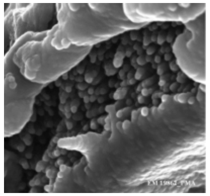
EUROPEAN SPALLATION SOURCE

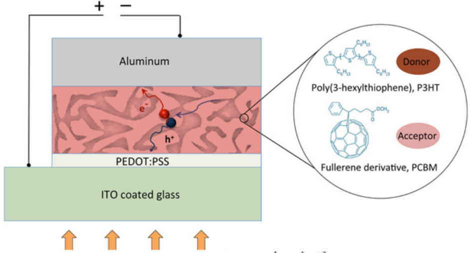
Soft Matter and Surface Science at ESS

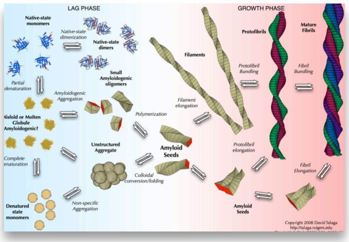
Hanna Wacklin
European Spallation Source ERIC
Physical Chemistry, Lund University

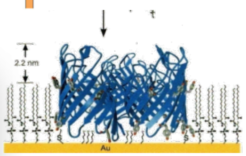
LUND UNIVERSITY

Huge Range of Science Complex Systems







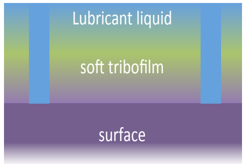


Cell membranes

Organic Photovoltaics

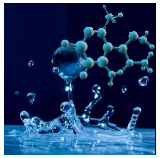
Sustainable Biomaterials

Biosensors



Lubrication Tribology

Ionic liquids



Surfactants & Polymers

Batteries and hydrogen storage

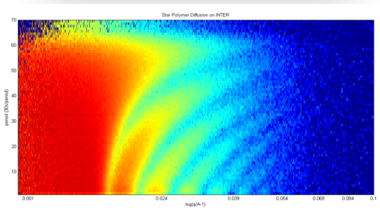
Drug development & delivery

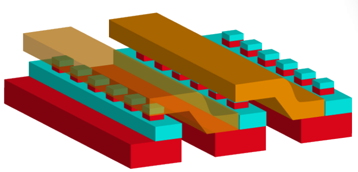
Thin film devices

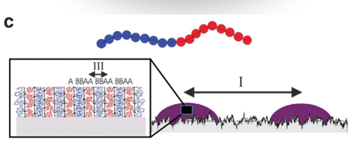
Self-assembly & Smart coatings

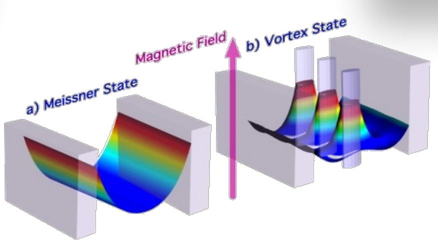
Neutron science

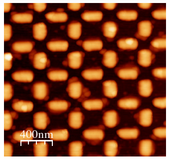
Nanomagnetism & superconductivity







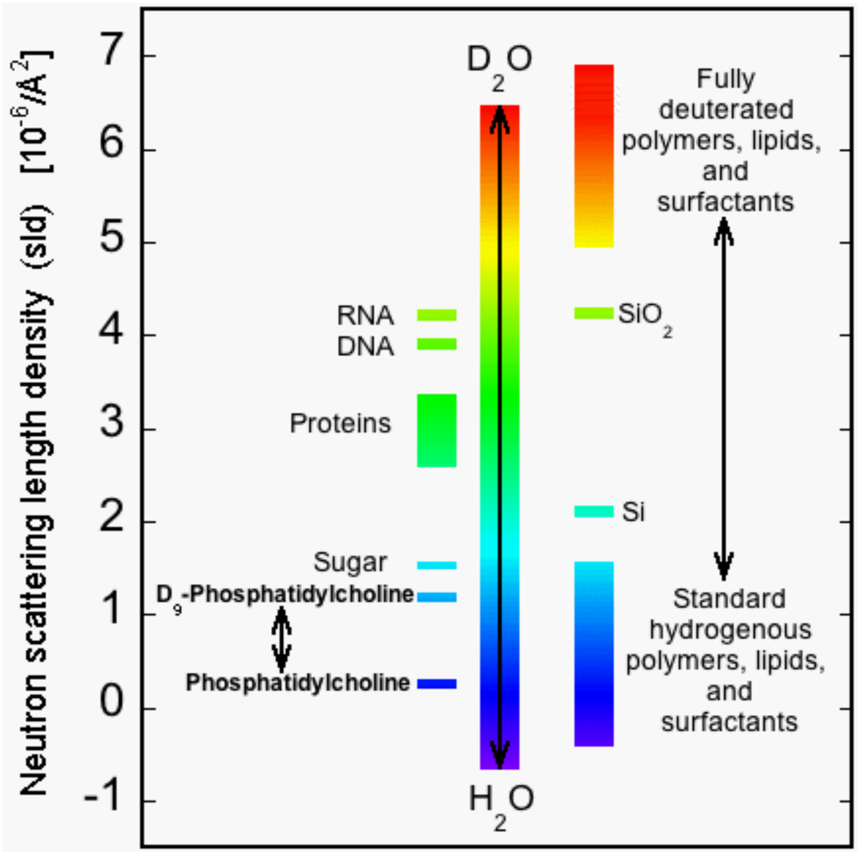




Why are neutrons useful for soft matter?

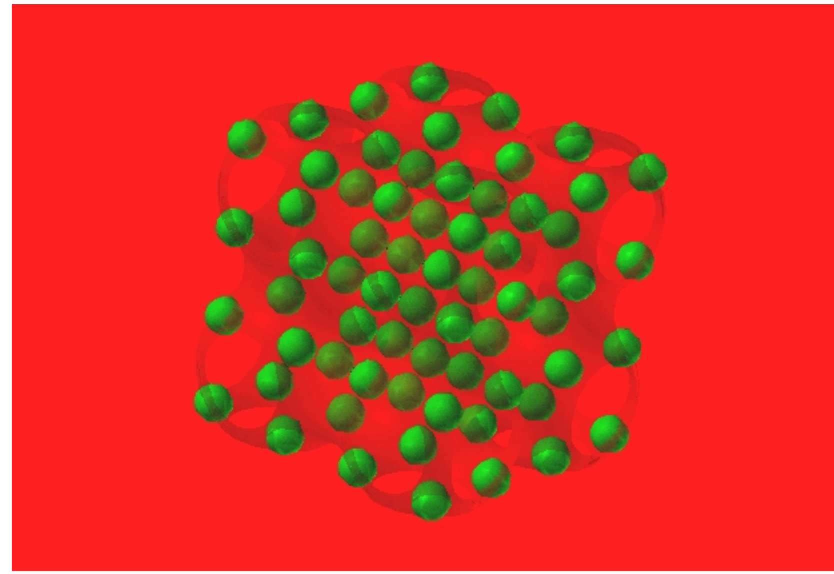
X-rays

What Neutrons "See"



12

9.6



e.g. proteins in a deuterated polymer matrix: by changing solvent from H_2O to D_2O can mask out lipid contribution.

Deuterium labeling can selectively highlight structural or dynamical features in complex materials

Neutrons have low energy (meV): no radiation damage/penetrating

ESS will help to solve many Soft Matter and Surface Science Challenges:

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- **Rapid data collection / short counting times to enable kinetics and high-throughput studies**
- Probe **broad size range** to examine **hierarchical structures**
- **Small samples for scanning, biological and complex samples**
- Integrated flexible **sample environment** for **non-equilibrium studies**
- Integration of **complementary techniques experimentally** and in **data analysis**

Main techniques: SANS, reflectometry, spectroscopy (QENS, INS, NSE) to probe *nano-scale structure and dynamics*

Initial Instrument Suite (15)

