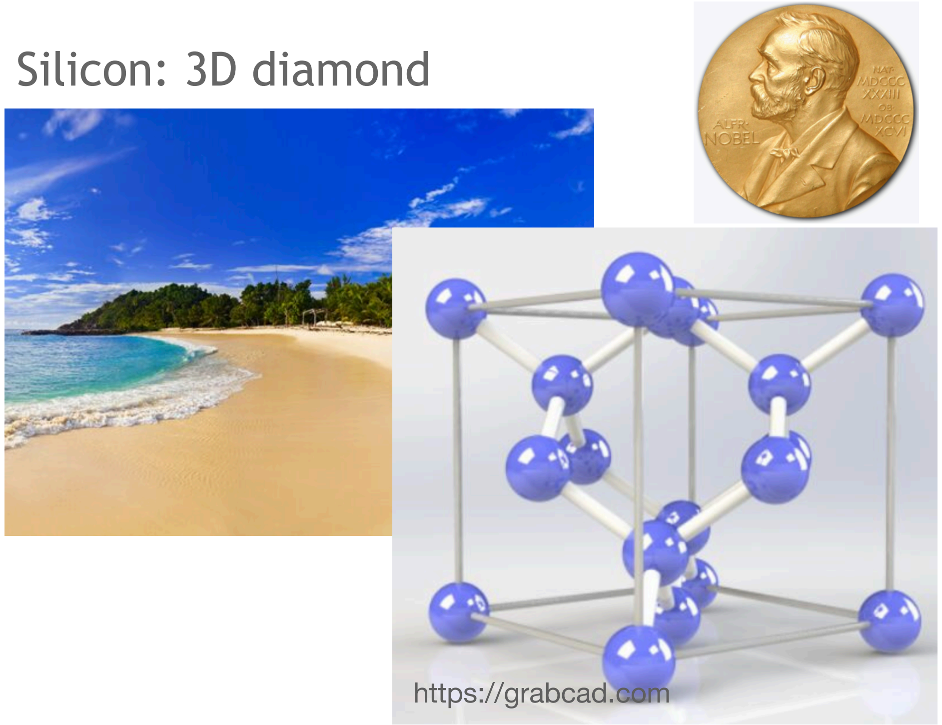
Probing material structures, dynamics and functionality with ESS instrumentation

PASCALE DEEN
SENIOR SCIENTIST FOR SPECTROSCOPY, EUROPEAN SPALLATION SOURCE
ADJUNCT ASSOCIATE PROFESSOR, NIELS BOHR INSTITUTE, UNIVERSITY OF COPENHAGEN

ESS : a vital facility for the study of materials science

Materials: what is the interest?

Silicon: 3D diamond



https://grabcad.com

High affinity to oxygen

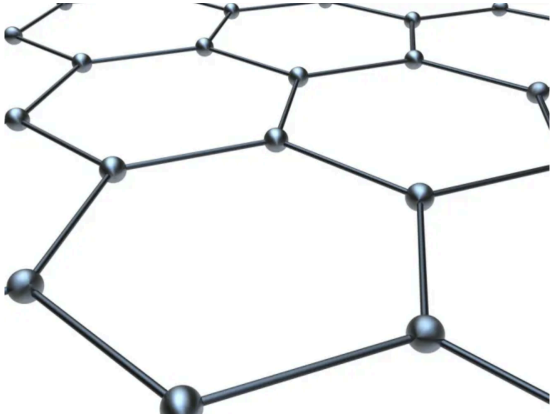
Pure form: 1823 (Jöns Jakob Berzelius, SE)

Semiconductor

Transistor technology (doping)

Electronics, computers: life as we know it.

Silicene: 2D honeycomb



Spin Orbit Interaction: Quantum Hall effect.

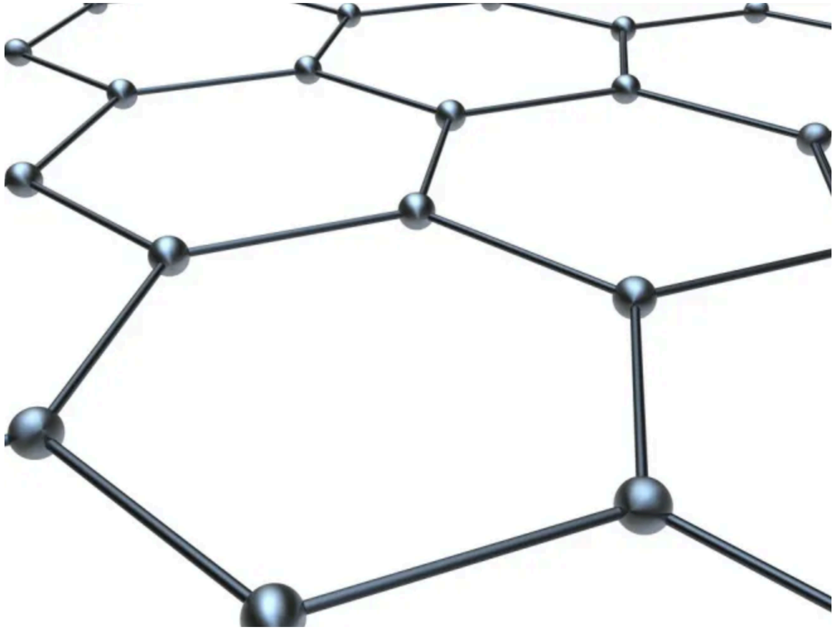
Novel quantum state: Topological insulator.

Surface: Conducting edge states topologically protected from backscattering

Great potential for low power consumption electronics

Unusual structures

Graphene : 2D Carbon layers



Enhanced electrical, optical, thermal and mechanical properties:

Electrochemical & Biochemical sensors

Energy storage, batteries, solar cells

Graphene composites: enhance material strength and rigidity

© The Nobel Foundation. Photo: U. Montan

Andre Geim

Prize share: 1/2

© The Nobel Foundation. Photo: U. Montan

Konstantin Novoselov

Prize share: 1/2

Functionality of materials

What the atoms are ?

Where the atoms are ?

How are they bonded ?

Dynamics ?

Orbital and spin contributions ?

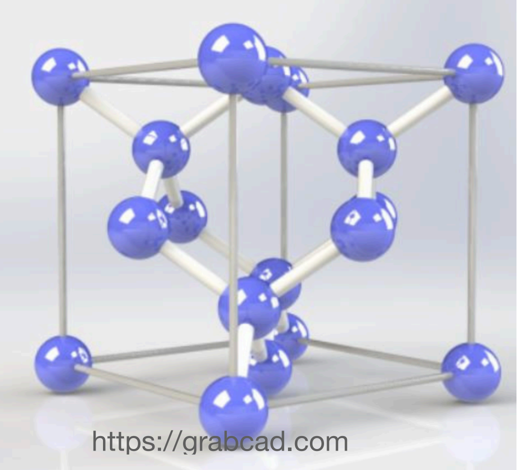
Exchange interactions ?

Phononic excitations ?

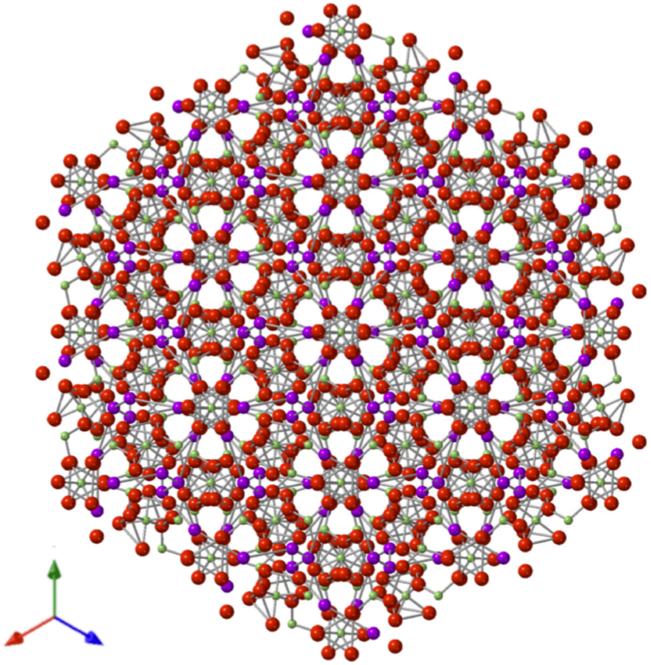
Diffusional behaviour ?

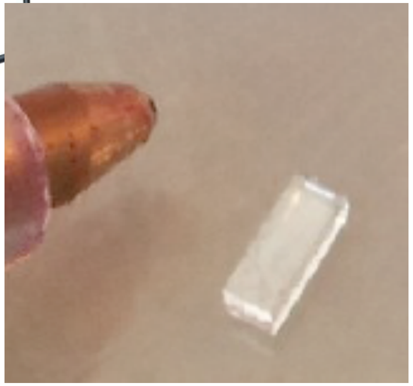
....

Probe??

