Diffraction instrumentation at ESS

26th June 2018

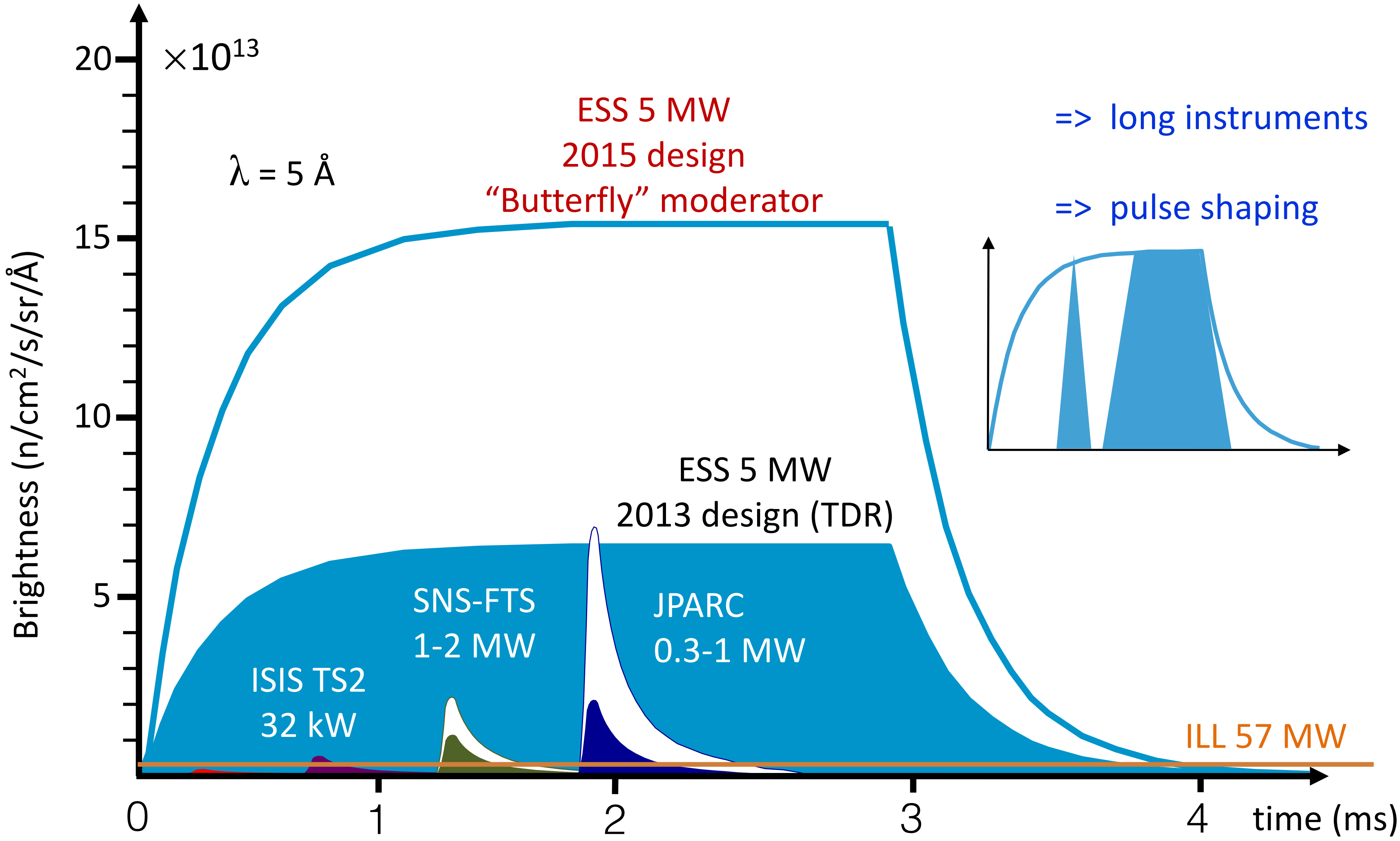
Werner Schweika, Neutron Instruments Division, European Spallation Source ERIC

