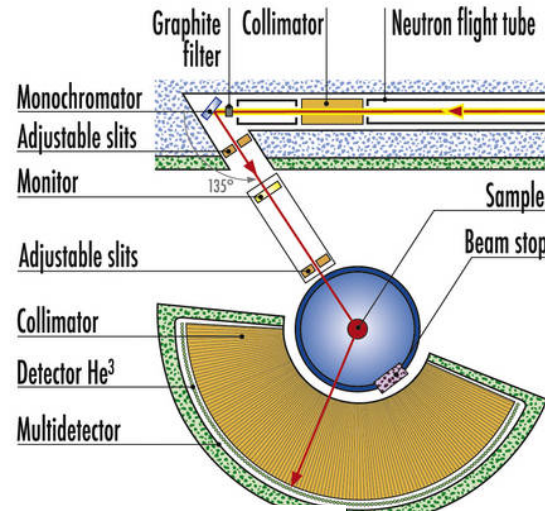
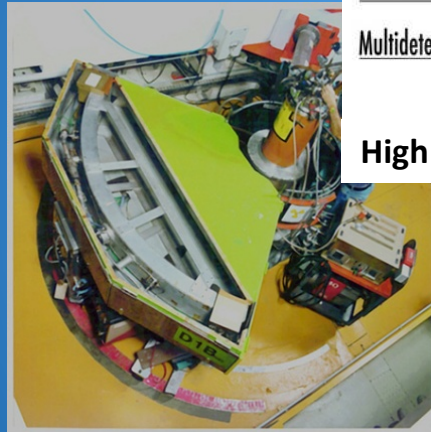
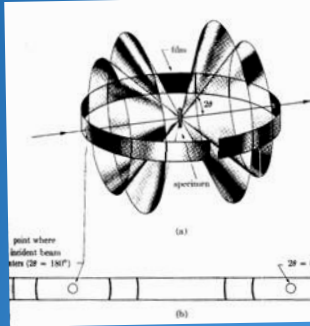
Magnetic refinement on D2B



