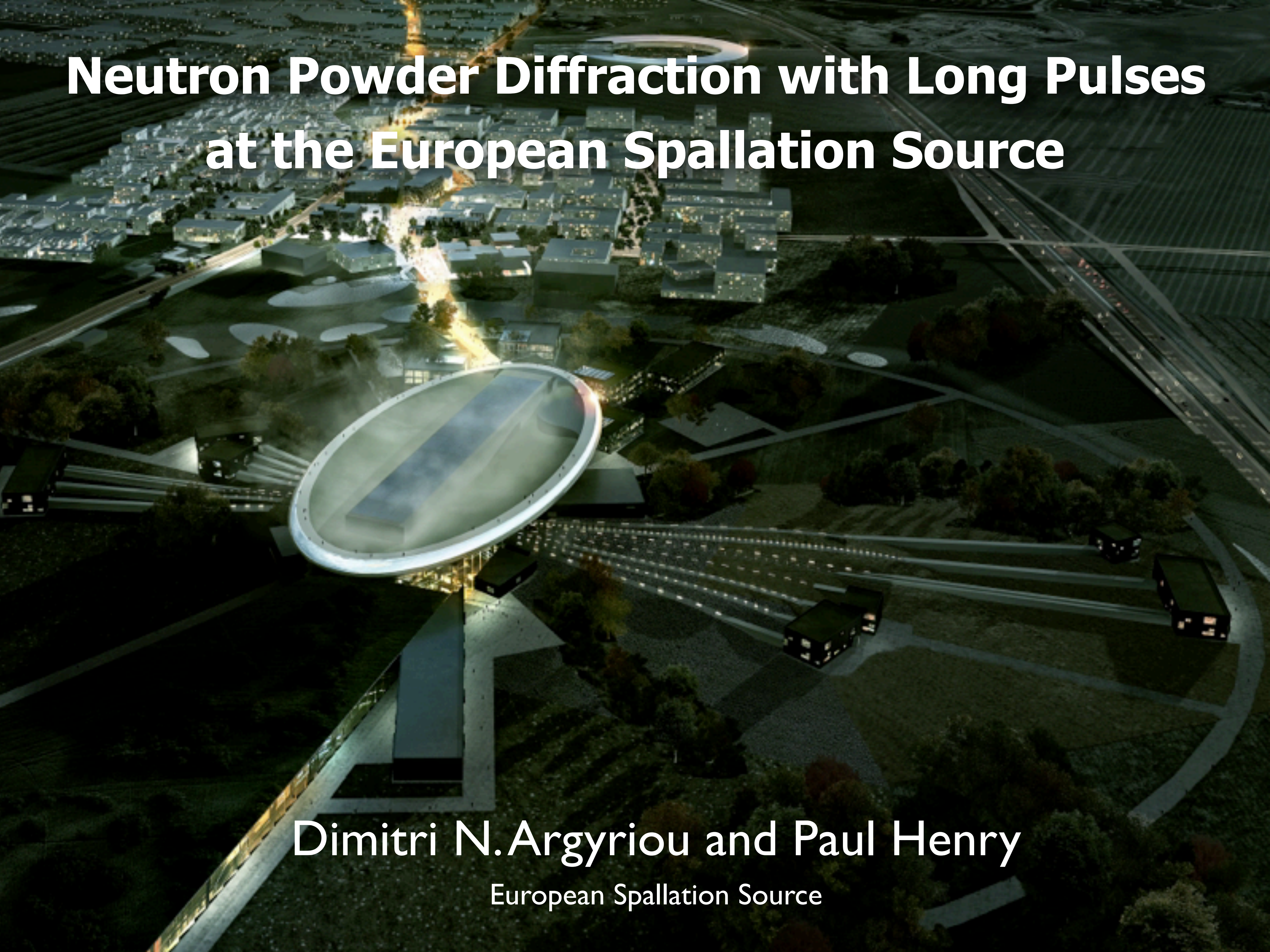
Neutron Powder Diffraction with Long Pulses at the European Spallation Source