2 SANS, 2 Reflectometers, 4 spectrometers

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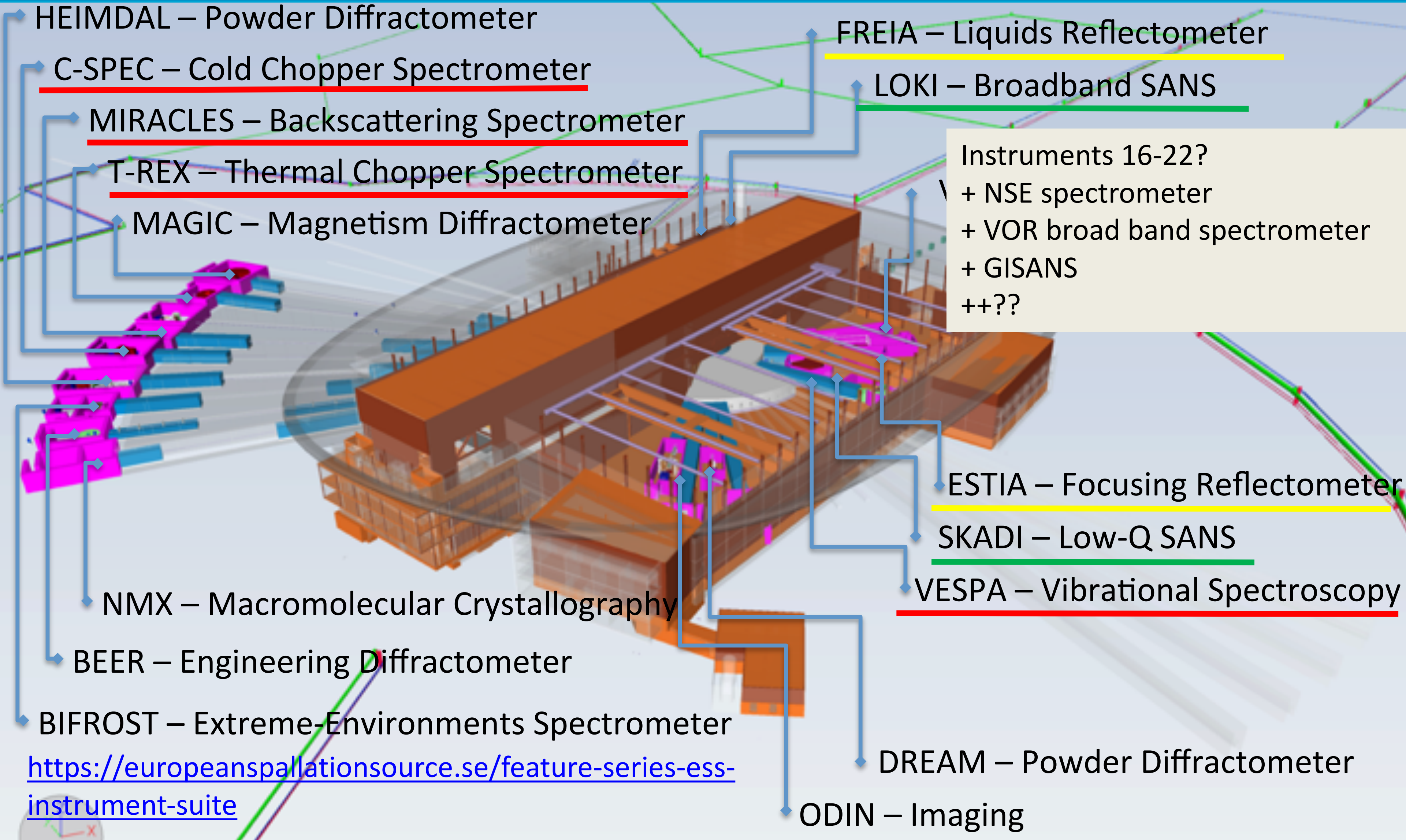


https://europeanspallationsource.se/feature-series-ess-instrument-suite

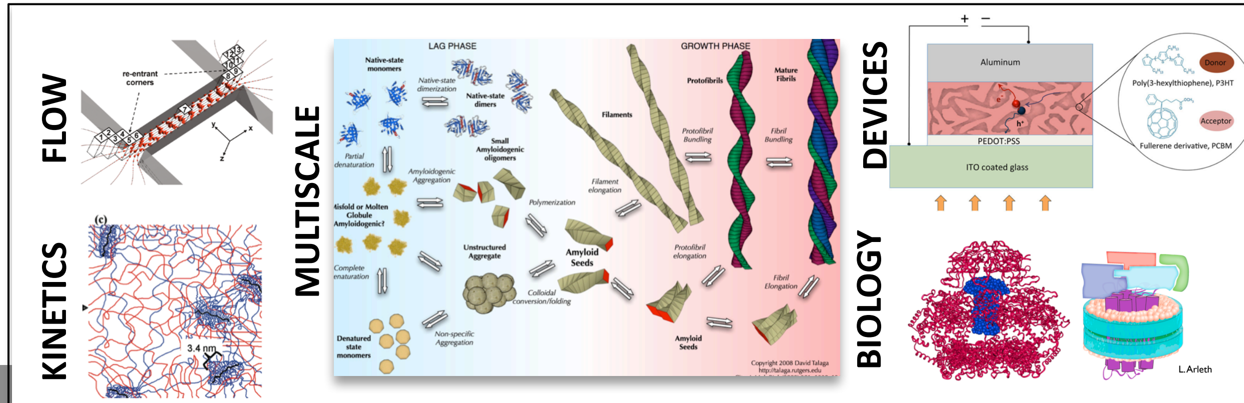
LoKI

SANS for Soft Matter, Materials & Bioscience

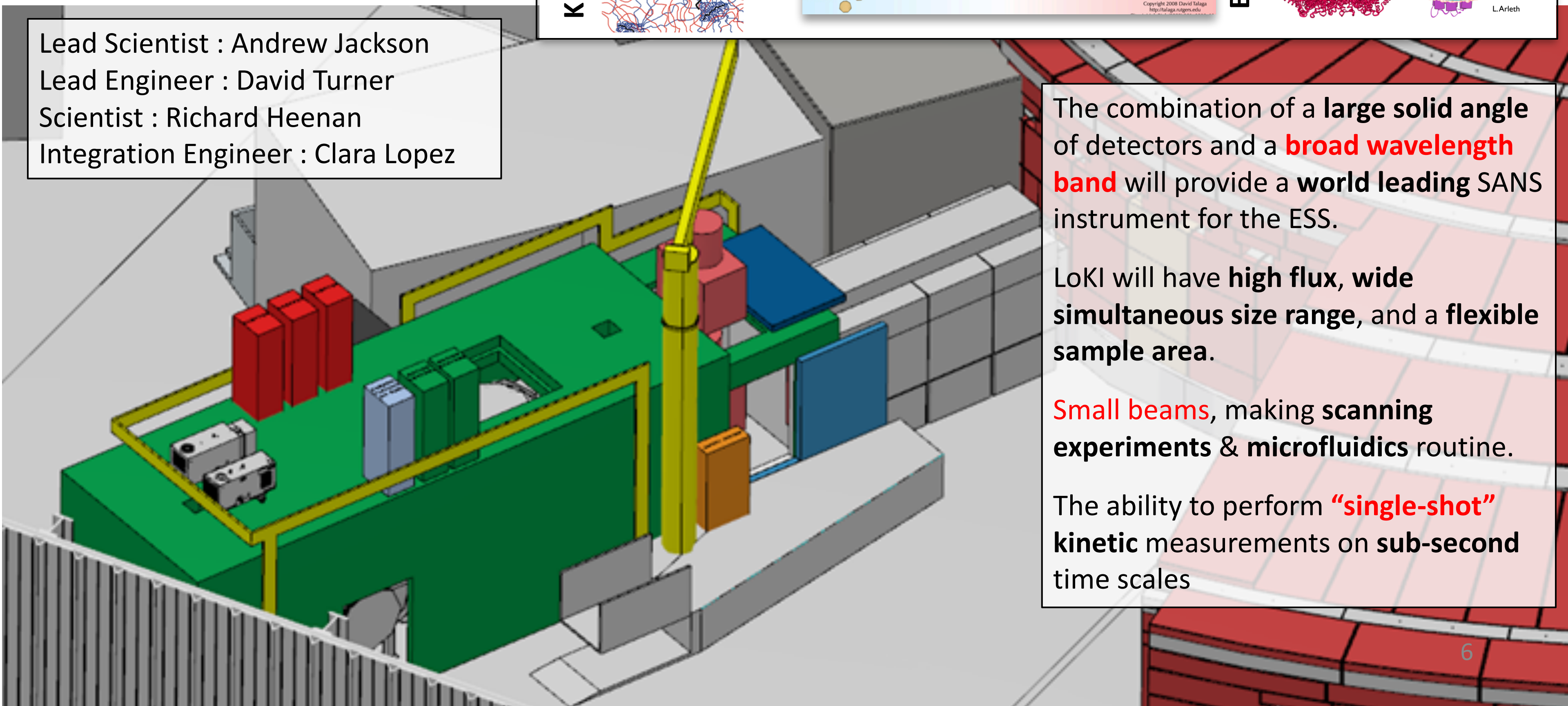
Science & Technology Facilities Council

EUROPEAN SPALLATION SOURCE

- High flux – up to 1×10^{9} n/cm^{2}/s
- 8 + 10 m collimation
- 14 Hz or 7 Hz operation
- Up to 20 Å bandwidth
- Option for resolution enhancing choppers



Lead Scientist : Andrew Jackson
Lead Engineer : David Turner
Scientist : Richard Heenan
Integration Engineer : Clara Lopez



The combination of a **large solid angle** of detectors and a **broad wavelength band** will provide a **world leading** SANS instrument for the ESS.

LoKI will have **high flux**, **wide simultaneous size range**, and a **flexible sample area**.

Small beams, making **scanning experiments** & **microfluidics** routine.

The ability to perform **"single-shot" kinetic** measurements on **sub-second** time scales

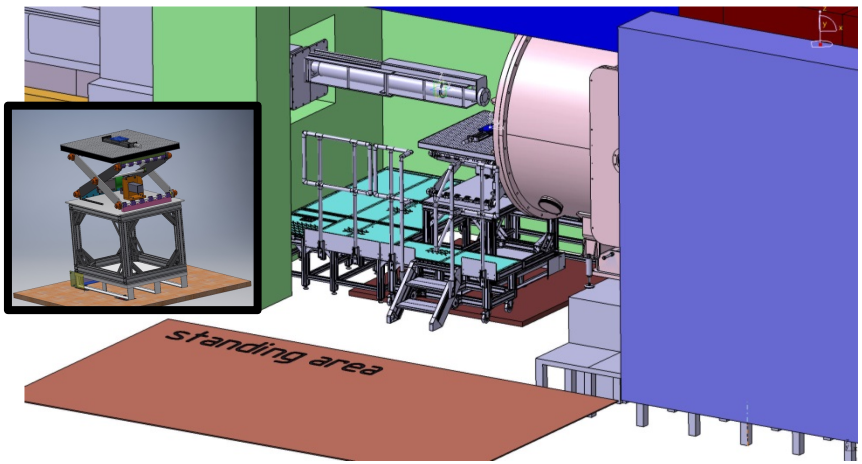
SKADI: Versatility, speed and resolution