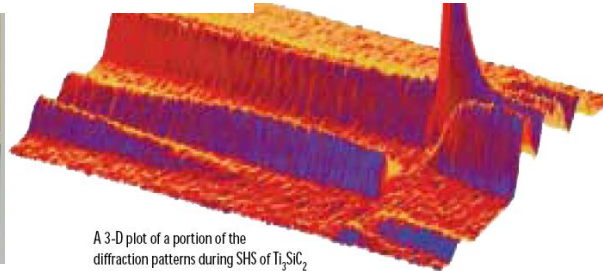
Outline

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

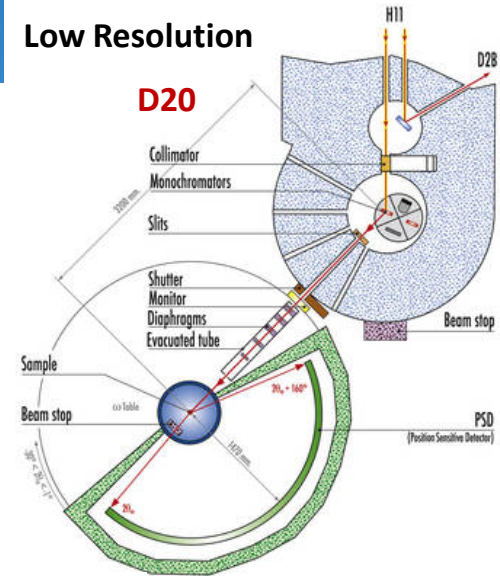


High Resolution D2B



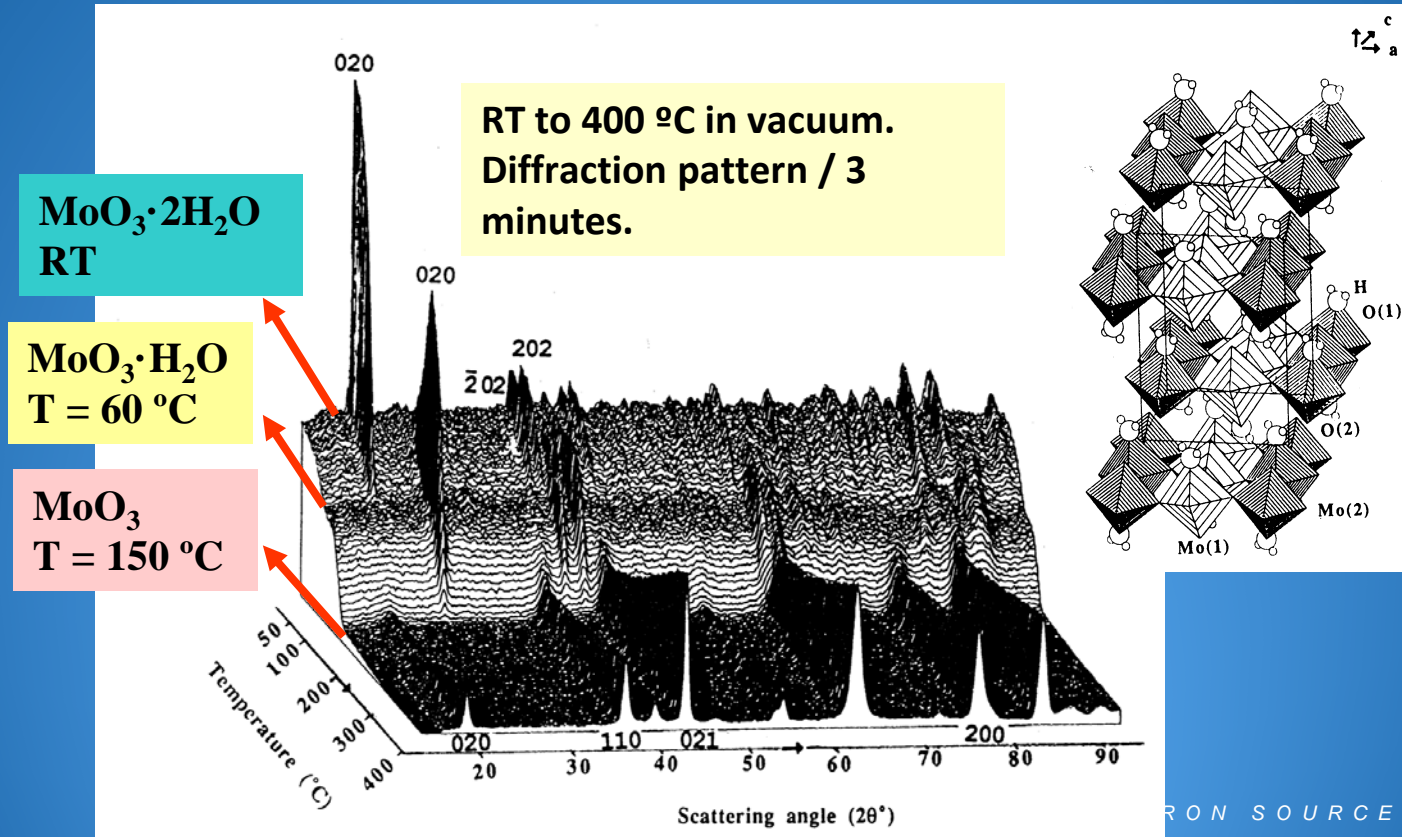
A 3-D plot of a portion of the diffraction patterns during SHS of Ti_3SiC_2 as the reaction progresses (left to right)