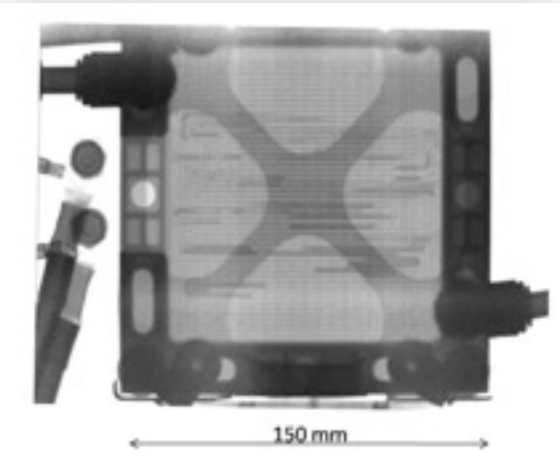


Dimitri N. Argyriou and Paul Henry

European Spallation Source

Neutrons are special

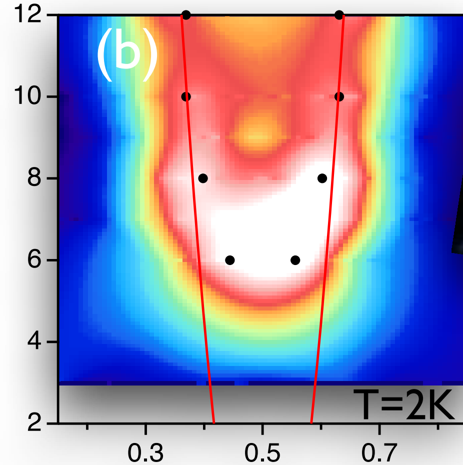
Charge neutral
Deeply penetrating



Li motion in fuel cells

Help build electric cars

S=1/2 spin
probe directly magnetism

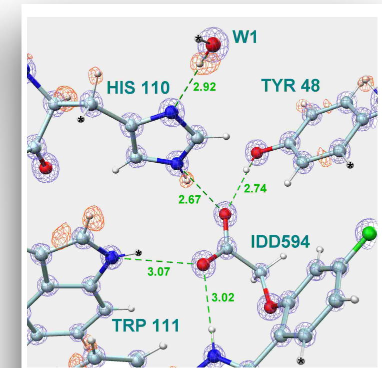


Test AdS/CFT correspondence

Solve the puzzle of High-Tc superconductivity

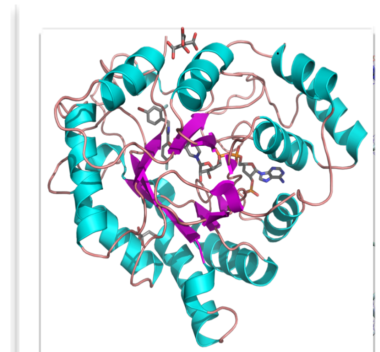
Efficient high speed trains

Nuclear scattering
Sensitive to light elements and isotopes



Active-site region of hAR

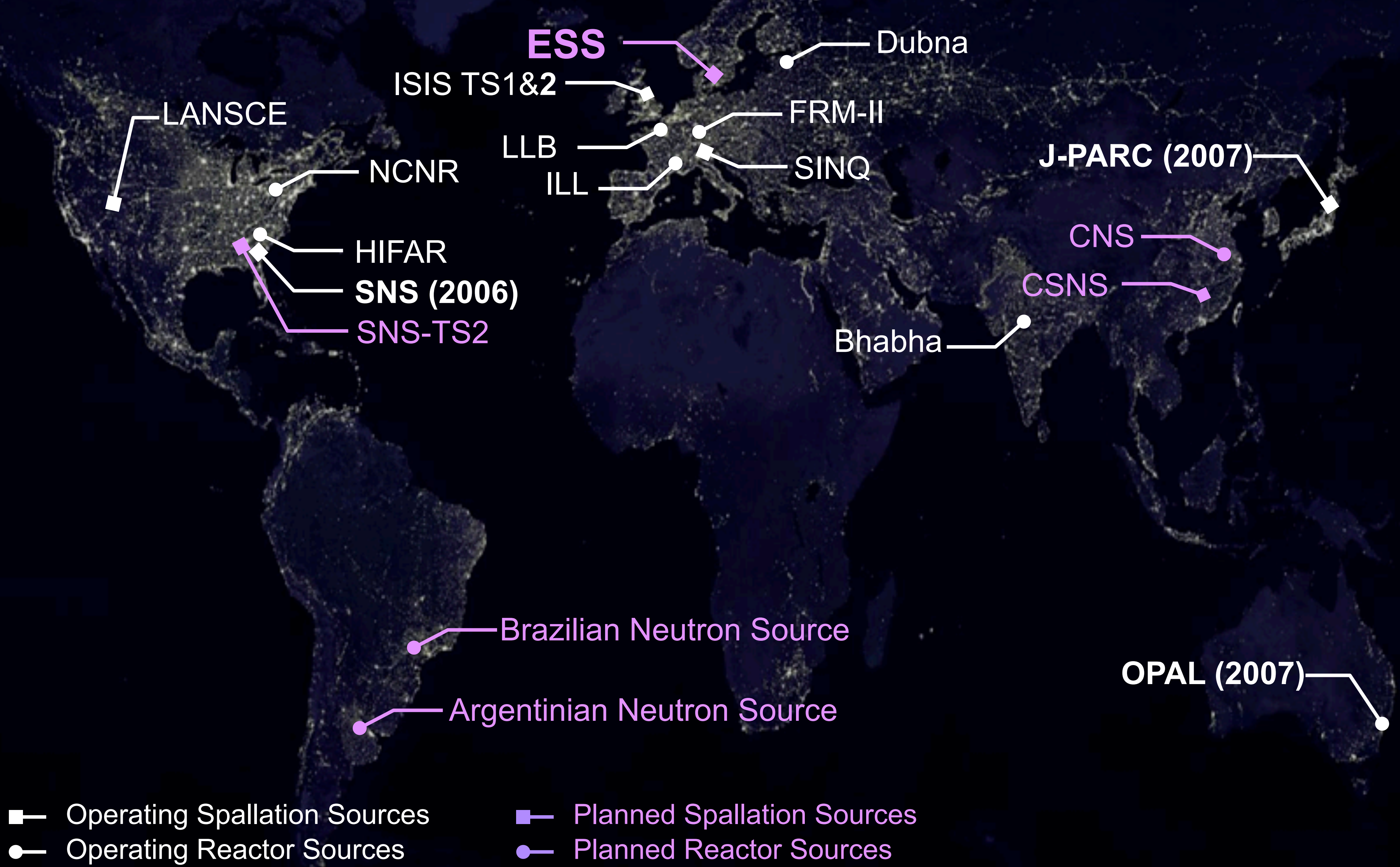
Actives sites in proteins



Tertiary structure of hAR

Better drugs

Major National and International Neutron Sources



First neutron instruments

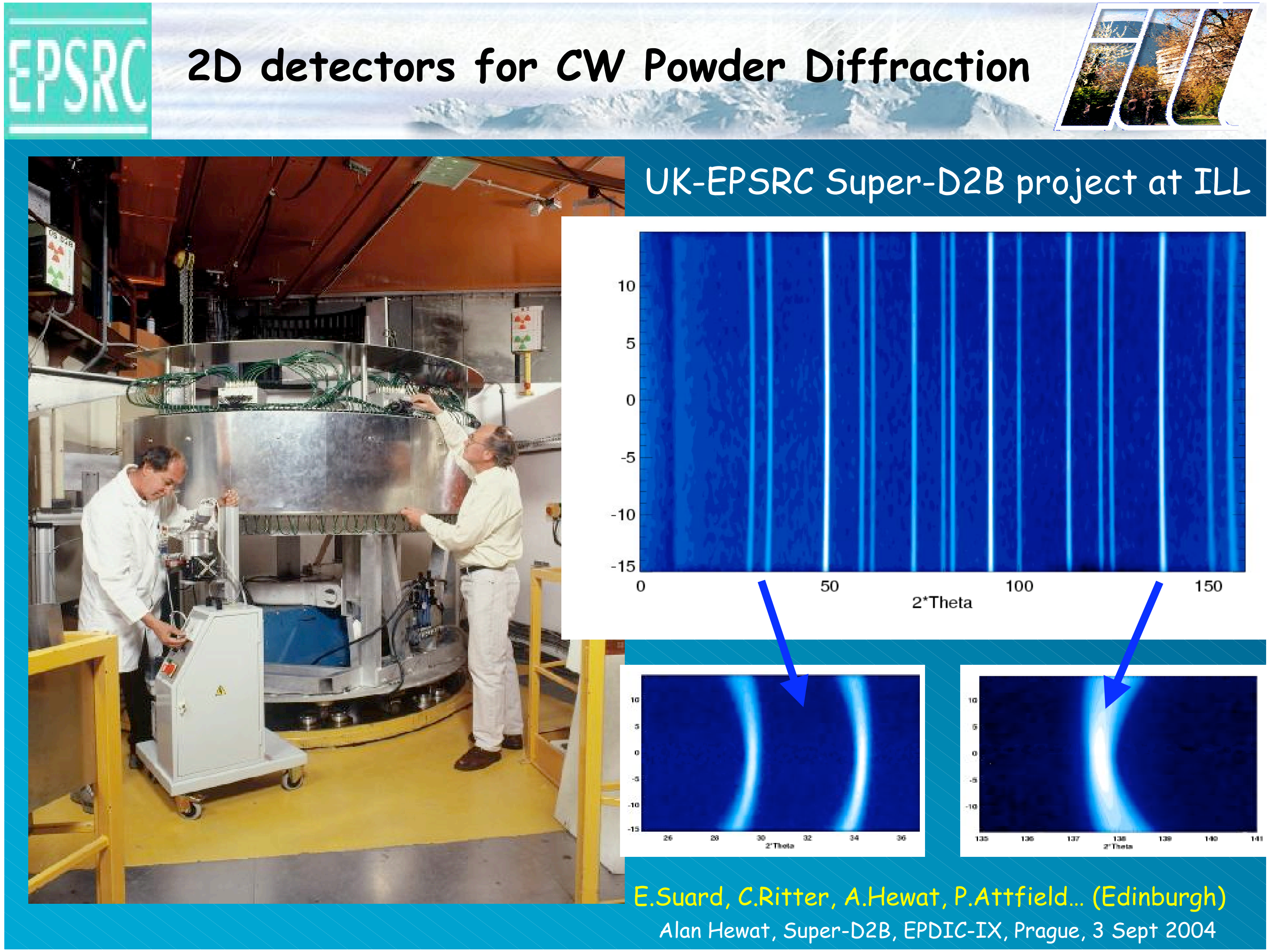
1950s

D1B (ILL)

D1A / D2B (ILL)

1970s

The Next Generation at ILL

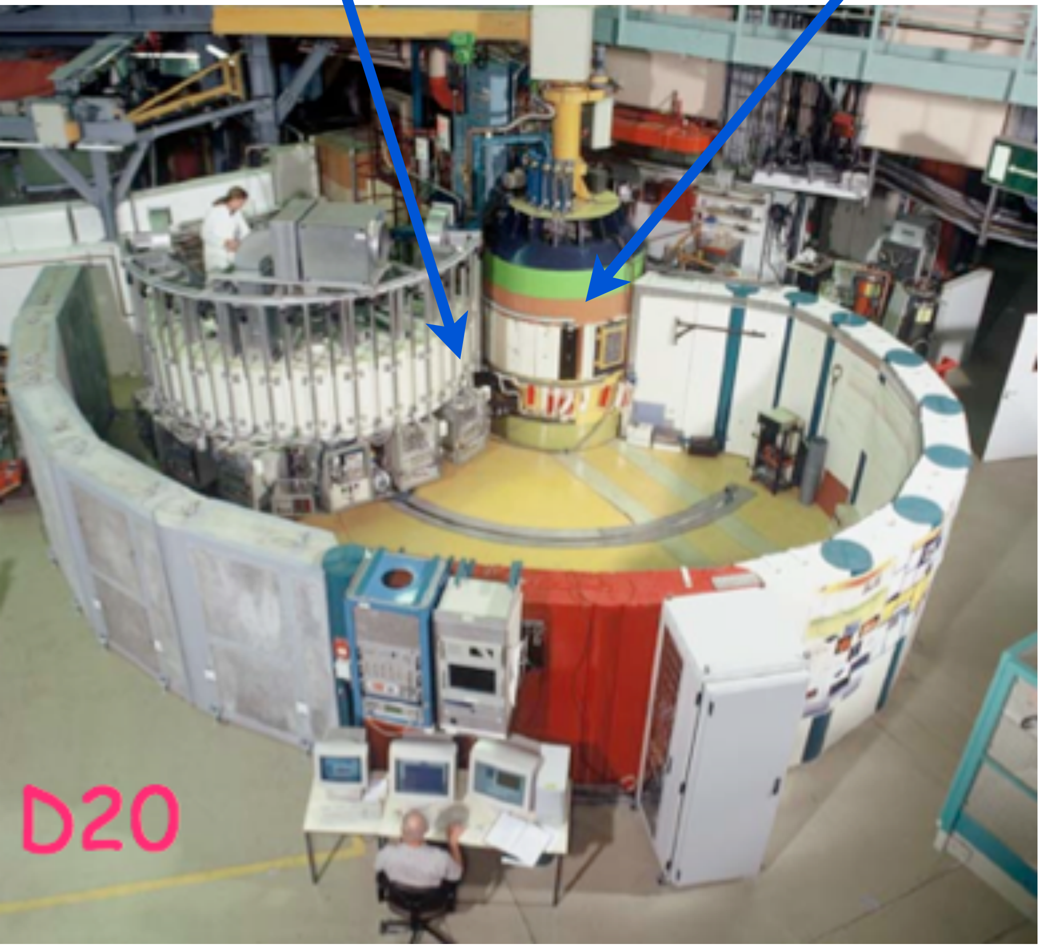


- 128 Detectors
- Relative long detectors
- Need to integrate over Debye Scherer Rings

D20 at ILL

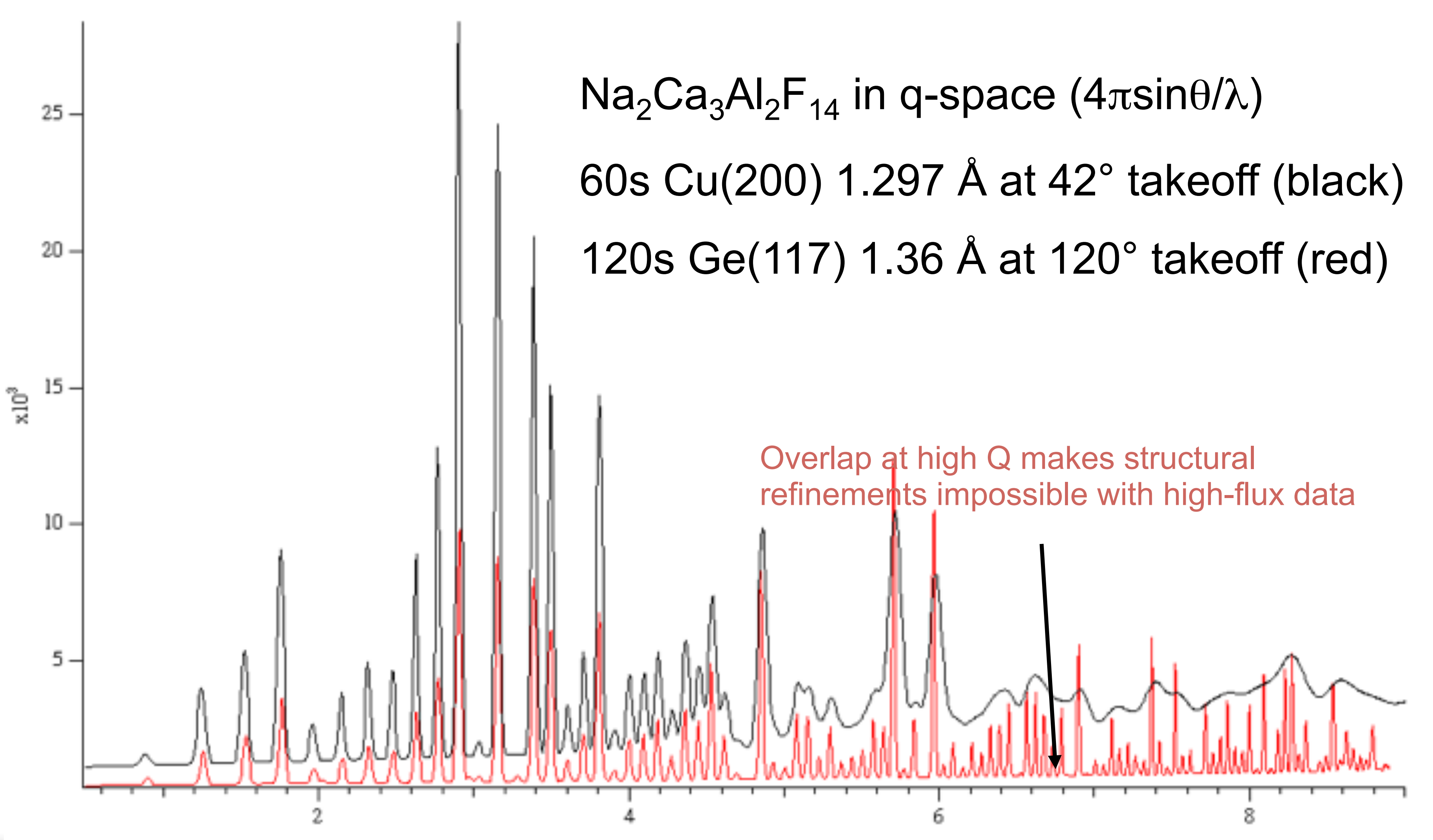
Continuous 120 deg detector

Monochromator Drum

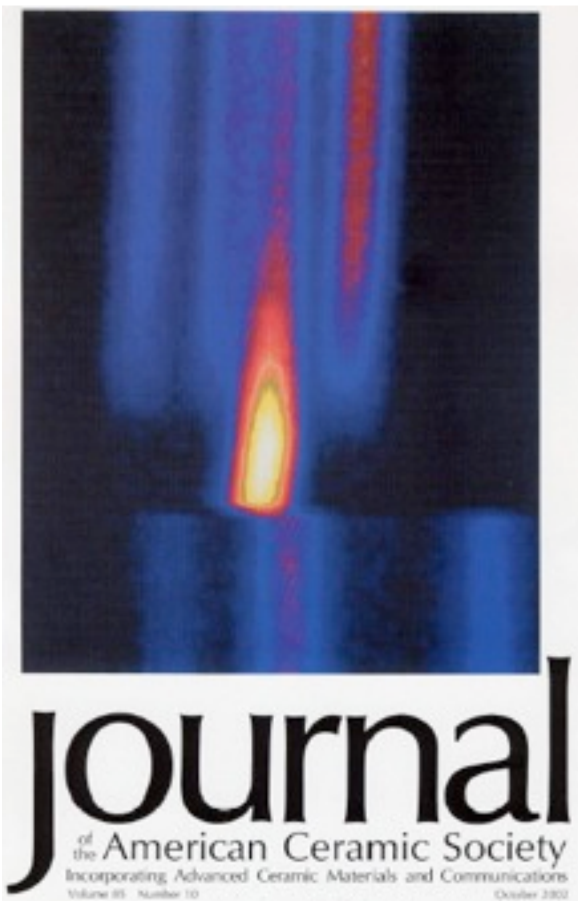


- Large monochromator viewing a large beam cross section
- Various λ available
- Large continuous coverage micro-strip detector
  - Major technological challenge
- Relative ease in changing take-off angle from low to high resolution.
- Flexible set-up in special environments
  - Pressure cells
  - Reaction cells
  - Furnaces ...