JÜLICH FORSCHUNGSZENTRUM

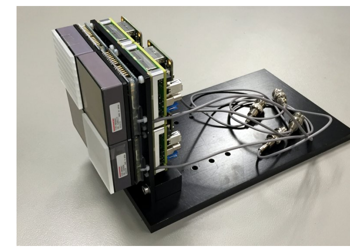
- World leading flux: ~5x10^{8} neutrons/s cm^{2} at sample position
- 20, 14, 8 m collimation (option for 4m)
- 14 or 7 Hz operation
- 5 -10 Å bandwidth
- wavelength resolution freely tuneable between 8 and 1%
- polarization and spin-flipper included for polarized scattering

FZJ/LLB collaboration with FZJ lead
Lead Scientist: Sebastian Jaksch (FZJ)
Scientist: Jacques Jestin (LLB)
Lead Engineers: Romuald Hanslik (FZJ) / Sylvain Désert (LLB)

Generalized Mounting System for SE



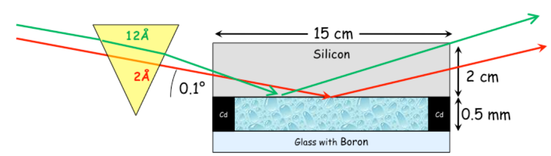
SoNDe Detector System



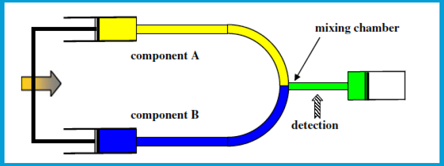
- high-flux
- high-resolution
- modular
- measurement of direct beam

GISANS/GINSES options:

prism, resonator



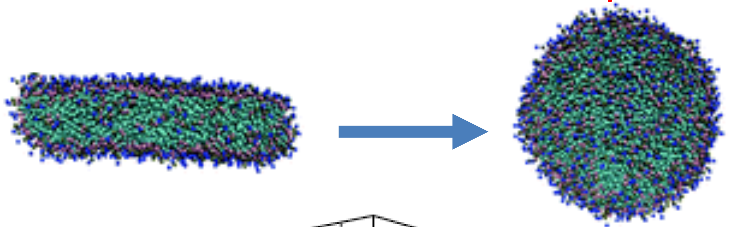
Non-equilibrium studies

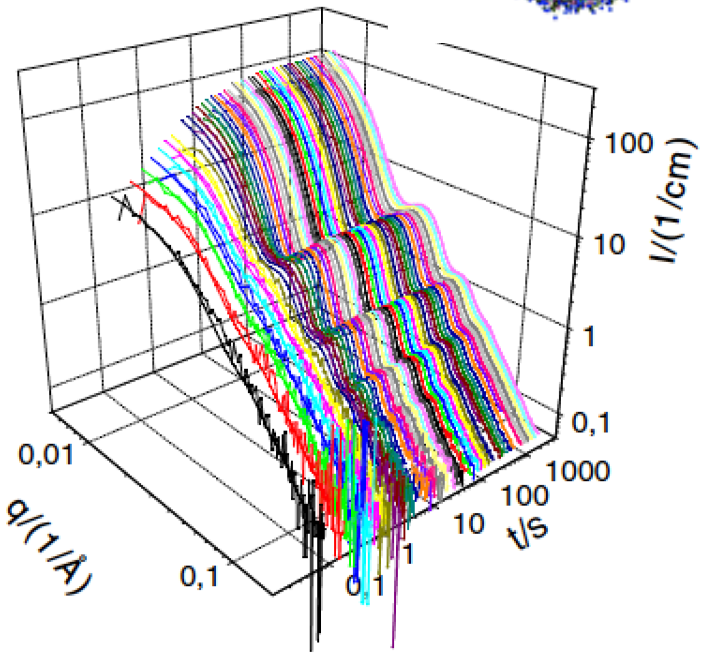


- Stopped-flow SANS: self-assembly and exchange kinetics

<u>Disc to vesicle transition:</u> 50-100ms shots repeated 10-25 times

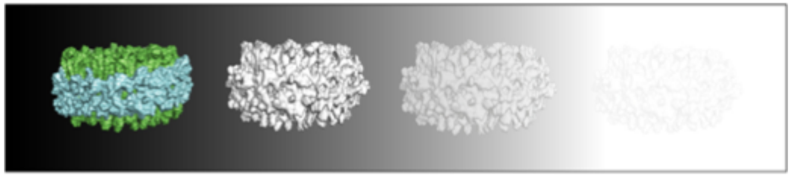
@ESS: SKADI/Loki: less need to repeat

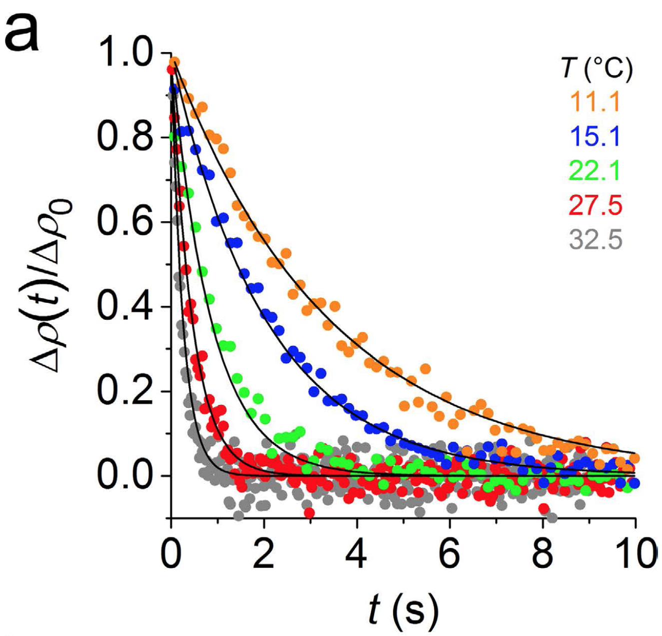




Bressel et al., Colloid Polym Sci (2010) 288:827–840

Neutron contrast (deuteration) to detect lipid transfer between nanodiscs:





Cuevas Arenas et al., Scientific Reports 7, 45875-1-45875-8 (2017)

8

Microfluidic SANS

“As the flux of neutron facilities continually increases, and with the advent of next generation pulsed sources (e.g. European Spallation Source, ESS, due to go live within the next 2–6 years), pressure on more effective and precise sample environments will only increase.”

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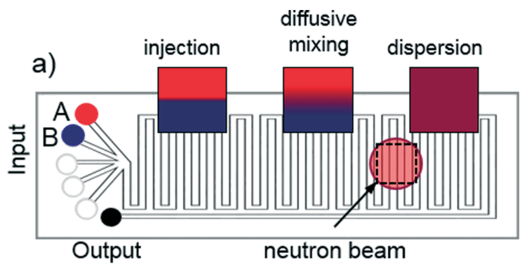
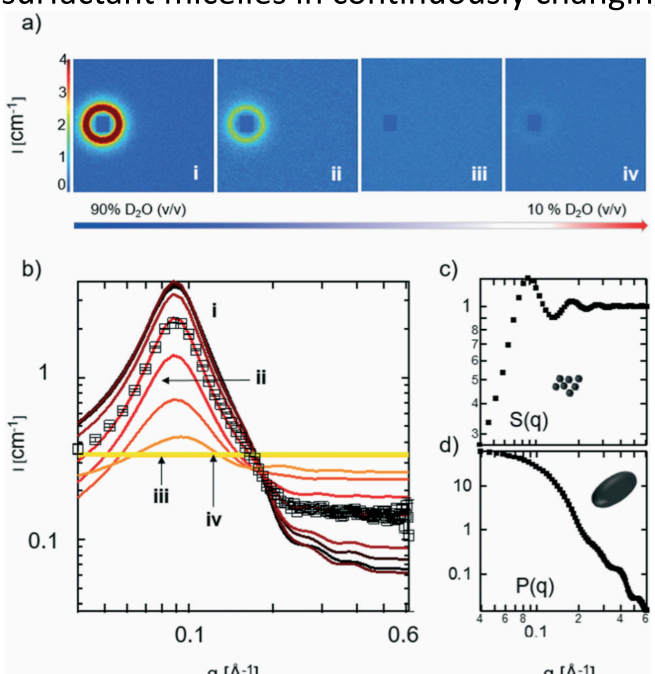
- High Throughput Mixing & Tailored Flow Geometry

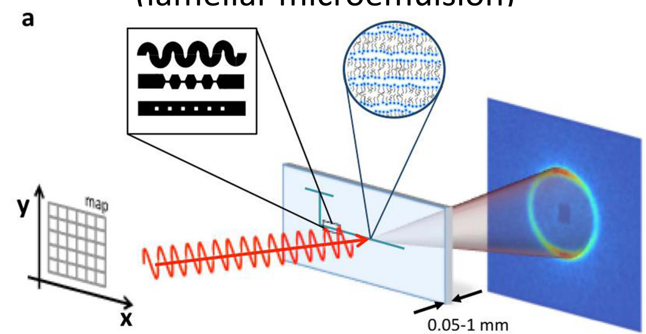


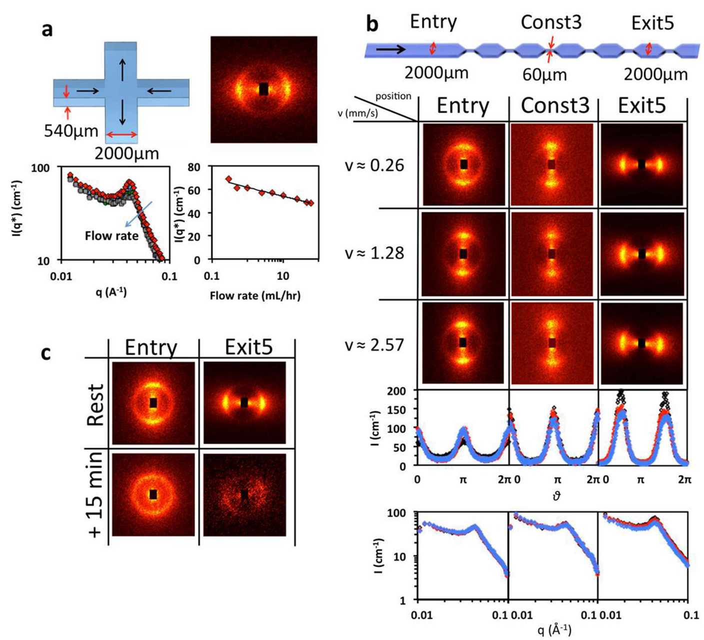
SDS surfactant micelles in continuously changing contrast:



Adamo, M., Poulos, A. S., Miller, R. M., Lopez, C. G., Martel, A., Porcar, L., & Cabral, J. T. (2017). *Lab Chip*, *17*(9), 1559–1569.