Low Resolution

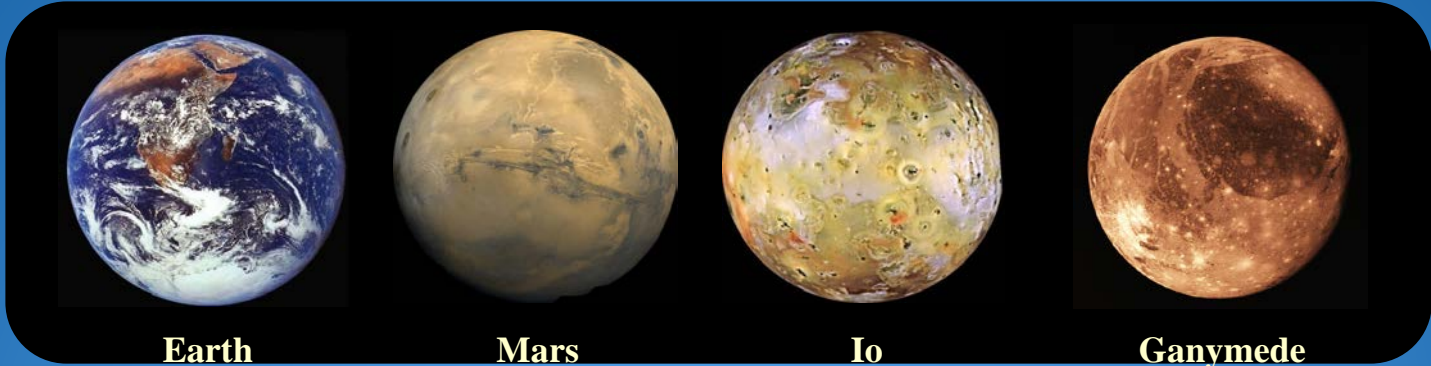


Real time powder diffraction on D1B

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



Earth

Mars

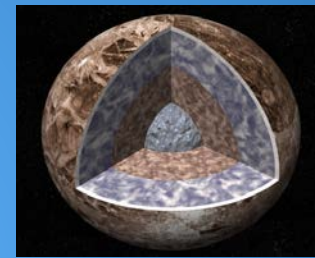
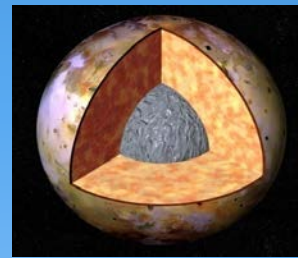
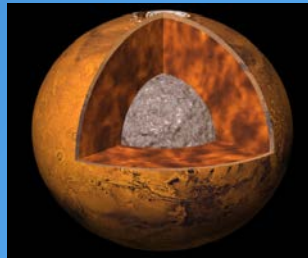
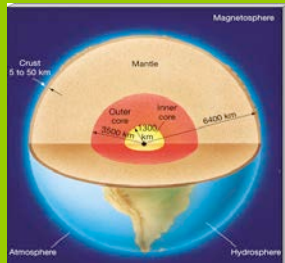
Io

Ganymede

What they have in common ...?

Metallic cores

Proximity in Solar System

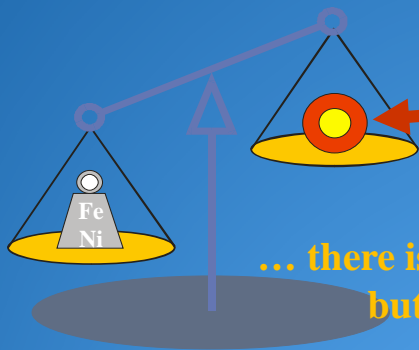


Fluid Outer Core: from 3000 to 5200 km

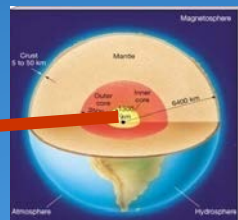
Primarily Fe with some Ni

Some Applications: Liquid state

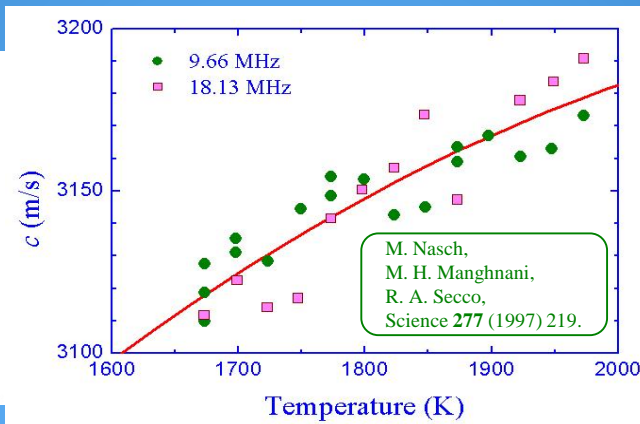
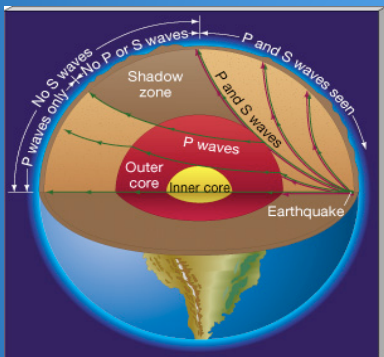
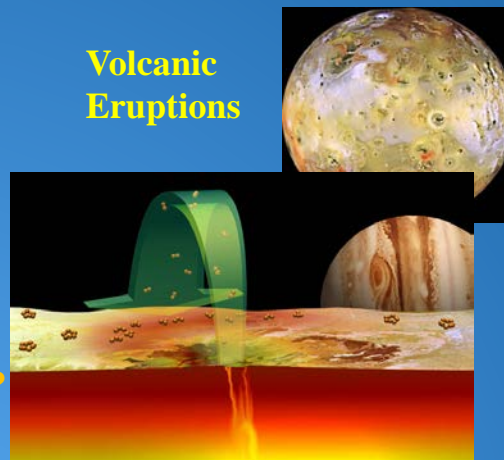
But ρ is 5-10% less than pure Fe+Ni ...



... there is some light element, but which? S? O? H? Si?