Flexible resolution on D20



Fast data collection / processes

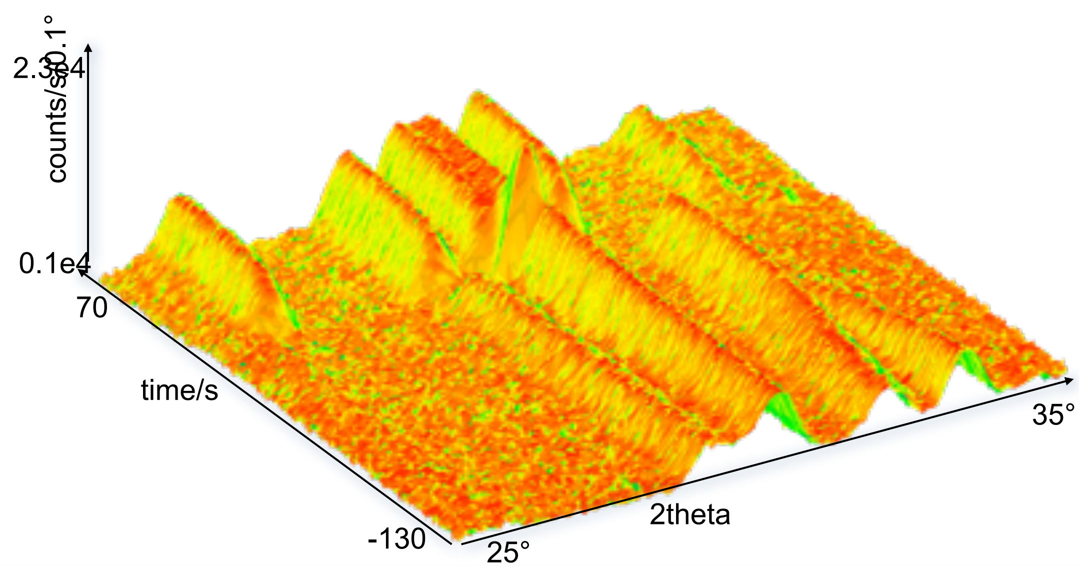


Ti_3SiC_2 made by hot isostatic pressing is expensive

In-situ investigation of thermal explosion synthesis (TES)

Initiate by heating from 850-1050 °C at 100 °C/min

Acquisition time 500 ms (300 ms deadtime)



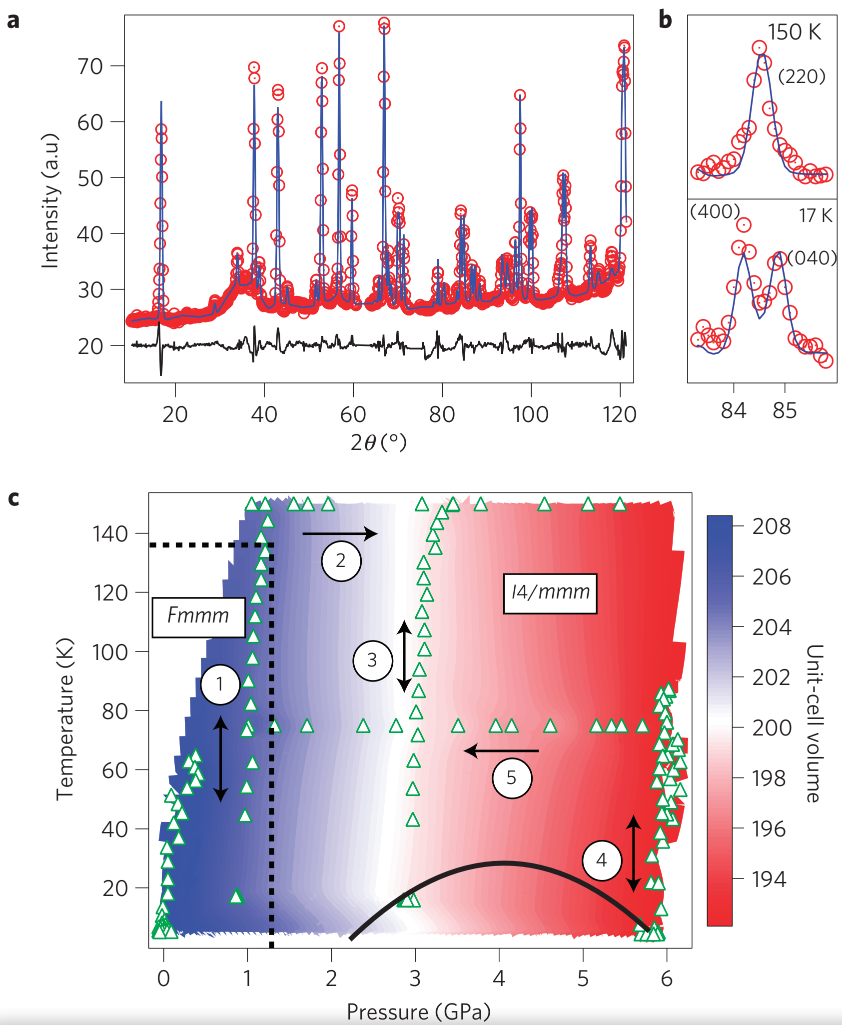
D.P.Riley et al. J. Am. Ceramic. Soc. 2002, 2417-2424.

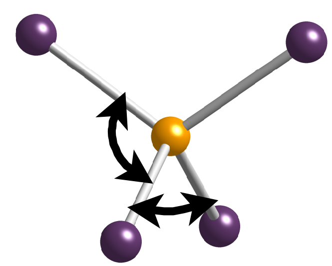
nature materials
LETTERS
PUBLISHED ONLINE: 3 MAY 2009 | DOI: 10.1038/NMAT2443

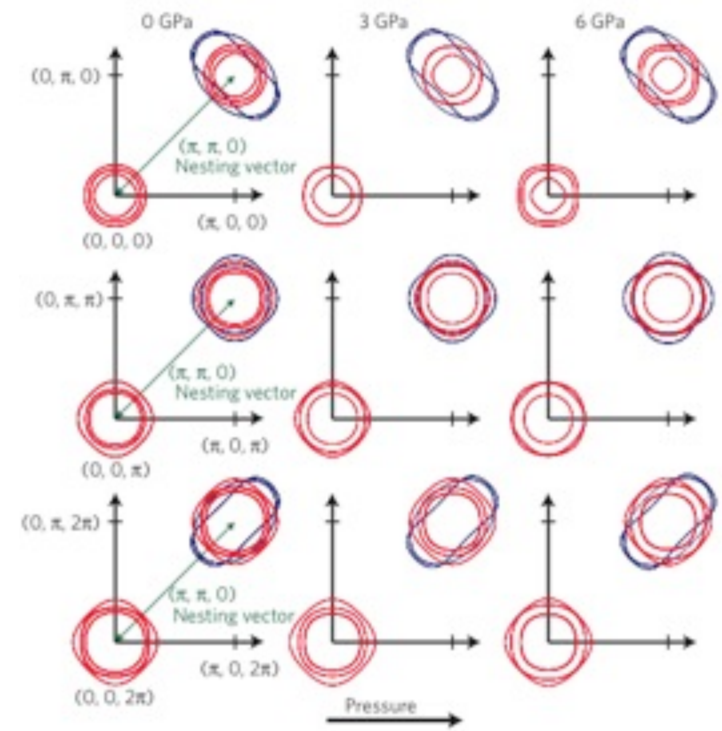
Powder Diffraction and Modeling

Similarities between structural distortions under pressure and chemical doping in superconducting $BaFe_2As_2$

Simon A. J. Kimber[1]*, Andreas Kreyssig[2,3], Yu-Zhong Zhang[4], Harald O. Jeschke[4], Roser Valentí[4], Fabiano Yokaichiya[5], Estelle Colombier[2], Jiaqiang Yan[2], Thomas C. Hansen[5], Tapan Chatterji[6], Robert J. McQueeney[2,3], Paul C. Canfield[2,3], Alan I. Goldman[2,3] and Dimitri N. Argyriou[1]*

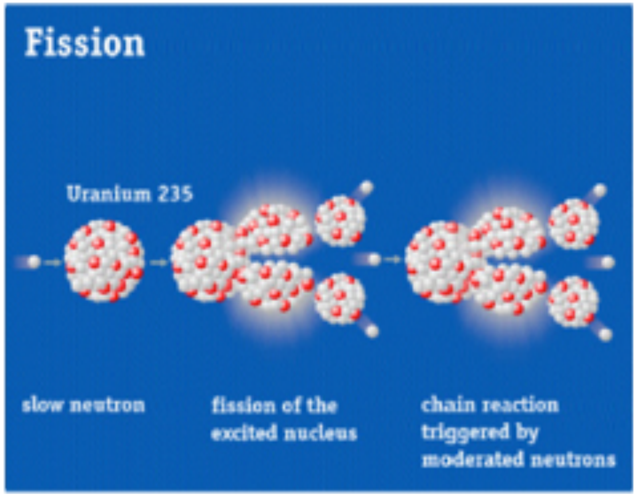






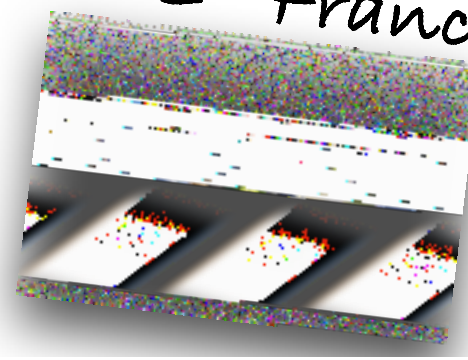
Experiment band-structures showing that the Fermi-surface nesting destabilizes with pressure (like with doping x) to allow superconductivity to emerge.

Two ways to produce Intense Neutron Beams Safely



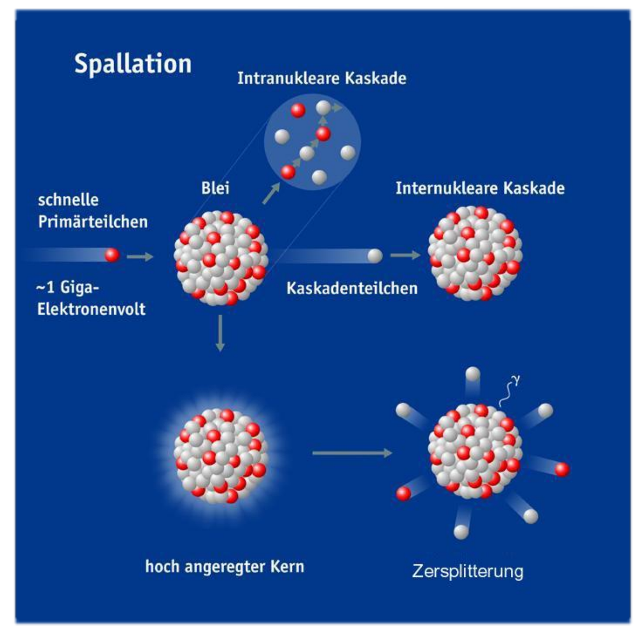
Fission:One neutron in, three neutrons out
How: Use a nuclear reactors

