Neutron scattering at high pressures: opportunities in geology

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Universidad Zaragoza

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Outline

- Introduction
- Basic concepts
- Instruments
- Examples
- How to apply for beam time

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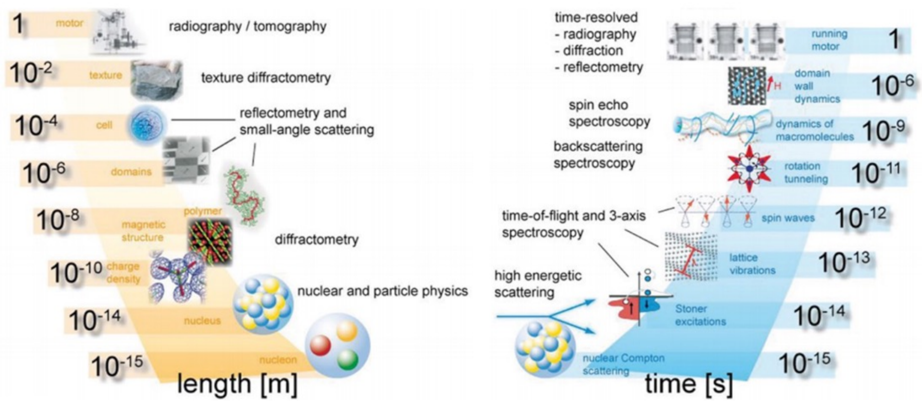
Introduction
Accesible Length and Energy scales



T. Brückel

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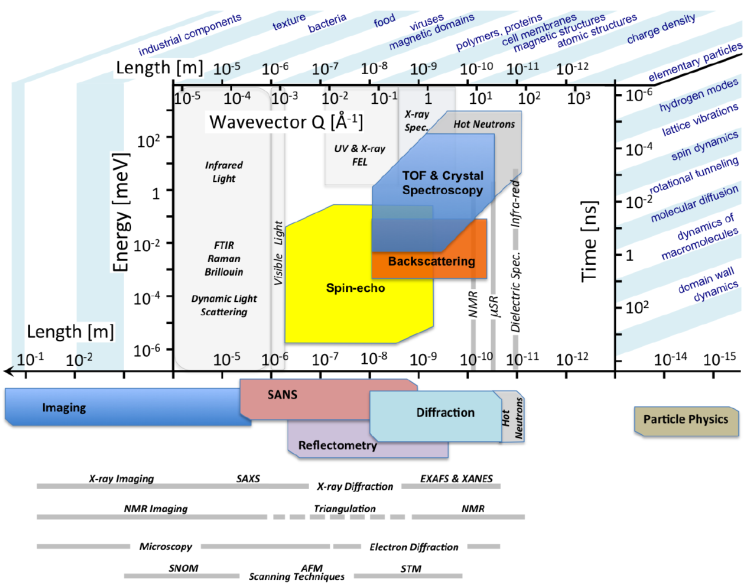
Complementarity



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Outline

- Introduction
- **Basic concepts**
- Instruments
- Examples
- How to apply for beam time

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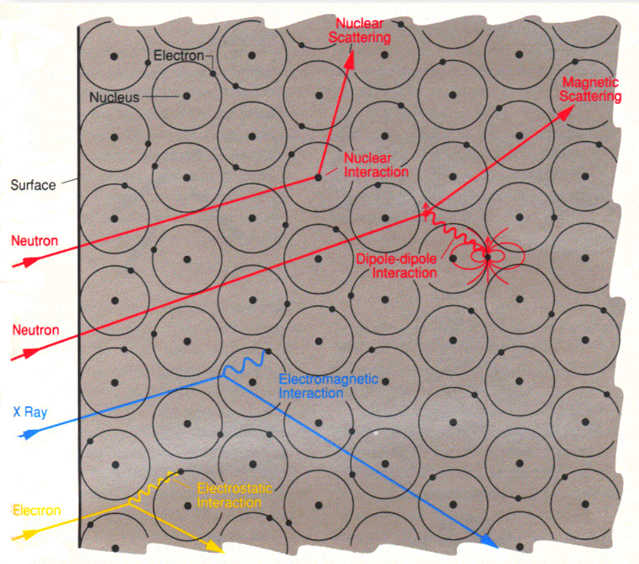
Probing the matter



R Pynn

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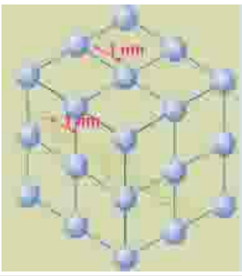
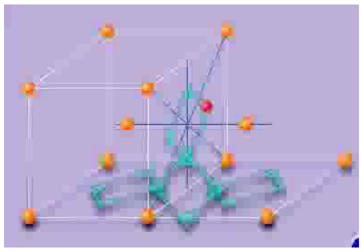
Basic concepts

Properties

- **Neutrons have similar wavelengths that the interatomic distances and therefore they can diffract**
  - Wide range of wavelengths (0.05 to 20 Å)
  - To observe different length scales

- **Same energy range that the atomic and electronic processes (meV a eV) with the possibility to detect changes less than μeV**
  - Tunneling,
  - Rotations
  - Vibrations
  - Electronic Transitions
  - Diffusion.





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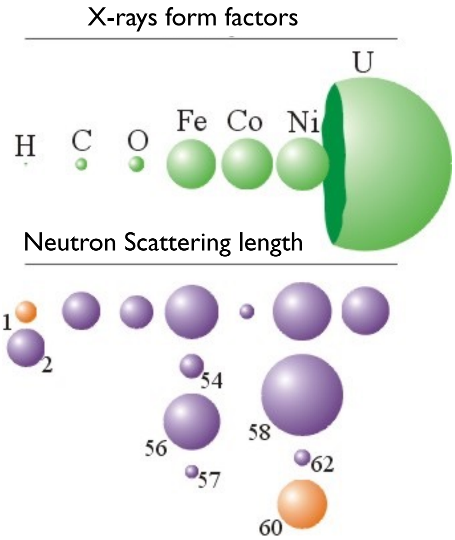
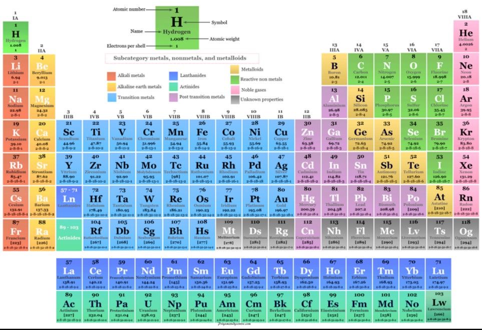
Basic concepts

Properties

- **The interaction neutron-matter is *via* nucleus (*strong interaction*) and not with the electronic cloud**
  - Scattering length for atoms does not depend on the atomic number Z
    - Easy to see light atoms (H, Li) in presence of heavy atoms
    - Easy to distinguish next neighbors atoms in the periodic table





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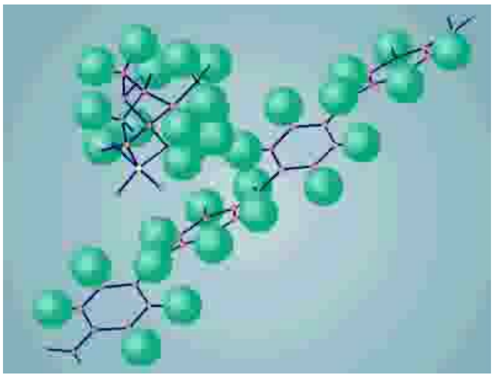
Basic concepts
Properties