User operation will start end of 2023
Impressions from the construction site

EUROPEAN SPALLATION SOURCE

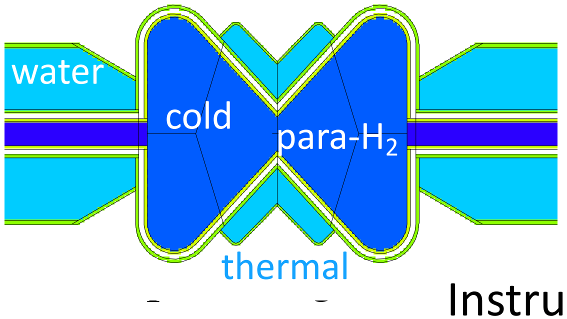
ESS: long-pulse 14Hz

superior flux & brightness



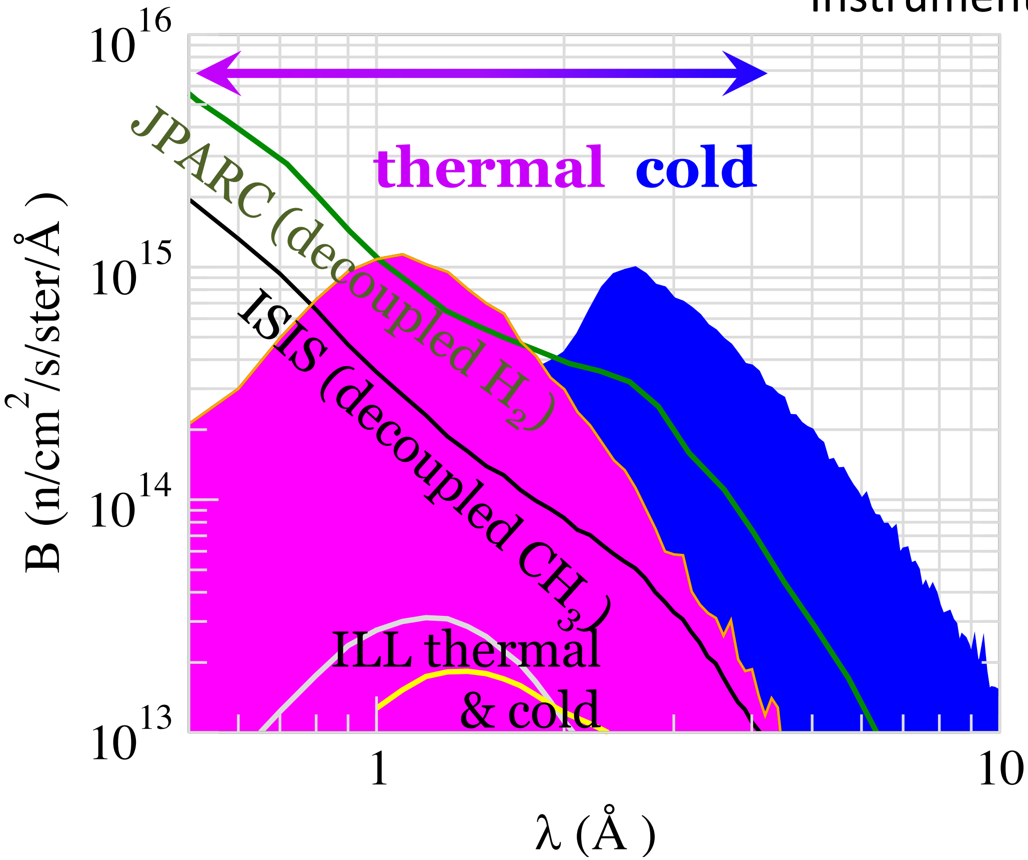
ESS “Butterfly” Moderator

peak brightness



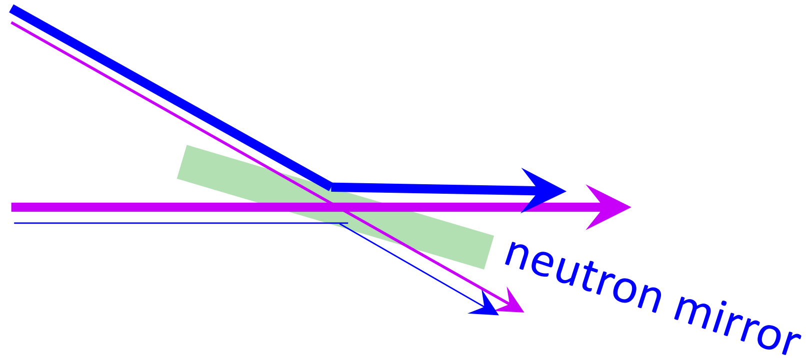
height reduced to 3 cm

Instruments can choose thermal or cold moderator

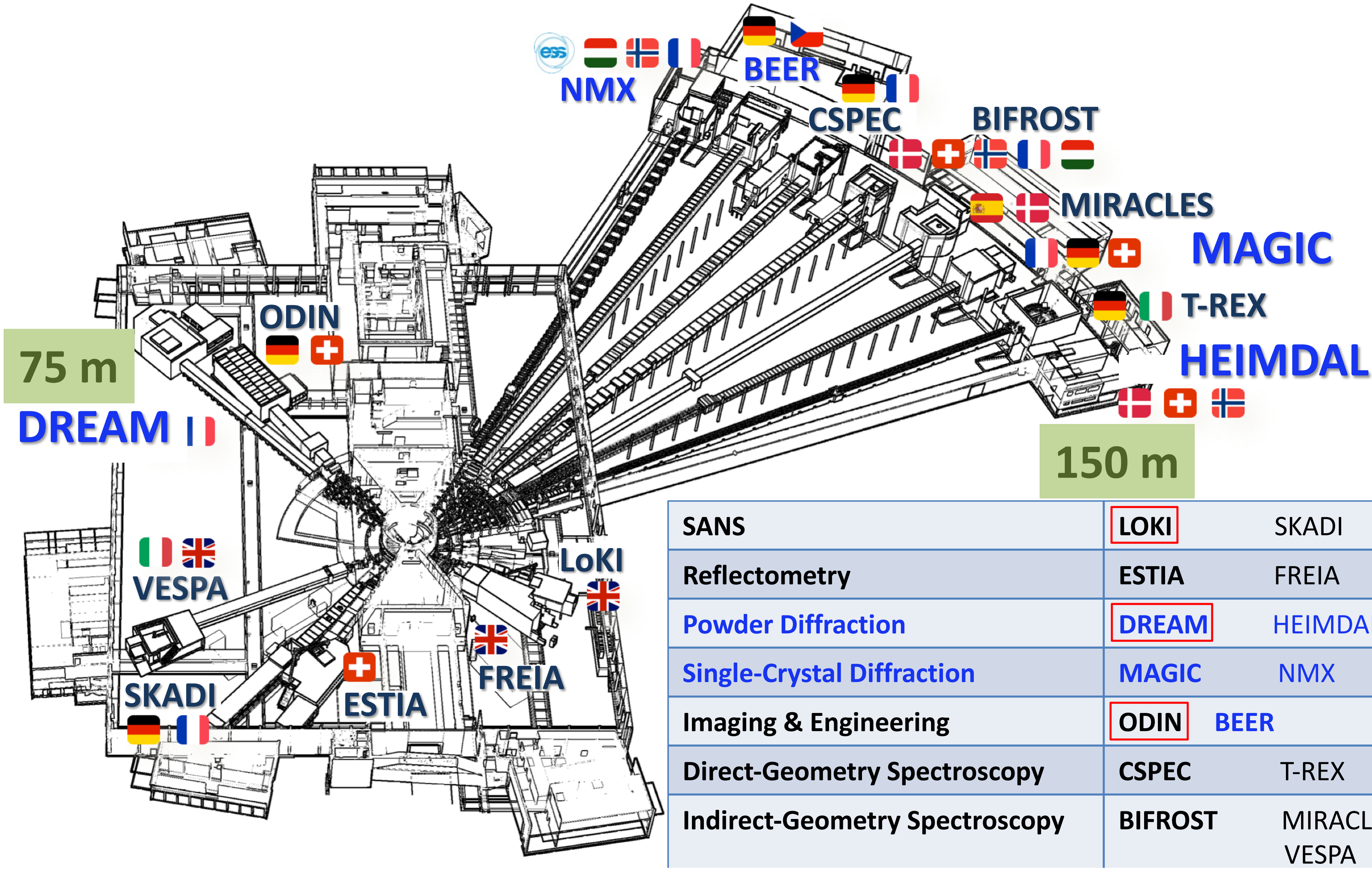


DREAM views simultaneously the peak flux of both

the trick is a “bi-spectral switch”



Instrument Suite

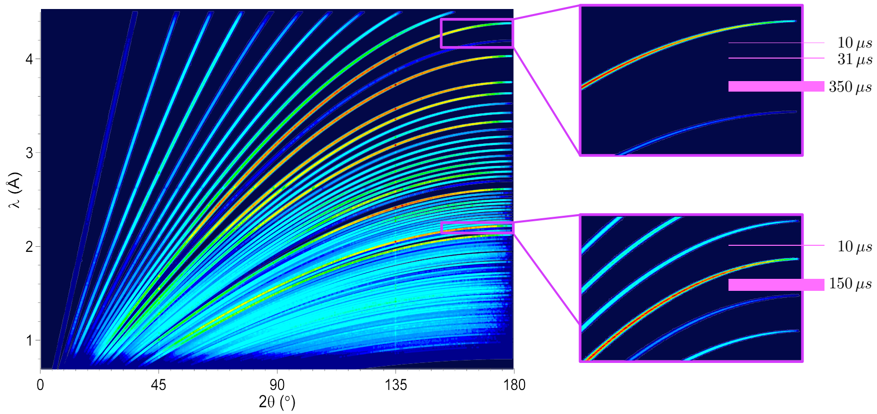


SANS	LOKI	SKADI
Reflectometry	ESTIA	FREIA
Powder Diffraction	DREAM	HEIMDAL
Single-Crystal Diffraction	MAGIC	NMX
Imaging & Engineering	ODIN	BEER
Direct-Geometry Spectroscopy	CSPEC	T-REX
Indirect-Geometry Spectroscopy	BIFROST	MIRACLES VESPA

powder diffraction

very high intensity compared to existing instruments
very flexible resolution due to pulse shaping

DREAM thermal and cold (+ nm-SANS)



HEIMDAL thermal (+SANS)
multiple length scales

MAGIC polarized

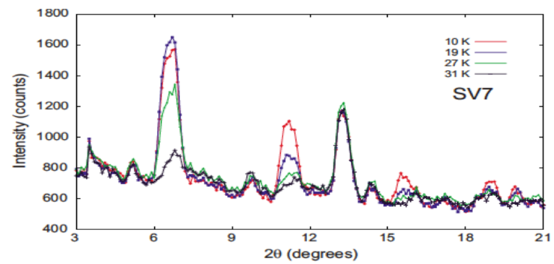
separating
magnetic neutron scattering
… and incoherent H …

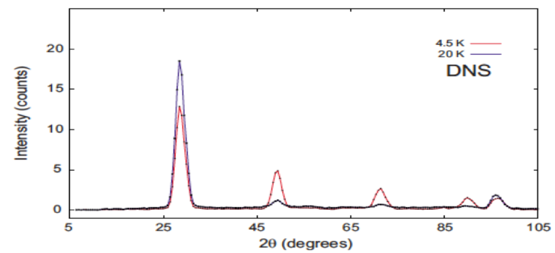
These instruments have new ^{10}B - detectors

* high efficiency and
* count rate capability
- 2D (3D) resolution
  single crystal diffraction
  texture

2D Rietveld

J. Appl. Cryst. 48 (2015) 1627
J. Appl. Cryst. (2017) in press





Krott et al, PRB 80 (2009), 024117

powder & texture

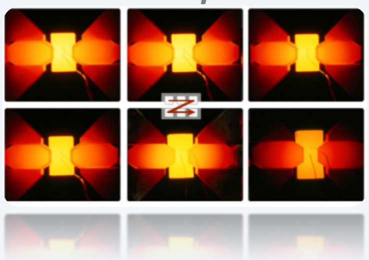
EUROPEAN SPALLATION SOURCE

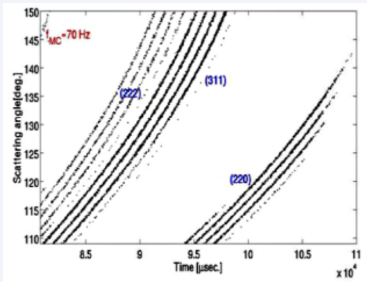
Engineering Diffractometer

BEER thermal and cold

Materials under stress

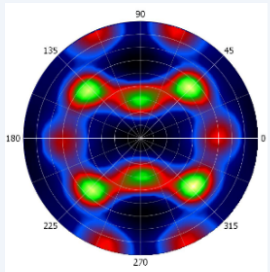
in-situ in operando





modulation technique

TOF Powder diffraction



Texture measurements

Imaging & SANS **in future**

J. Fenske (HZG, Germany) P. Beran (NPI, Czech Republic)

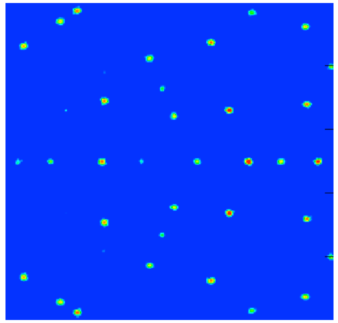
TOF Laue diffraction => 3d Q space

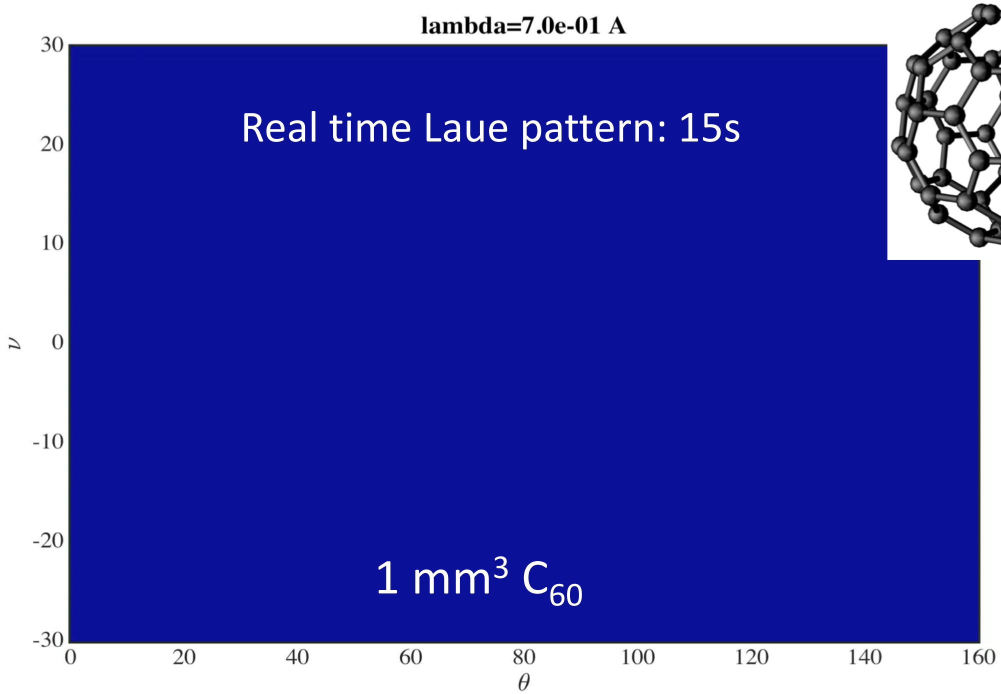
EUROPEAN SPALLATION SOURCE

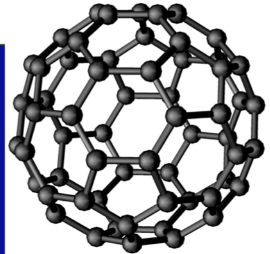
Instruments for single crystal diffraction

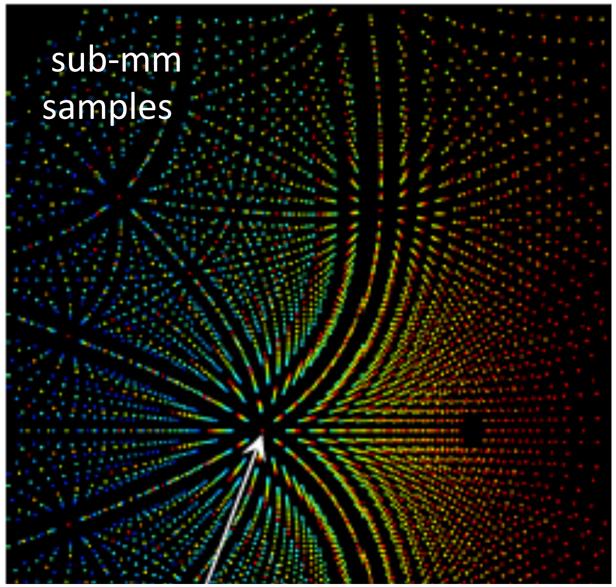
MAGIC dedicated for magnetism - **polarized**

DREAM unpolarized / higher resolution / 3D PDF (HEIMDAL)