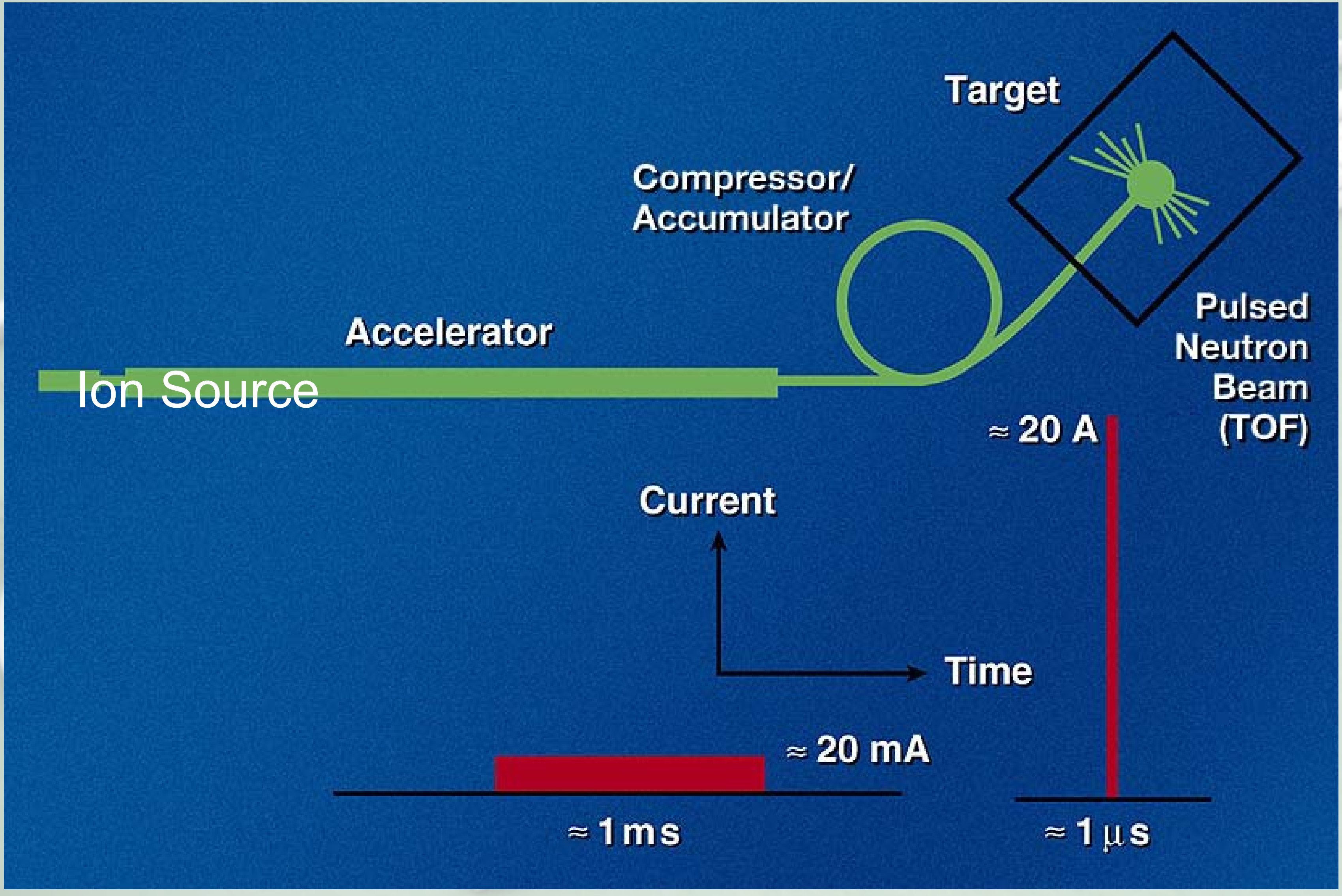
ILL - France



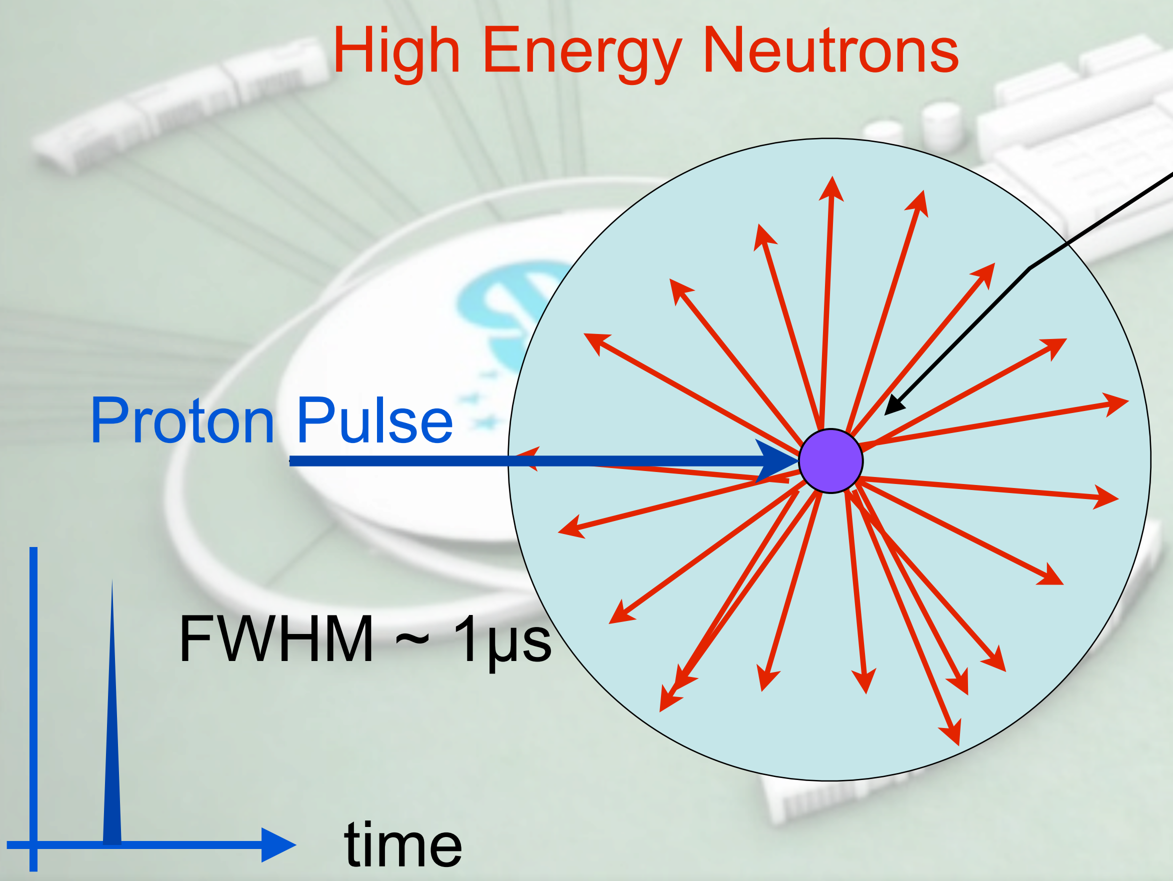
Spallation: Up to 30 neutrons per proton !
How: Use an accelerator to propel a proton onto on a tungsten element target

ESS-Sweden

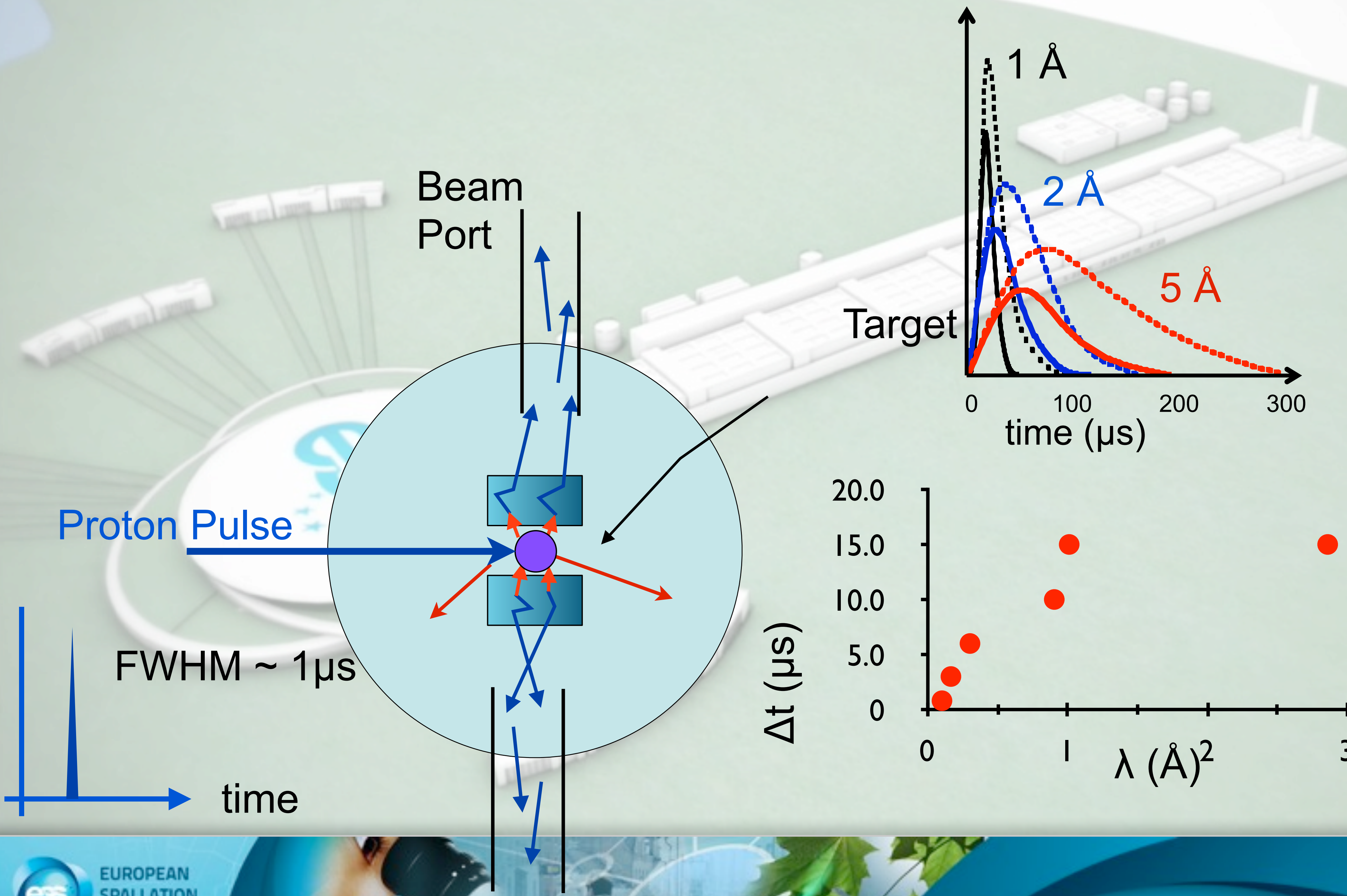



Target
Compressor/
Accumulator
Accelerator
Ion Source
Pulsed
Neutron
Beam
(TOF)
≈ 20 A
Current
Time
≈ 20 mA
≈ 1 ms
≈ 1 μs