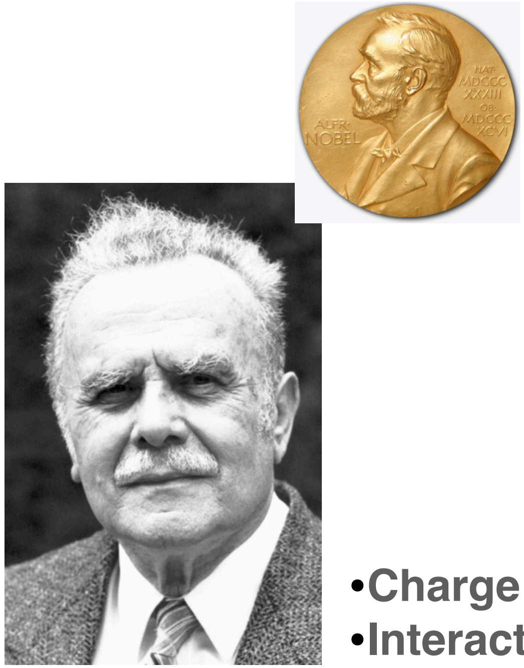

https://grabcad.com


https://epthinktank.eu/





Neutrons for the study of materials



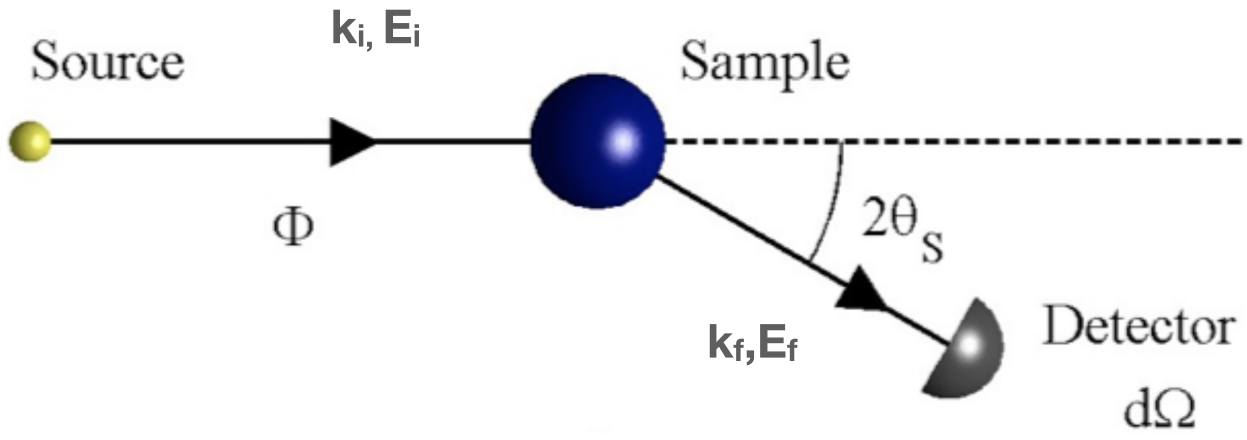
Bert Brockhouse
Nobel Laureate (1994)

The neutron is ideal for the study of condensed matter:

It might be said that, if the neutron did not exist, it would need to be invented.

- **Charge = 0**
- **Interaction with atomic nuclei via the strong force. Scattering varies with isotope.**
- **Magnetic, S = 1/2 , μ = 1.91 (unpaired electrons : dipole interactions)**
- **S = 1/2 = Polarisation analysis**
- **Wavelength ~ atomic length, energy scale of interatomic dynamics**
- **Calculate interaction potential directly**
- Mean lifetime = 880 seconds
- Mass = 1.69e-27 kg.

Calculate interaction potential directly



A. Boothroyd.

Experiment **Theory**

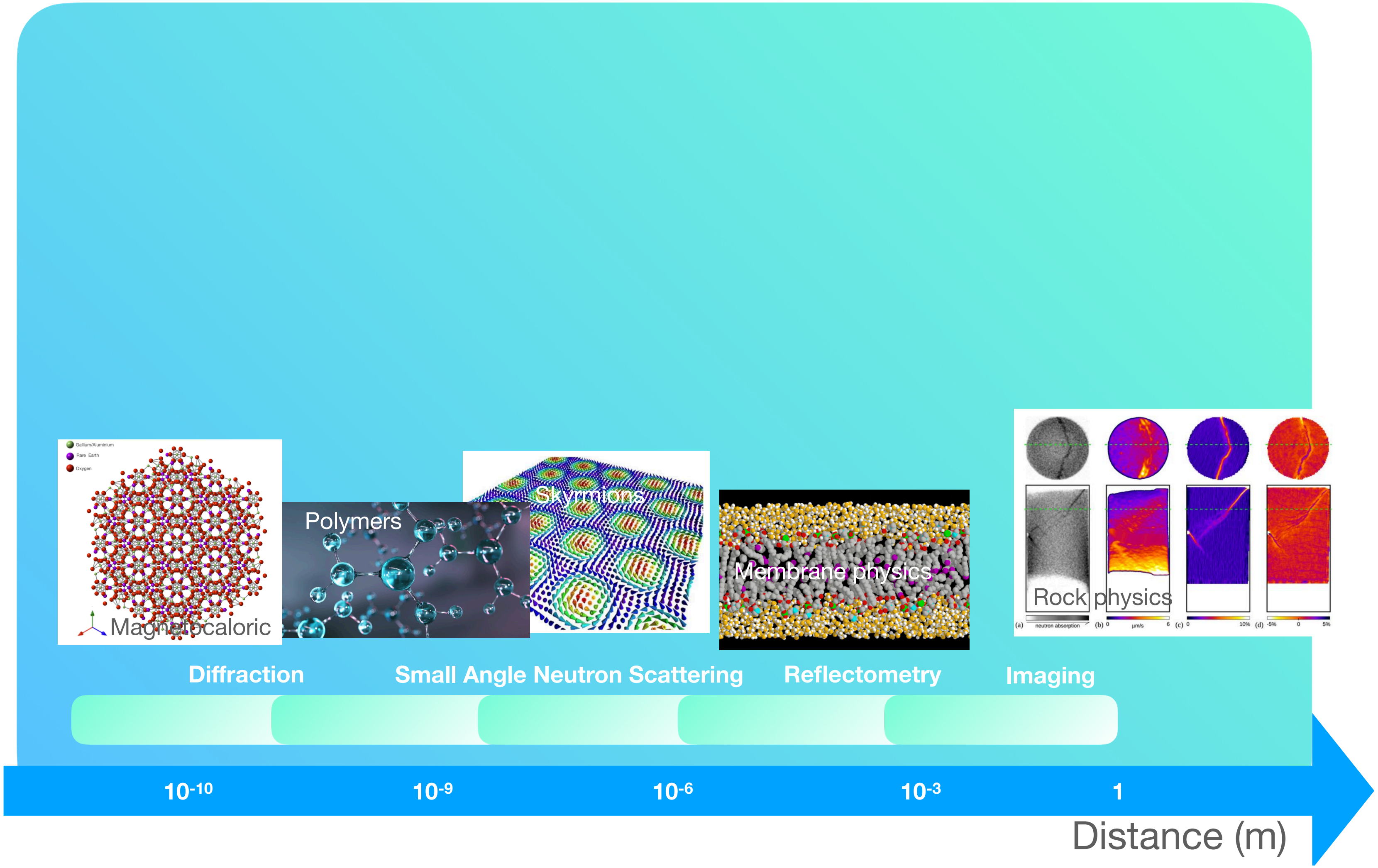
$$\left(\frac{d^2\sigma}{d\Omega dE}\right)_{\lambda_i\rightarrow\lambda_f} = \frac{k_f}{k_{\text{i}}}\left(\frac{m_n}{2\pi\hbar^2}\right)^2 |\mathbf{k_f}\lambda_f|V|\mathbf{k_i}\lambda_i|\delta\left(E_{\lambda_i} - E_{\lambda_f} + \hbar\omega\right)$$

Probe both length scales and energy scales (dynamics)

0.0 – 5 meV	Cold (Slow) neutrons
5 - 100 meV	Thermal neutrons
100 meV–400 meV	Epithermal neutrons

Spatial and time(energy) scale of materials = functionality

What can neutrons probe



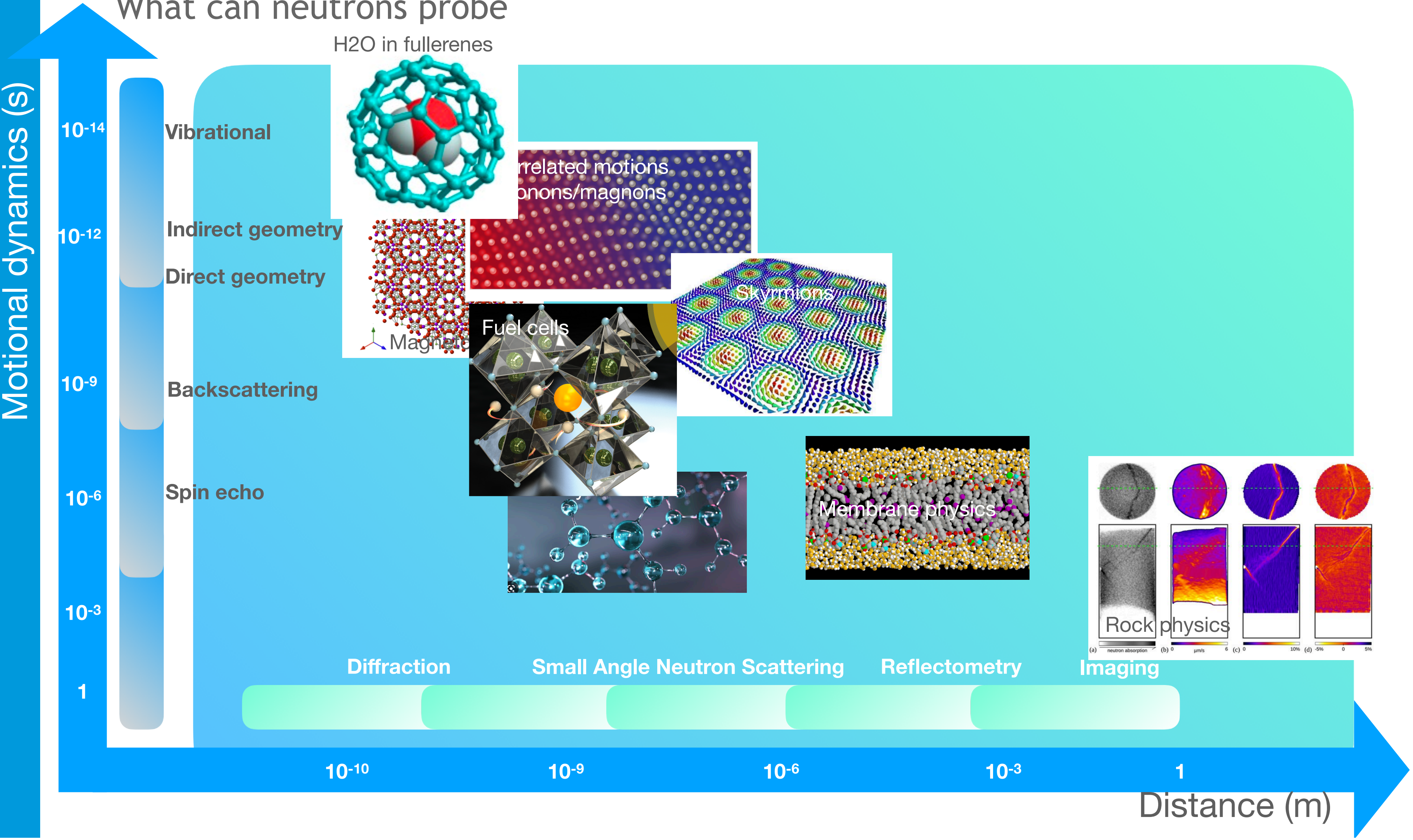
Diffraction **Small Angle Neutron Scattering** **Reflectometry** **Imaging**