The dependence of the scattering length with the nuclei do that different isotopes of the same element scatter in a very different way (isotopic substitution and contrast experiments)



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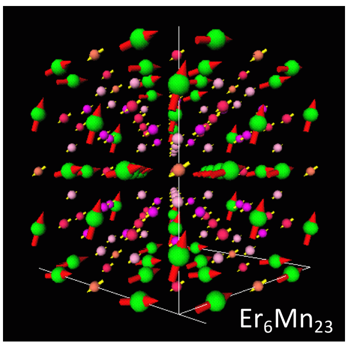
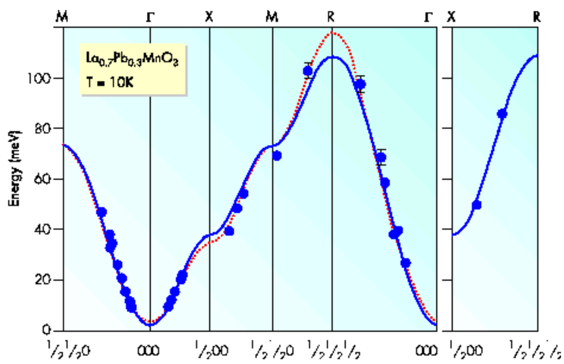
Basic concepts
Properties

Neutrons carry magnetic moment then they "see" (via the magnetic dipolar interaction) the magnetism present in the matter; unpaired electrons, nuclei

- To study magnetic structures
- Magnetic excitations





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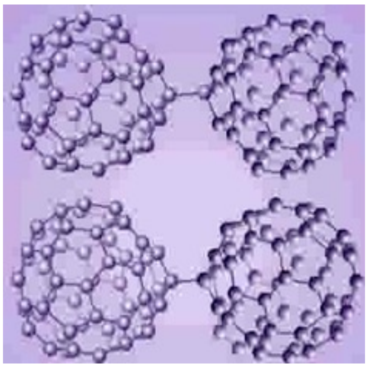
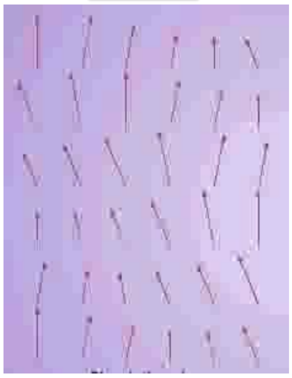
Basic concepts
Properties

- **With neutrons we can measure simultaneously the structure and the dynamics**

Phonons



Magnons



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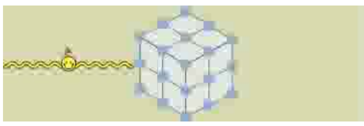
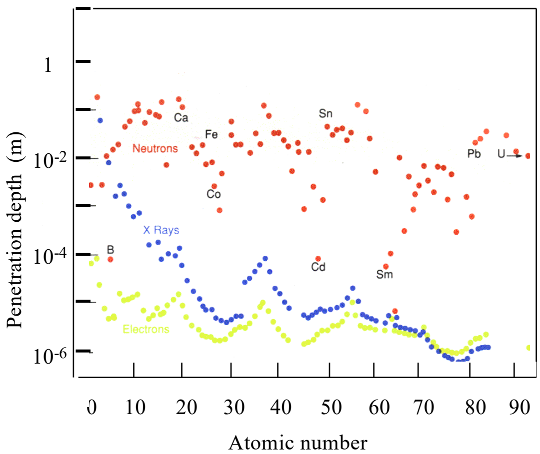
Basic concepts
Properties

Penetration. Neutron enters into materials without problems

- X Ray, EM , optical methods are surface probes.
- Hard exp. conditions
  - Cryostats
  - Furnaces
  - Pression clamps
  - Electrochemical cells
  - Etc…





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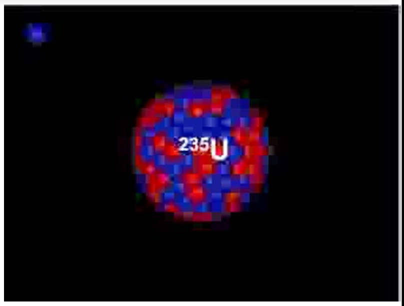
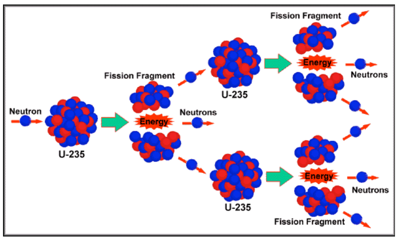
Basic Concepts
Neutron production: nuclear reactors





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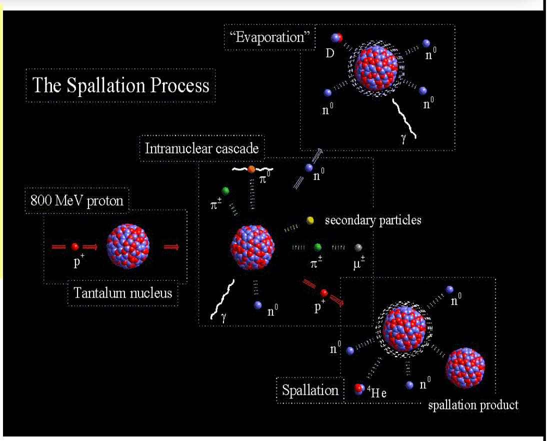
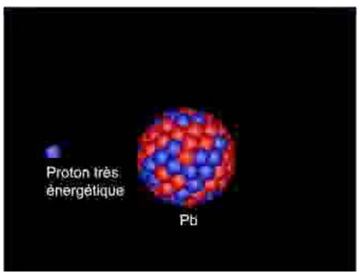
Basic Concepts
Neutron production: spallation sources

- H^+ accelerated to 1~5 GeV
- Target W, Pb, Hg…
- Average 20 to 25 neutrons / H^+
- 50 to 16 Hz Pulses
- Pulse time ~ µs to ms
- Pulsed Flux 3.7 10^{16} ns^{-1} (ISIS)





- SNS @ ORNL
- ISIS @ United Kingdom
- MLF @ JPARC

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Basic Concepts
Neutron production: spallation sources

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Basic Concepts
Neutron production: spallation sources

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Basic Concepts
Neutron production: spallation sources

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The European spallation source, ESS

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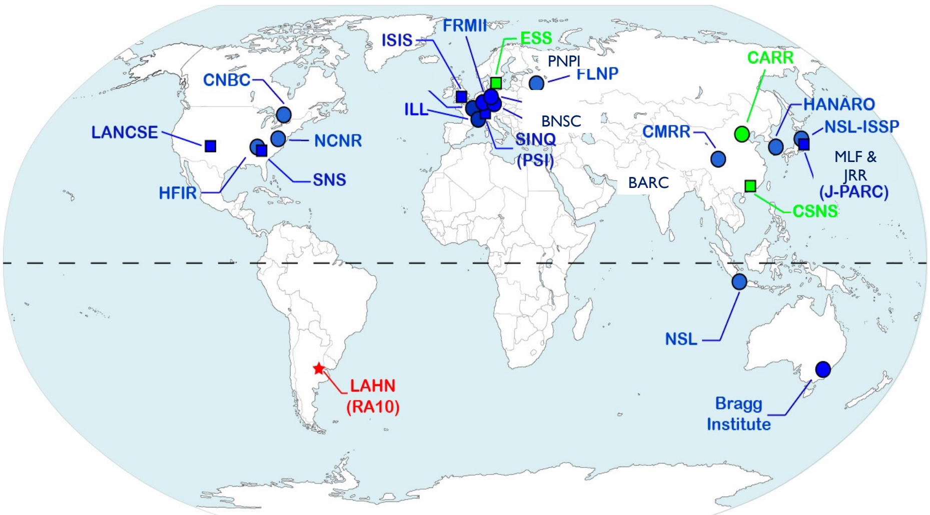
Some neutron sources