Moderators

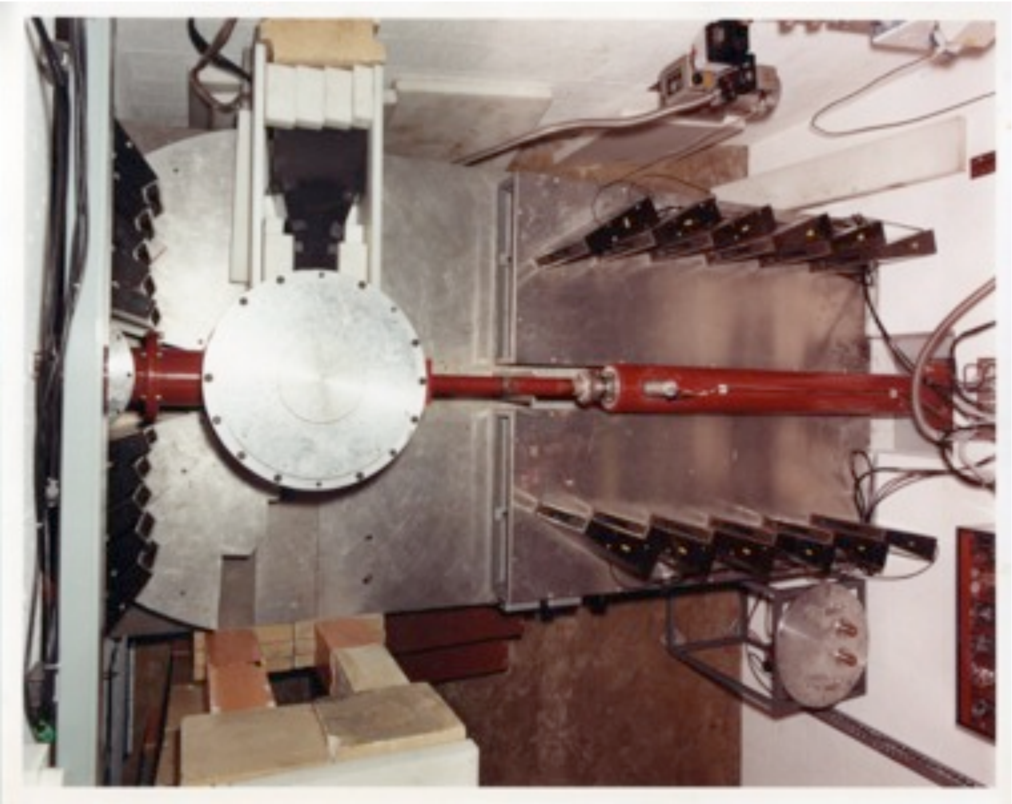


Moderators



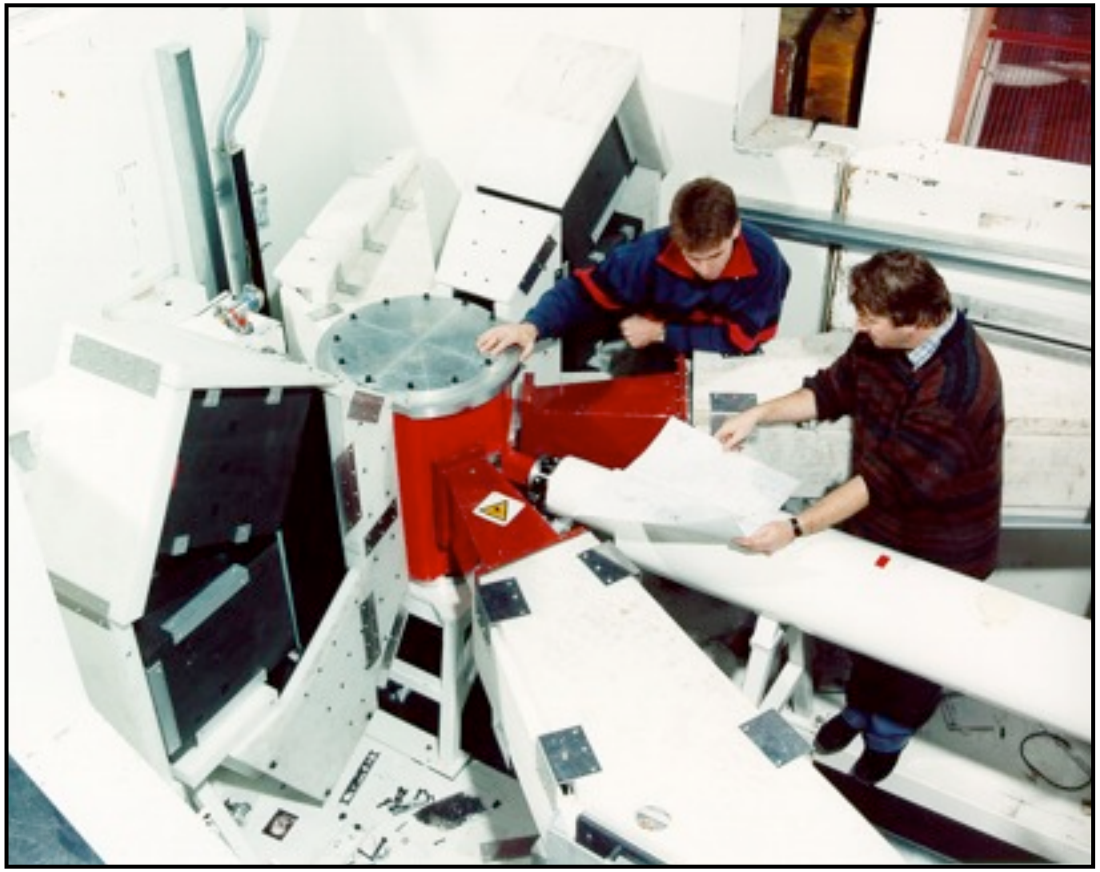
Beam
Port
Proton Pulse
FWHM ~ 1μs
time
Target
1 Å
2 Å
5 Å
0
100
200
300
time (μs)
20.0
15.0
10.0
5.0
0
Δt (μs)
0
1
2
3
λ (Å)

POLARIS + HRPD (ISIS)

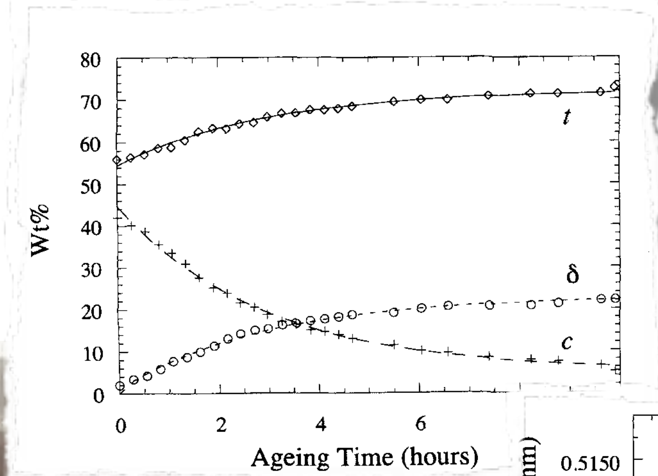


POLARIS 1991

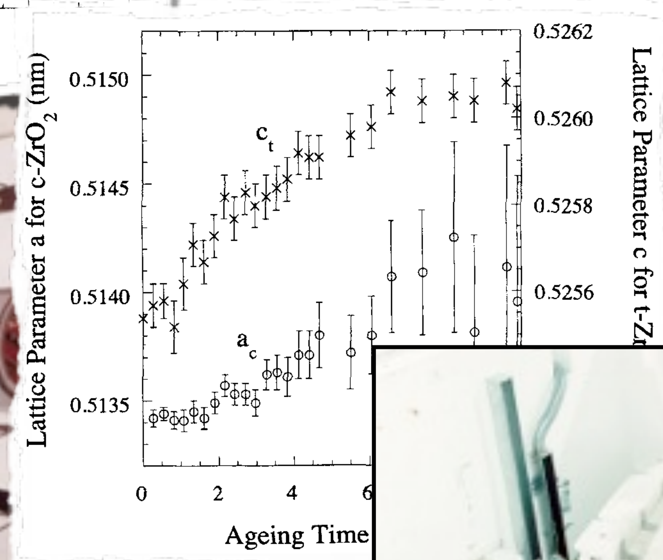
POLARIS
1996-2010

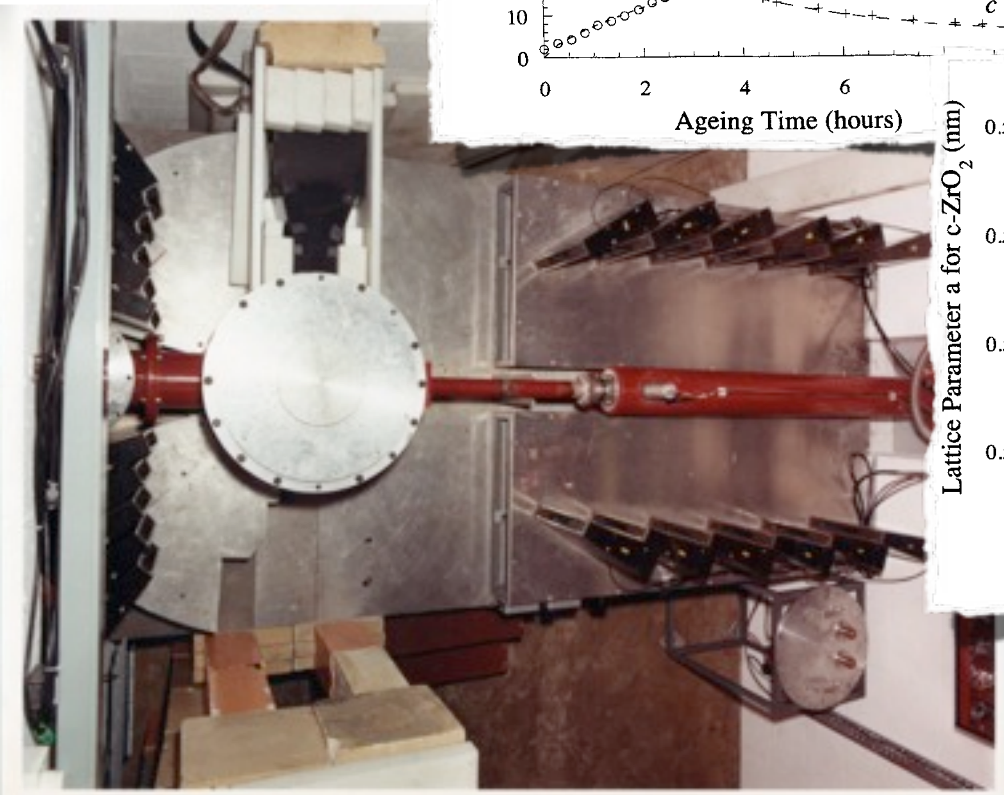


POLARIS (ISIS)



Y-Partially stabilized zirconia
Argyriou, Howard and Smith (1994)
15 minute data from 10g sample