NMX for macromolecular crystallography

Esko Oksanen

Hydrogen positions

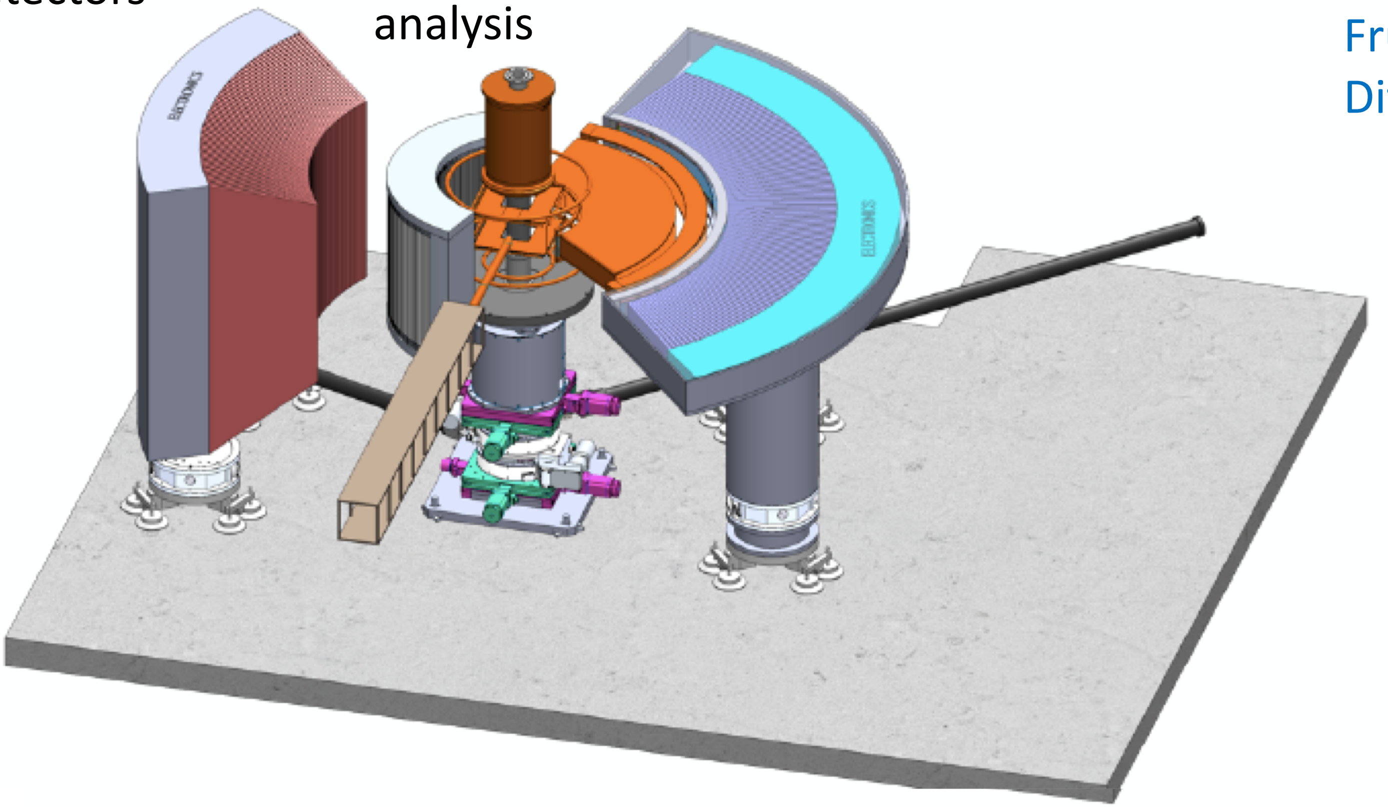
MAGIC

polarized cold & thermal beam

Magnetic structures
Spin densities
Local susceptibilities
Frustrated magnetism
Diffuse scattering

Detectors

Polarization
analysis



DREAM

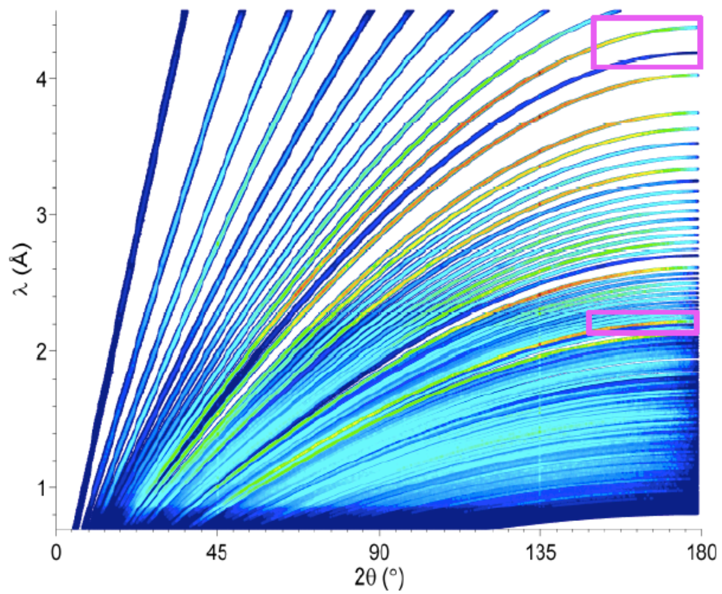
Diffraction **R**esolved by **E**nergy and **A**ngle **M**easurements

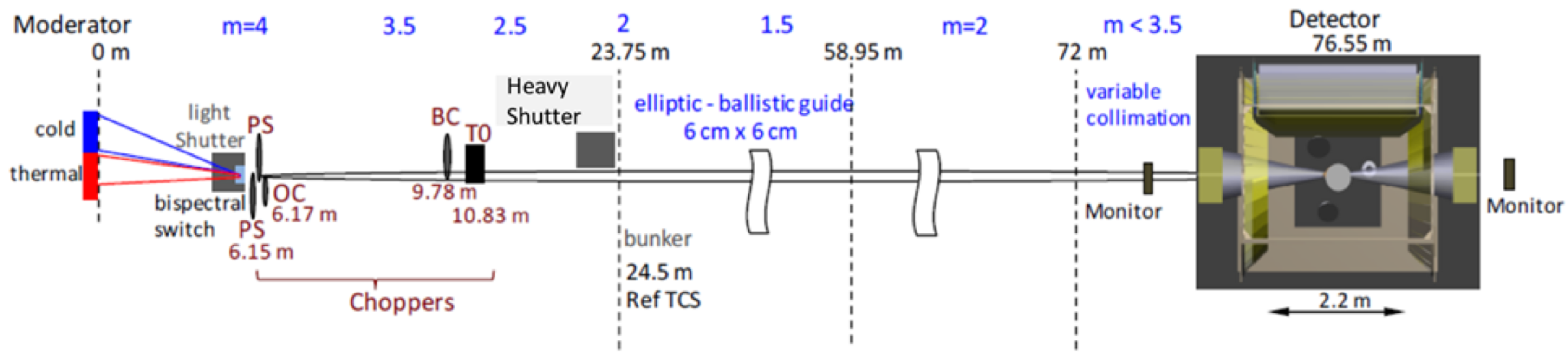
- General use powder diffractometer with novel capabilities, which will outperform in its first stage existing instruments by factor of 10 on day one

- In-kind contribution to ESS from Germany (FZJ – 75 %) and France (LLB – 25 %)

- One of the first 3 instruments to be built at ESS

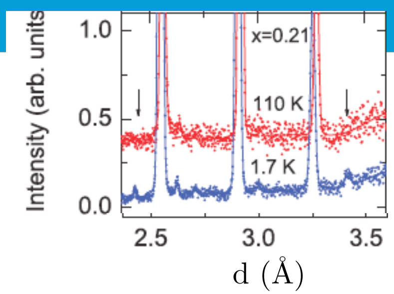
$$\lambda = 2d\sin\theta$$





DREAM Science Case

$Ba_{1-x}K_xFe_2As_2$



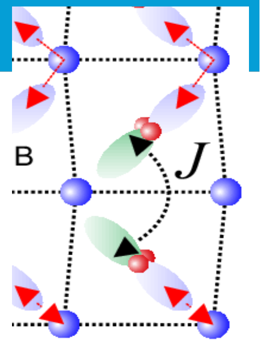
weak moments
T-x-H-p
phase diagrams

Magnetism

Superconductivity
Multiferroics

Functionality

orbital ordering
charge ordering
distortion
magnetic exchange

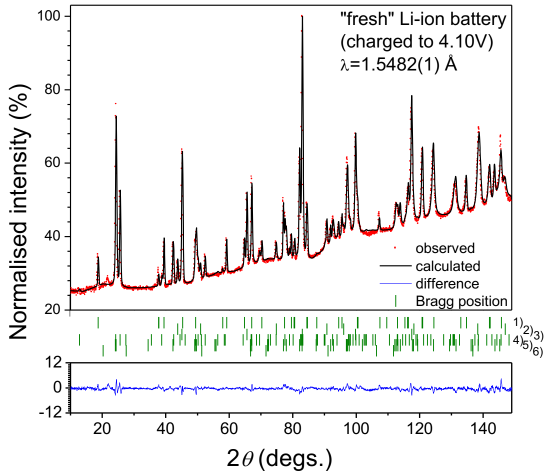


new compounds
TM - 0 – F – N -

Energy research

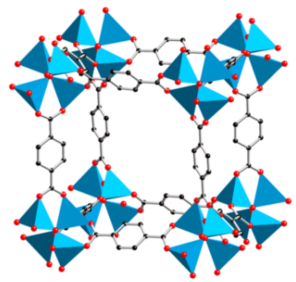
Batteries Li, H

multiphase
in-operandi
real-time

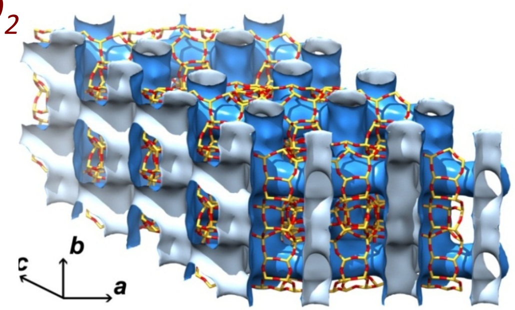


MOFS

metal-organic
framework structures



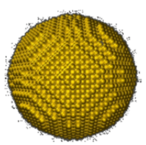
molecular sieves CO_2
H_2 *- storage*
catalysts