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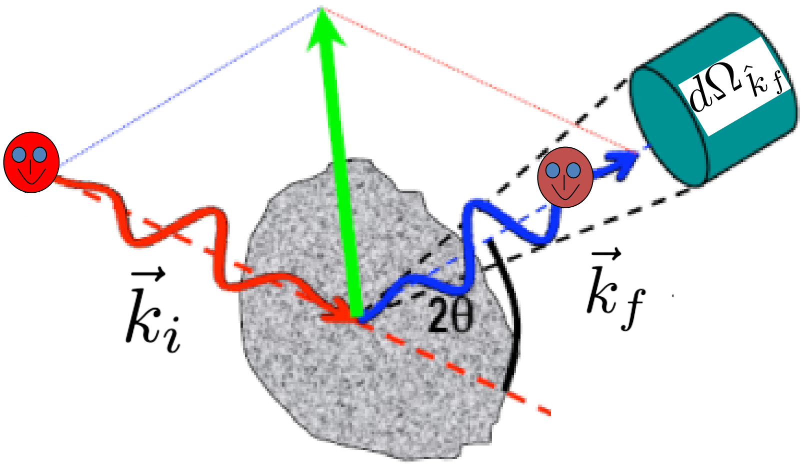
Exploring the matter with neutron scattering

$$\vec{q} = \vec{k}_f - \vec{k}_i$$



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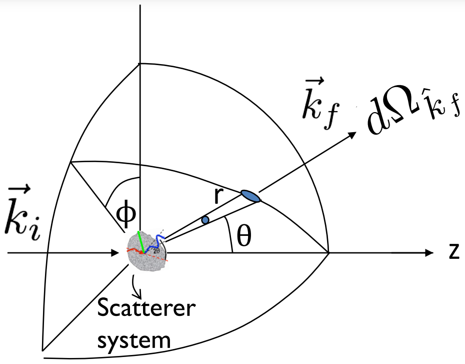
Scattering cross section

- **Double partial differential cross section**



$$\frac{d^2\sigma}{d\Omega_{\hat{k}_f}\, dE_f} = \frac{\text{Number of scattered neutrons per second in a solid angle } d\Omega_{\hat{k}_f} \text{ in the direction given by } \hat{k}_f \text{ with final energy between } E_f \text{ and } E_f + dE_f}{\varphi\ d\Omega_{\hat{k}_f}\, dE_f}$$

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Neutron scattering basic theory

- Consideremos un caso en el que la aproximación dipolar es válida y hagamos uso de las expresiones siguientes.

$$\delta(E_\lambda - E_{\lambda'} + \hbar\omega) = \frac{1}{2\pi\hbar}\int_{-\infty}^{\infty} \exp\{i(E_{\lambda'} - E_\lambda)t/\hbar\}\exp(-i\omega t)dt$$

$$\exp(i\mathbf{H}t/\hbar)|\lambda\rangle = \exp(iE_\lambda t/\hbar)|\lambda\rangle$$

- Llegamos a

$$\sum_{\lambda\lambda'} p_\lambda \langle\lambda|\exp(-i\boldsymbol{\kappa}\cdot\mathbf{R}_{l'd'})\mathbf{J}^{\alpha}_{l'd'}|\lambda'\rangle\langle\lambda'|\exp(i\boldsymbol{\kappa}\cdot\mathbf{R}_{ld})\mathbf{J}^{\beta}_{ld}|\lambda\rangle\delta(E_\lambda - E_{\lambda'} + \hbar\omega)$$

$$= \frac{1}{2\pi\hbar}\int_{-\infty}^{+\infty}\langle\exp\{-i\boldsymbol{\kappa}\cdot\mathbf{R}_{l'd'}(0)\}\mathbf{J}^{\alpha}_{l'd'}(0)\exp\{i\boldsymbol{\kappa}\cdot\mathbf{R}_{ld}(t)\}\mathbf{J}^{\beta}_{ld}(t)\rangle\exp(-i\omega t)dt$$

- Y la sección eficaz

$$\left(\frac{d^2\sigma}{d\Omega dE'}\right) = \frac{(\gamma r_0)^2}{2\pi\hbar}\frac{k'}{k}\sum_{\alpha\beta}(\delta_{\alpha\beta} - \hat{\kappa}_\alpha\hat{\kappa}_\beta)\sum_{l'd'}\sum_{ld}\frac{1}{4} g_{d'} g_d F^{*}_{d'}(\boldsymbol{\kappa})F_d(\boldsymbol{\kappa})\times$$

$$\int_{-\infty}^{+\infty}\langle\exp\{-i\boldsymbol{\kappa}\cdot\mathbf{R}_{l'd'}(0)\}\exp\{i\boldsymbol{\kappa}\cdot\mathbf{R}_{ld}(t)\}\rangle\langle\mathbf{J}^{\alpha}_{l'd'}(0)\mathbf{J}^{\beta}_{ld}(t)\rangle\exp(-i\omega t)dt$$

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Bibliography

- G. L. Squires, "*Thermal neutron scattering*", Cambridge Universtity Press, 1978
- S.W. Lovesey, "*Theory of neutron scattering from Condensed Matter*", Vol I y II Oxford University Press, 1986
- Javier Campo and Víctor Laliena "*Neutron Scattering in Magnetism: Fundamentals and Examples*" https://doi.org/10.1007/978-3-030-70443-8_14

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Diffraction

Structure factors

$$I(\vec{q}) = \frac{(2\pi)^3}{V_0} \sum_{\vec{H}} \delta(\vec{H} - \vec{q}) |N_{\vec{q}}|^2$$

Diffraction

$$N_{\vec{q}} = \sum_{d} \Psi_d \, e^{i2\pi\vec{q}\cdot\vec{r}_d} e^{-W_d(\vec{q})}$$

Nuclear Structure factor

$$\Psi_d(\vec{q}) = b_d$$

Neutrons *Scattering lenght*

$$V_d(\vec{r}_d) = \frac{2\pi\hbar^2}{m} b_d \delta(\vec{r}_d)$$

$$\Psi_d(\vec{q}) = f_d(\vec{q})$$

XR *Form factors*

$$V_d(\vec{r}_d) \propto \rho_d(\vec{r}_d)$$

Debye-Waller factor

$$W_d(\vec{q}) = \langle \{\vec{q} \cdot \vec{u}_d(0)\}^2 \rangle$$

$$T_d(\vec{q}) = e^{-W_d(\vec{q})}$$

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General expression for NON polarised neutrons in crystals

Nuclear structure factor

$$\frac{d\sigma_{\mathrm{coh}}^{(\mathrm{n,e})}}{d\Omega_{\hat{k}_f}} = \frac{N_c}{V}\sum_{\vec{H}}\delta(\vec{q}-\vec{H})|N_{\vec{q}}|^2. \qquad N_{\vec{q}} = \sum_{d}^{N_{\mathrm{uc}}} b_d\, T_d(\vec{q})\, \mathrm{e}^{\mathrm{i}2\pi\vec{q}\cdot\vec{r}_d}$$