Volcanic Eruptions

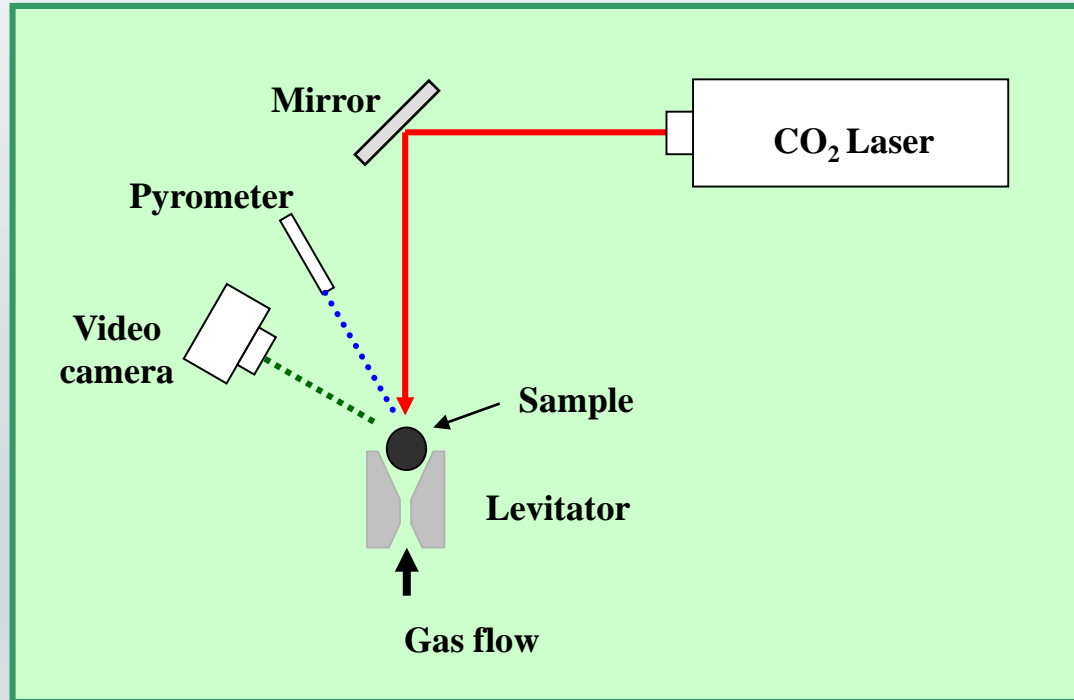


Hypothesis:

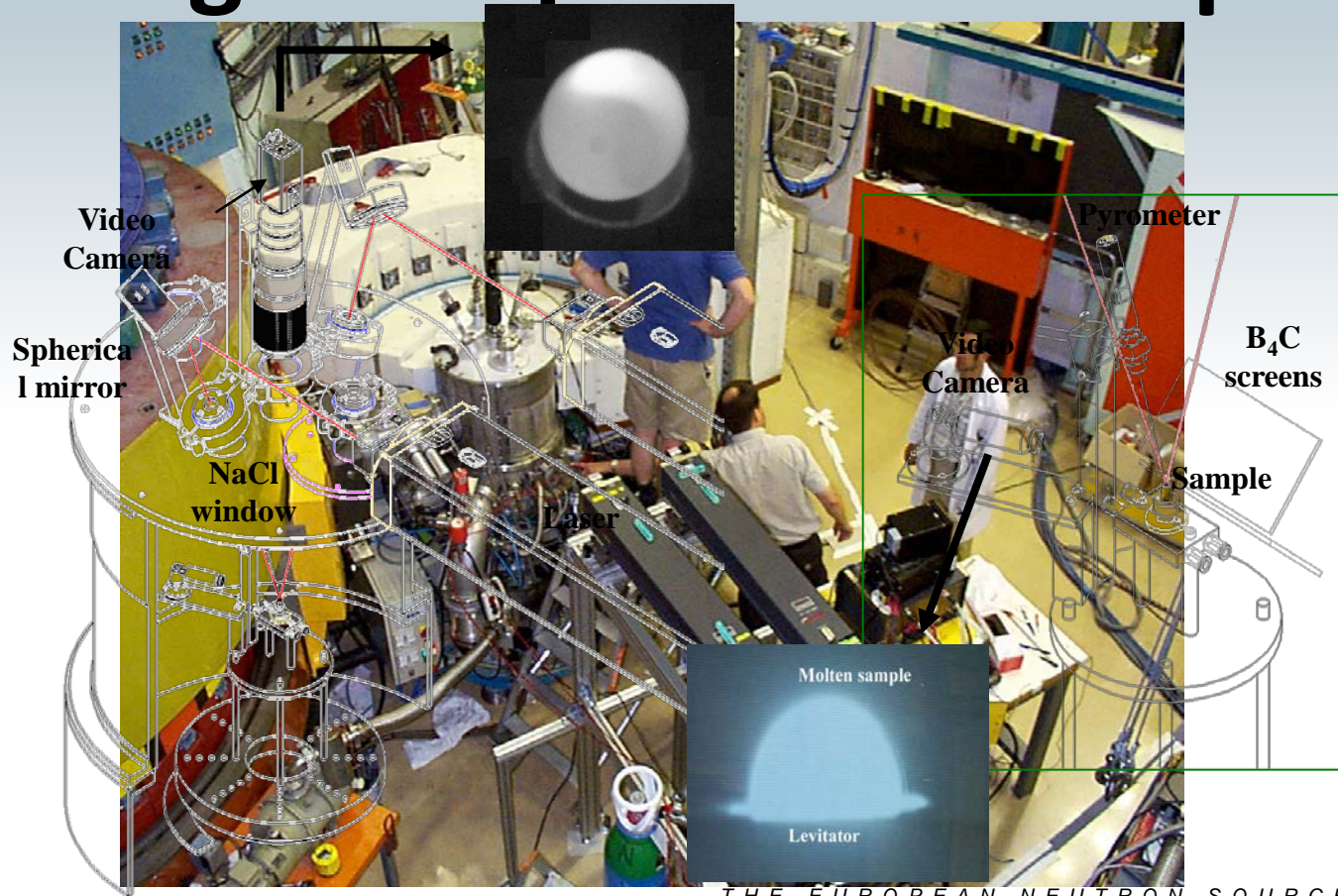
The light element helps aggregating clusters, which in turn are disaggregated by heating the system.