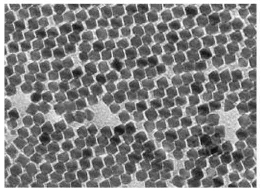


Large unit cells materials

Thermoelectrics
structure + ADPs

Nanostructures





magnetic nanoparticles
core-shell structures

=> small samples

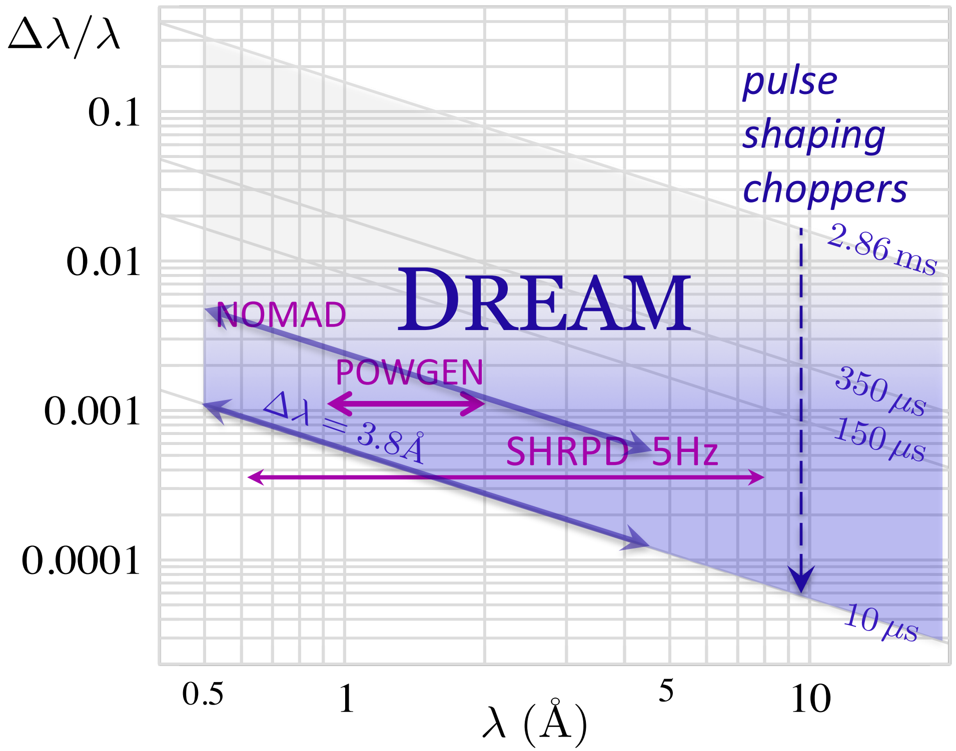
Resolution

Back scattering

flexible Pulse shaping

=> from low resolution to even world **best resolution**

symmetric pulse shape



note: *Δλ* ~ const at short-pulse spallation sources

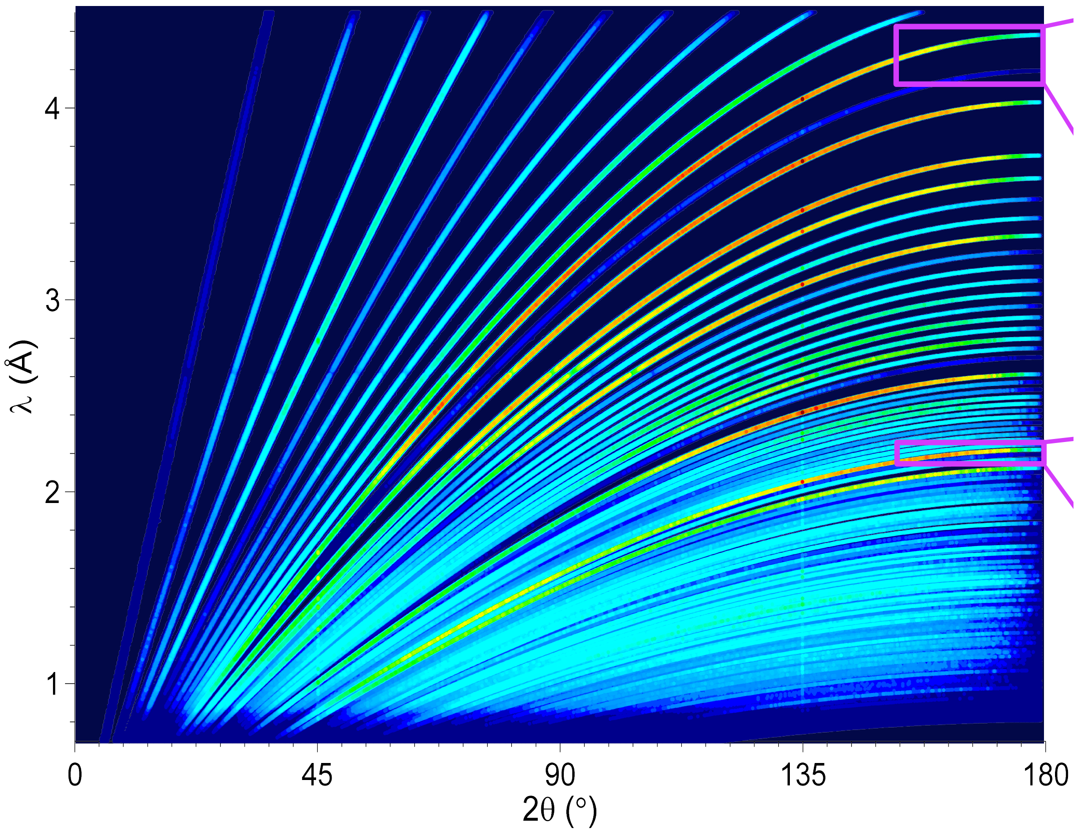
DREAM can combine the virtues of NOMAD, POWGEN and SHRPD