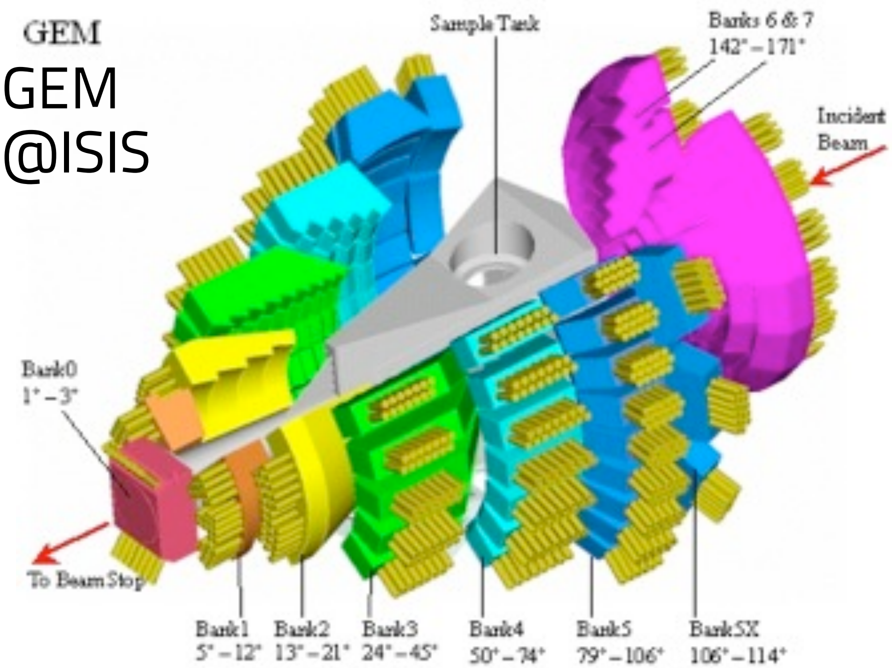


POLARIS 1991

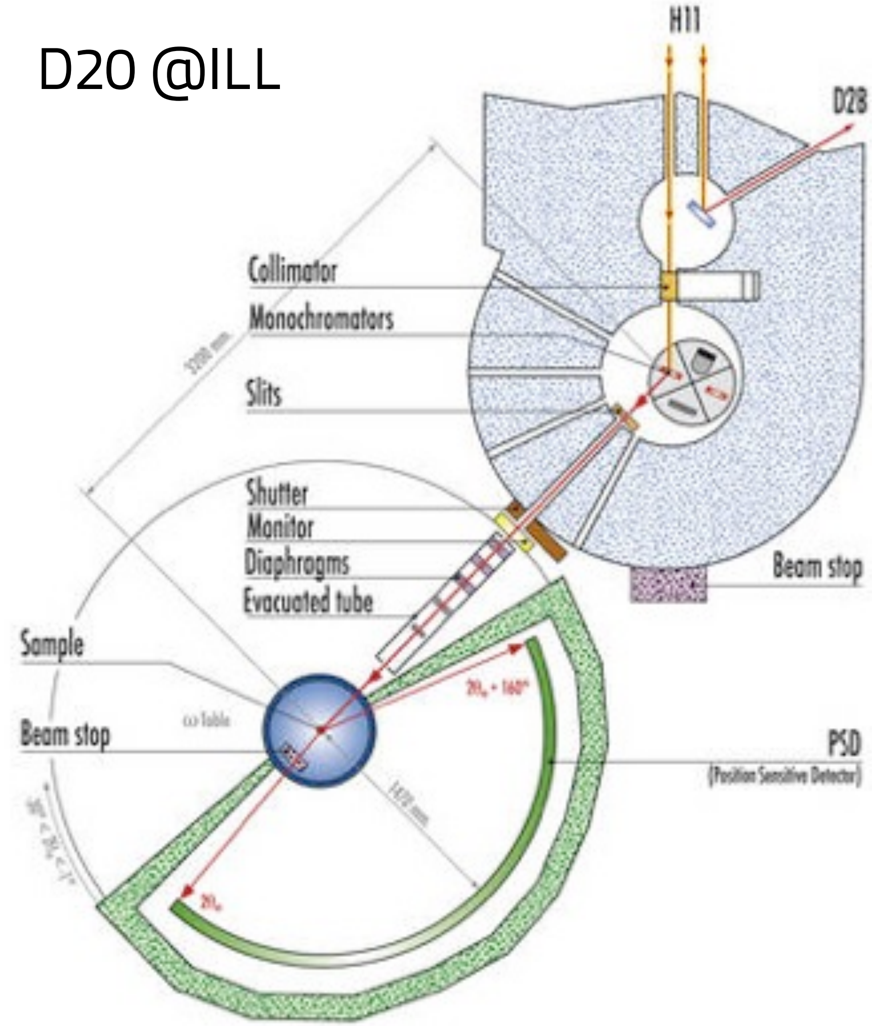
POLARIS
1996-2010

Step change in instrumentation

GEM
@ISIS



D20 @ILL



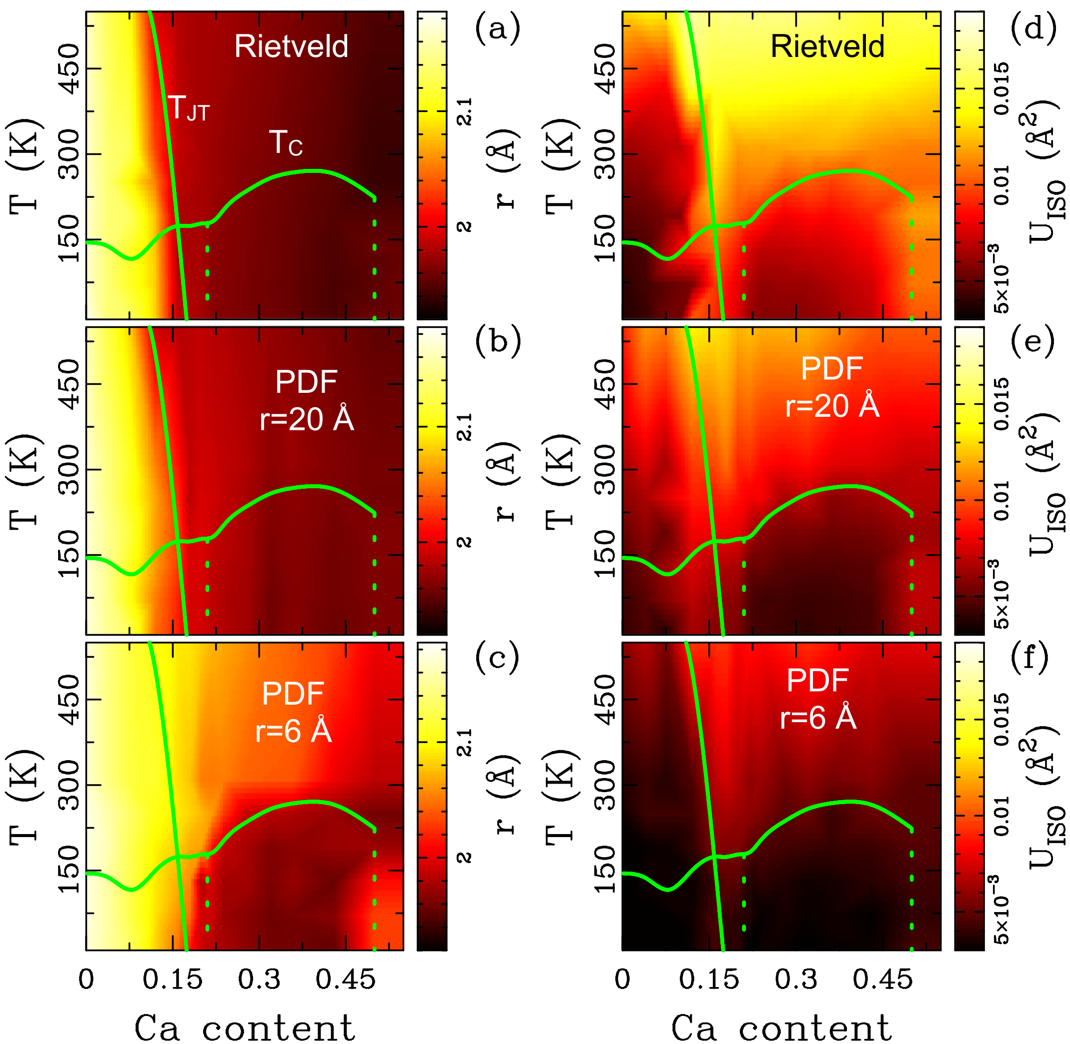
- High count rate / efficiency
- Large detector arrays
- Optimised beam transport
- Flexible resolution

late 1990s

PRL 98, 137203 (2007) PHYSICAL REVIEW LETTERS week ending 30 MARCH 2007

Understanding the Insulating Phase in Colossal Magnetoresistance Manganites: Shortening of the Jahn-Teller Long-Bond across the Phase Diagram of $La_{1-x}Ca_xMnO_3$

E. S. Božin,[1] M. Schmidt,[2] A. J. DeConinck,[1] G. Paglia,[1] J. F. Mitchell,[3] T. Chatterji,[4] P. G. Radaelli,[2] Th. Proffen,[5] and S. J. L. Billinge[1]



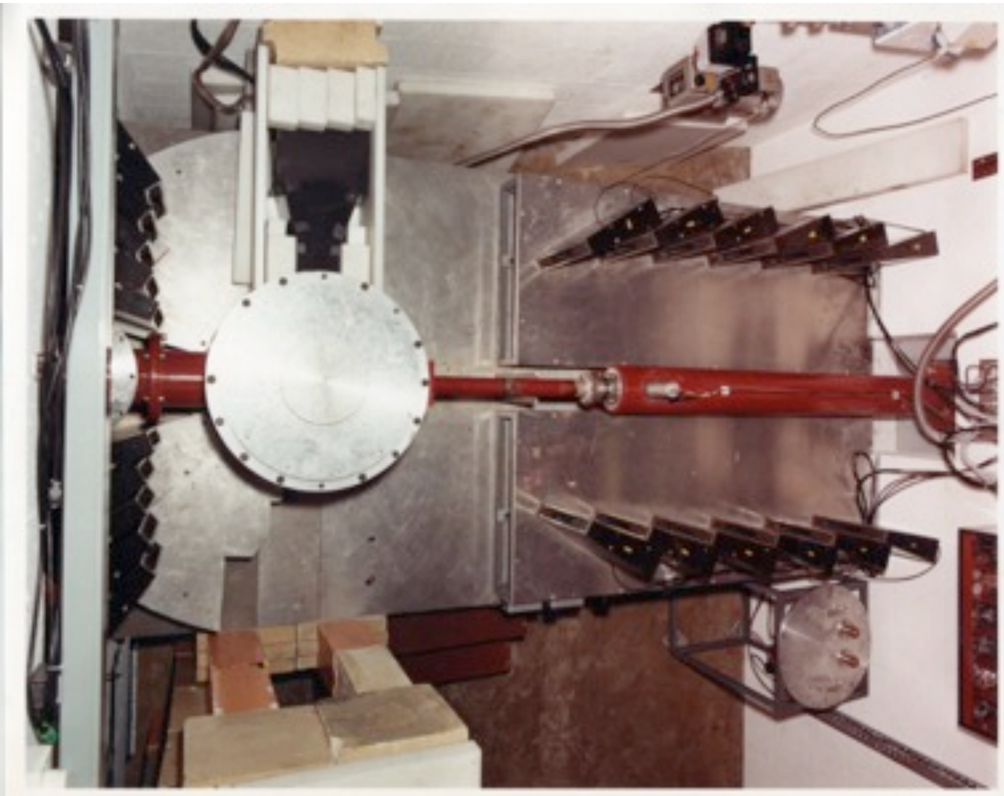
- High throughput and high quality data allows for detailed parametric investigation as a function of composition, temperature, pressure etc

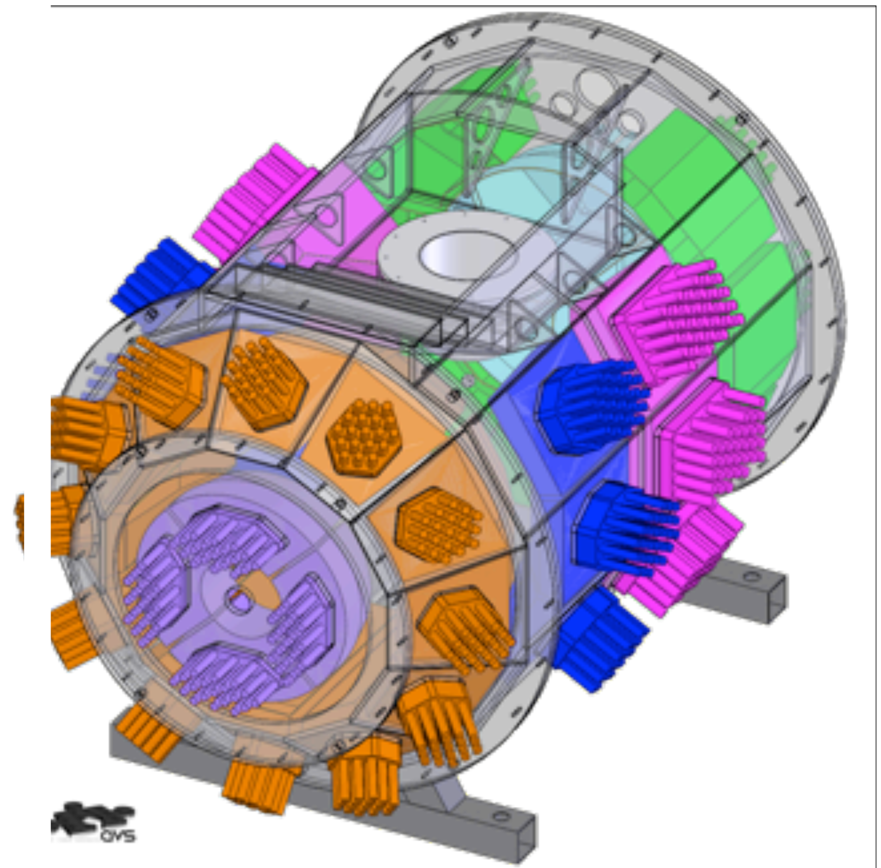
- Was *tour de force*, now routine !