Magnetic Interaction Vector

$$\frac{d\sigma_{\mathrm{coh}}^{(\mathrm{m,e})}}{d\Omega_{\hat{k}_f}} = \frac{N_c}{V}\sum_{\vec{H}\vec{K}}\delta(\vec{q}-\vec{H}-\vec{K})\,|\vec{M}_{\perp\vec{q}}|^2 \qquad \vec{M}_{\perp\vec{q}} = \hat{q}\times(\vec{M}_{\vec{q}\vec{K}}\times\hat{q})$$

$$I_{\vec{q}} = |N_{\vec{q}}|^2 + |\vec{M}_{\perp\vec{q}}|^2$$

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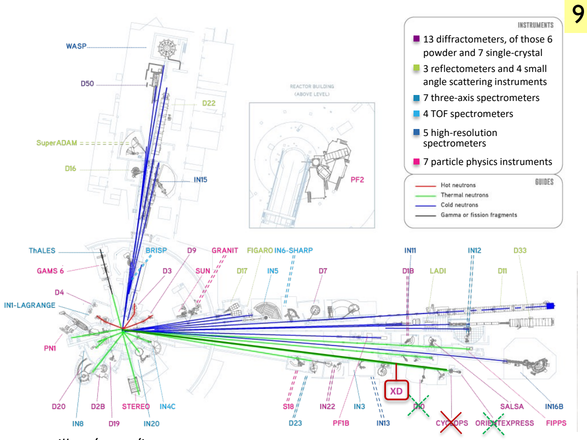
Outline

- Introduction
- Basic concepts
- **Instruments**
- Examples
- How to apply for beam time

ILL instrument suit

29 instruments
9 CRG instruments



www.ill.eu/users/instruments

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Spanish instruments at the ILL: XtremeD and D1B

Two Spanish instruments at the ILL (XtremeD and D1B) where we could perform "*high pressure*" experiments

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In brief

In brief

A **dedicated** diffractometer optimized for extreme conditions (**high P, high H**, low and high T) but with **flexibility** to accommodate a broad range of studies

Technically:

A flexible powder diffractometer with single crystal capabilities, with a variable focused beam on the sample, optimized signal/background ratio, big solid angle position-sensitive detector, and dedicated sample environment

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What is around?

How XtremeD compares?

In the general diffraction context:

- Less P than X-ray but essential for **light elements**, contrast, 'high quality' crystallography and **magnetism**

In the neutron facilities context:

- High intensity at low Q (w.r.t. most short pulse spallation instruments)
- Extended H & P range and **combination** of both parameters
- Both powders & single crystals
- Flexibility

In ILL's context:

- **Dedicated**
- Flux ~D20 but better signal to noise ratio and bigger solid angle
- Only single Xtal diffractometer at ILL with large 2D detector + high H (D19: space restrictions for high magnetic fields)

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Sample environment

cryostat

PE press

cryomagnet

HT furnace

pressure cell

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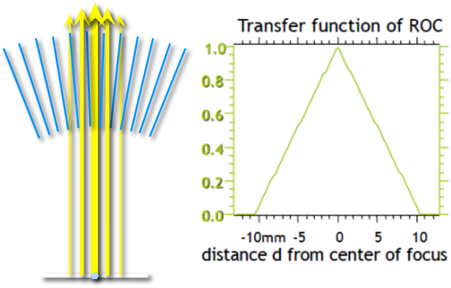
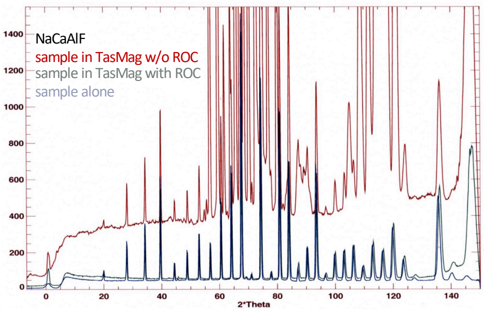
Collimation

Radial oscillating collimator



ROC & data quality



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XtremeD: current status

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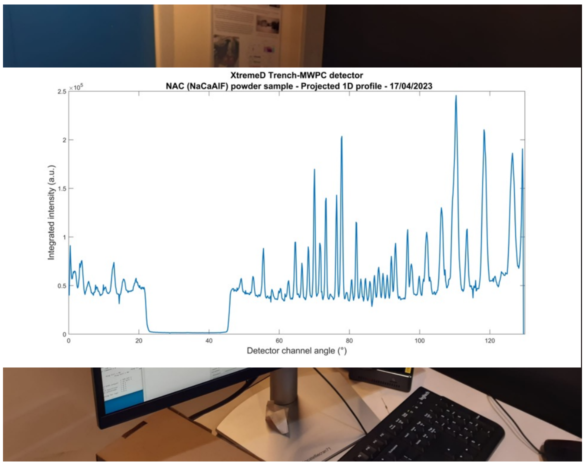
First neutrons

April 17, 2023



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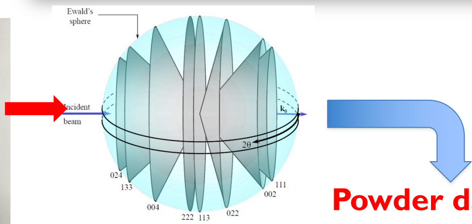
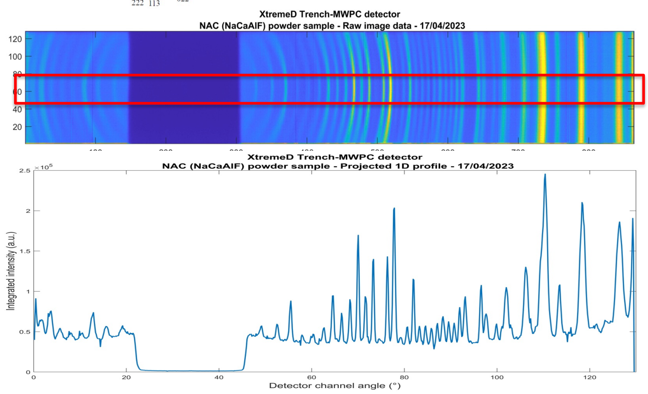
Powder diffraction



Powder data: 2D detector view



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XtremeD: resolution

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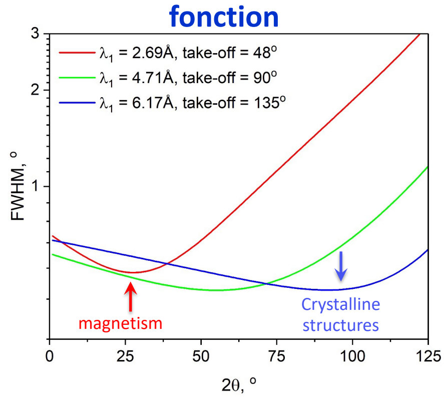
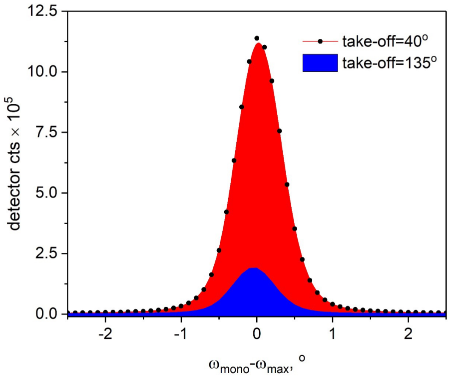
Instrument Resolution fonction



flux vs. take-off (HOPG)



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Applications: XtremeD vs. D20