DREAM - performance

full instrument MC simulations - VITESS

$Na_2Ca_3Al_2F_{14}$ cubic ($I2_13$) a = 10.257(1) Å   0.4 cm^3

Diffraction Resolved by Energy and Angle Measurements



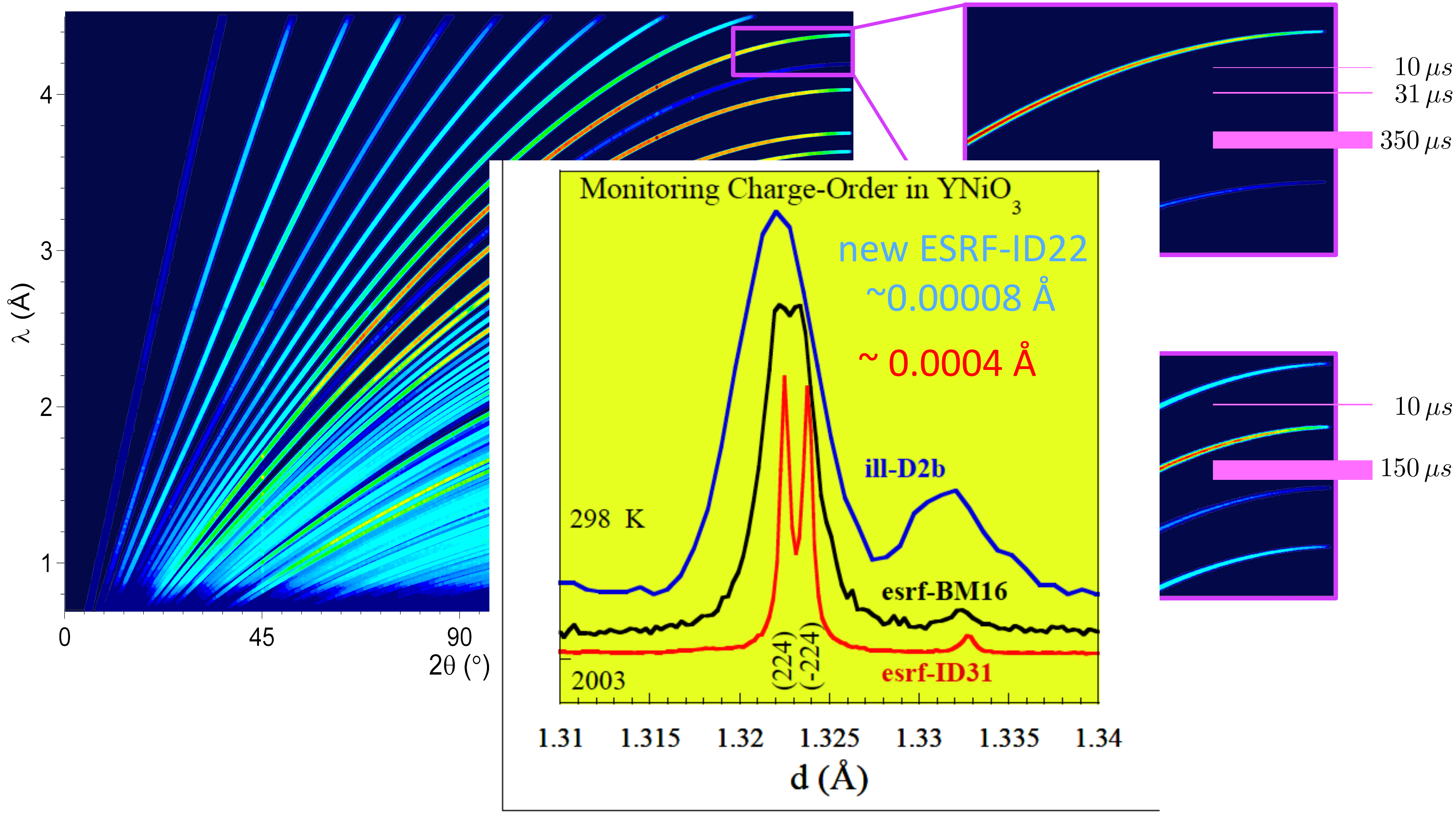
DREAM - performance

full instrument MC simulations - VITESS

$Na_2Ca_3Al_2F_{14}$ cubic ($I2_13$) a = 10.257(1) Å   0.4 cm^3

high resolution in backscattering

Δd = 0.00028 Å



DREAM - performance

full instrument MC simulations - VITESS

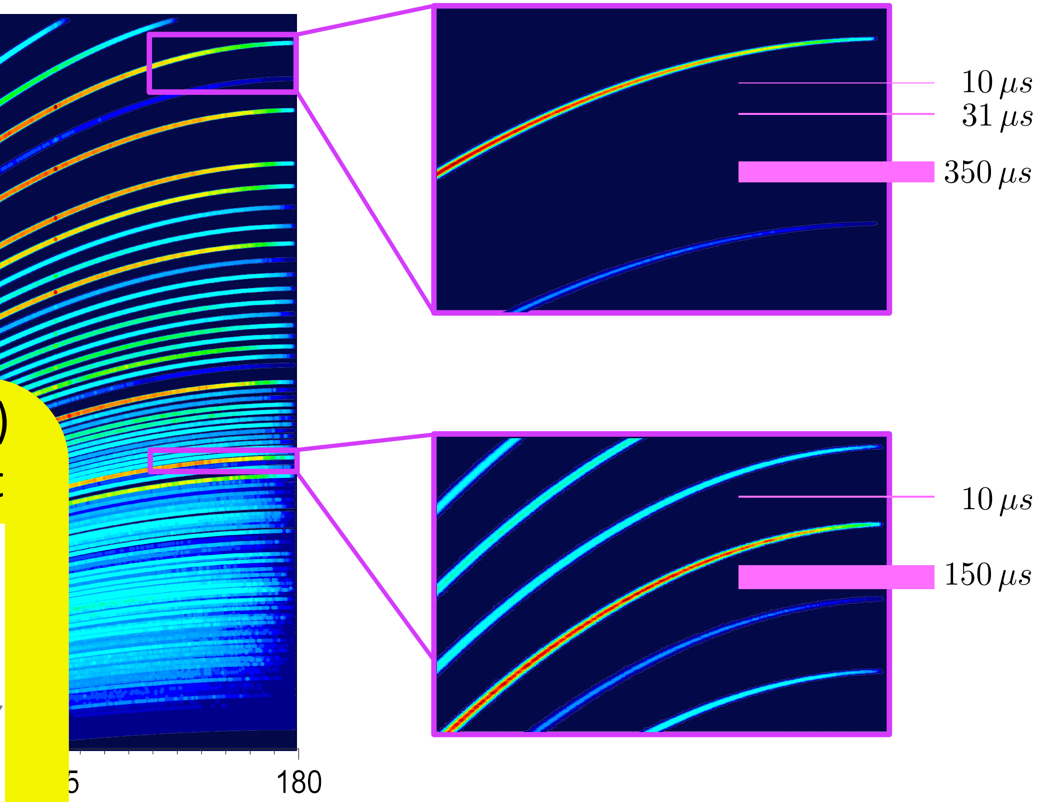
$Na_2Ca_3Al_2F_{14}$ cubic ($I2_13$) a = 10.257(1) Å   0.4 cm^3

high intensity

- in a single strong peak up to **500000 n/pulse** (14 Hz)
- **ms resolution** in phase studies of chemical reactions

high resolution in backscattering

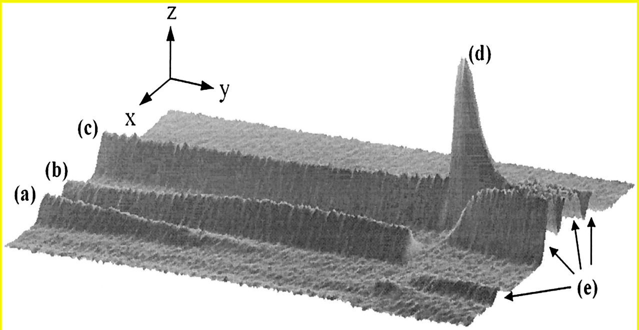
Δ*d* = 0.00028 Å



time resolution 0.9 s Kisi&Riley (2002)

300 ms +0.8 ms readout

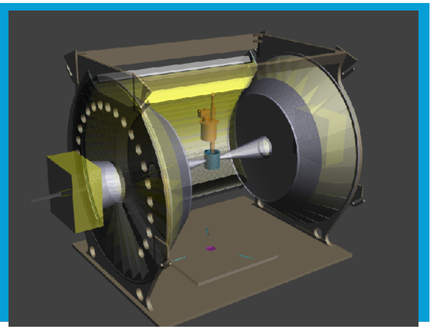
D20



How to deal best with the varying resolution function?