10^{-10} 10^{-9} 10^{-6} 10^{-3} 1

Distance (m)

Spatial and time(energy) scale of materials = functionality

What can neutrons probe



Motional dynamics (s)

10-14

10-12

10-9

10-6

10-3

1

Vibrational

Indirect geometry

Direct geometry

Backscattering

Spin echo

H2O in fullerenes

rrelated motions
onons/magnons

Magnet

Skyrmions

Fuel cells

Membrane physics

Rock physics

Diffraction

Small Angle Neutron Scattering

Reflectometry

Imaging

10-10

10-9

10-6

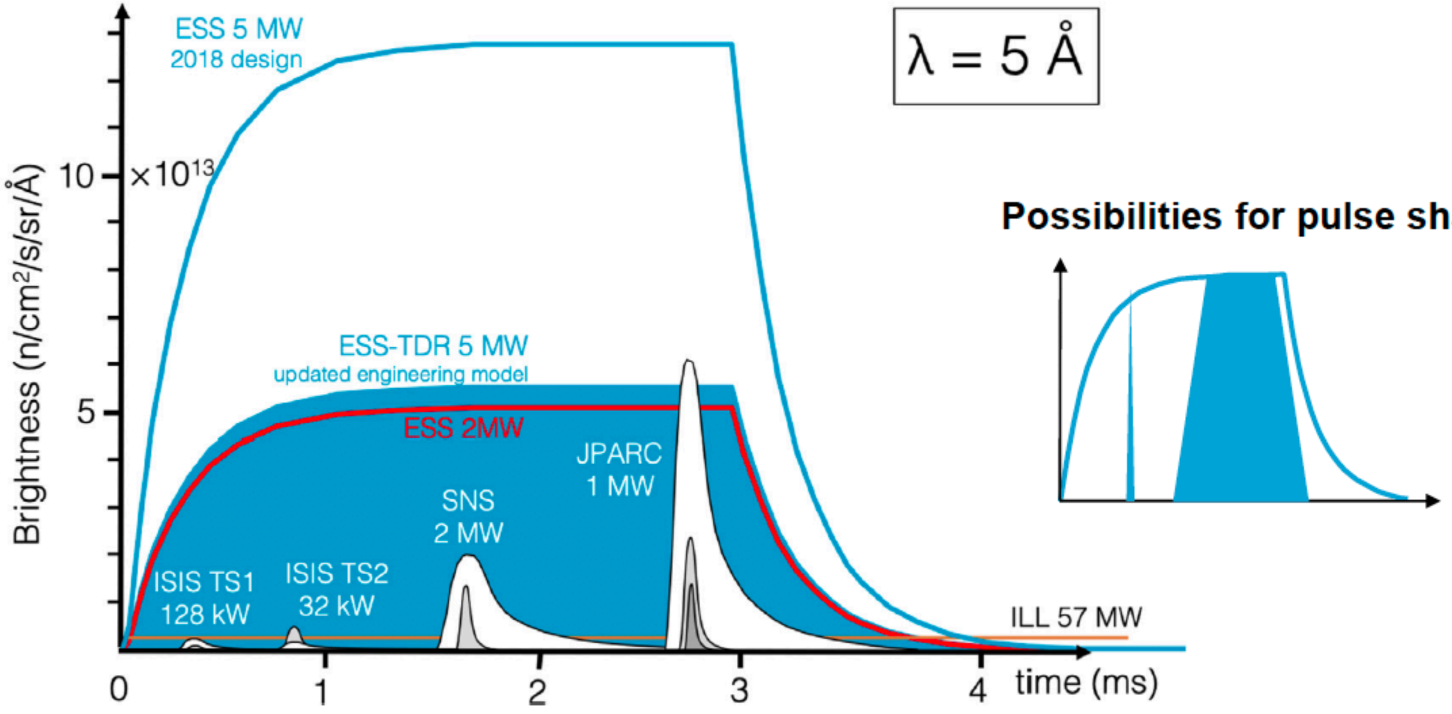
10-3

1

Distance (m)

Long pulse, short pulse and reactor sources.
More flux needed

Time average equal to ILL: flux on sample for all instruments is greater



New opportunities:

Smaller samples.

Weak effects.

Parametric studies.

Kinetic behaviour.

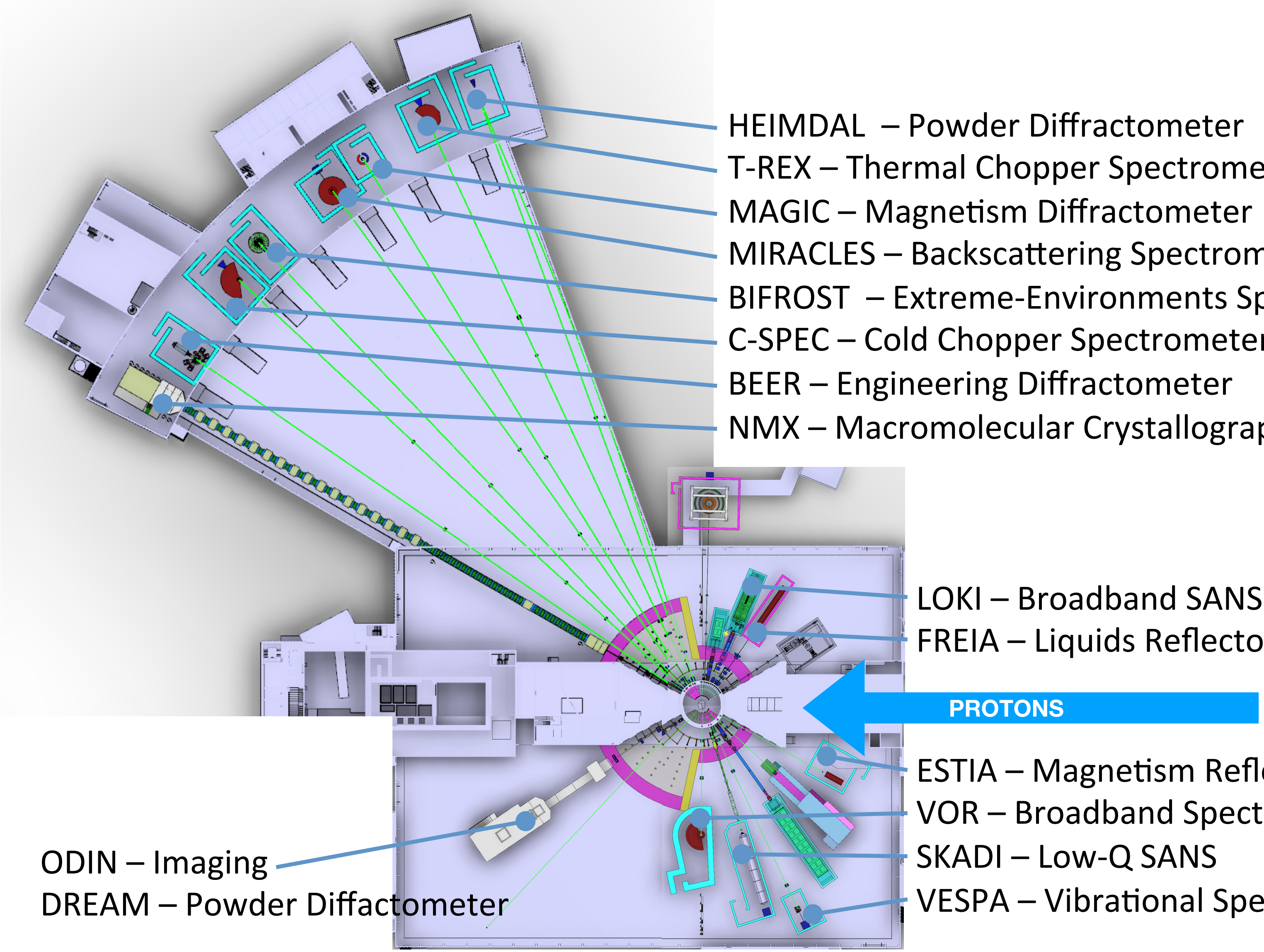
ESS Instrument suite (phase 1)
15 instruments under construction