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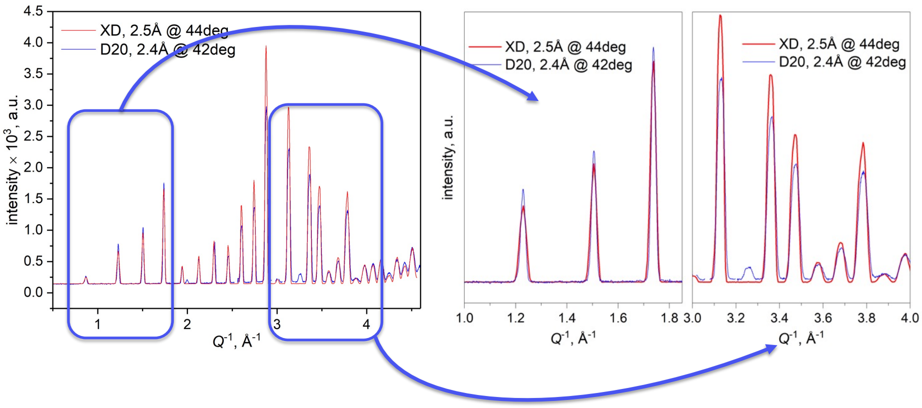

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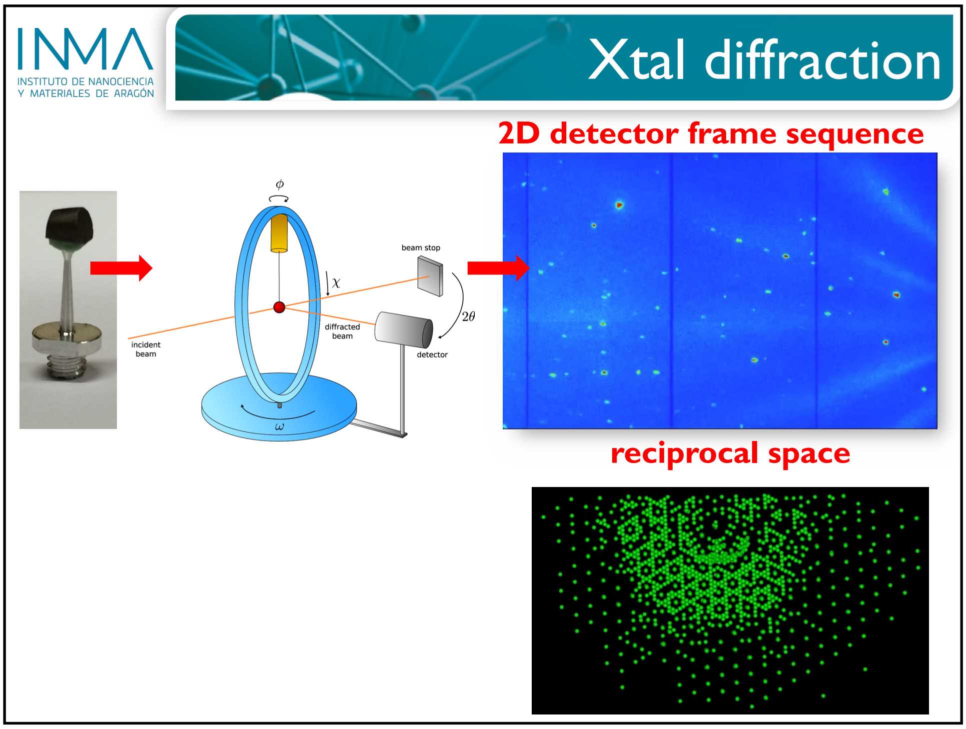
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Xtal diffraction
2D detector frame sequence
φ
χ
beam stop
2θ
incident beam
diffracted beam
detector
ω
reciprocal space

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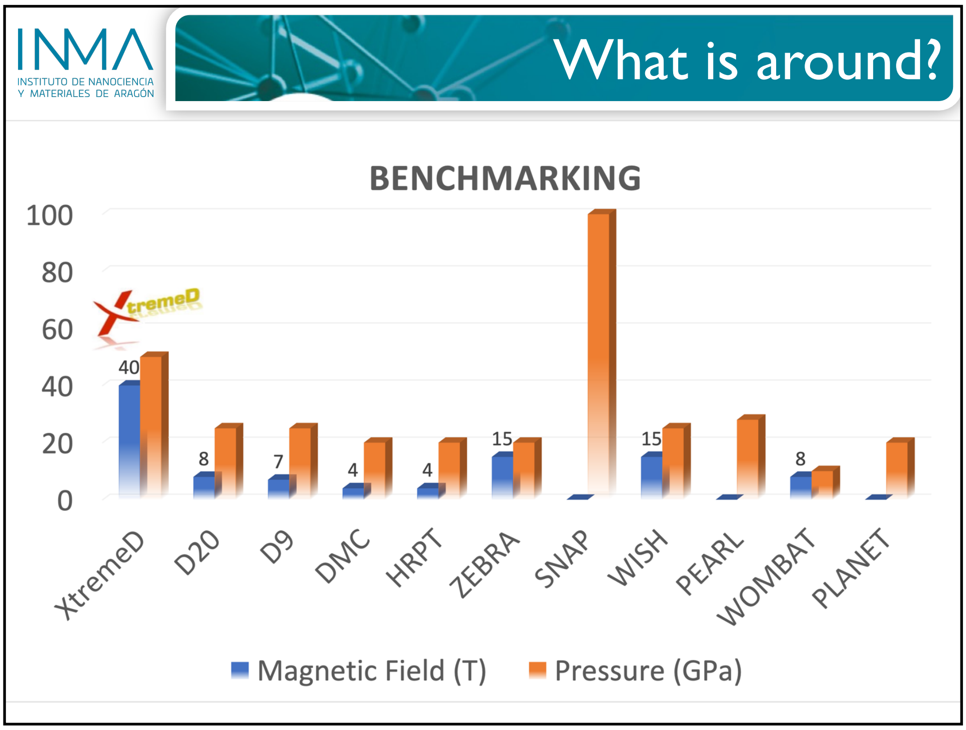
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What is around?
BENCHMARKING
100
80
60
40
20
0
XtremeD
40
8
7
4
4
15
15
8
XtremeD
D20
D9
DMC
HRPT
ZEBRA
SNAP
WISH
PEARL
WOMBAT
PLANET
Magnetic Field (T)
Pressure (GPa)

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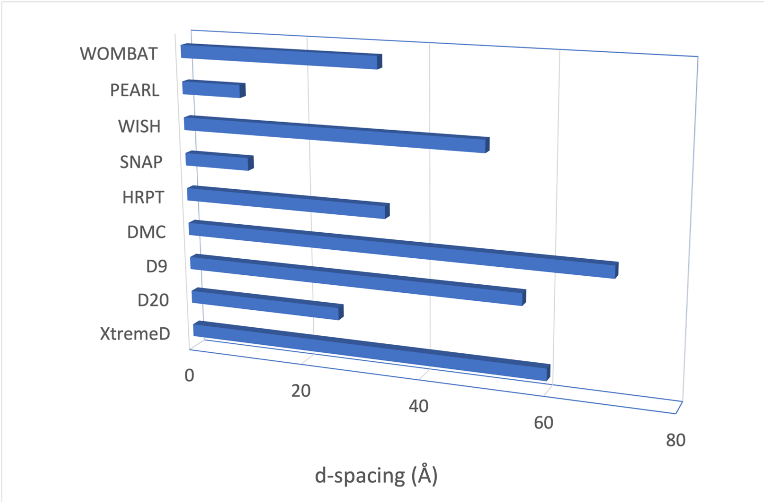
What is around?