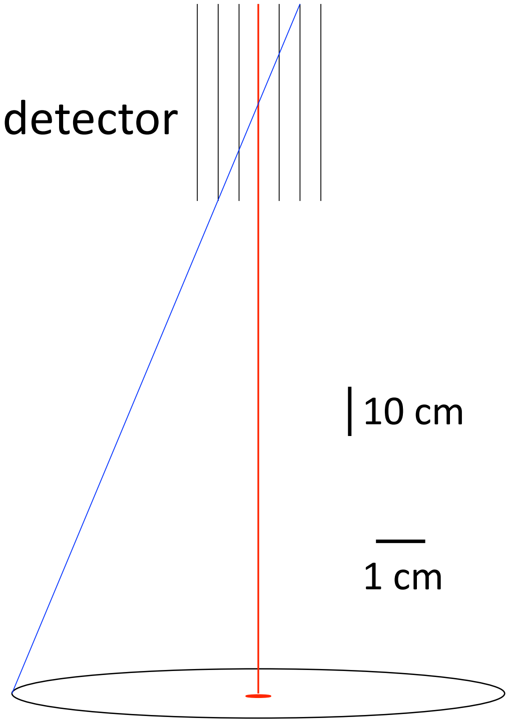
Using 2D and 3D detector information

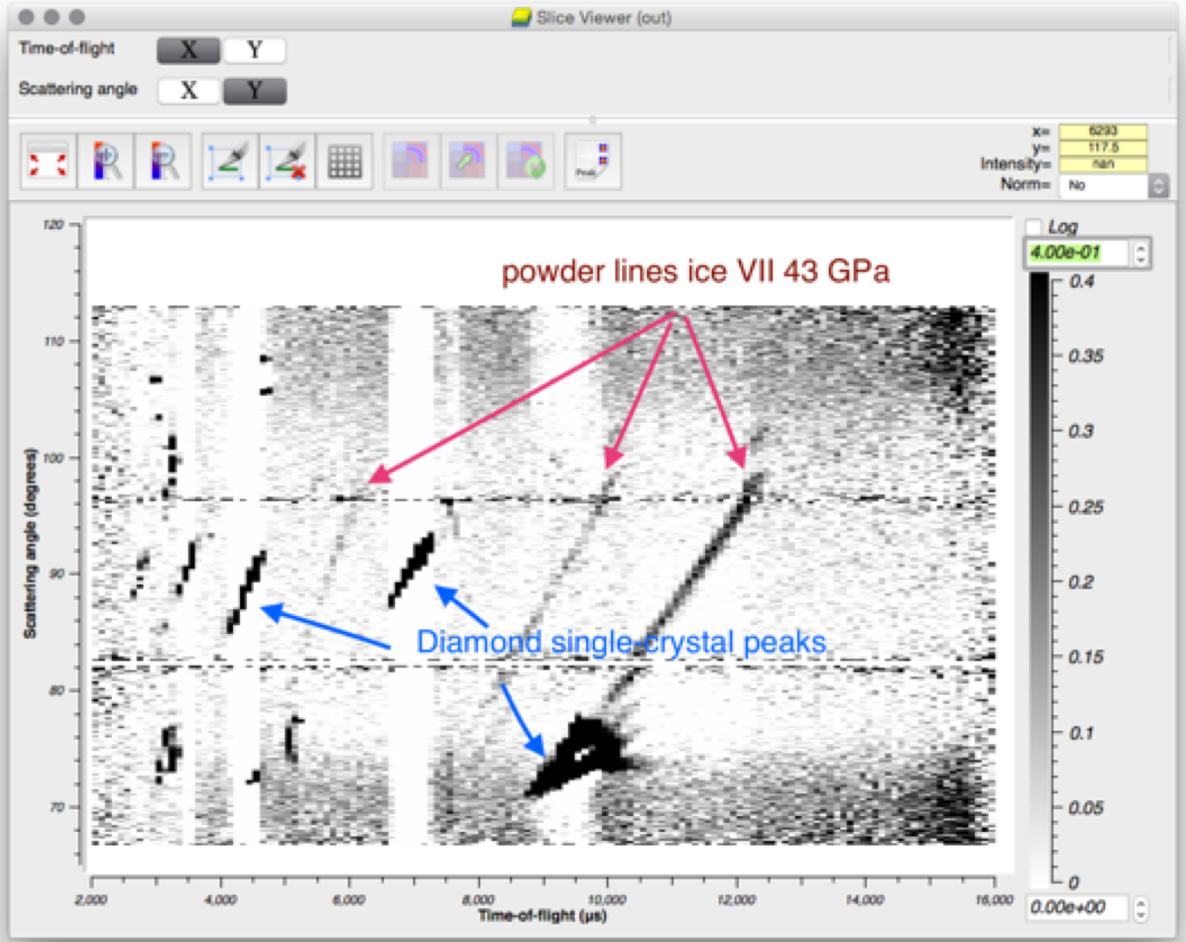
High pressure – very small samples



a great help for identifying **weak signals** in large background

intrinsic collimation
& back-tracing



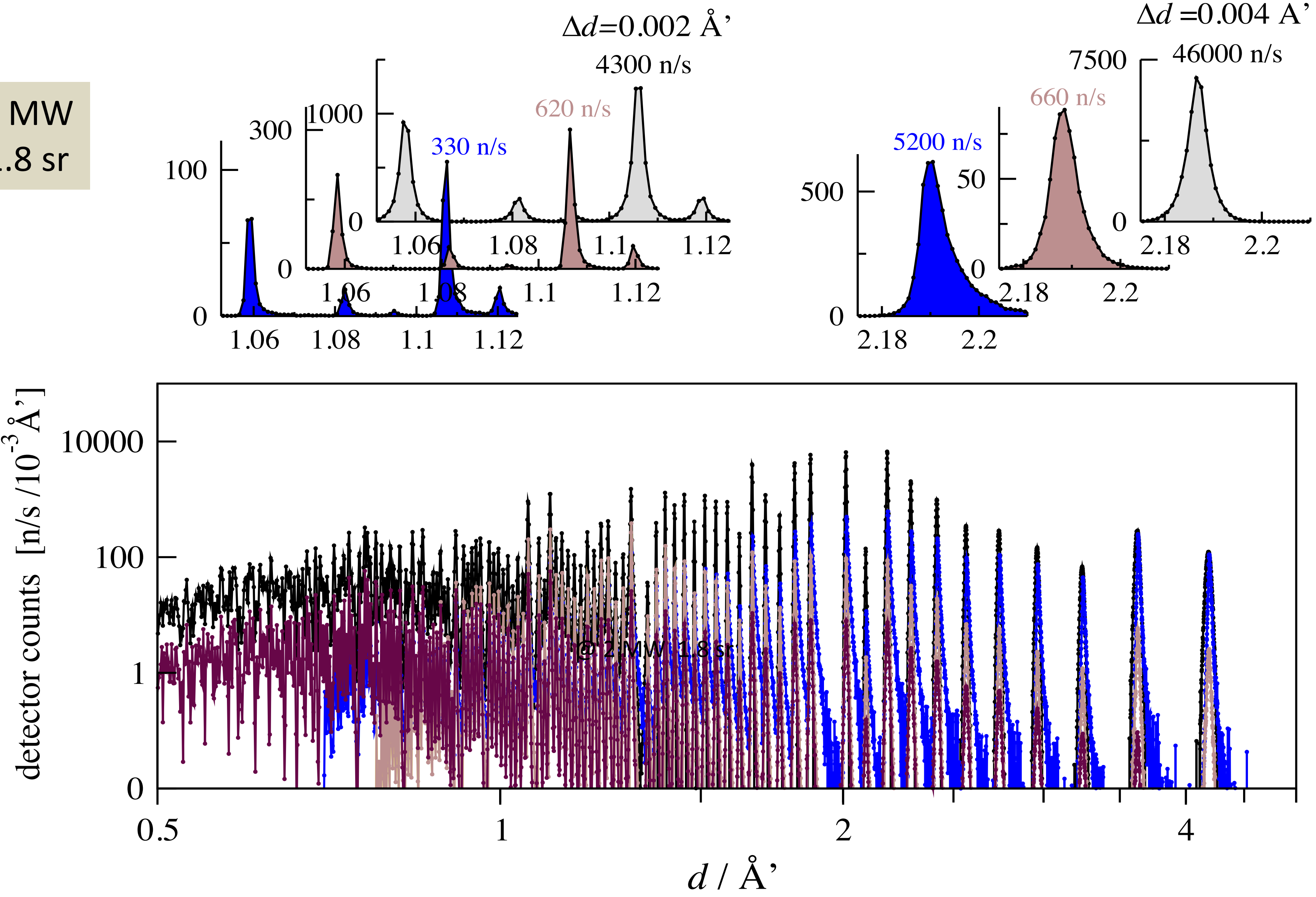


Courtesy of Malcolm Guthrie

Benchmarking full instrument MC simulations VITESS

reference $Na_2Ca_3Al_2F_{14}$ cubic ($I2_13$) a = 10.257(1) Å

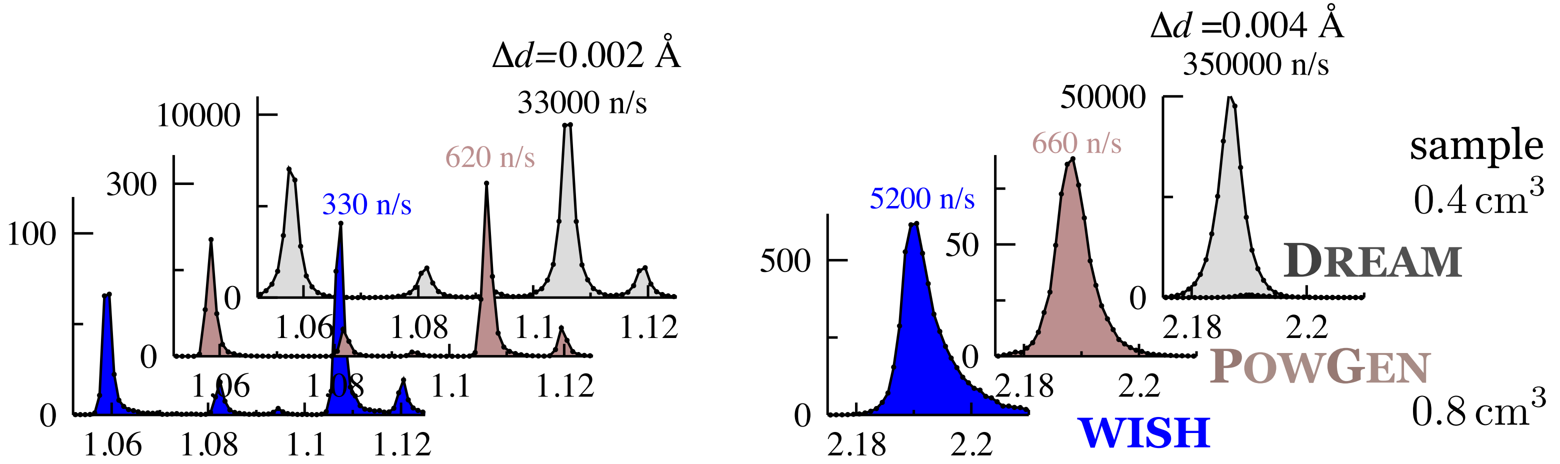
2 MW
1.8 sr

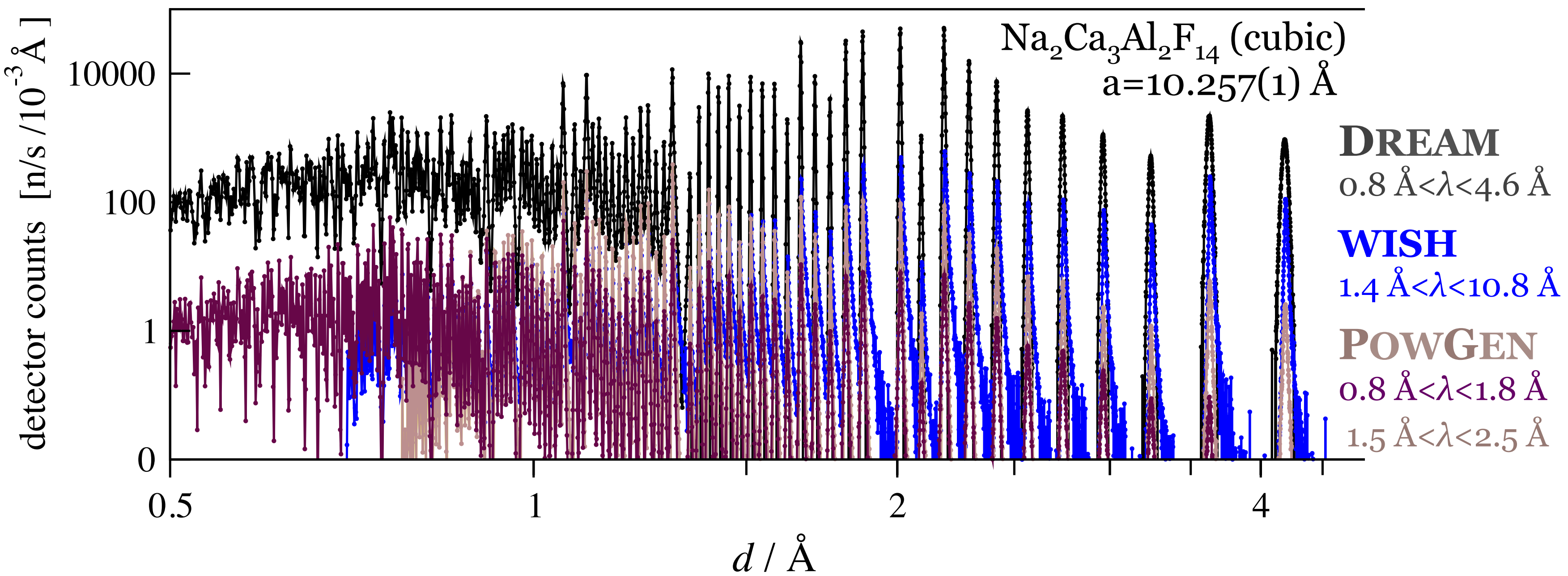


Benchmarking full instrument MC simulations VITESS

reference $Na_2Ca_3Al_2F_{14}$ cubic (I2$_1$3) a = 10.257(1) Å

5 MW
5.2 sr

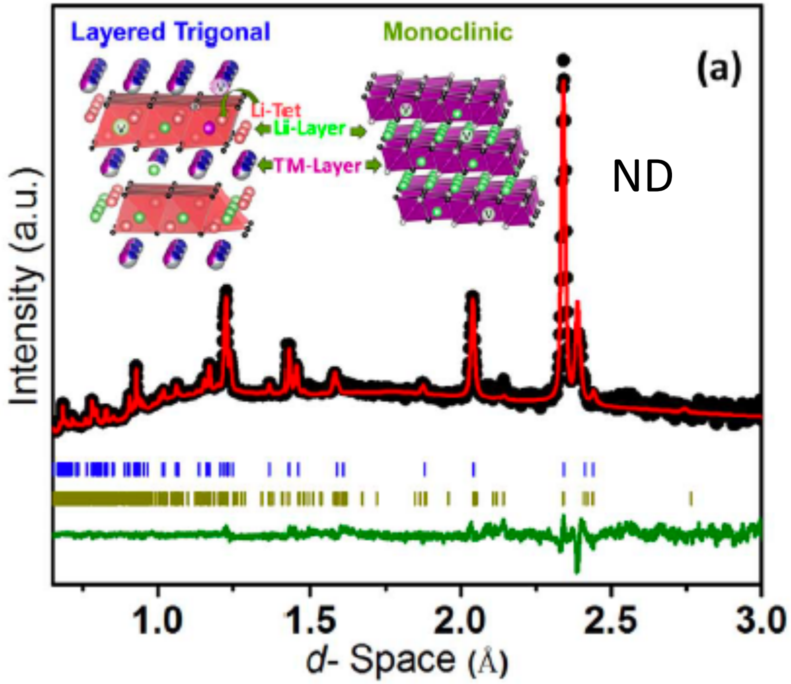




from Mikhail Feygenson DREAM

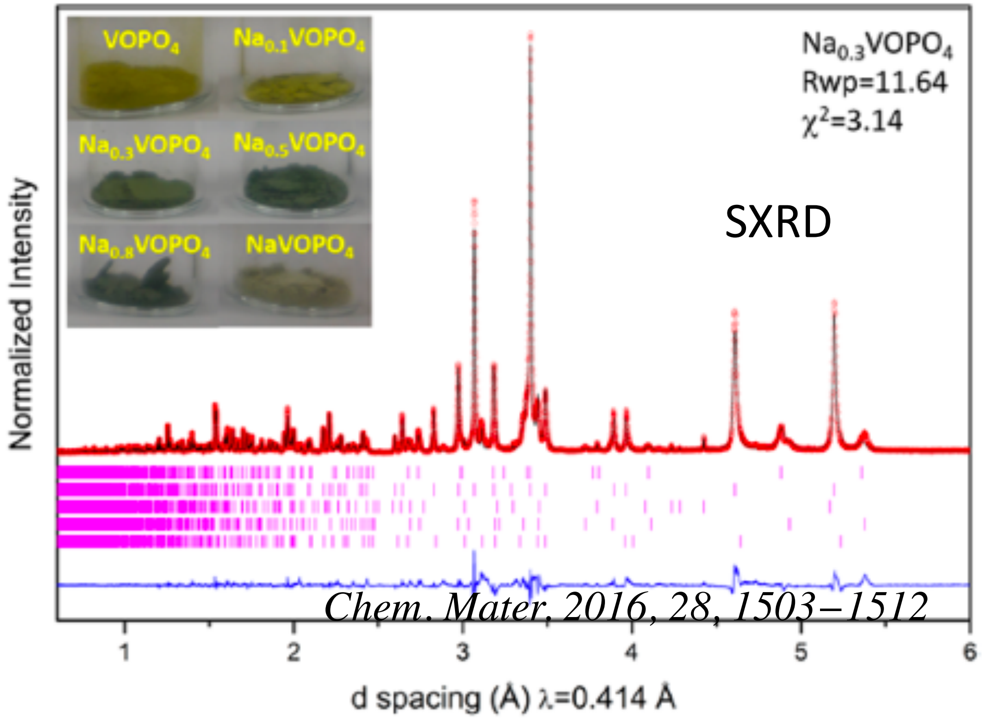
High Resolution relevant for energy materials