Levitation of Liquids

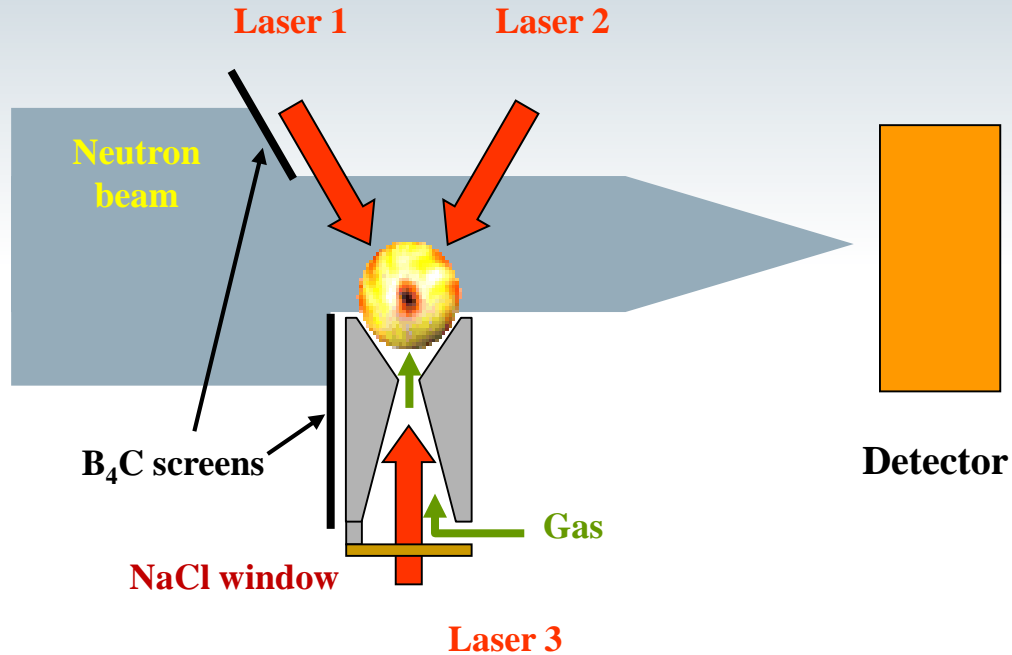
Principle of the aerodynamic levitation and laser heating