Short Pulse Spallation source = Inaccessibility to low Q-reflections
VERY IMPORTANT FOR MAGNETISM



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Ringwoodite [$(Mg,Fe^{2+})_2SiO_4$ spinel]

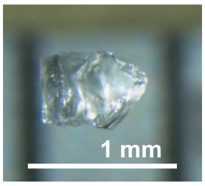
research papers

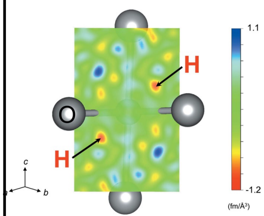
STRUCTURAL SCIENCE
CRYSTAL ENGINEERING
MATERIALS

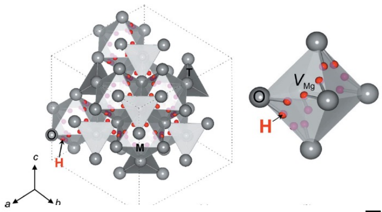
ISSN 2052-5206

Determination of hydrogen site and occupancy in hydrous Mg_2SiO_4 spinel by single-crystal neutron diffraction

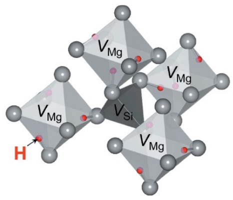
Narangoo Purevjav,[a*] Takuo Okuchi,[a] Xiaoping Wang,[b] Christina Hoffmann[b] and Naotaka Tomioka[c]







Refined crystal structure of hydrous ringwoodite along [111] and hydrogen sites in a vacant Mg site.



The most plausible model (3H+ at 192*i* sites within an Mg-vacant octahedron)

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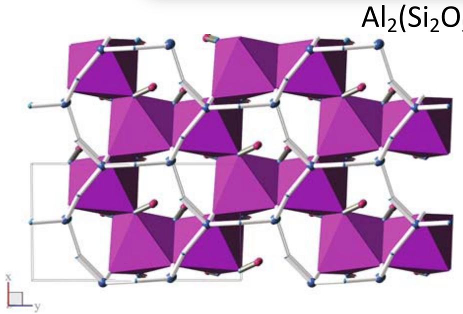
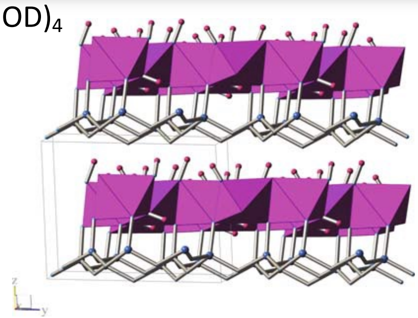
Kaolinite [$Al_2(Si_2O_5)(OD)_4$]

$Al_2(Si_2O_5)(OD)_4$





Crystal structure of kaolinite as refined from neutron diffraction. Layer slab with the $[AlO_6]$-octahedra in dark shading showing hydroxyl terminations between layers (left) and the excess hydroxyl between tetrahedral silicate layer and those oxygens that are not charge balanced (right) are shown.

Neutrons clearly identify the location of the protons

E. Akiba, et al. Clays Clay Miner. 45, 781 (1997)

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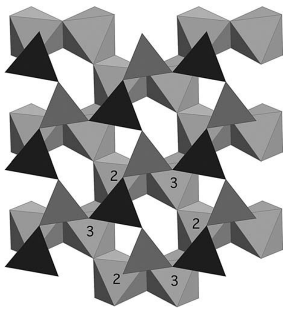
Cation order-disorder

- Cation order-disorder in Olivines and Spinels at high temperature
  - Fe-Ni, Mn-Fe, Mg-Al, Ni-Mn, Ni-Mg, etc…
- Mg/Al Ordering in Dioctahedral Micas
  - $K_{0.9}[Mg_{0.58}Al_{1.43}][Si_{3.57}Al_{0.43}]O_{10}(OH)_2$
- Al/Si and Mg/Al ordering in high-temperature amphibole
  - $NaCa_2[Mg_4Al]$ $[Si_6Al_2]O_{22}(OH)_2$,



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other

- Mechanical Behavior of Geological Materials
  - if the deformation experiment is performed on a neutron beam line, the deformation behavior within the sample can be monitored as it is being deformed from diffraction patterns collected at different stages of the deformation
- Texture determination
  - neutron texture goniometry affords true volume texture measurements of relatively large (up to several cm^3) isometric samples

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High Pressure

What would be the *neutrons at HP* contribution?

The study of these systems implies systematic studies of **hydrogen bonding and clustering, host-guest interactions in clathrate hydrates, the role of water in crystals and cation order disorder phase transitions.**

- Planetary ices (water, methane, ammonia) and clathrates
- Segregation and related phenomena in water solutions at extreme conditions
- Mineral hydration: implications for the water cycle in Earth crust and mantle
- Hydrothermal reactions
- Cation order-disorder

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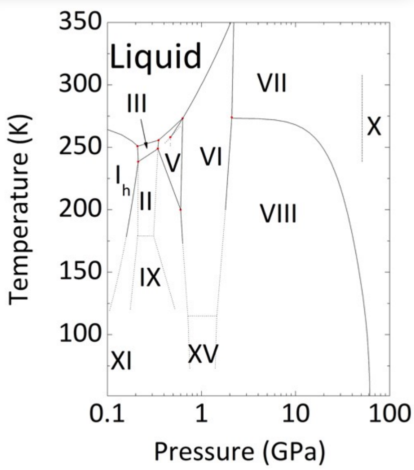
Simple molecules	D_2O, D_2, NH_3, CH_4
Larger molecules	KDP
Hydrides	MgH_2, gamma-CoH, MgD_2 : TiD_2 mixture, Fe hydrides, LiD, NaD, AlD_3
perovskite hydrides	$Na_{1-x}Li_xMgH_3$, the ternary hydrides Mg_2NiH_4 hydride and Mg_3CuH_x, Laves phase hydrides
Hydroxides	MOH family (M = Li,Na,K,...), $M(OH)_2$ hydroxides (M = Mn, Fe, Co, Ni, Cd, Mg and Ca)

Guthrie, J. Phys.: Condens. Matter 27 (2015) 153201



Currently 16 distinct crystalline phases of ice have been measured experimentally.

Pruzan P 1998 The Phase Diagram of H_2O

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Hydrogen atoms

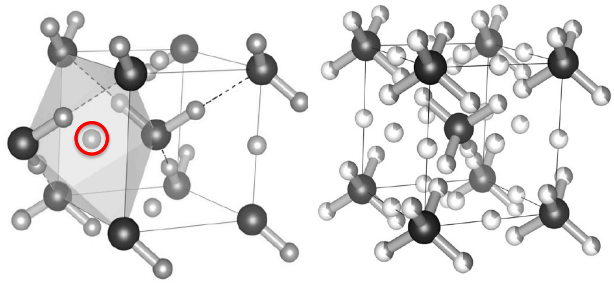
PNAS

Neutron diffraction observations of interstitial protons in dense ice

Malcolm Guthrie[a,1], Reinhard Boehler[a], Christopher A. Tulk[b], Jamie J. Molaison[b], António M. dos Santos[b], Kuo Li[a], and Russell J. Hemley[a,1]

Location of hydrogen atoms

25-50 GPa
RT



Dense ices:
- Starting of the destabilisation of water molecule.
- This phase, precursor of superionic behaviour (ice X)?
- Intermediate dissociation, with **H occupying of interstitial sites**

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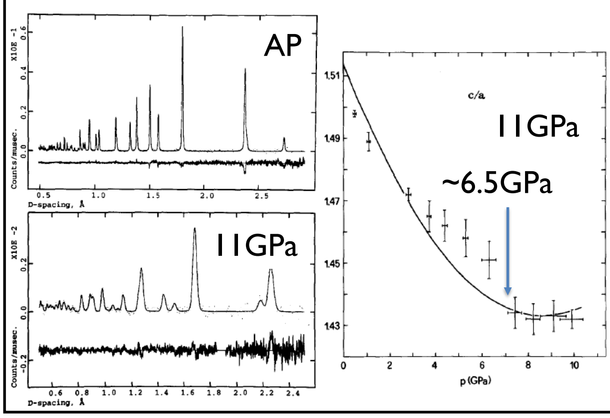
Phase transformations

Static Compression and H Disorder in Brucite, $Mg(OH)_2$, to 11 GPa: a Powder Neutron Diffraction Study

M. Catti[1], G. Ferraris[2], S. Hull[3], A. Pavese[4]

PHYSICS AND CHEMISTRY OF MINERALS

Polaris @ISIS



The onset of H disorder, and a jump of the c/a ratio vs. pressure at ~6.5 GPa, may be related to a second-order phase transition consistent with Raman results.