Flow processing of complex fluids:
(lamellar microemulsion)





C.G. Lopez, T. Watanabe, A. Martel, L. Porcar, J.T. Cabral, Scientific Reports, 5 (2015) 7727.

Weakly scattering bio-engineered samples

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PLOS | BIOLOGY

RESEARCH ARTICLE

The in vivo structure of biological membranes and evidence for lipid domains

Jonathan D. Nickels[1,2,3©], Sneha Chatterjee[2,4©], Christopher B. Stanley[2], Shuo Qian[2], Xiaolin Cheng[5,6], Dean A. A. Myles[2], Robert F. Standaert[1,2,4,6]*, James G. Elkins[4,7]*, John Katsaras[1,2,3]*

1 Shull Wollan Center—A Joint Institute for Neutron Sciences, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States of America, 2 Biology and Soft Matter Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States of America, 3 Department of Physics and Astronomy, University of Tennessee, Knoxville, Tennessee, United States of America, 4 Biosciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States of America, 5 Center for Molecular Biophysics, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States of America, 6 Department of Biochemistry & Cellular and Molecular Biology, University of Tennessee, Knoxville, Tennessee, United States of America, 7 Department of Microbiology, University of Tennessee, Knoxville, Tennessee, United States of America

© These authors contributed equally to this work.
* standaertrf@ornl.gov (RFS); elkinsjg@ornl.gov (JGE); katsarasj@ornl.gov (JK)

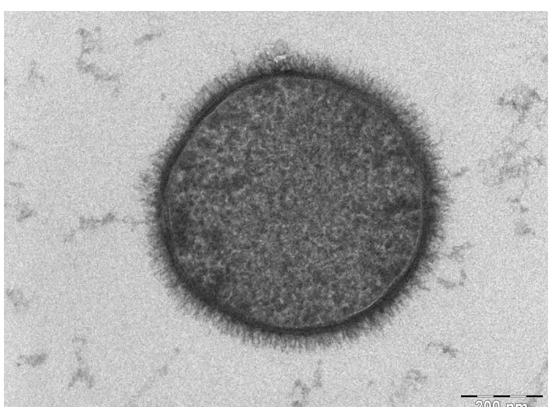
Check for updates

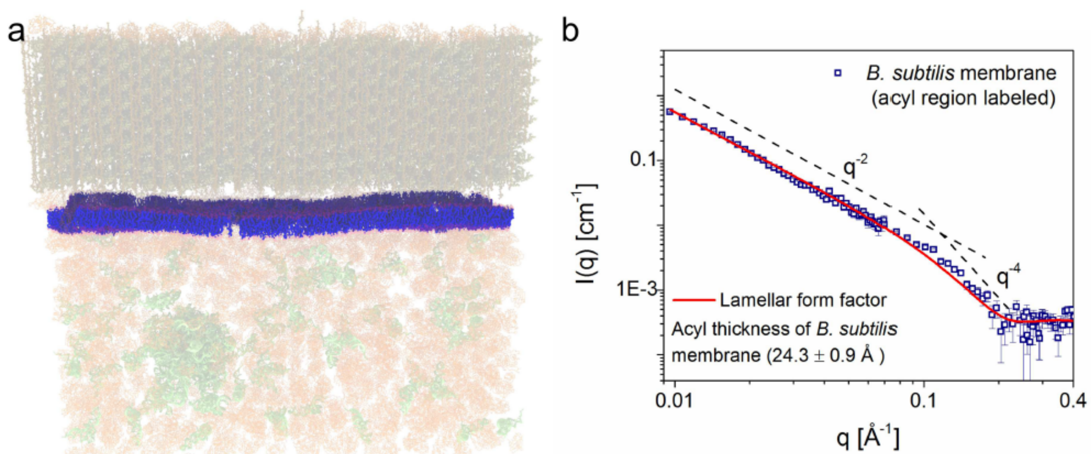
SANS from perdeuterated living cells (B. subtilis) with hydrogen labelled cell membrane

- 4h on BioSANS (ORNL) @ 5mg/ml cells

-> shorter counting times/lower concentration

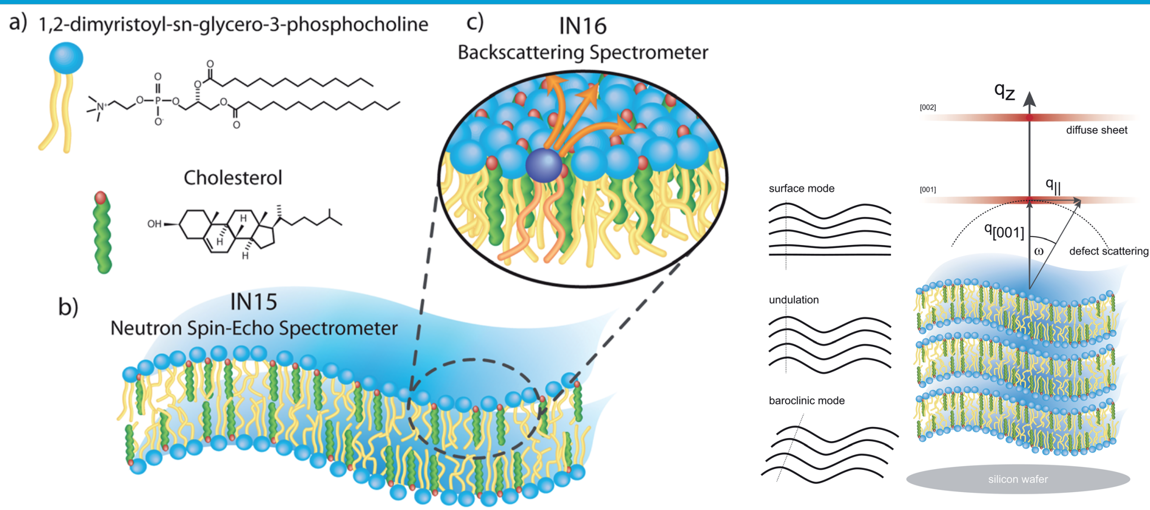
-> other "crowded" environments



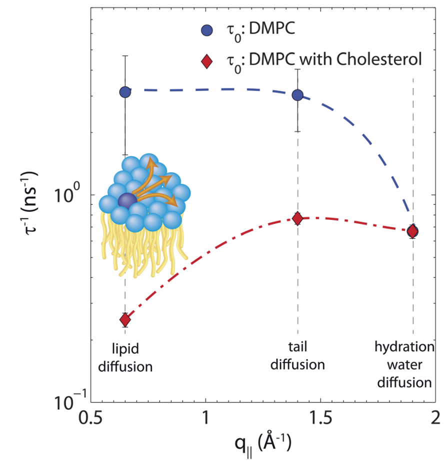


Lipid dynamics in membranes

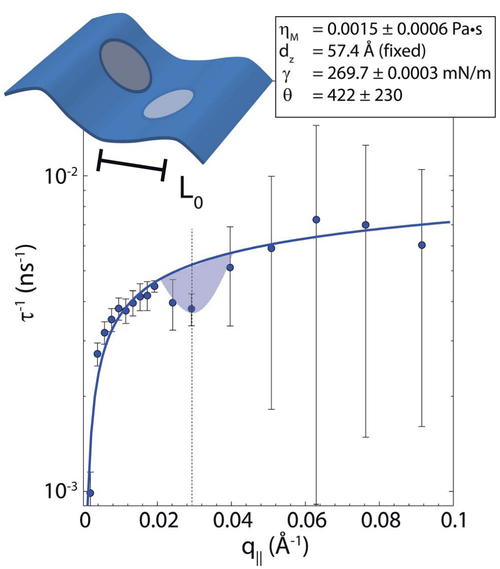
Armstrong, C. L.; Häußler, W.; Seydel, T.; Katsaras, J.; Rheinstädter, M. C. *Soft Matter* **2014**, *10* (15), 2600–2612.