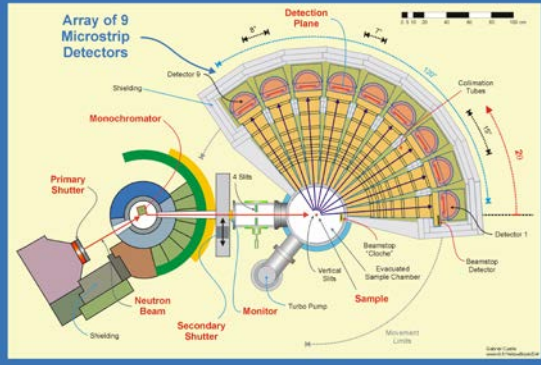
High Temperature Setup



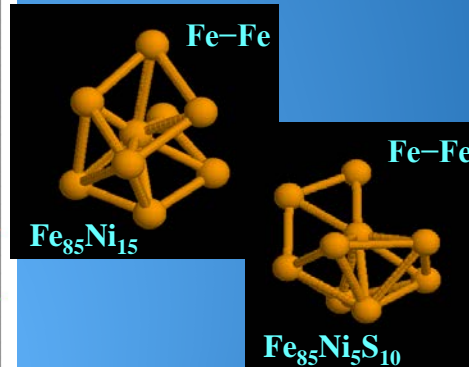
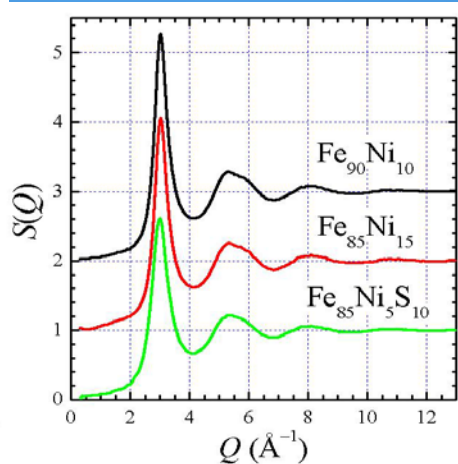
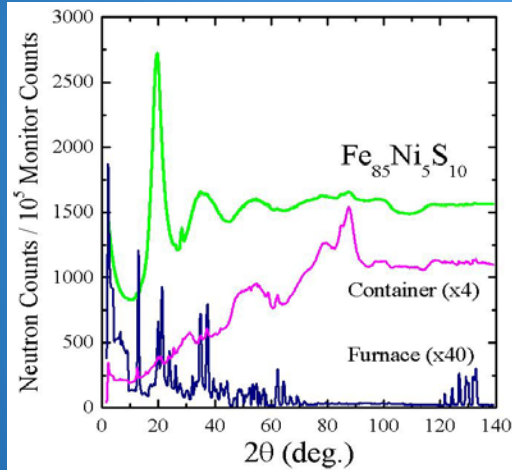
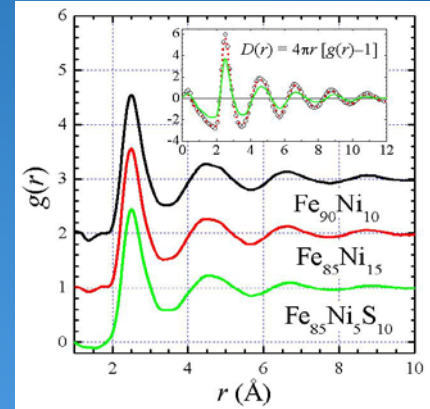
Three Lasers Setup



Some Applications: Liquid state

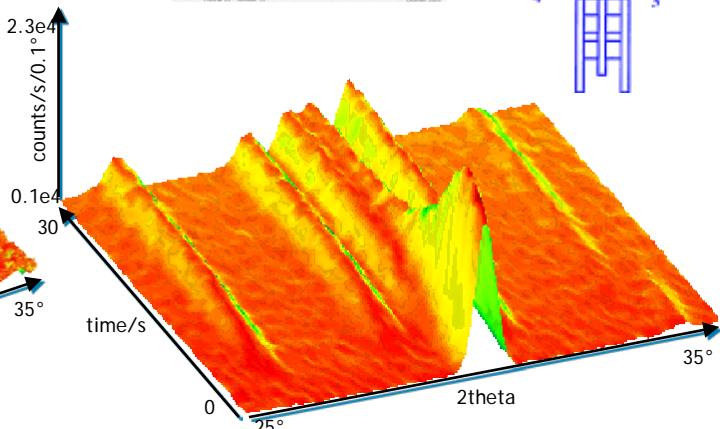
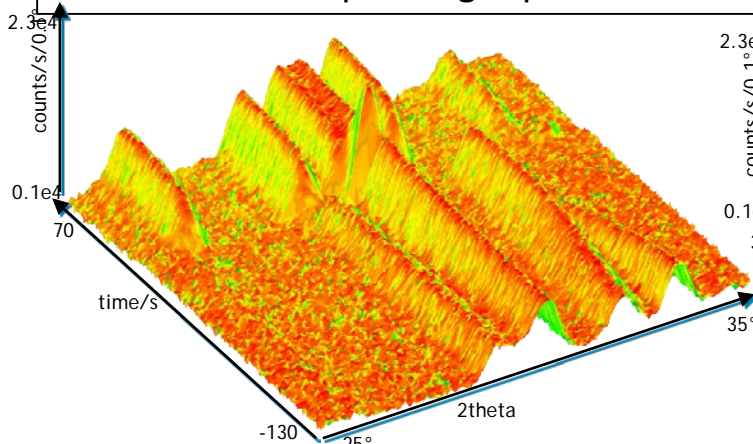
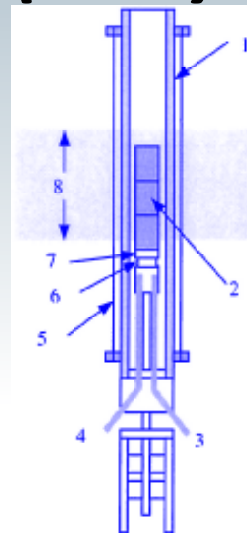
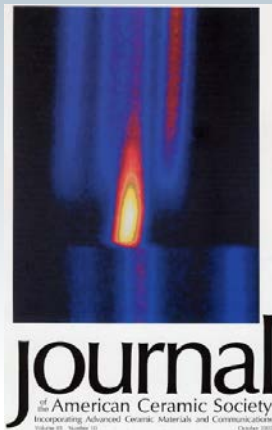


FeNi and FeNiS alloys
 Liquid state
 High temperature
 Special furnace
 Two-axis diffractometer