Large-Scale Structures	**ODIN** Imaging Instrument	🌿 🫧 🧲 🔨 🏺
	SKADI General Purpose SANS	🧪 🫧 🧲 🔋
	LoKI Broadband SANS	🌿 🫧
	Surface Scattering	🌿 🫧 🧪 🧲
	FREIA Horizontal Reflectometer	🌿 🫧 🧪
	Estia Vertical Reflectometer	🧲 🧪 🔋 🫧 🌿
Diffraction	**HEIMDAL** Powder Diffractometer	🧪 🔋 🔨 🧲
	DREAM Powder Diffractometer	🧲 🧪 🔋 🔨
	Monochromatic Powder Diffractometer	🧪 🔋 🧲
	BEER Engineering Diffractometer	🔨 🧪 🔋
	Extreme Conditions Diffractometer	🔋 🔨 🧲 🧪
	MAGiC Magnetism Diffractometer	🧲 🧪
	NMX Macromolecular Diffractometer	🌿 🧲

Spectroscopy	**CSPEC** Cold Chopper Spectrometer	🌿 🫧 🧪 🧲
	Broadband Spectrometer	🌿 🫧 🧪 🧲
	T-REX Thermal Chopper Spectrometer	🧲 🧪 🔋
	BIFROST Crystal Analyser Spectrometer	🧲 🧪 🔋 🔨
	VESPA Vibrational Spectroscopy	🧲 🧪 🔋
	MIRACLES Backscattering Spectrometer	🌿 🫧 🧪 🔋
	High-Resolution Spin-Echo	🌿 🫧 🧪 🔋
	Wide-Angle Spin-Echo	🌿 🫧 🧲 🔋
	Particle Physics Beamline	⚛

🌿	life sciences	🧲	magnetism & superconductivity
🫧	soft condensed matter	🔨	engineering & geo-sciences
🧪	chemistry of materials	🏺	archeology & heritage conservation
🔋	energy research	⚛	particle physics

ESS instrument suite (day 1): broad ranging



HEIMDAL – Powder Diffractometer
T-REX – Thermal Chopper Spectrometer
MAGIC – Magnetism Diffractometer
MIRACLES – Backscattering Spectrometer
BIFROST – Extreme-Environments Spectrometer
C-SPEC – Cold Chopper Spectrometer
BEER – Engineering Diffractometer
NMX – Macromolecular Crystallography
LOKI – Broadband SANS
FREIA – Liquids Reflectometer
ESTIA – Magnetism Reflectometer
VOR – Broadband Spectrometer
SKADI – Low-Q SANS
VESPA – Vibrational Spectroscopy
ODIN – Imaging
DREAM – Powder Diffactometer

Overview of some instruments

Ready for Beam on Target 2025 (Estimate 6-7 ready for 2025)

ODIN : Imaging

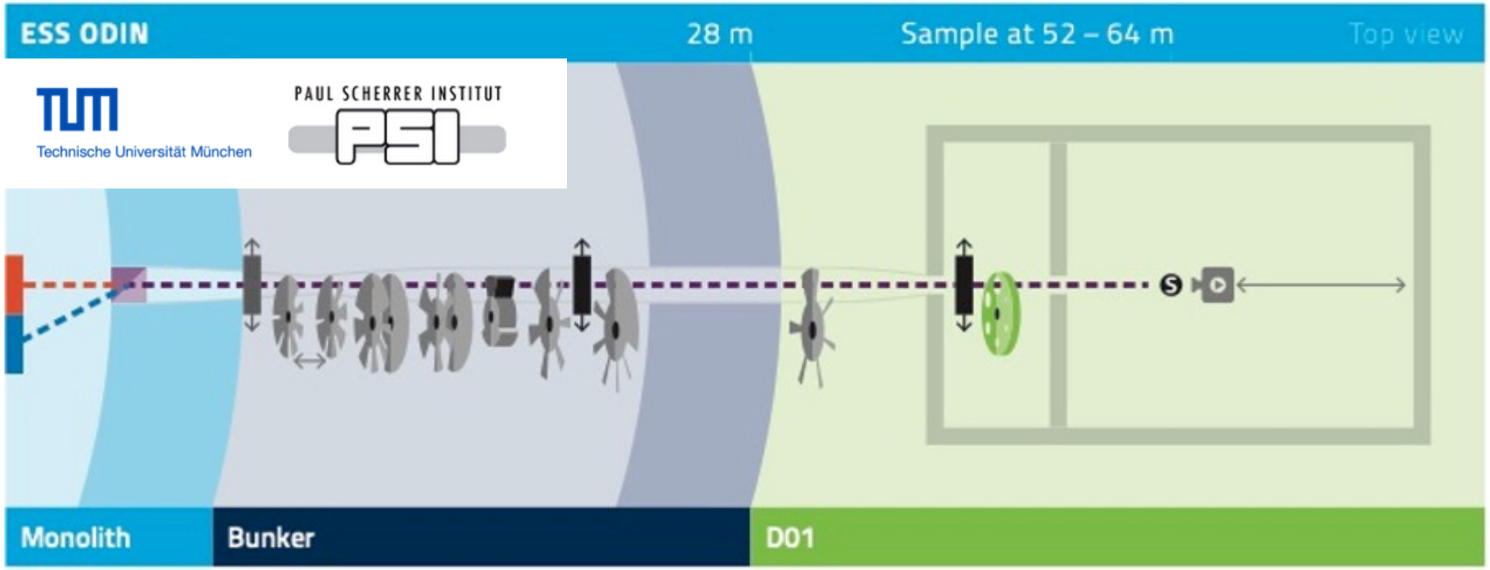
LOKI : Small angle neutron scattering (SANS)

DREAM : Bispectral Powder Diffractometer

BIFROST : Extreme Environment Cold Neutron Spectrometer

ODIN: Multi purpose imaging beamline

Scientists: Aureliano Tartaglione, Manuel Morgano



10 choppers to tune wavelength resolution : cover real space length scales from nm to mm range

Quick facts:

Moderator: Bispectral

Primary flightpath: 50 m

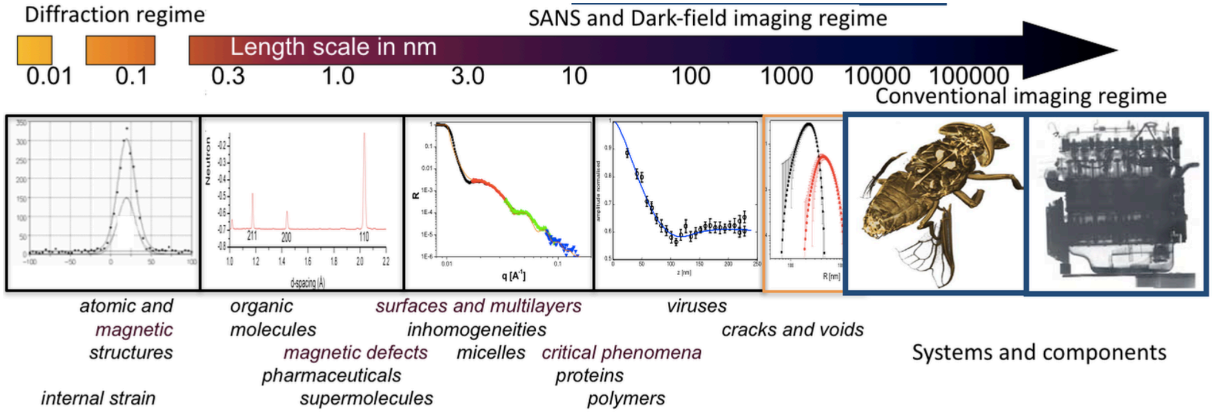
Secondary flightpath: 2 - 14 m

Wavelength range :1 - 10 Å

Field of view : 20 cm²

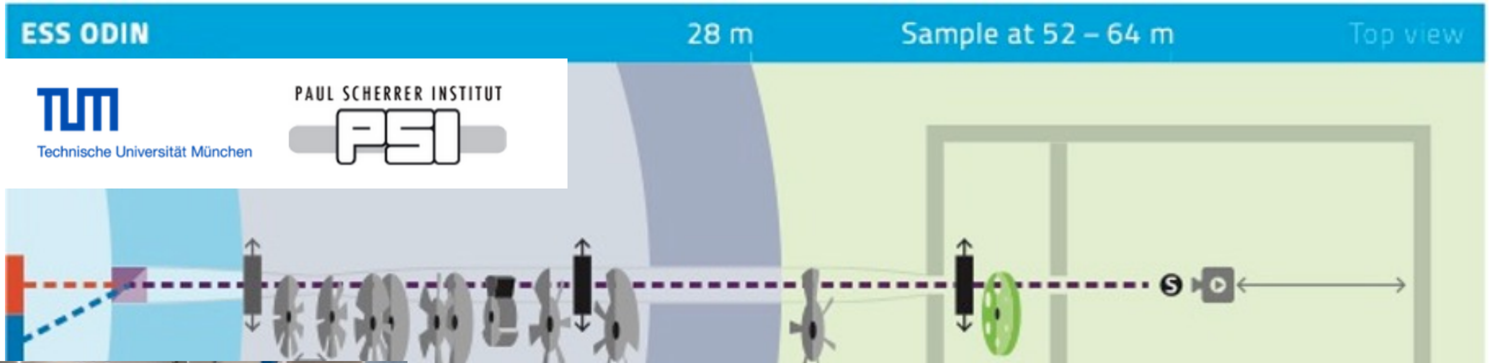
Spatial resolution < 10 μm

Polarisation analysis possible.