GEM@ISIS

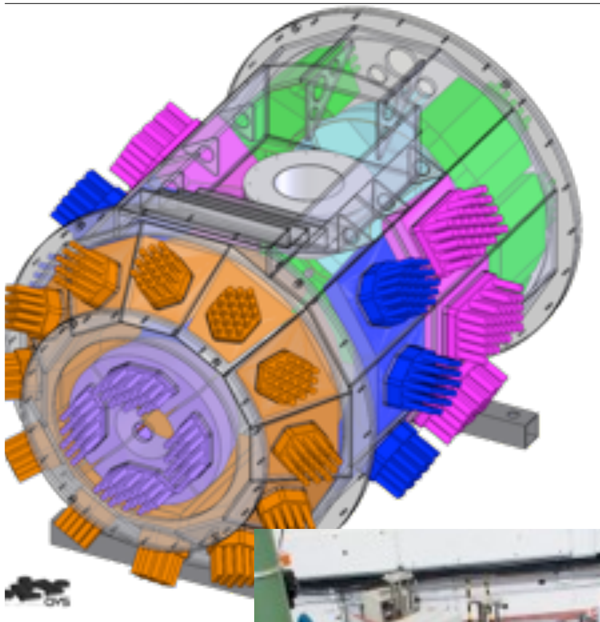
EUROPEAN SPALLATION SOURCE

1995 cf. 2013

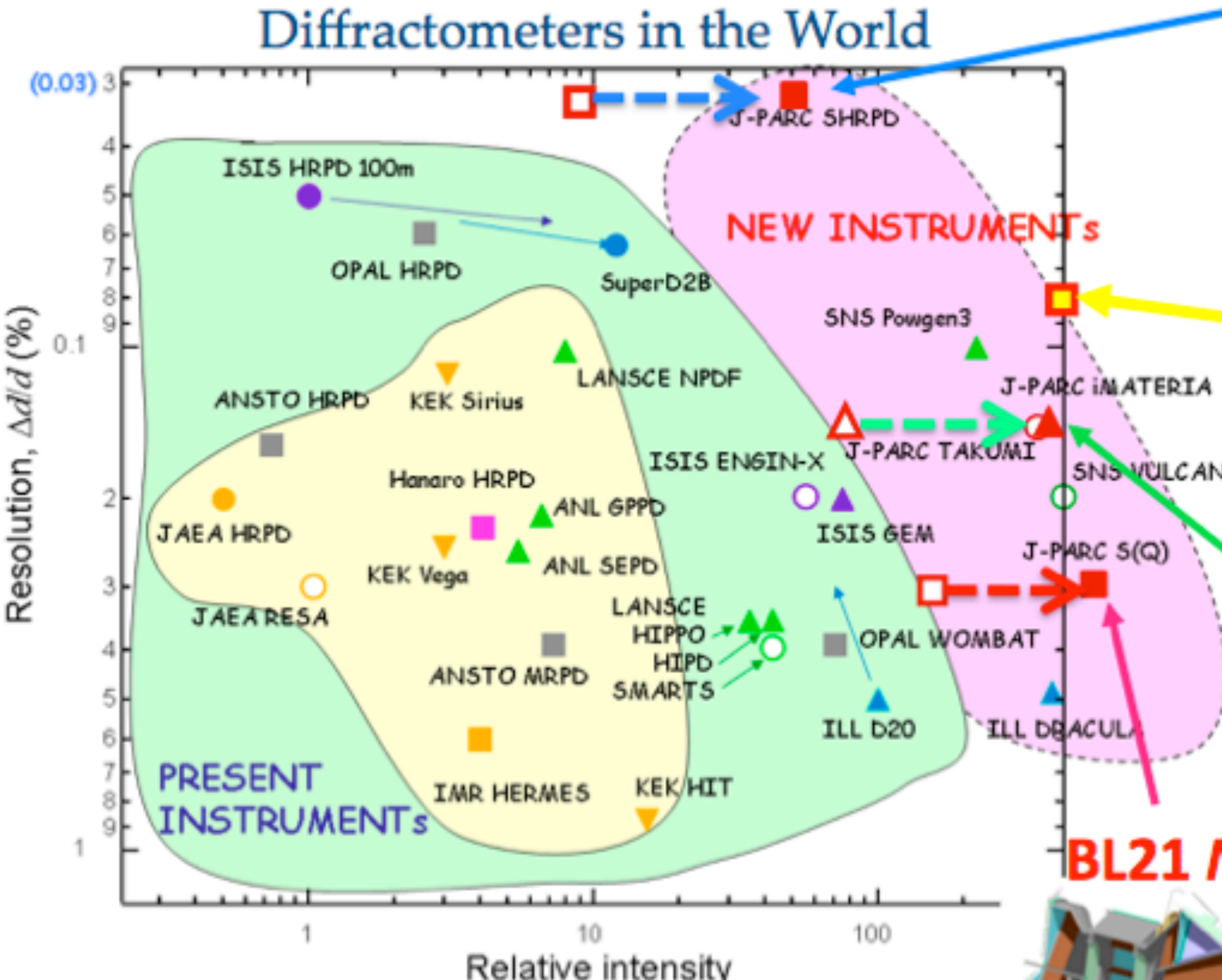




- 1995 500 mg 24+ hrs
- 2013 500 mg 15-20 minutes with increased Q-range



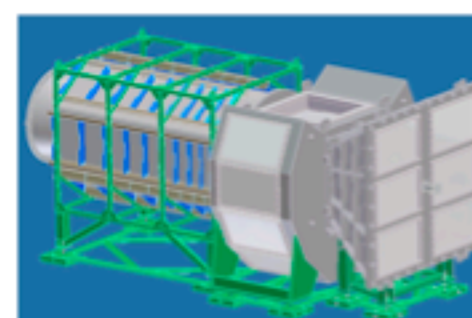
Polycrystalline Diffractometers Resolution & Intensity



BL08 *SuperHRPD*

BL09 *SPICA*

BL20 *iMATERIA*



BL21 *NOVA*

Several instruments to be constructed at once ,,,
Relation to other facility is important,,,

Requirements in Modern NPD

- Flexible Instrumentation
  - -trade flux for resolution
  - -Match resolution to problem
  - -Match Q-range to system of interest
- Smaller samples
  - -Isotopic substitution
  - -Extreme conditions (high pressure/ high magnetic fields/simultaneous measurements)
- Fast data collection
  - -Parametric studies of phase diagrams
  - -In situ study of reactions
  - -Kinetic studies
- New technical developments
  - -Polarisation
  - -Hydrogenous materials

Reactor and Spallation Sources have distinct advantages and disadvantages

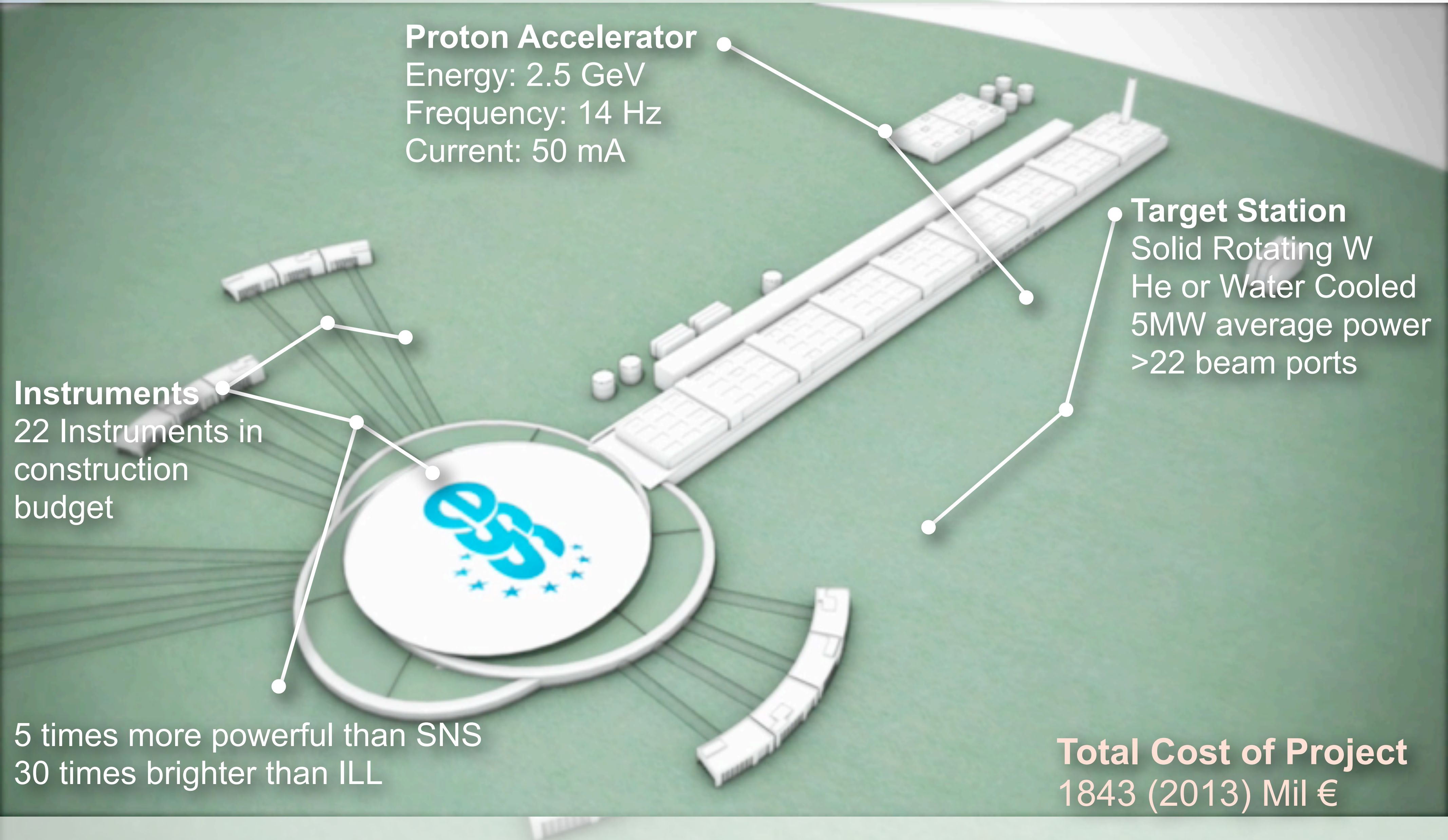
- Take-off Angle
- Moderator pulse shape

Need for higher source fluxes, how ?

Advanced data acquisition electronics and methods to track fast reactions.

- micro-secs possible from the perspective of flux
- Problems in using large detector arrays in this way

Polarisation is flux intensive !