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

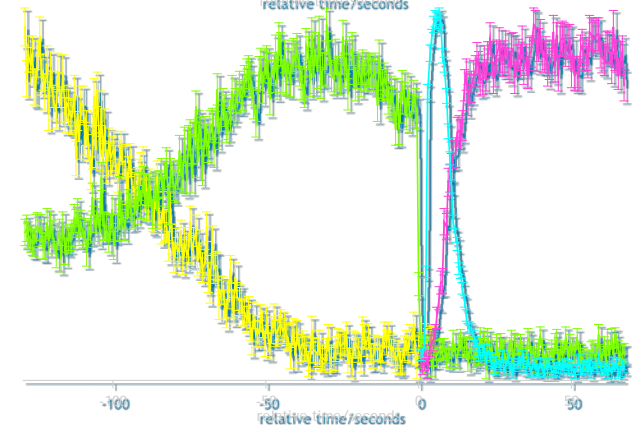
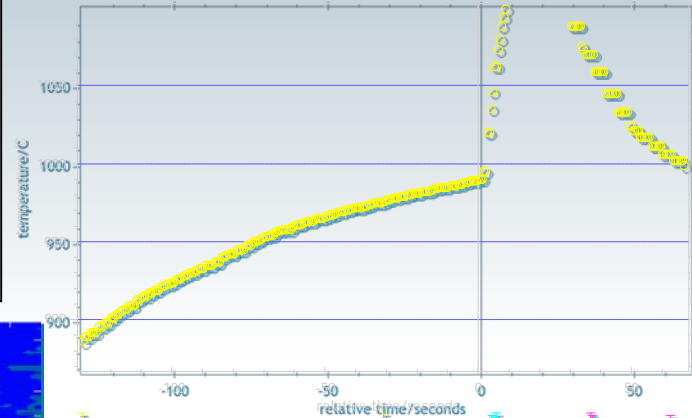
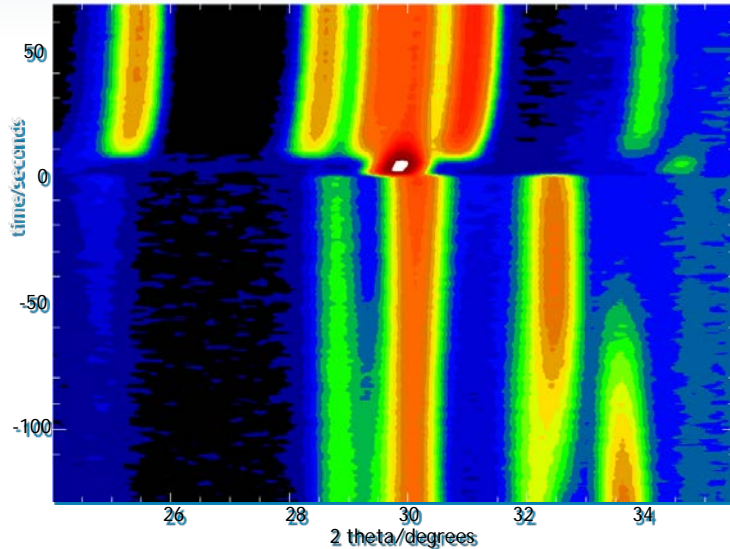


Thanks to
Thomas Hansen

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

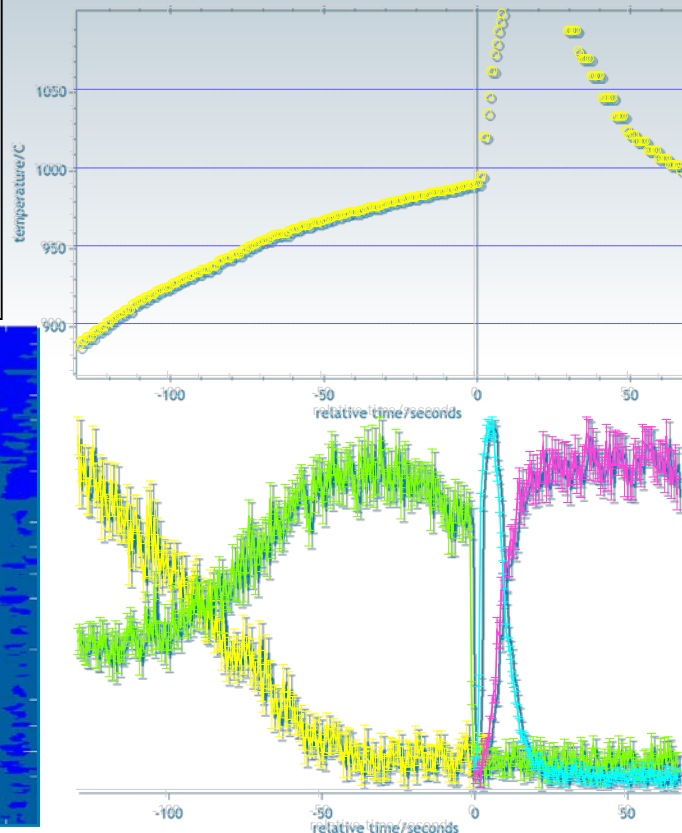
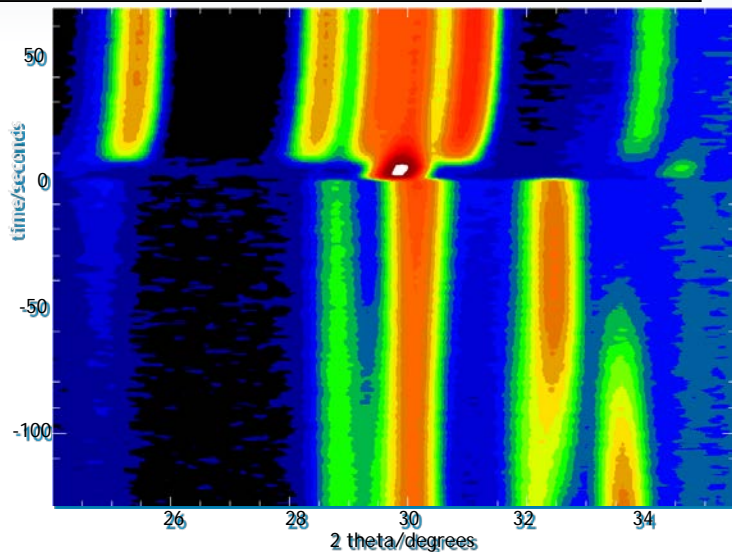
SHS: pre-ignition