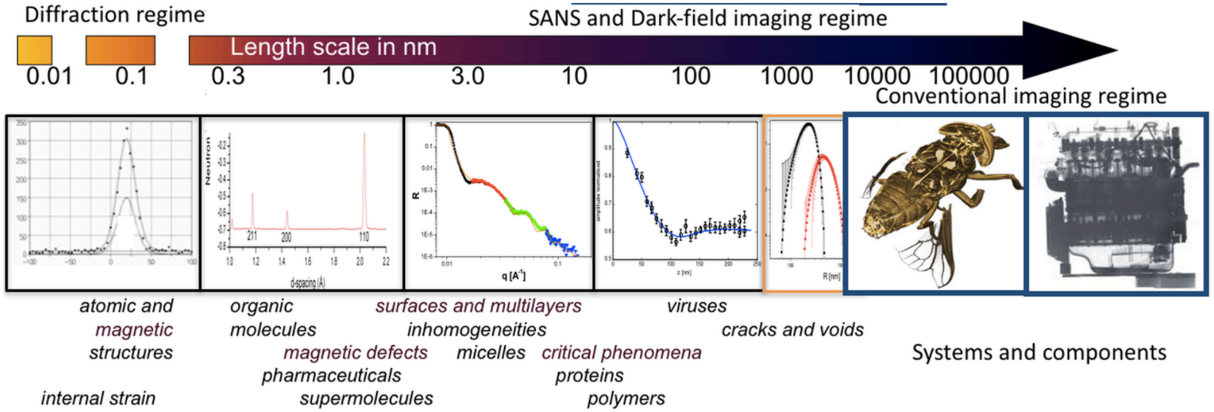


ODIN: Multi purpose imaging beamline

Scientists: Aureliano Tartaglione, Manuel Morgano



ength resolution : cover real space length scales from nm to mm range



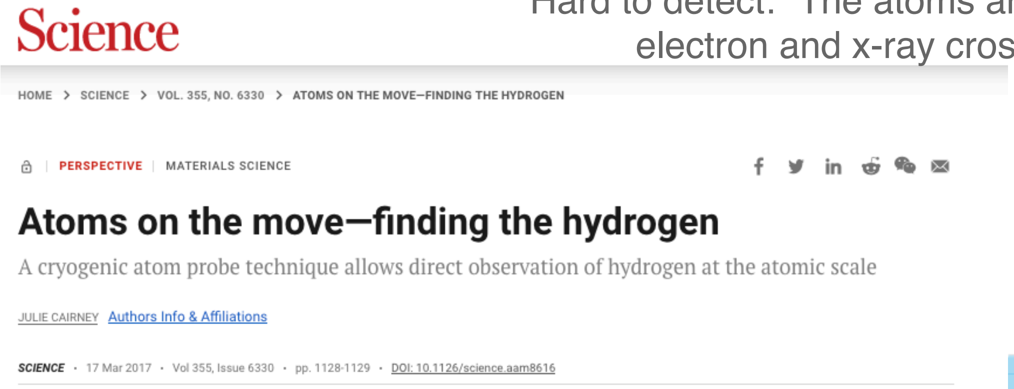
ODIN: Science

Oakland Bay Bridge: San Franscico: Catastrophic failure of shear steel bolts. Attributed to embrittlement.

Cheesegrater building London: bolts failing due to embrittlement. 3000 bolts replaced: cost of £6 m.

R. Woracek.

Hard to detect: “The atoms are small, light, mobile, have a small electron and x-ray cross section and diffuse easily.”



Science

HOME > SCIENCE > VOL. 355, NO. 6330 > ATOMS ON THE MOVE—FINDING THE HYDROGEN

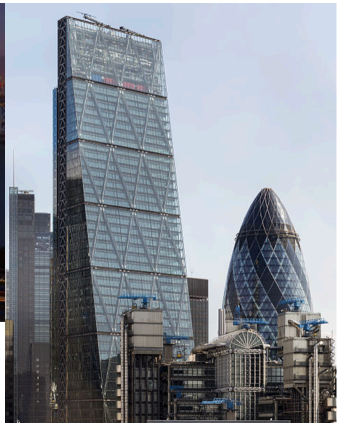
PERSPECTIVE | MATERIALS SCIENCE

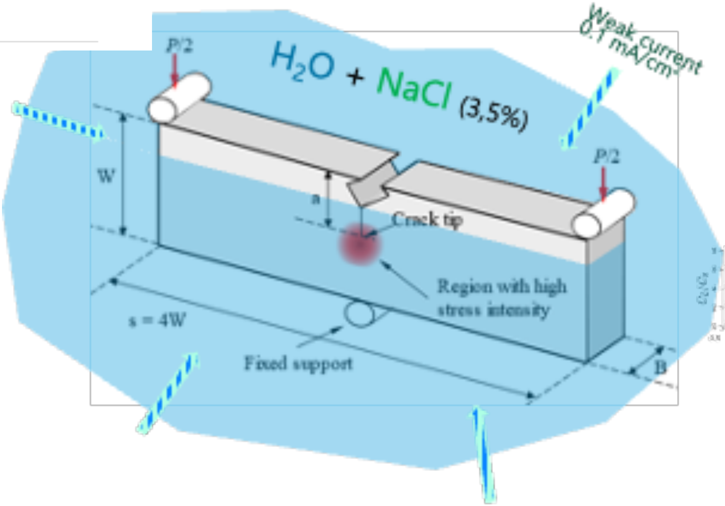
Atoms on the move—finding the hydrogen

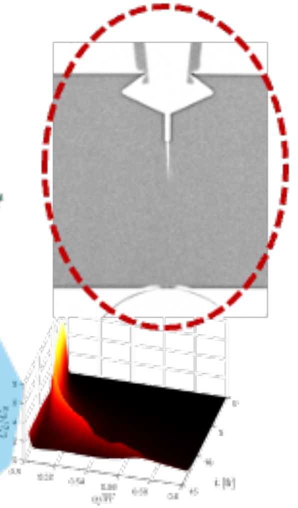
A cryogenic atom probe technique allows direct observation of hydrogen at the atomic scale

JULIE CAIRNEY Authors Info & Affiliations

SCIENCE • 17 Mar 2017 • Vol 355, Issue 6330 • pp. 1128-1129 • DOI: 10.1126/science.aam8616



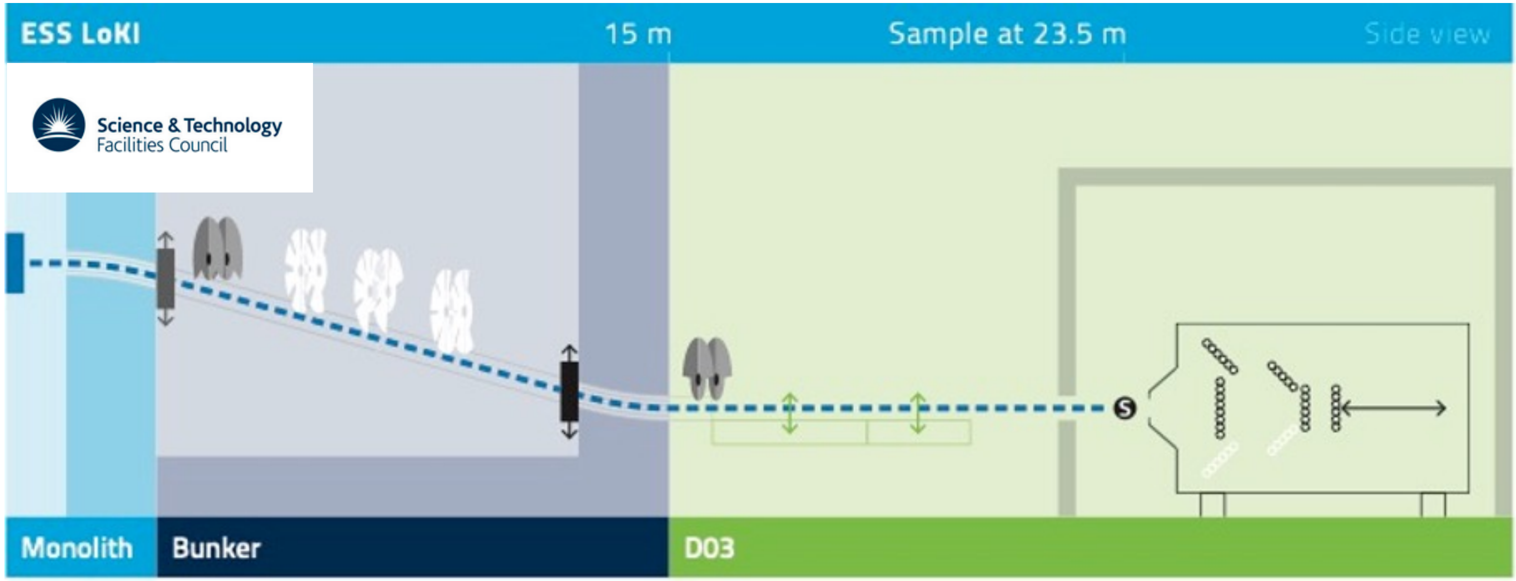




The ‘first ever’ in-situ imaging of H in steel under operational conditions!?!
(data still under final analysis)