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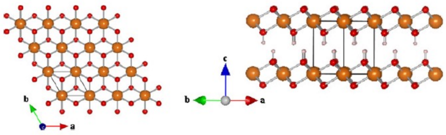
Effect of the H-D isotopes

Phys Chem Minerals (2010) 37:741–749
DOI 10.1007/s00269-010-0372-5

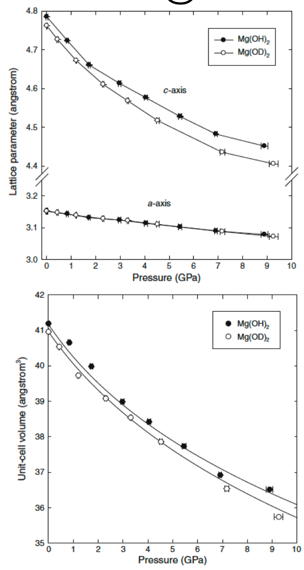
ORIGINAL PAPER

High-pressure neutron diffraction study on H–D isotope effects in brucite

Juske Horita · António M. dos Santos · Christopher A. Tulk · Bryan C. Chakoumakos · Veniamin B. Polyakov



WAND@HFIR



- a axis, parallel to the sheets of Mg–O octahedra, decrease only slightly with pressure with no effect of H–D substitution.
- c axis of $Mg(OD)_2$ is shorter and may exhibit greater compressibility with pressure than that of $Mg(OH)_2$.
- brucite preferentially incorporate deuterium over hydrogen under pressure

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Symmetrization O-H···O

SCIENTIFIC REPORTS

OPEN

Direct observation of symmetrization of hydrogen bond in δ-AlOOH under mantle conditions using neutron diffraction

Received: 18 June 2018
Accepted: 1 October 2018
Published online: 19 October 2018

Asami Sano-Furukawa[1], Takanori Hattori[1], Kazuki Komatsu[2], Hiroyuki Kagi[2], Takaya Nagai[3], Jamie J. Molaison[4], António M. dos Santos[4] & Christopher A. Tulk[4]

Theoretical studies have indicated that symmetrization triggers an increase in the bulk modulus and that it modifies the seismic wave velocity

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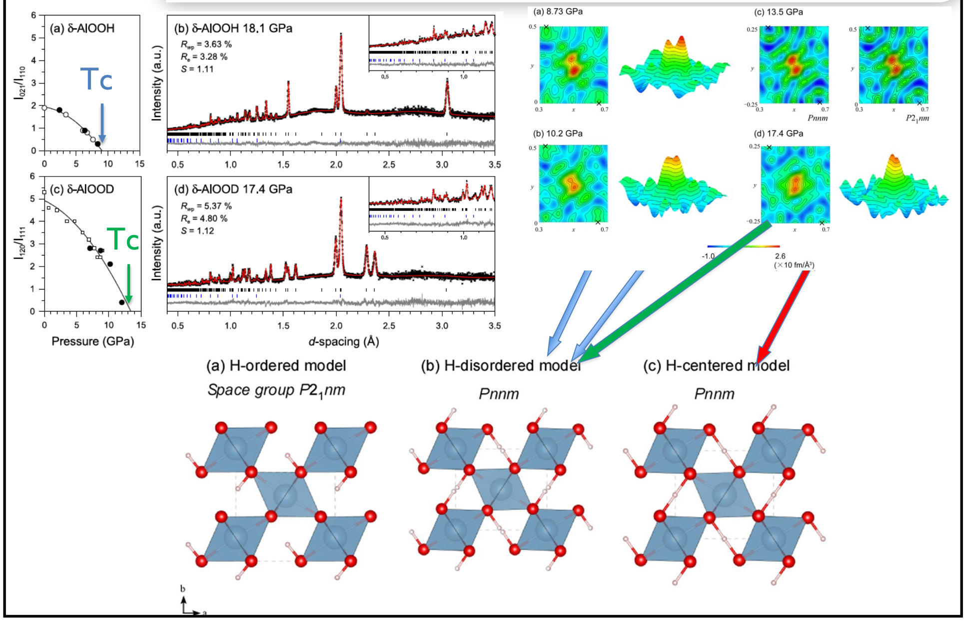
Symmetrization O - H ··· O



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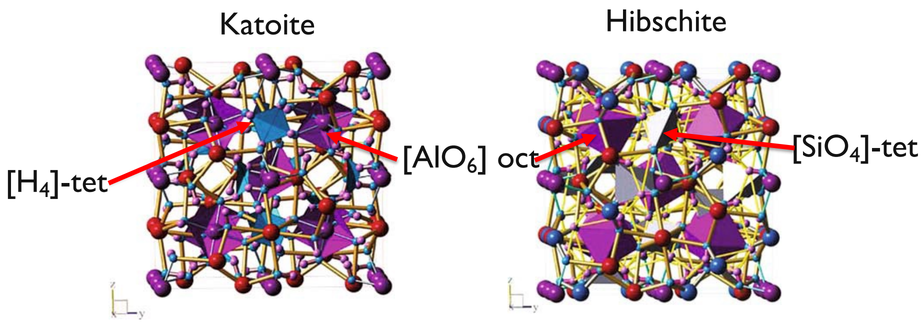
Hydrogarnets

- Hydrogarnet katoite, $Ca_3Al_2(O_4D_4)_3$, is used as a model for incorporation of (OH) into garnets and has been proposed as water carriers



in katoite the $[SiO_4]$-tet is almost completely replaced by the $[H_4O_4]$-tet, in hibschite, $Ca_3Al_2(SiO_4)_{2.30}(OD)_{2.8}$, still contains considerable amounts of residual $[SiO_4]$

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High Pressure and Temperature

Physics and Chemistry of Minerals (2019) 46:459–469
https://doi.org/10.1007/s00269-018-1016-4

ORIGINAL PAPER

Crystal structure change of katoite, $Ca_3Al_2(O_4D_4)_3$, with temperature at high pressure

Atsushi Kyono[1] · Masato Kato[1] · Asami Sano-Furukawa[2] · Shin-Ichi Machida[3] · Takanori Hattori[2]

effect of hydrogen on the katoite's phase transition under high-pressure and high-temperature conditions,

→ → high-temperature neutron diffraction measurements at about 8 GPa where it is expected that the phase transition from *Ia3d* to another one is observed

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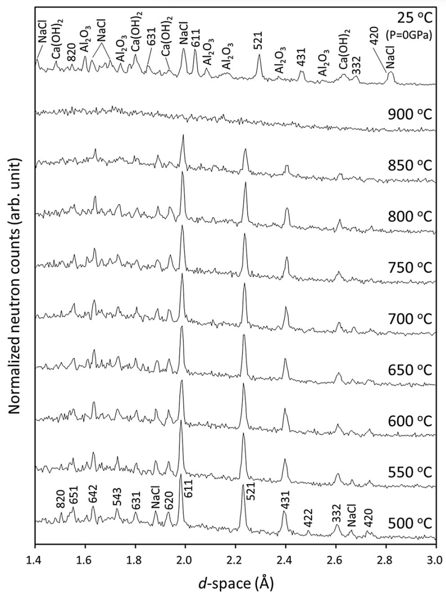
Thermal decomposition of katoite at high pressure



At 850 °C, all peaks corresponding to katoite start to decrease in intensity

the peaks from NaCl also disappear

katoite virtually unchanged during heating from 25 to 800 °C,

The sample pressure increased gradually from 7.1 to 7.8 GPa on heating up to 850 °C.