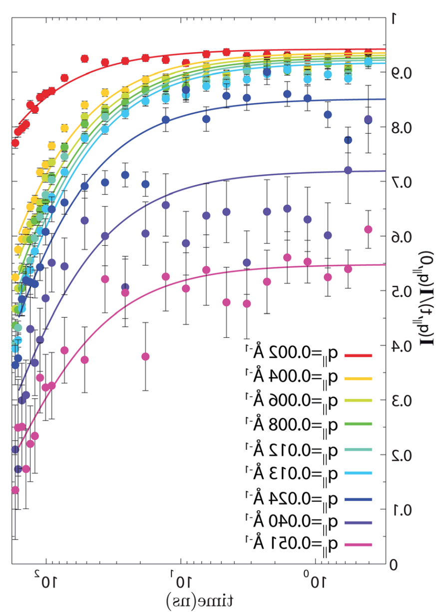


Backscattering spectroscopy (QENS):



NSE spectroscopy:





@ ESS: smaller samples will allow broader range of materials studies

- MIRACLES Backscattering/QENS
- NSE spectrometer (TBD)
- SKADI: NSE in grazing incidence geometry (GINSES)

Lipid dynamics in nanorafts: SANS + neutron spin-echo spectroscopy

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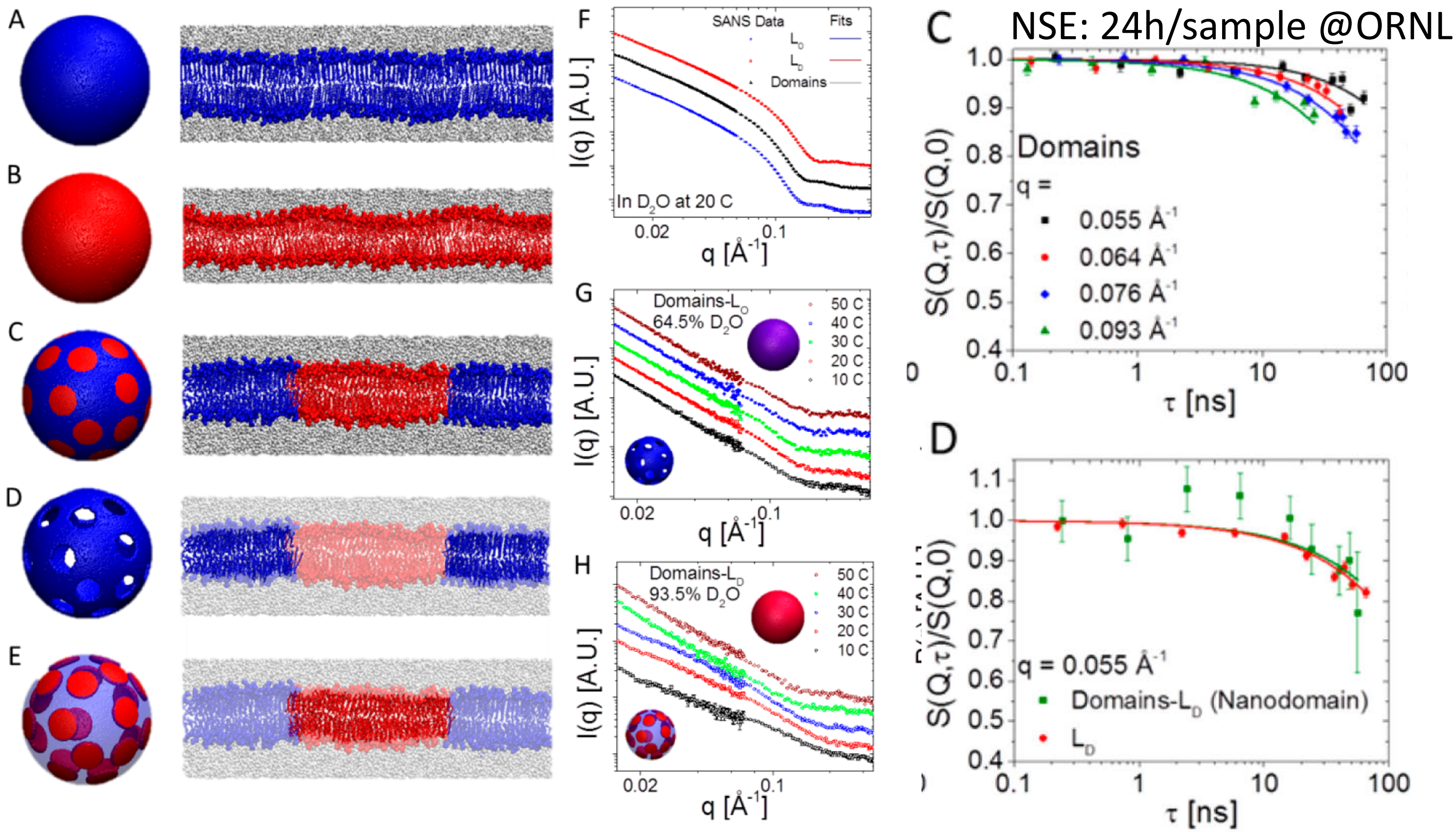
DSPC-POPC-Cholesterol K_{ld} = 17.3 ± 3.2 k_BT, K_{lo} = 196.0 ± 42.5 k_BT

Bending rididigy of nanodomains K_{rafts} (13nm) = 18.4 ± 9.8 k_BT



Nickels et al., JACS 137 (2015) 15772-15780, http://dx.doi.org/10.1021/jacs.5b08894.

Estia *a polarised focusing reflectometer for small samples*

Paul Scherrer Institut
Switzerland
Jochen Stahn Artur Glavic

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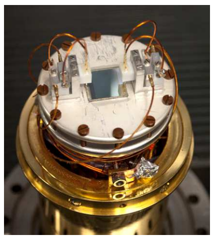
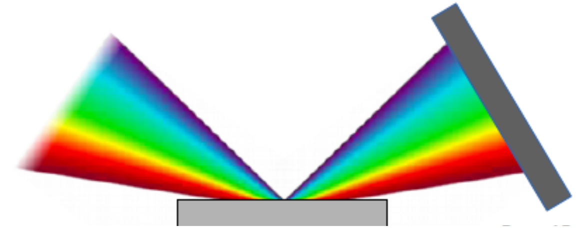
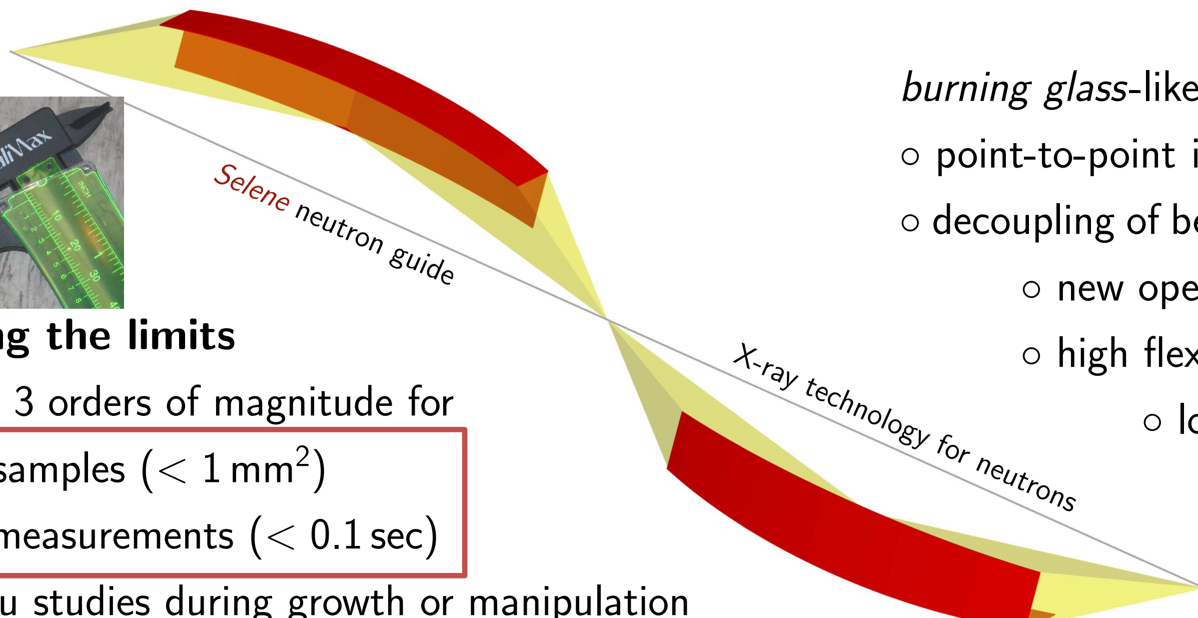
for the investigation of the
chemical and magnetic depth-profile near surfaces
and of lateral correlations and structures



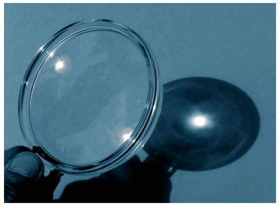


functional devices:	*spin-valves, spintronics*
diffusion processes:	*Li batteries, corrosion protection*
multifunctional materials:	*interface-coupled electric and magnetic properties*
towards *real* materials:	*raster-scanning of bent, faceted or multi-domain surfaces*



burning glass-like **neutron guide**

- point-to-point imaging
- decoupling of beam size and divergence
  - new operation modes
  - high flexibility
    - low background



pushing the limits

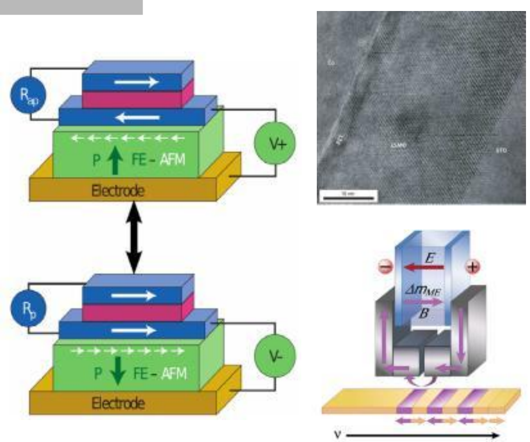
by 2 to 3 orders of magnitude for

- tiny samples ($< 1\,\text{mm}^2$)
- fast measurements ($< 0.1\,\text{sec}$)
- in-situ studies during growth or manipulation