Loki: Small angle neutron scattering

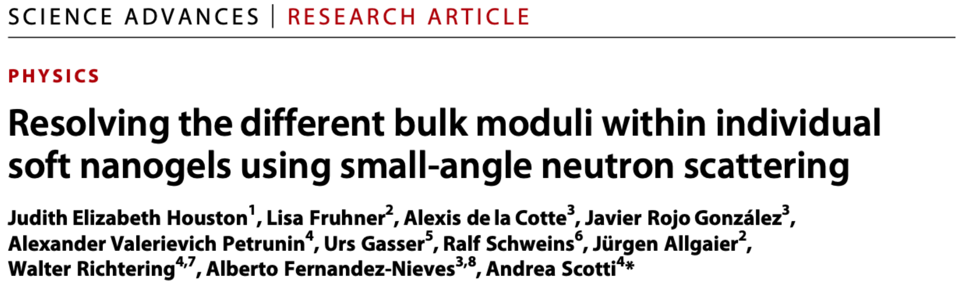
Lead scientist : Judith Houston



Quick facts:
Moderator: Cold
Primary flightpath : 23.5 m
Secondary flightpath : 1.5, 3, 5-10 m
Wavelength range: 2 - 22 Å
Bandwidth 7.5 Å [L2 = 10 m], 10 Å [L2 = 5 m]
Sample beam size : 8 x 12 mm^2
Q-range 0.005 - 2 Å^{-1} (Standard mode)
Q-range 0.002 - 2 Å^{-1} (Pulse skipping mode)

Probing softness with SANS

Houston et al., Sci. Adv. 8, eabn6129 (2022)

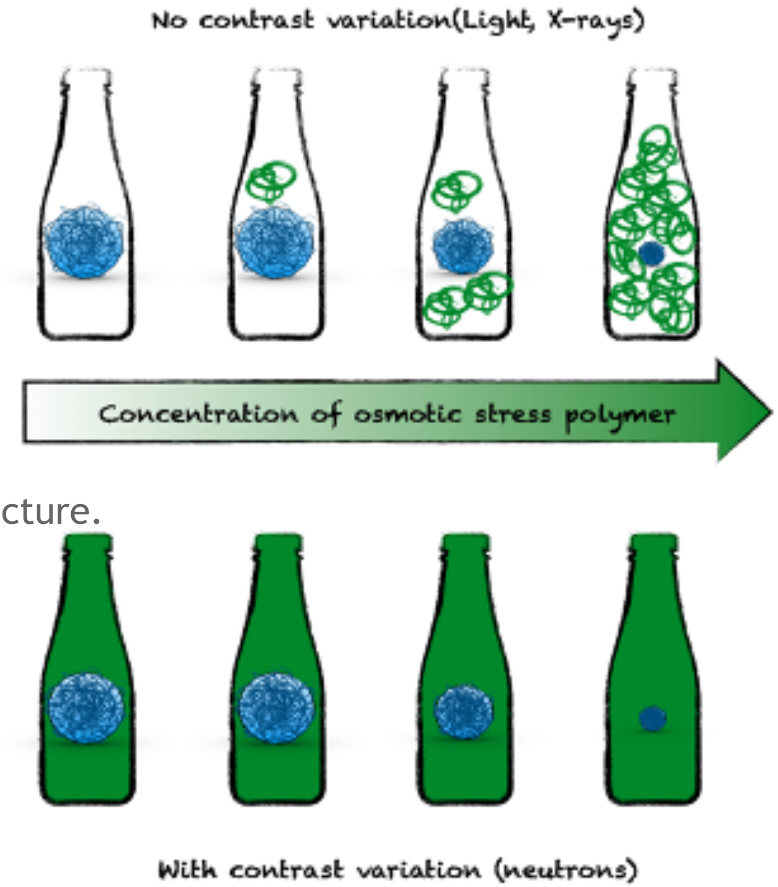


SCIENCE ADVANCES | RESEARCH ARTICLE

PHYSICS

Resolving the different bulk moduli within individual soft nanogels using small-angle neutron scattering

Judith Elizabeth Houston[1], Lisa Fruhner[2], Alexis de la Cotte[3], Javier Rojo González[3], Alexander Valerievich Petrunin[4], Urs Gasser[5], Ralf Schweins[6], Jürgen Allgaier[2], Walter Richtering[4,7], Alberto Fernandez-Nieves[3,8], Andrea Scotti[4]*

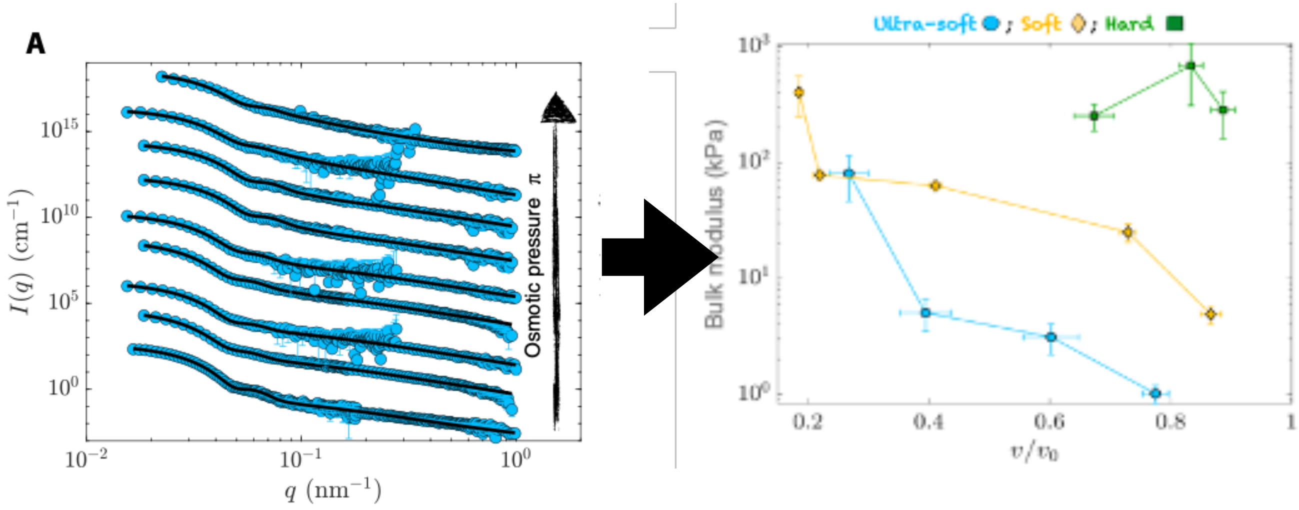


Quantify softness in real systems: viruses, proteins, blood cells.

Colloid achitecture characterised by softness.

Softness: blood clotting, virus infectivity.

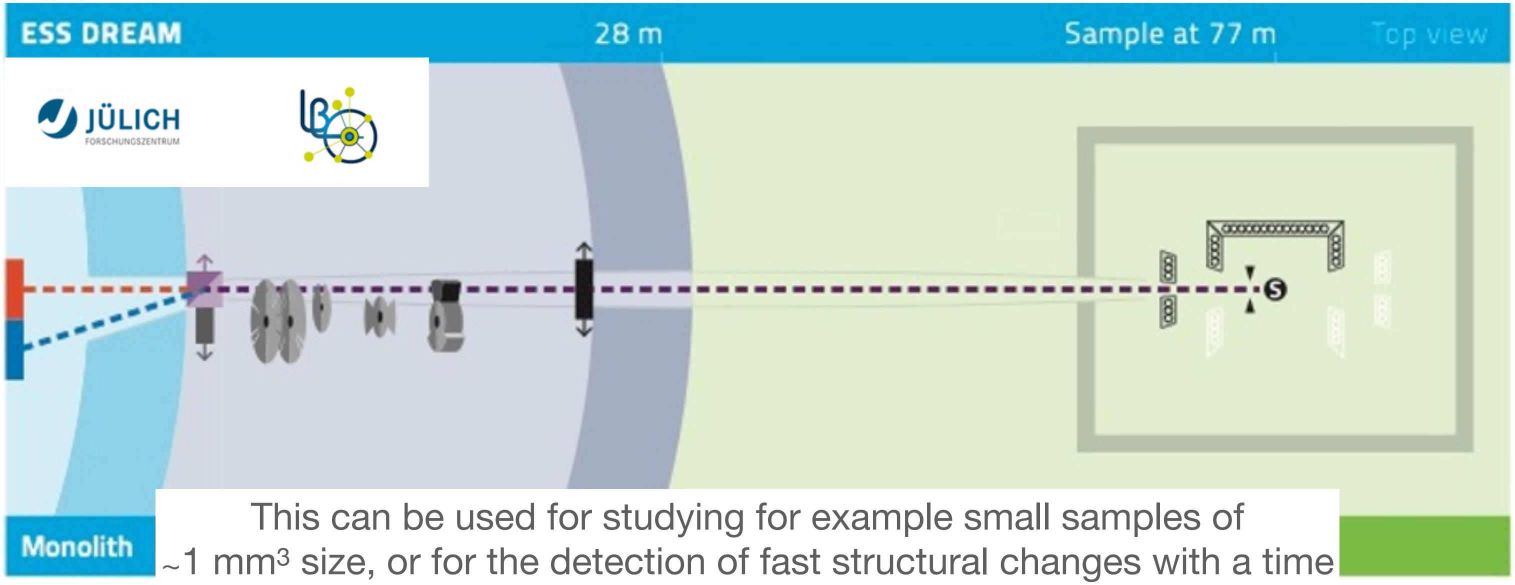
Bulk modules: real environments, accessed via contrast variation = true architecture.



LOKI: Time dependence of clotting ..

DREAM: Powder diffractometer

Lead scientist : Mikhail Feygenson



This can be used for studying for example small samples of ~1 mm^3 size, or for the detection of fast structural changes with a time resolution in the ms-range for samples as large as 0.5 cm^3.