After cooling to RT and subsequent decompression to ambient pressure, the peaks of katoite reappear, together with those of corundum Al_2O_3 and portlandite $Ca(OH)_2$.

high pressure strongly suppresses the dehydration of katoite.

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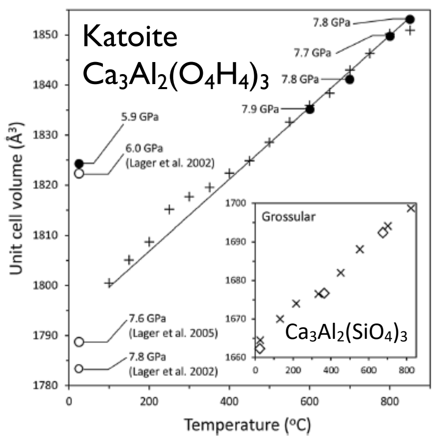
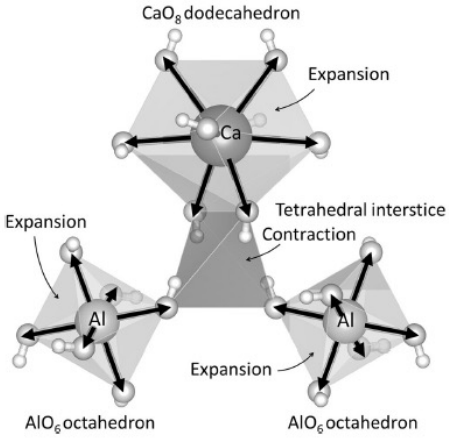
P-T phase diagram





By a further increase in pressure, hydrous silicates expand their stable temperature region to the higher temperatures. This study revealed that katoite remains fundamentally stable over a wide P–T area, at least up to 8 GPa and 850 °C.

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Magnetism of rare earths at high pressure

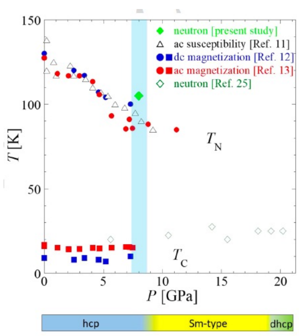
scientific reports

Check for updates

OPEN

Revisiting the magnetic structure of Holmium at high pressure by using neutron diffraction

M. Pardo-Sainz[1,2], F. Cova[1], J. A. Rodríguez-Velamazán[3], I. Puente-Orench[1], Y. Kousaka[4], M. Mito[5] & J. Campo[1]✉



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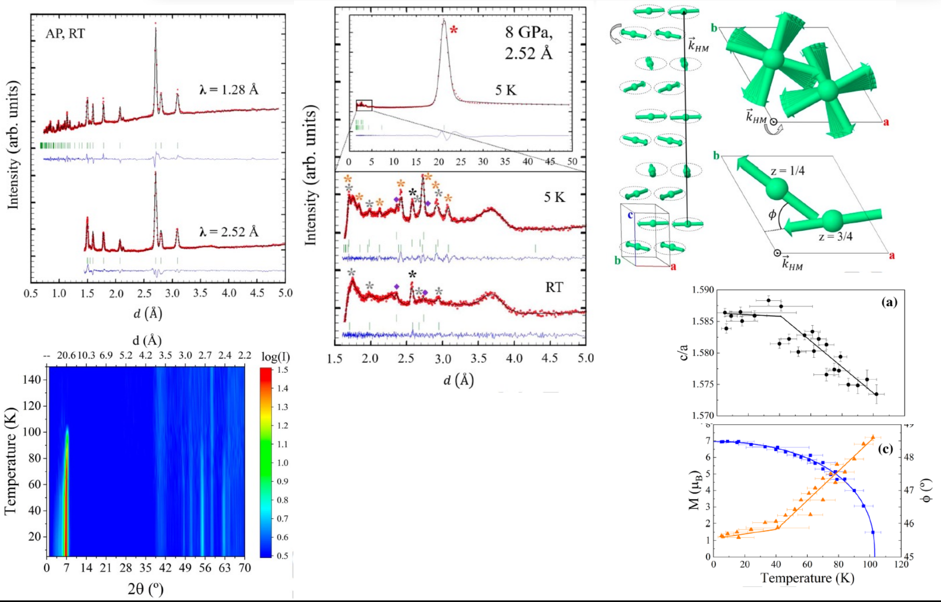

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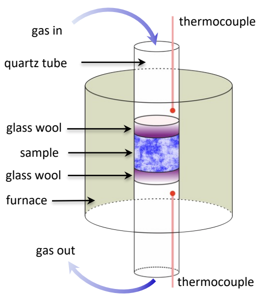
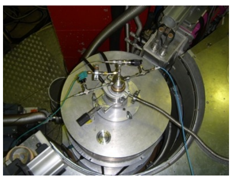
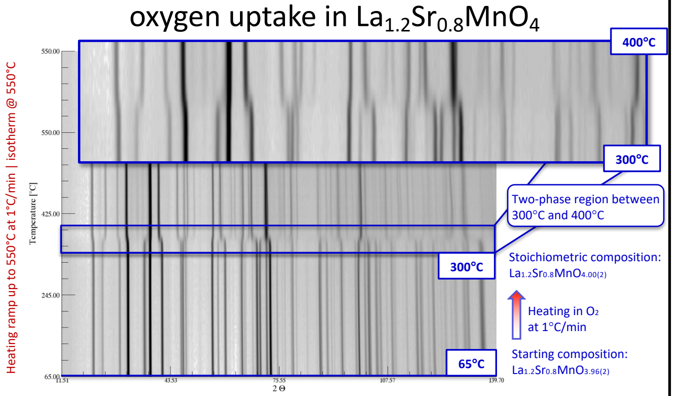
“*in situ*” studies





oxygen uptake in $La_{1.2}Sr_{0.8}MnO_4$



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Outline

- Introduction
- Basic concepts
- Instruments
- Examples
- How to apply for beam time

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How to apply for beamtime

- Do you have a good idea for neutron scattering?
- Is neutron scattering the only technique to solve the problem?
- Do you have any previous experiments relevant for the proposal?
- Did you contact any scientist specialist in NS to help you with the more technical questions? (Local Contact…)
- Did you think in the expected results of the experiment?
- How will you analyze your data?
- How did you estimate the requested beamtime?
- Which is the best instrument and neutron source?
- Etc…

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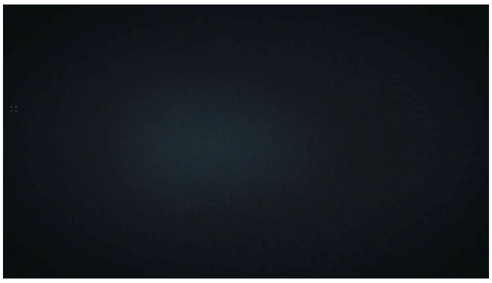
Thanks

Thank you very much for your attention !!

Our webpage m4.unizar.es



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