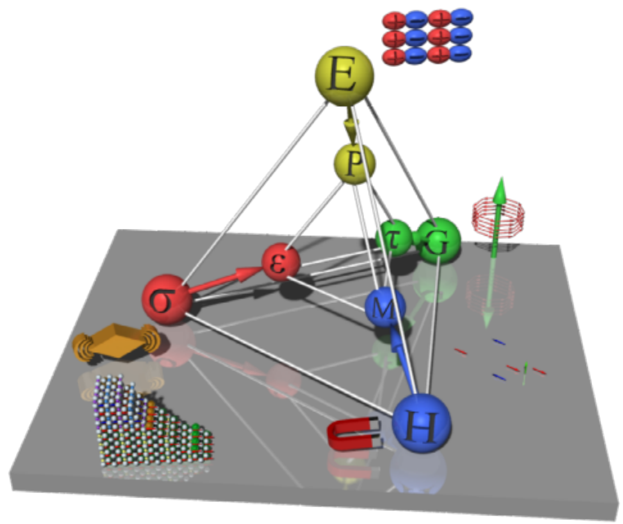
Artur Glavic

Broad range of science

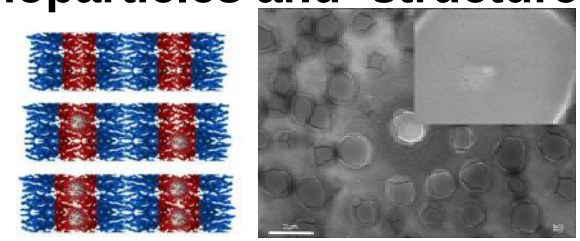
Spin-tronics Devices



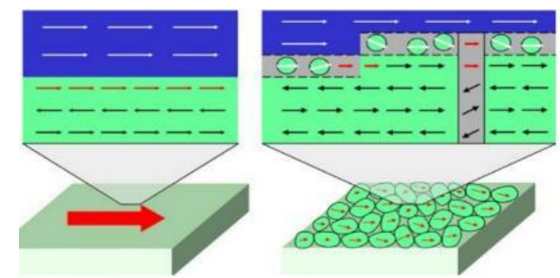
Multiferroics



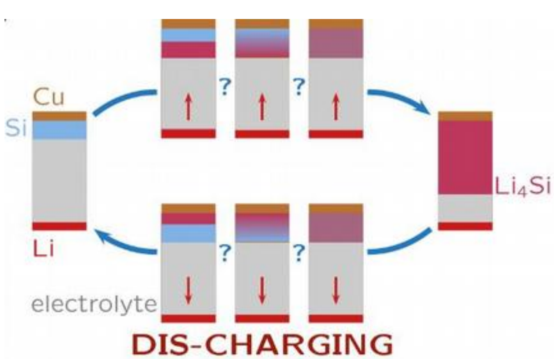
Nanoparticles and -structures



Exchange Bias

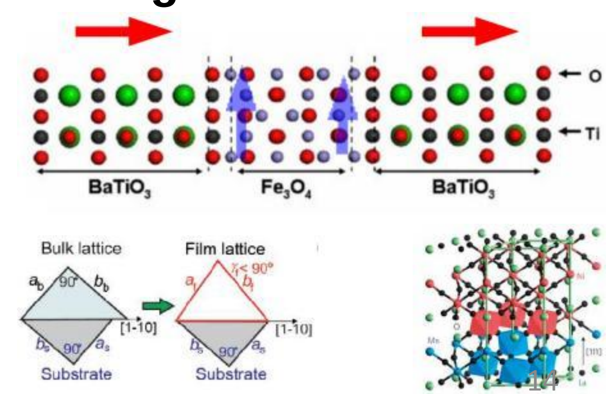


Batteries



Liquid Interfaces

Emergent Phaenomena



FREIA Horizontal Reflectometer

(H.Wacklin, ESS – in-kind partner ISIS Neutron facility, STFC, UK)

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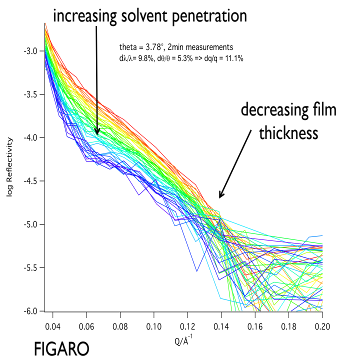
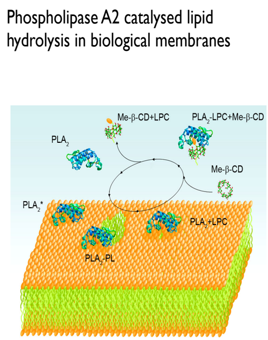
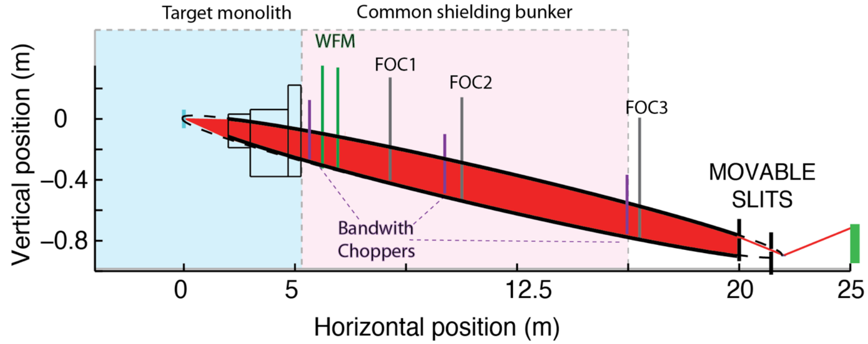
Time-resolved experiments, free- liquid interfaces.

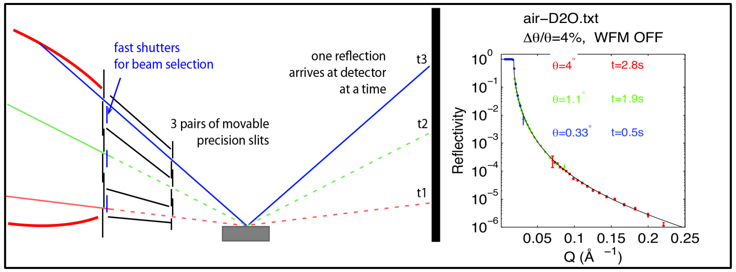


FIGARO



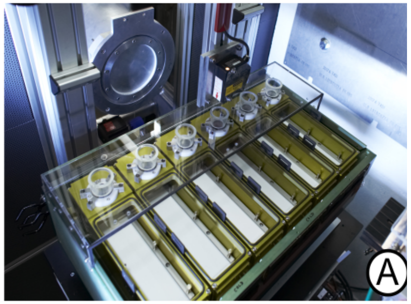


No movement of sample – broad Q-range

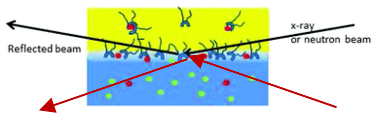


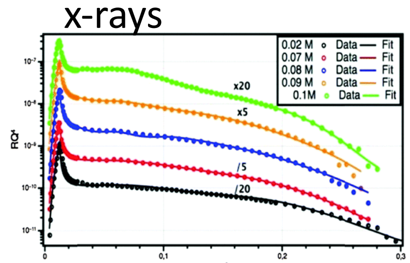
Kinetics at interfaces: Lipid remodeling by enzymes – broad Q-range needed.

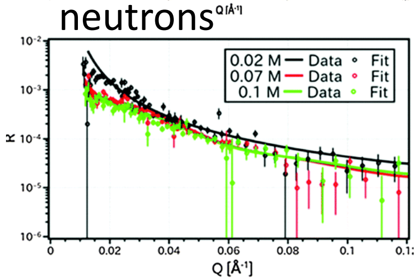
Free liquid interfaces:



Liquid-liquid interfaces:



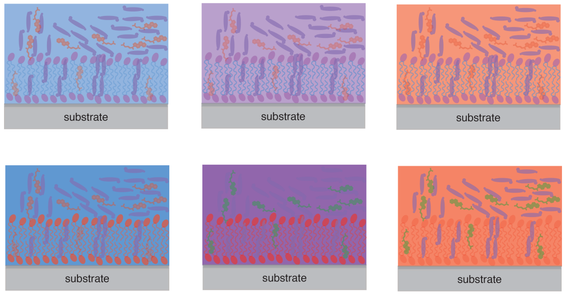


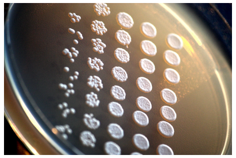


High throughput studies:

h-lipid d-sterol

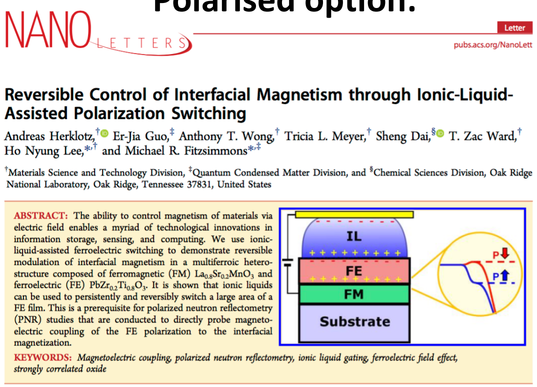
d-lipid h-sterol





Screening drug resistance/ virulence factors

Polarised option:


NANO LETTERS

Letter

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Reversible Control of Interfacial Magnetism through Ionic-Liquid-Assisted Polarization Switching

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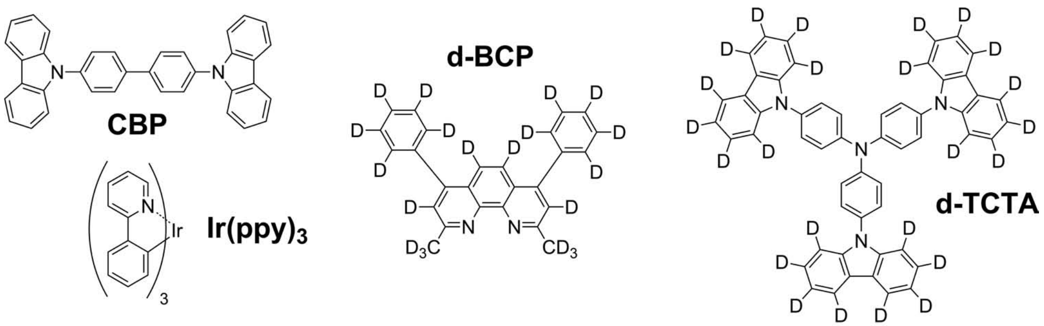
ABSTRACT: The ability to control magnetism of materials via electric field enables a myriad of technological innovations in information storage, sensing, and computing. We use ionic-liquid-assisted ferroelectric switching to demonstrate reversible modulation of interfacial magnetism in a multiferroic heterostructure composed of ferromagnetic (FM) $La_{0.8}Sr_{0.2}MnO_3$ and ferroelectric (FE) $PbZr_{0.2}Ti_{0.8}O_3$. It is shown that ionic liquids can be used to persistently and reversibly switch a large area of a FE film. This is a prerequisite for polarized neutron reflectometry (PNR) studies that are conducted to directly probe magnetoelectric coupling of the FE polarization to the interfacial magnetization.