Quick facts:

Moderator: Bispectral

Primary flightpath: 76.5 m

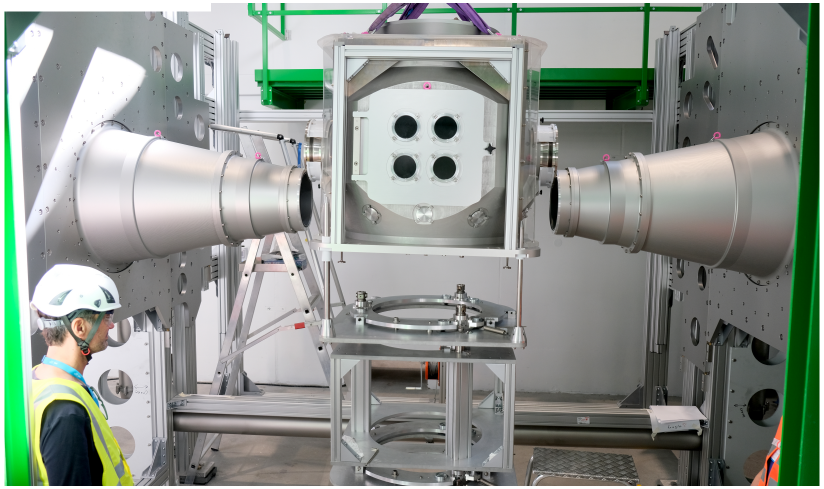
Secondary flightpath: 1.1 or 2.5 m

Wavelength range : 0.5 - 4 Å

Sample beam size : down to 1 mm^2

Detector coverage: 1.82 Sr (5.12 Sr, 46% of 4π)

d-space resolution: $3x10^{-4}$ - 2.5×10^{-2}



DREAM Science

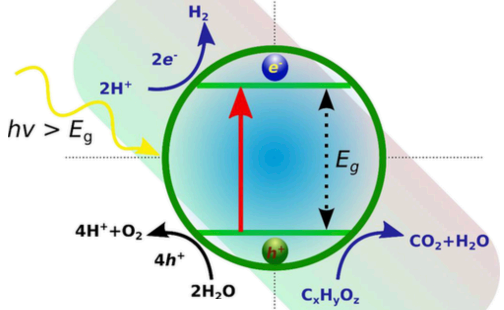

Inorganic Chemistry
Article
pubs.acs.org/IC

Average and Local Crystal Structures of $(Ga_{1-x}Zn_x)(N_{1-x}O_x)$ Solid Solution Nanoparticles

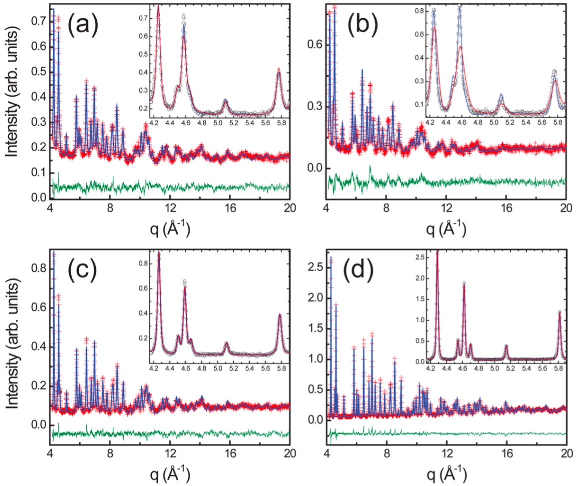
Mikhail Feygenson,[*,†] Joerg C. Neuefeind,[†] Trevor A. Tyson,[‡] Natalie Schieber,[§] and Wei-Qiang H

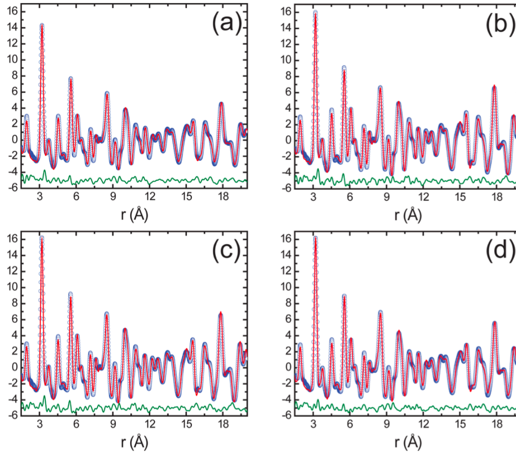
DOI: 10.1021/acs.inorgchem.5b01605 Inorg. Chem. 2015, 54, 11226–11235

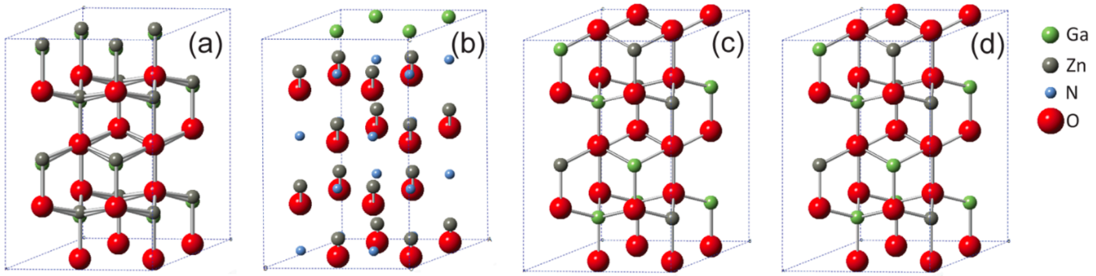
https://doi.org/10.1002/cctc.201601659



- Renewable energies: solar water splitting photocatalysis: source of hydrogen fuel.
- The most promising photocatalyst is the (Ga1−xZnx)(N1−xOx) solid solution of GaN and ZnO.
- Various nanoparticles 10−27 nm and x = 0.075−0.51, varying electronic structure bandgap (Eg).
- Disorder alters the structure and reduces Eg.



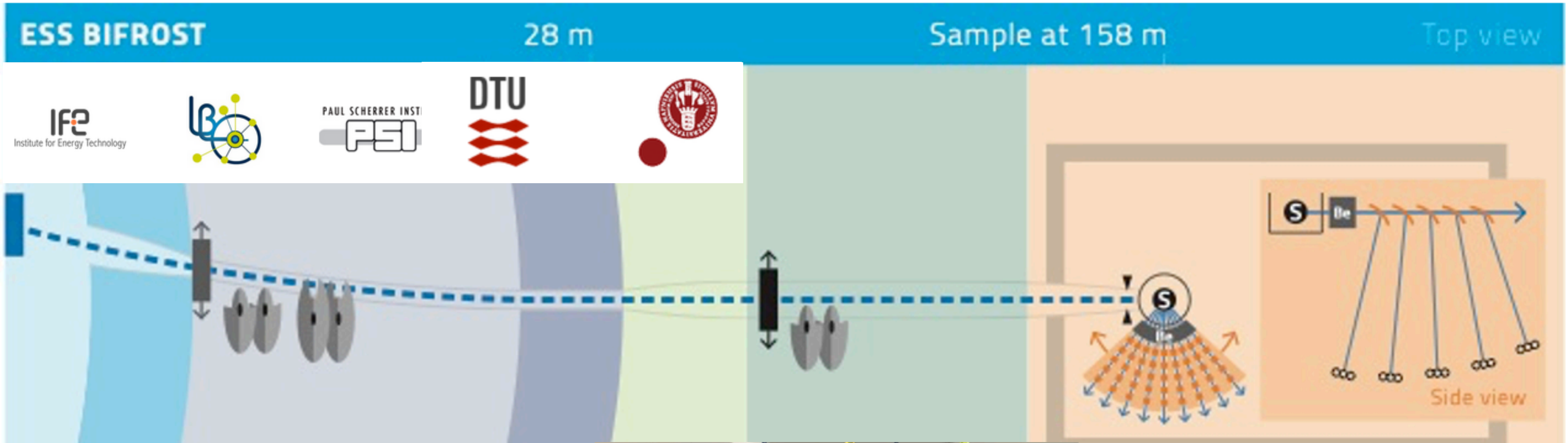


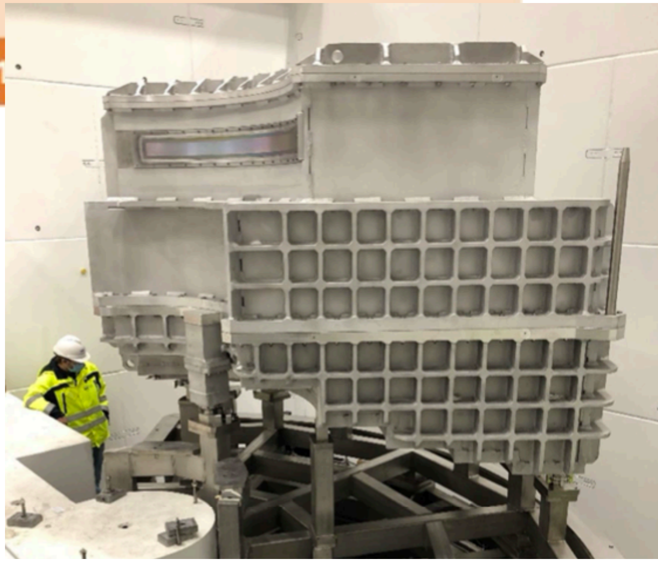


DREAM: Parametric studies, time dependent behaviour.