$Li_{1.2}Mn_{0.55}Ni_{0.15}Co_{0.1}O_2$ cathode Li-ion



Nano Energy 36 (2017) 76–84

β-$NaVOPO_4$ cathode for sodium-ion batteries



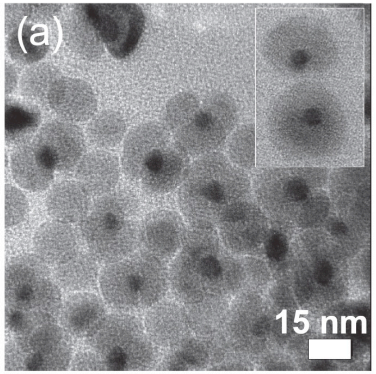
Chem. Mater. 2016, 28, 1503−1512

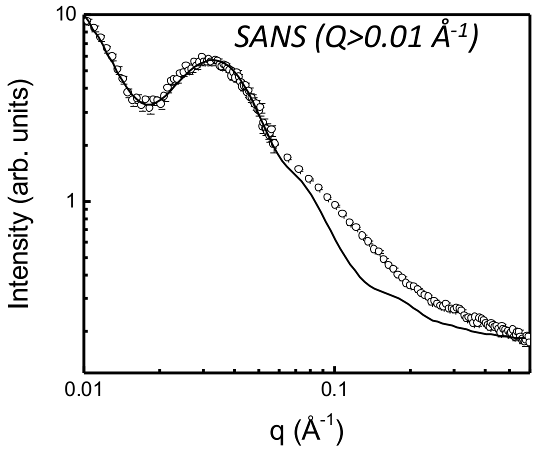
- Multi-phase materials with many overlapping peaks
- Energy materials often consist of amorphous phases which can not be refined by Rietveld method

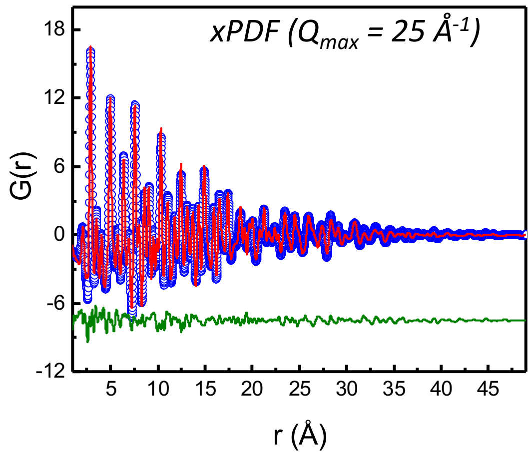
from Mikhail Feygenson DREAM

Nanostructures
Powder diffraction + PDF + nm-SANS

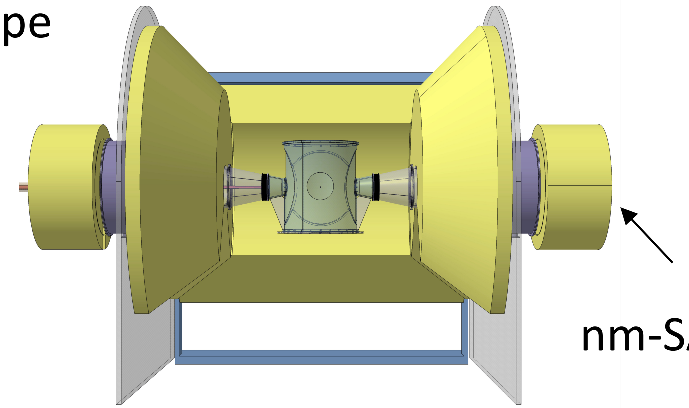
Enhanced catalysis in Fe_3O_4 – Au dumbbell nanoparticles *M. Feygenson et al, PRB (2015)*



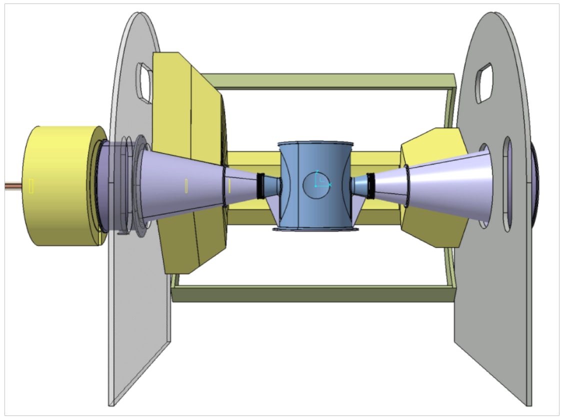




Full scope



nm-SANS



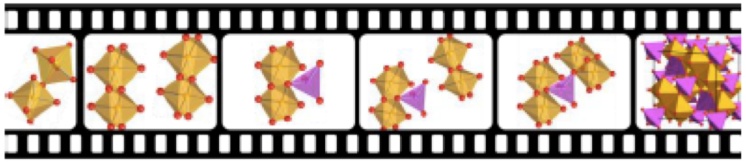
Initially

from HEIMDAL presentation at STAP 2017

HEIMDAL science case

EUROPEAN SPALLATION SOURCE

Next Generation Powder Diffractometer for for *In-Situ/In-Operandi Studies*



Science cases:

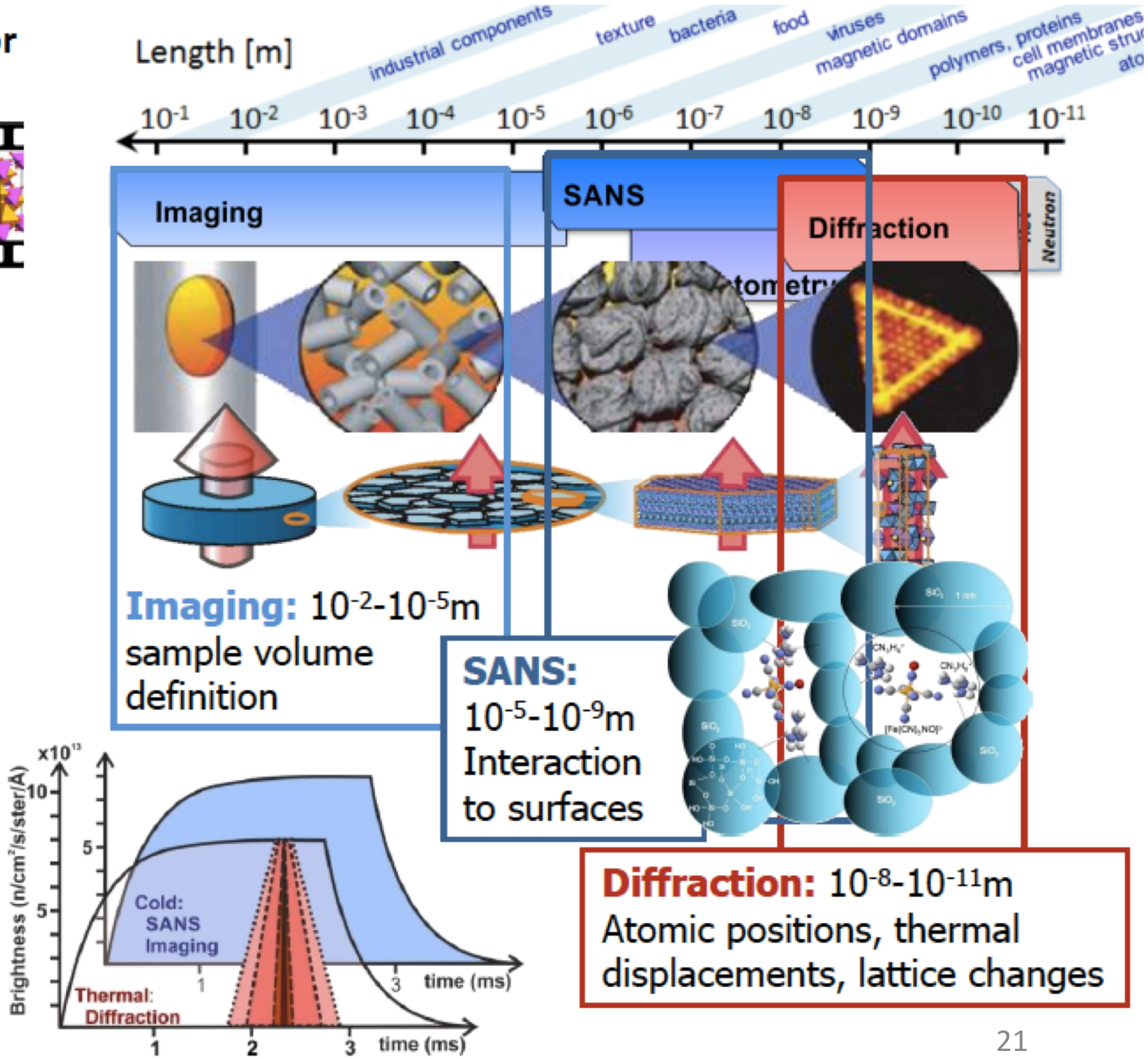
- Energy materials, catalysts, cement
- Hierarchical systems, biominerals
- Structure of functional materials
- Crystallization/growth
- Magnetic phases
- Nanomaterials

Virtues:

- High flux, low background
- Simple data treatment, ease of use

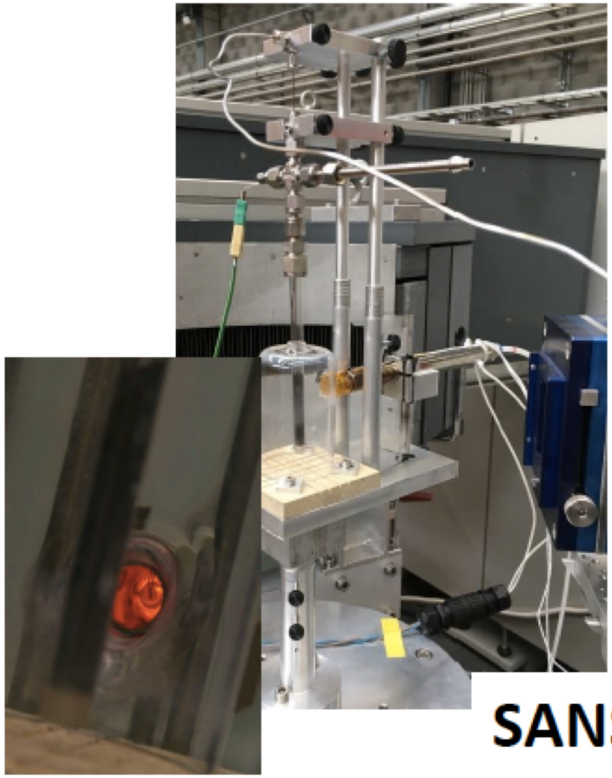
Flexible flux/resolution:

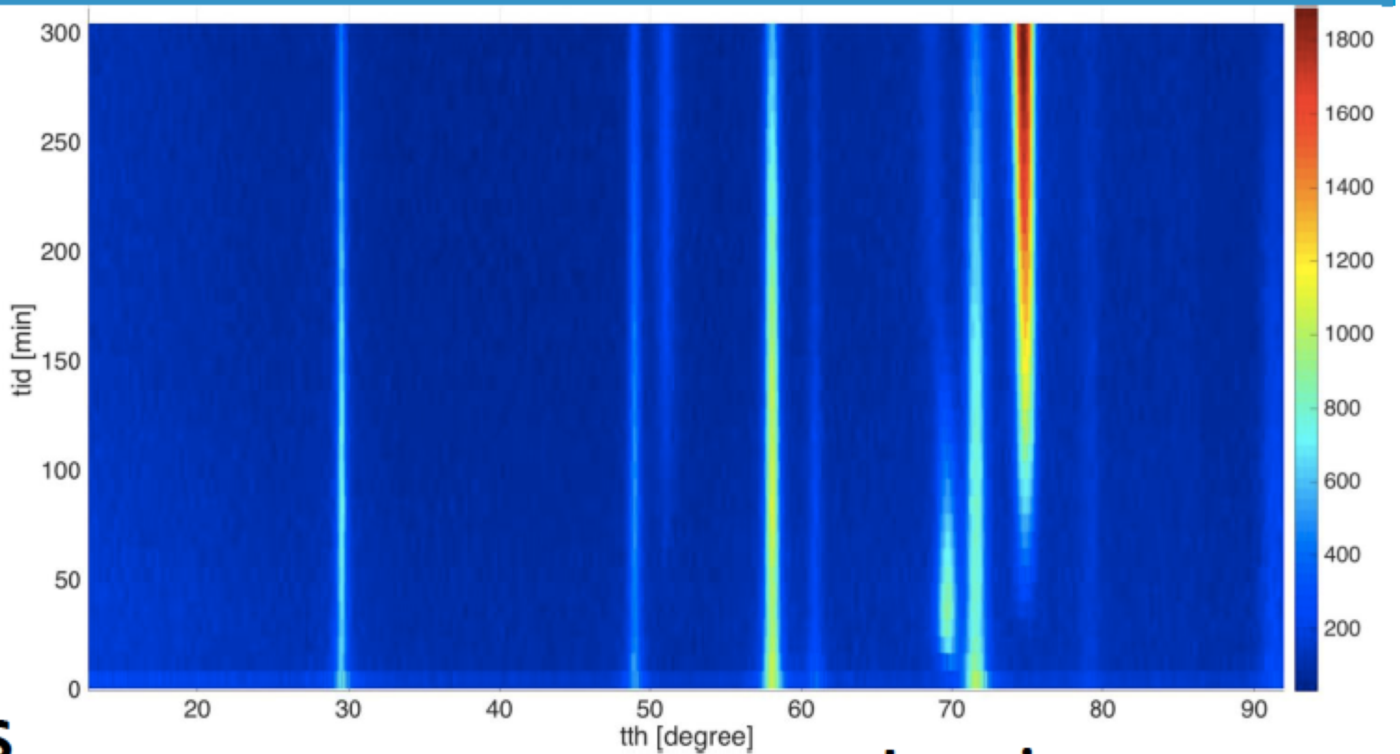
Easily adjustable during the experiment



from HEIMDAL presentation at STAP 2017

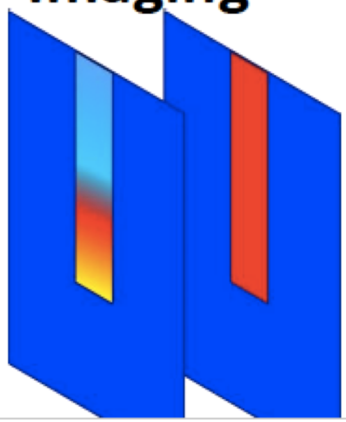
Reduction experiments





SANS

Imaging



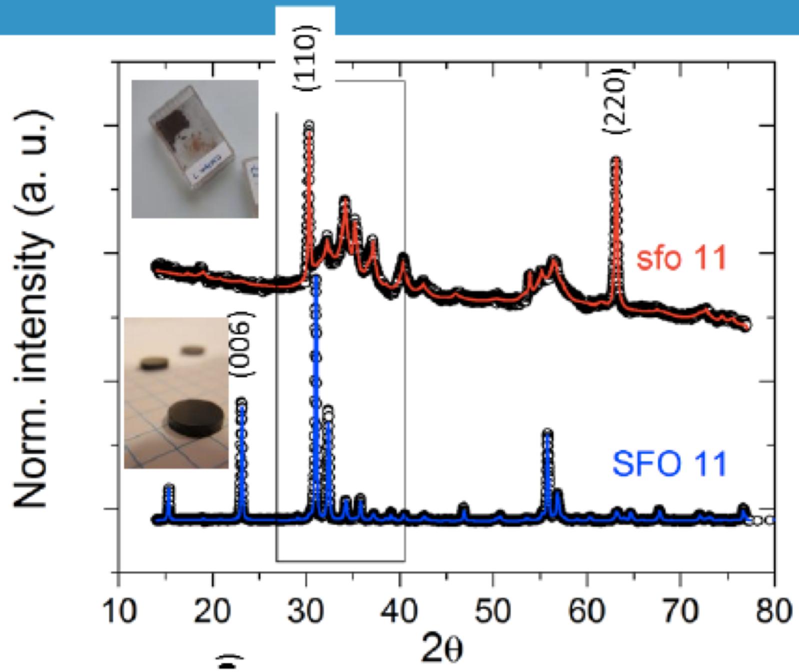
Follow chemical process:

α-Fe_2O_3 -> Fe_3O_4 **Very fast reaction –possibly topotatic**

Jakob Ahlburg, Cecilia Granados, Henrik Lyder Andersen, Pelle Garbus, Emmanuel, Lukas Keller

from HEIMDAL presentation at STAP 2017

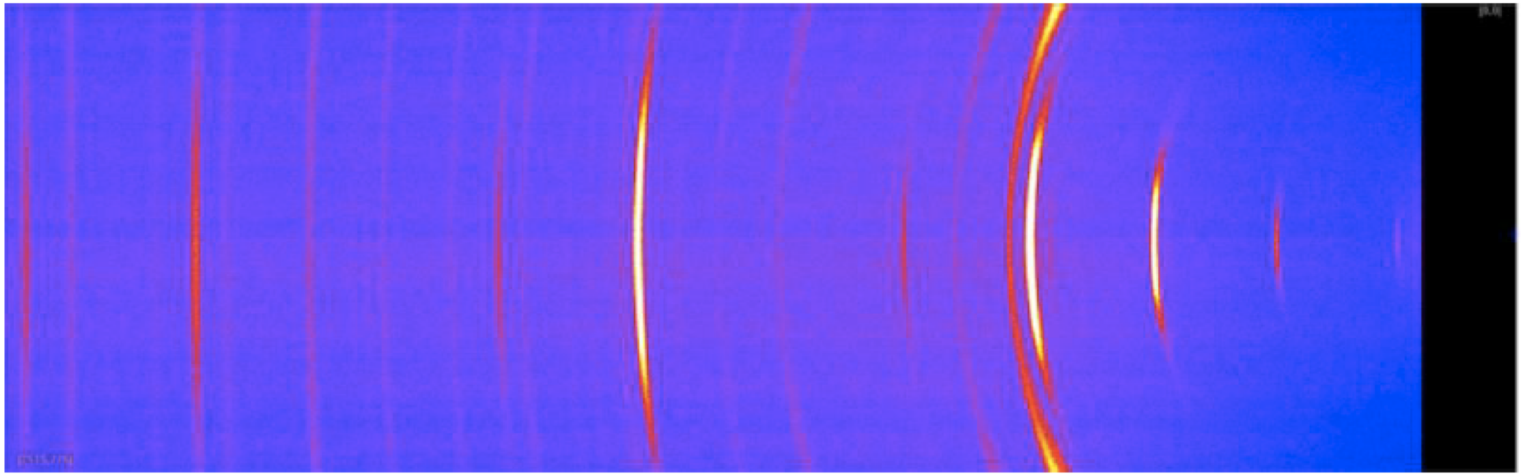
Texture



HEIMDAL provides:

- Atomic structure
- Phase composition
- Texture
- Particle morphology – full scope

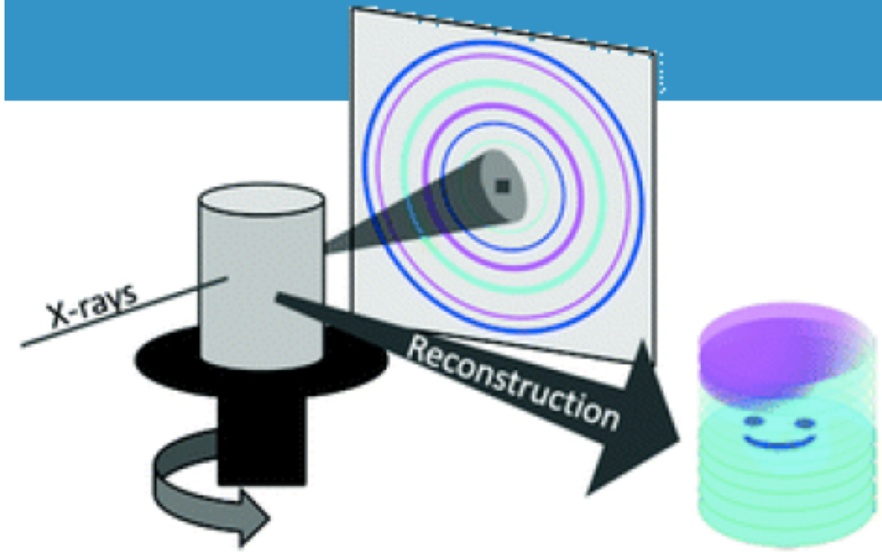
- **During compaction at elevated:**
  - Temperature (1000 °C)
  - Pressure (0.1 GPa)



x 7.50 in

from HEIMDAL presentation at STAP 2017

Diffraction tomography



Three-dimensional distribution of polymorphs and magnesium in a calcified underwater attachment system by diffraction tomography

Hanna Leemreize[1], Jonathan D. Almer[2], Stuart R. Stock[3] and Henrik Birkedal[1]

(*a*)