IL
FE
FM
Substrate

KEYWORDS: *Magnetoelectric coupling, polarized neutron reflectometry, ionic liquid gating, ferroelectric field effect, strongly correlated oxide*

Interdiffusion in OLEDs – neutron contrast:

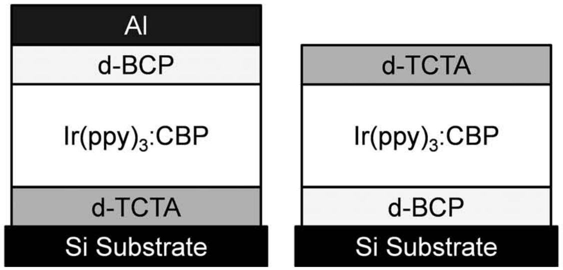
Mixing of components at high operating T reduces performance



Interdiffucion between OLED layers:

FREIA – faster scans with extended q-range – thinner films

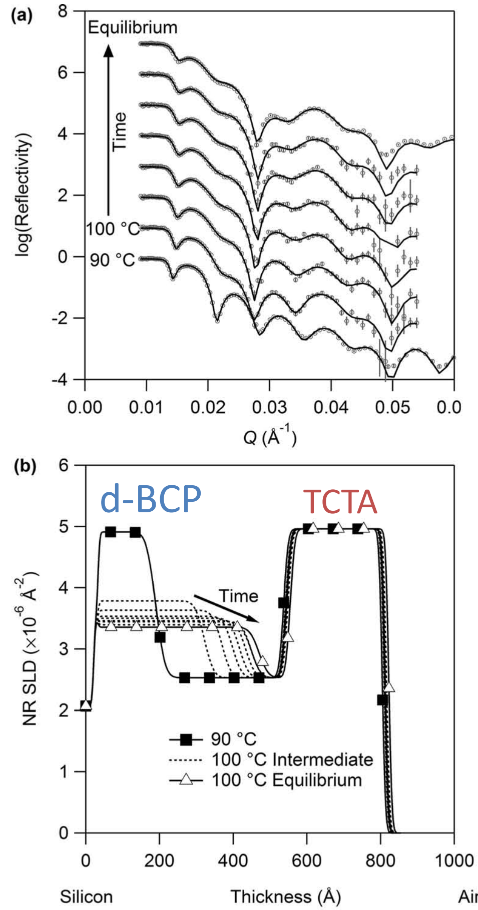
Film 1



TCTA: hole transport layer

photoluminescent layer

BCP: electron transport layer



OLED performance depends on compartmentalization of active layers. Operating temperatures cause interdiffusion and degradation of photoluminescence.

Good neutron contrast by deuterium labeling of TCTA and BCP allows determination of vertical concentration profile.

A.R.G. Smith etal. , Advanced Materials, 24 (2012) 822-826.

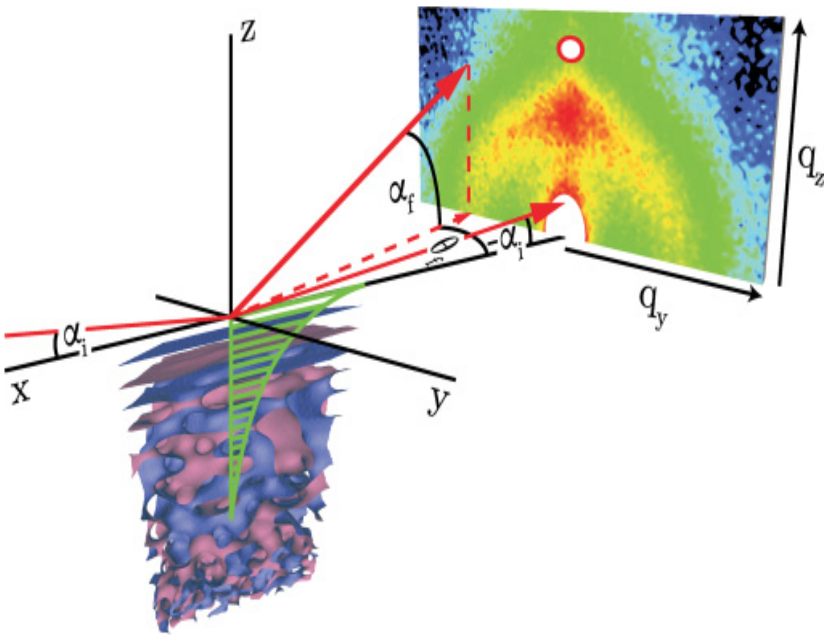
Grazing incidence SANS (GISANS)

-> small angle scattering of reflected beam = surface sensitive

Much shorter measurement times at ESS to make GISANS a routine technique

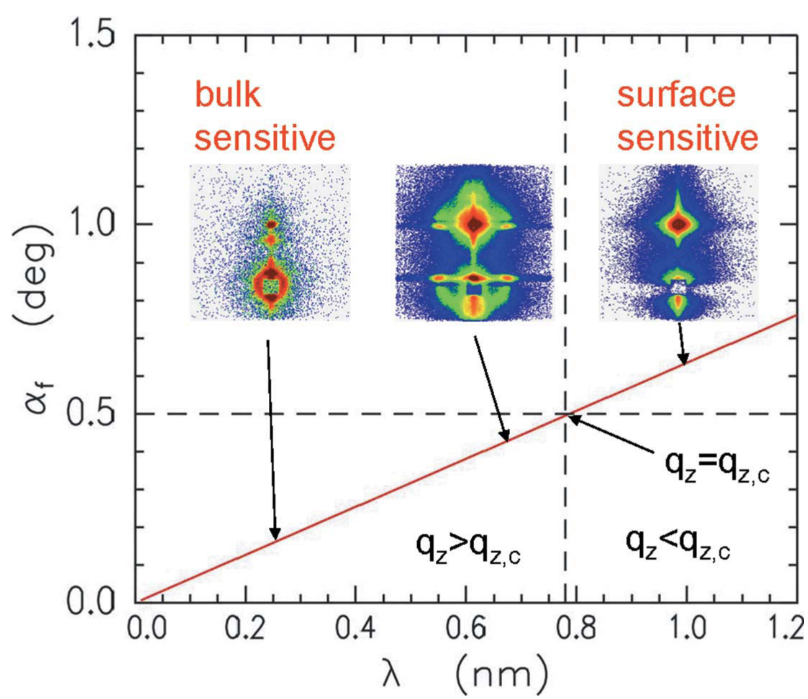
GISANS option @ SKADI, ESTIA

+ interest in dedicated instrument



H. Frielinghaus, M. Kerscher, O. Holderer, M. Monkenbusch, D. Richter, Phys. Rev. E, 85, (2012), 041408.

Surface sensitivity above critical wavelength

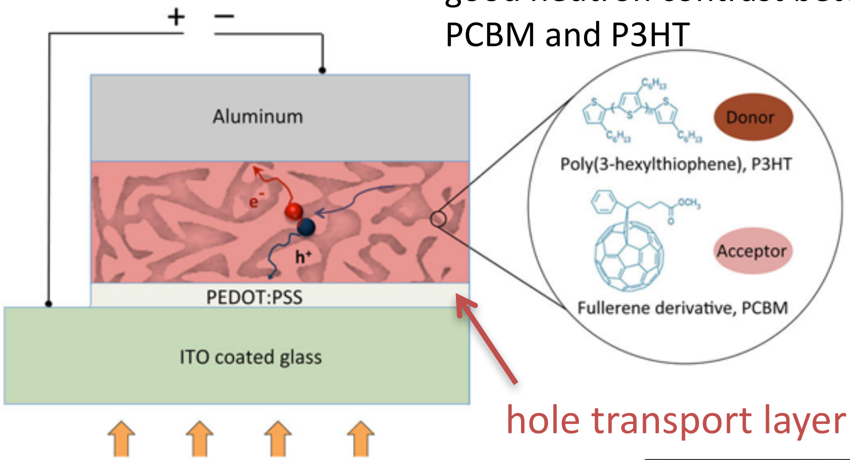


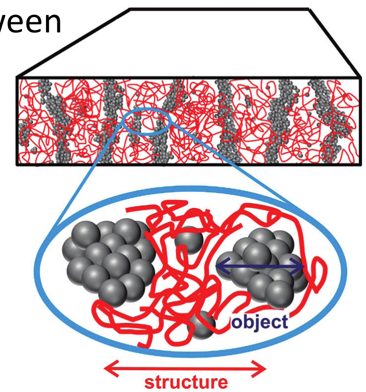
P. Mueller-Busch-Baum, Polymer Journal (2013) 45, 34–42