BIFROST Extreme Environment Cold Neutron Spectrometer

Lead scientist : Rasmus Toft-Petersen





Quick facts:

Moderator: Cold

Primary flightpath: 162 m

Sample- analyser flightpath: 1.5 - 1.7 m

Wavelength range :1.5 - 6 Å

Sample beam size : 1 cm^2

Detector coverage: 90º (2 setting)

Analyser energies: 2.7, 3.2, 3.8 4.4 and 5.0 meV

Energy resolution: Ef = 5 meV, omega = 0. = 50 - 190 µeV

Bifrost Science

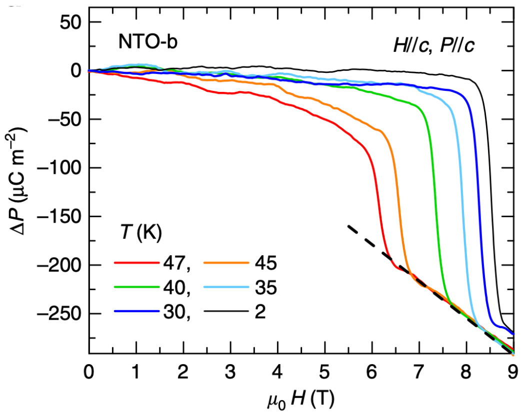
Multiferroic: coupling between structural, magnetic and electronic ordering in an insulator

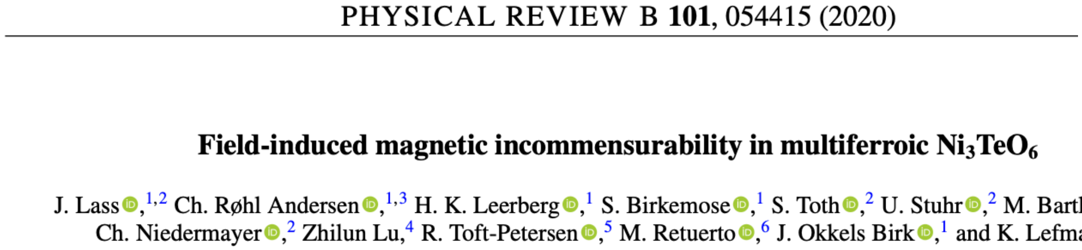
Control of magnetic order via application of electric field. Weak hysteresis, reversible.

Need large coupling strength and weak hysteresis for functional materials: capacitors, sensors, spintronics

Ni_3TeO_6

Nature : DOI: 10.1038/ncomms4201

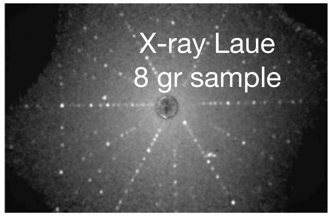


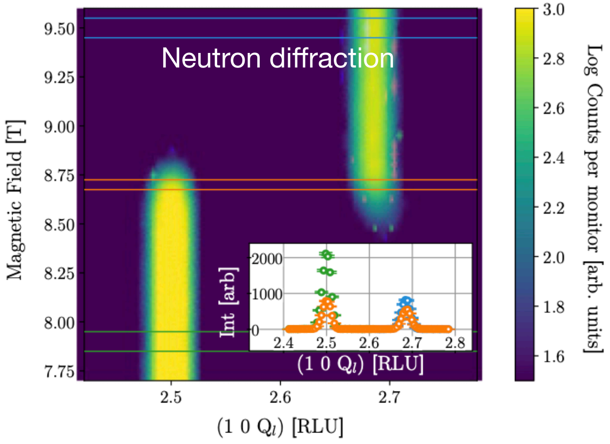


PHYSICAL REVIEW B **101**, 054415 (2020)

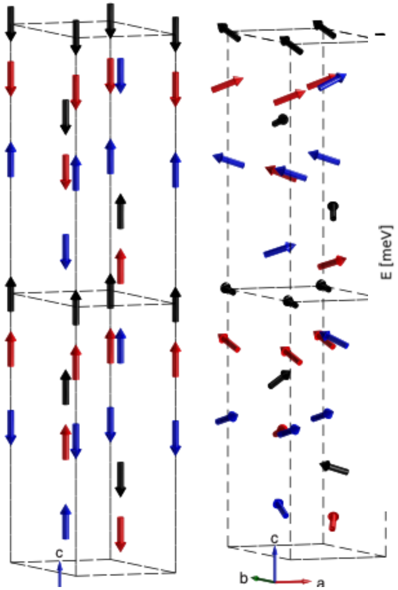
Field-induced magnetic incommensurability in multiferroic Ni_3TeO_6

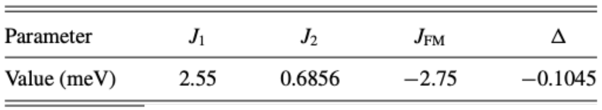
J. Lass,[1,2] Ch. Røhl Andersen,[1,3] H. K. Leerberg,[1] S. Birkemose,[1] S. Toth,[2] U. Stuhr,[2] M. Bartkowiak,[2] Ch. Niedermayer,[2] Zhilun Lu,[4] R. Toft-Petersen,[5] M. Retuerto,[6] J. Okkels Birk,[1] and K. Lefmann[1]


X-ray Laue
8 gr sample

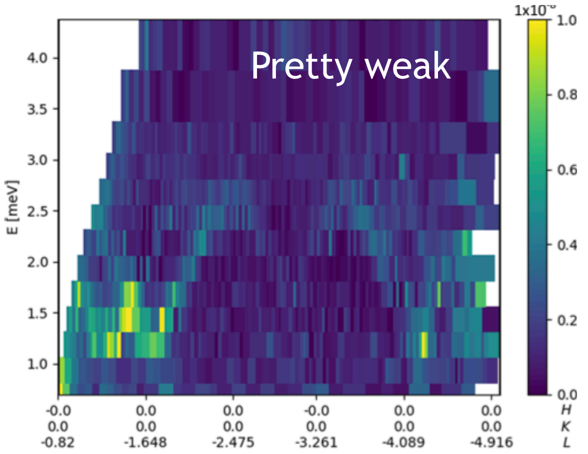

Neutron diffraction

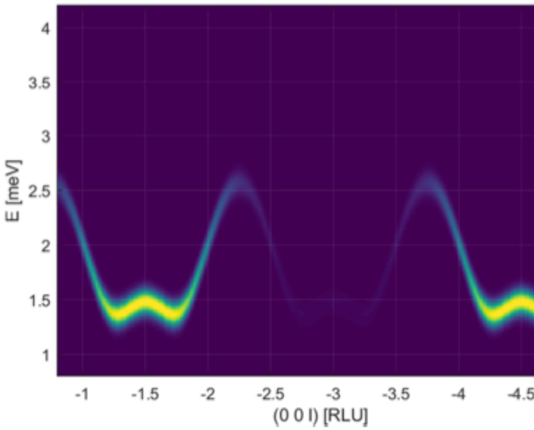
AF to conical spin spiral.





Parameter	J_1	J_2	J_{FM}	Δ
Value (meV)	2.55	0.6856	−2.75	−0.1045


Pretty weak



Bifrost: more flux: parametric studies .

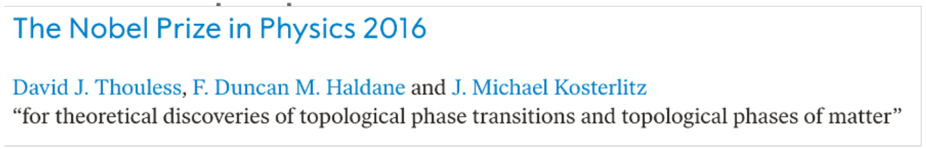
ESS: a source to deliver meV neutron for the study of materials

Neutrons probes directly **magnetic spins**.

High technology society: magnetic and electronic phenomena.

Magnetic spins:

- quantum computing / Classical
  = 200 sec/10 000 years (Google 2021)
- Superconductivity : lossless power transfer
- Magnetocaloric cooling : low carbon

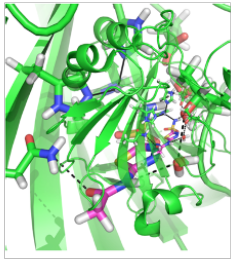


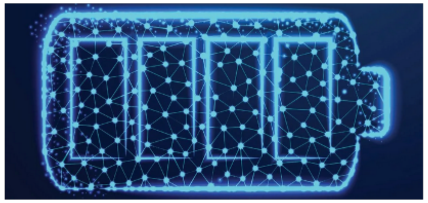
The Nobel Prize in Physics 2016

David J. Thouless, F. Duncan M. Haldane and J. Michael Kosterlitz
“for theoretical discoveries of topological phase transitions and topological phases of matter”

Neutrons: Probes directly light elements

(hydrogen, lithium)





- Biological processes: where hydrogen (H) atoms are and how they are transferred between biomacromolecules, solvent molecules, and substrates.
- Optimise diffusion in battery materials.

The Nobel Prize in Chemistry 2019

John B. Goodenough, M. Stanley Whittingham and Akira Yoshino “for the development of lithium-ion batteries”

Uniquely with the correct energy scale!!

MAX IV

LUNDS UNIVERSITET

LTH
LUNDS TEKNISKA HÖGSKOLA

DTU

MALMÖ UNIVERSITY

+ .....

ess
EUROPEAN SPALLATION SOURCE

pascale.deen@ess.eu

University affiliation for instrument scientists at ESS

Dan Mannix: CNRS, France, Aarhus University (under discussion).

Judith Houston: Lund University (under discussion).

Rasmus Toft-Peterson: DTU, Denmark.

Pascale Deen: Niels Bohr Institute, KU, Denmark.

Robin Woracek: Lund University, Sweden. (Under discussion)

Esko Oksanen: Lund University, Sweden. (Under discussion)

Manuel Morgano: PSI, Switzerland, Lund University (under discussion).

Daria Noferini: Considering various options.

Thomas Arnold: Bath University, UK.

Werner Schweika: Aachen University, Germany.

Andrew Jackson: Lund University, Sweden.

Mikhail Feygenson: Uppsala University, (under discussion).

Premek Beran: Uppsala University (under discussion).