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



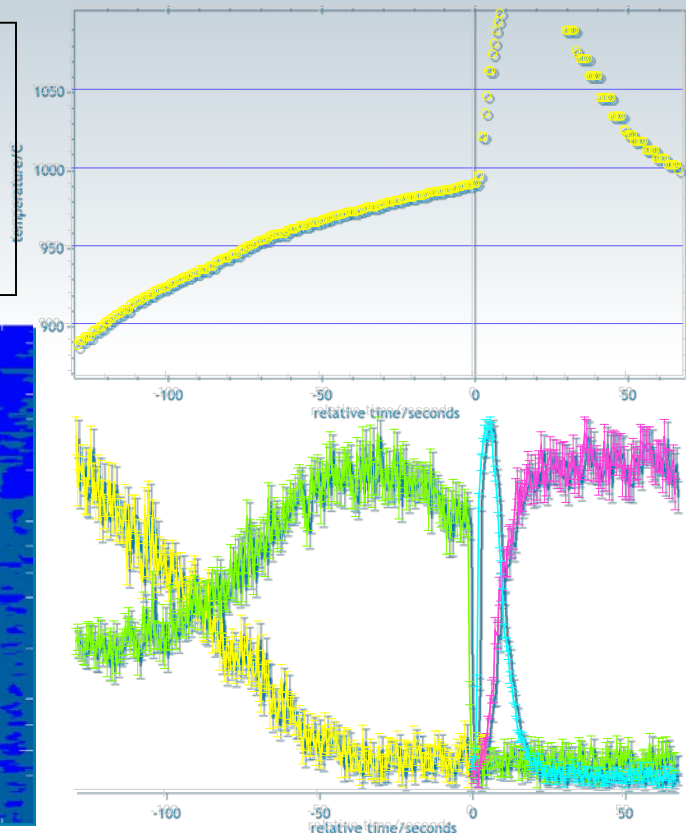
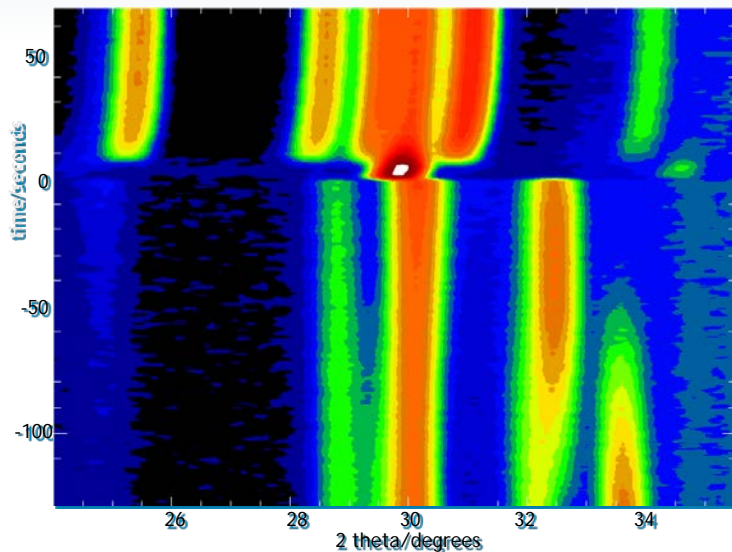
SHS: intermediate product

- 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



Thank you for your attention



I N S T

Facilities

Reference

Software

Conferences

Announcements

**Neutron
Scattering
Web**

www.neutron.anl.gov

Neutron Scattering Mailing Lists