GISANS: nanostructure in organic solar cells

Phase separation, composition, crystallinity using GISANS + GISAXS:

good neutron contrast between PCBM and P3HT



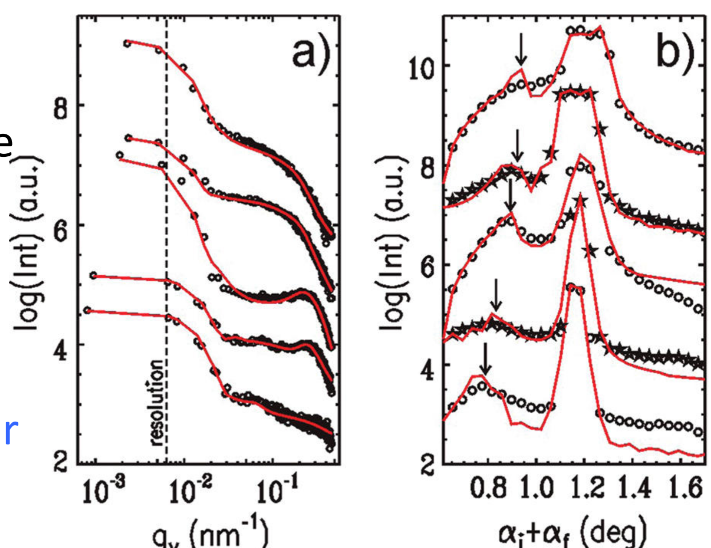


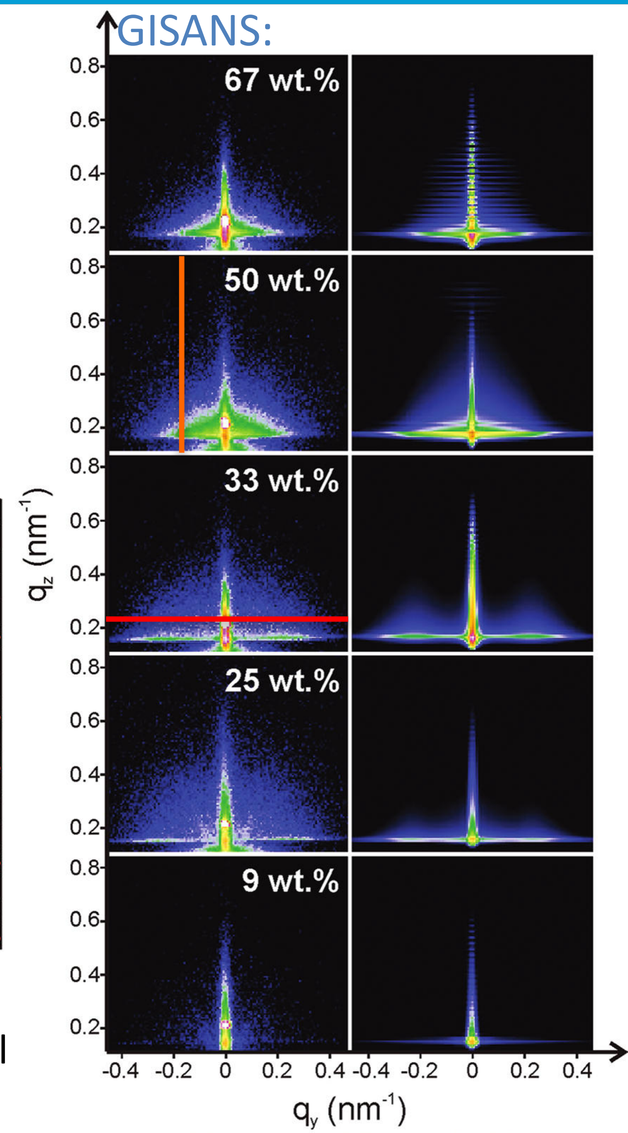
a) GISANS horizontal cuts: particle and domain size

b) GISANS vertical cuts:-> composition of phases

c) GISAXS: -> crystallinity

Increasing molecularly dispersed PCBM with higher content ≠ pure phases





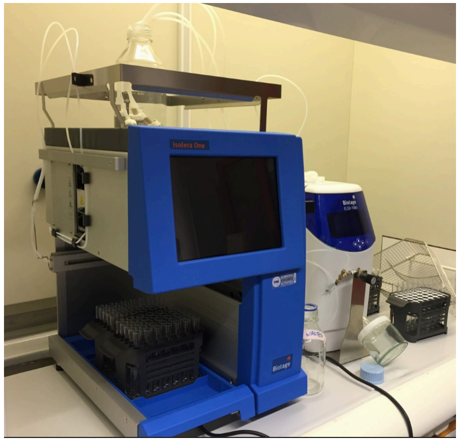
M.A. Ruderer, R. Meier, L. Porcar, R. Cubitt, P. Mueller-Buschbaum, Journal of Physical Chemistry Letters, 3 (2012) 683-688.

DEULAB at ESS: Contrast for neutron scattering

- Chemical deuteration and synthesis

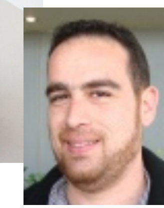
Synthesis, purification and characterisation of small molecules

- Enzyme technologies for synthesis of chiral/complex molecules
- Analysis and purification of lipids and fatty acids from cell cultures
- Derivatisation of biological molecules
- DEMAX Biological deuteration at Lund Protein Production facility.



Deunet

Peter



Tamim

Find out more: http://deuteration.net or follow @deuteration.net

European chemical deuteration network

NEUTRONS FOR SCIENCE

Science & Technology Facilities Council

JÜLICH FORSCHUNGSZENTRUM

Australian Government
Department of Education, Science and Training

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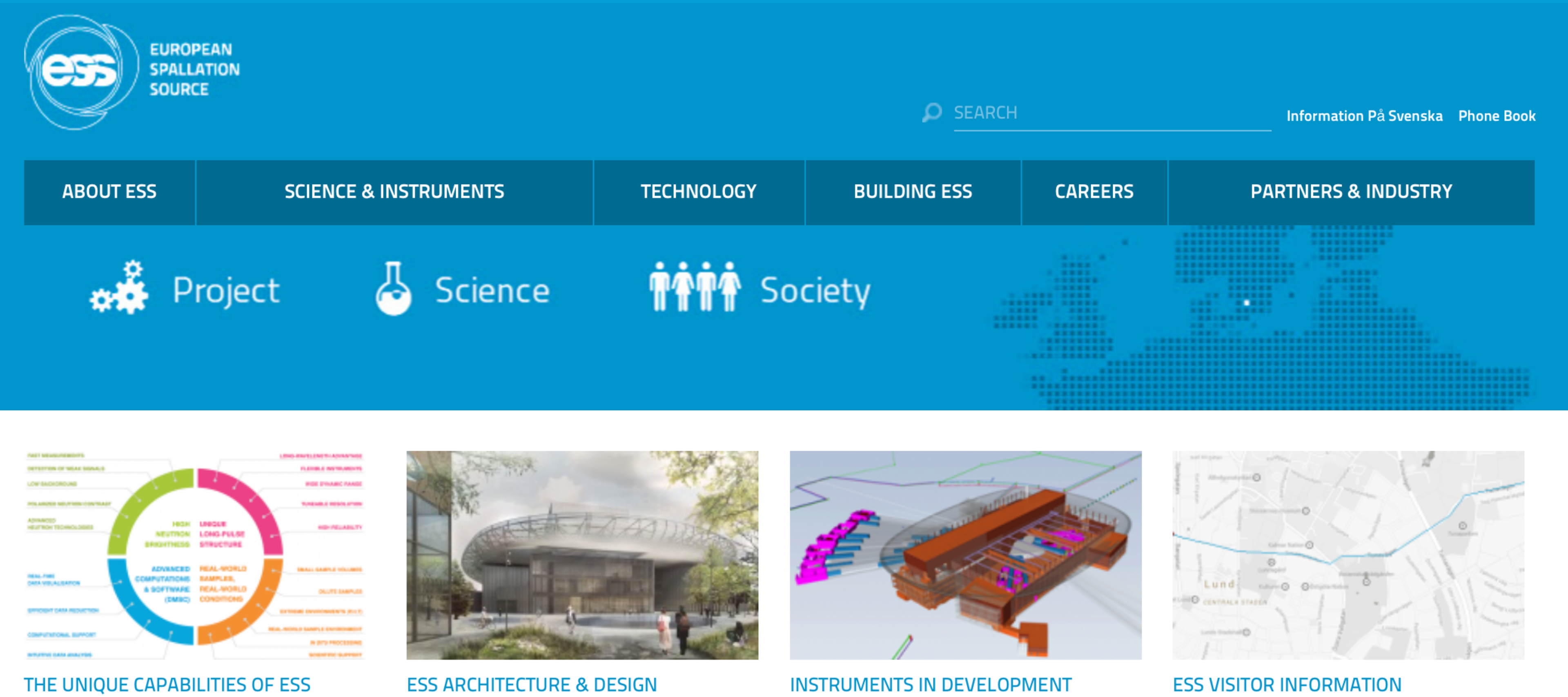
Conclusion:

The neutron flux and instrumentation at ESS will enable

- **neutron scattering form a broader range of soft materials and interfaces**
- **new experiments with faster time-resolution and smaller samples**
- **parametric studies**
- **in-situ and in-operando studies**
- **fundamental and applied research by academic and industrial users**

Thank you for your attention!



http://europeanspallationsource.se