Proton Accelerator
Energy: 2.5 GeV
Frequency: 14 Hz
Current: 50 mA
Target Station
Solid Rotating W
He or Water Cooled
5MW average power
>22 beam ports
Instruments
22 Instruments in construction budget
5 times more powerful than SNS
30 times brighter than ILL
Total Cost of Project
1843 (2013) Mil €

An International Collaboration

Sweden,
Denmark and Norway:
50% of construction and
20% of operations costs

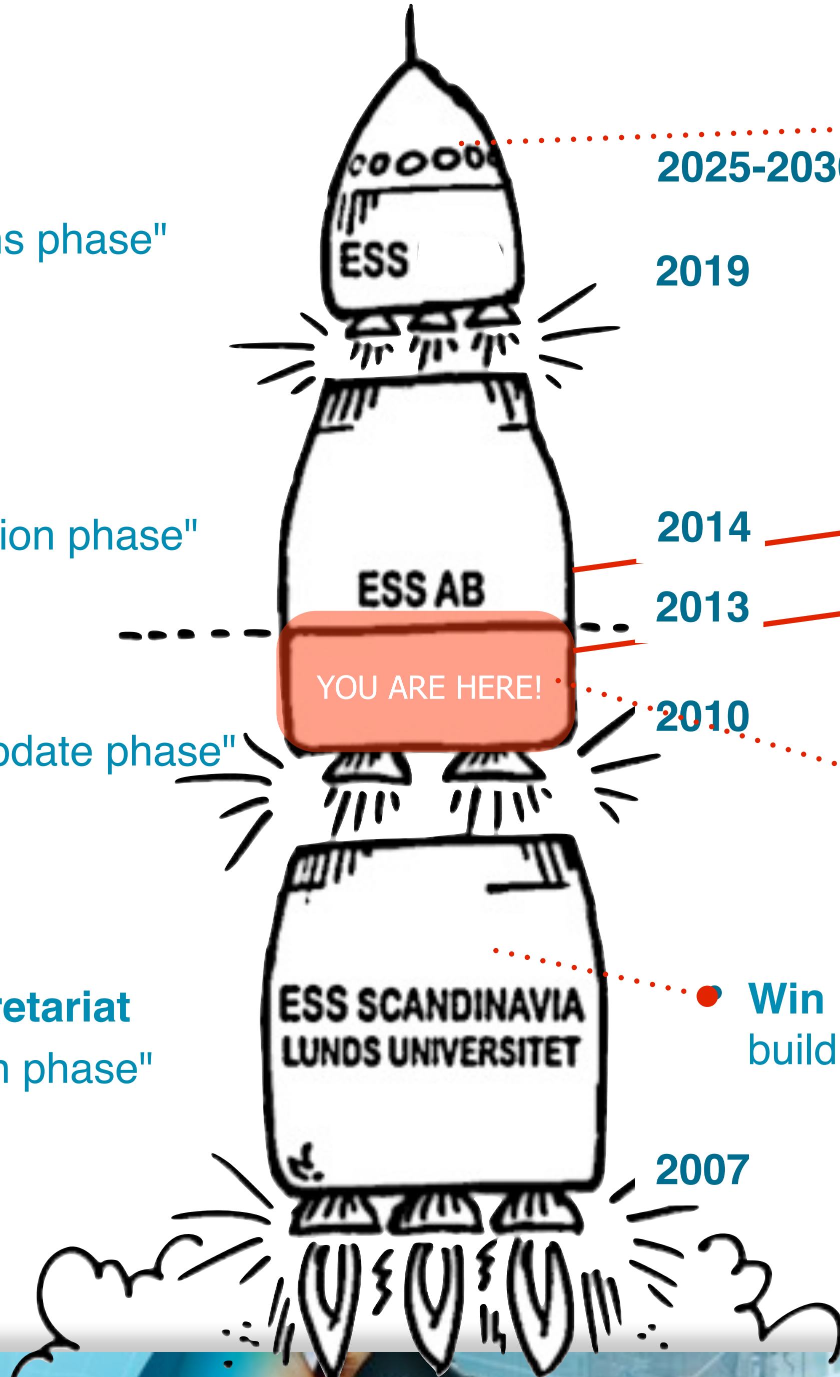
European partners
pays the rest

ESS
- "operations phase"

ESS AB
- "construction phase"

ESS AB
- "design update phase"

ESSS Secretariat
- "campaign phase"



2025-2030 — **ESS reaches full operational capability at 5MW and 22 instruments**

2019

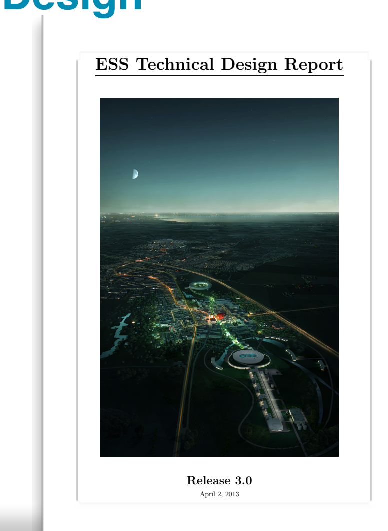
2014 — **Start Civil construction**

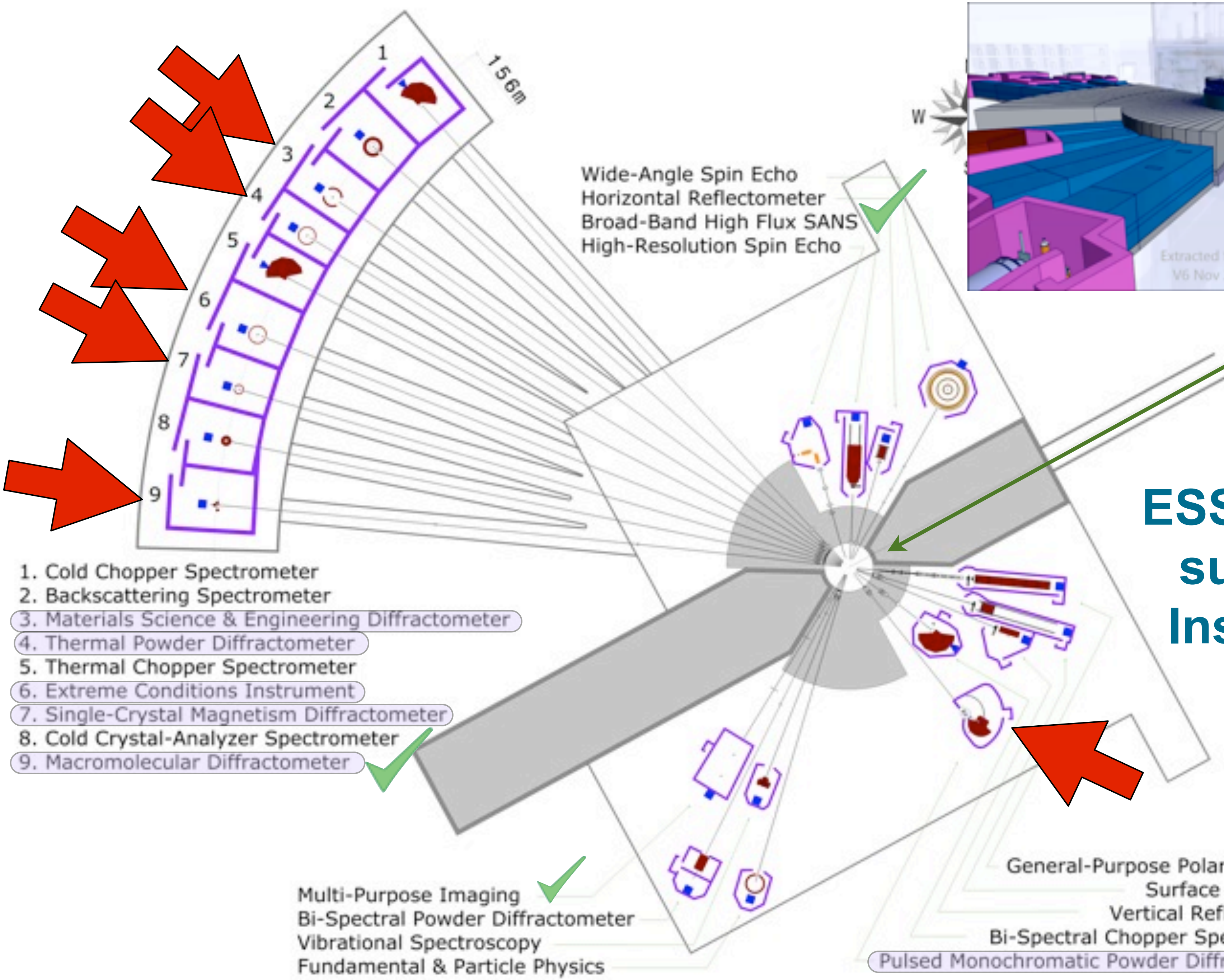
2013 — **Deliver Technical Design Review**
- **Costing Report**
- **Cost Book**

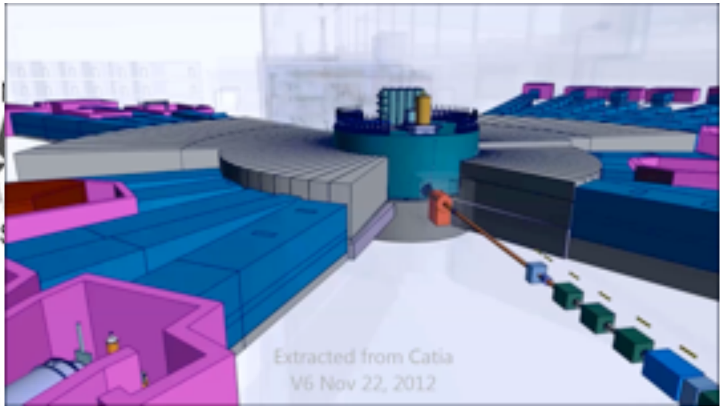
2010 — **Deliver Conceptual Design Review**

Win international support for building ESS in Lund, Sweden

2007

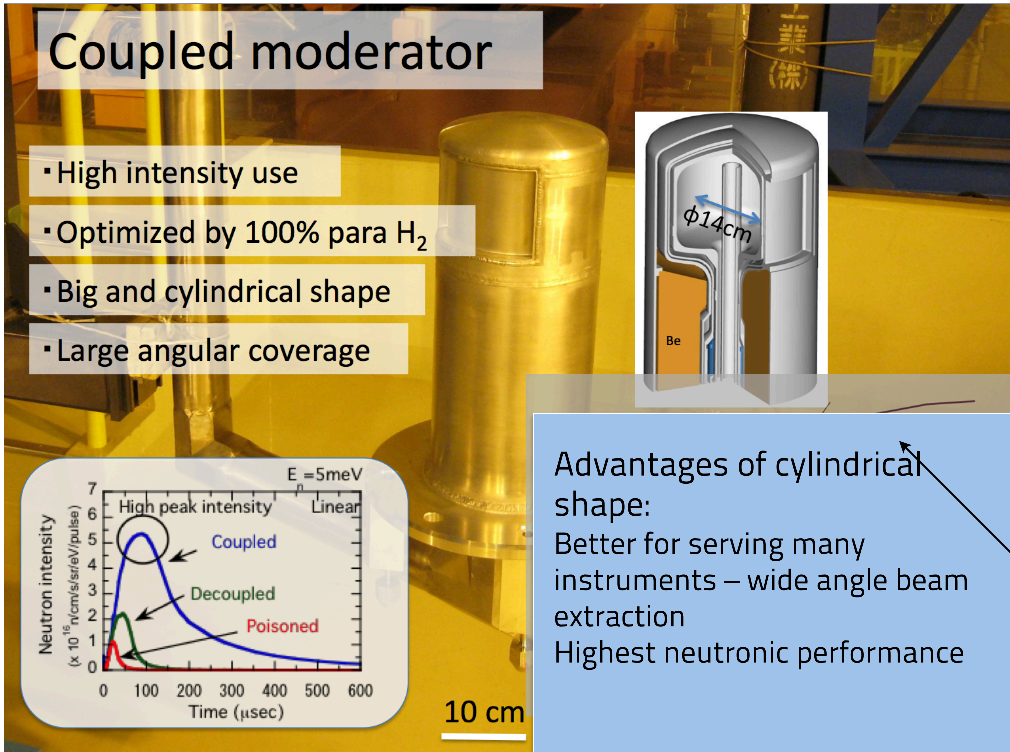




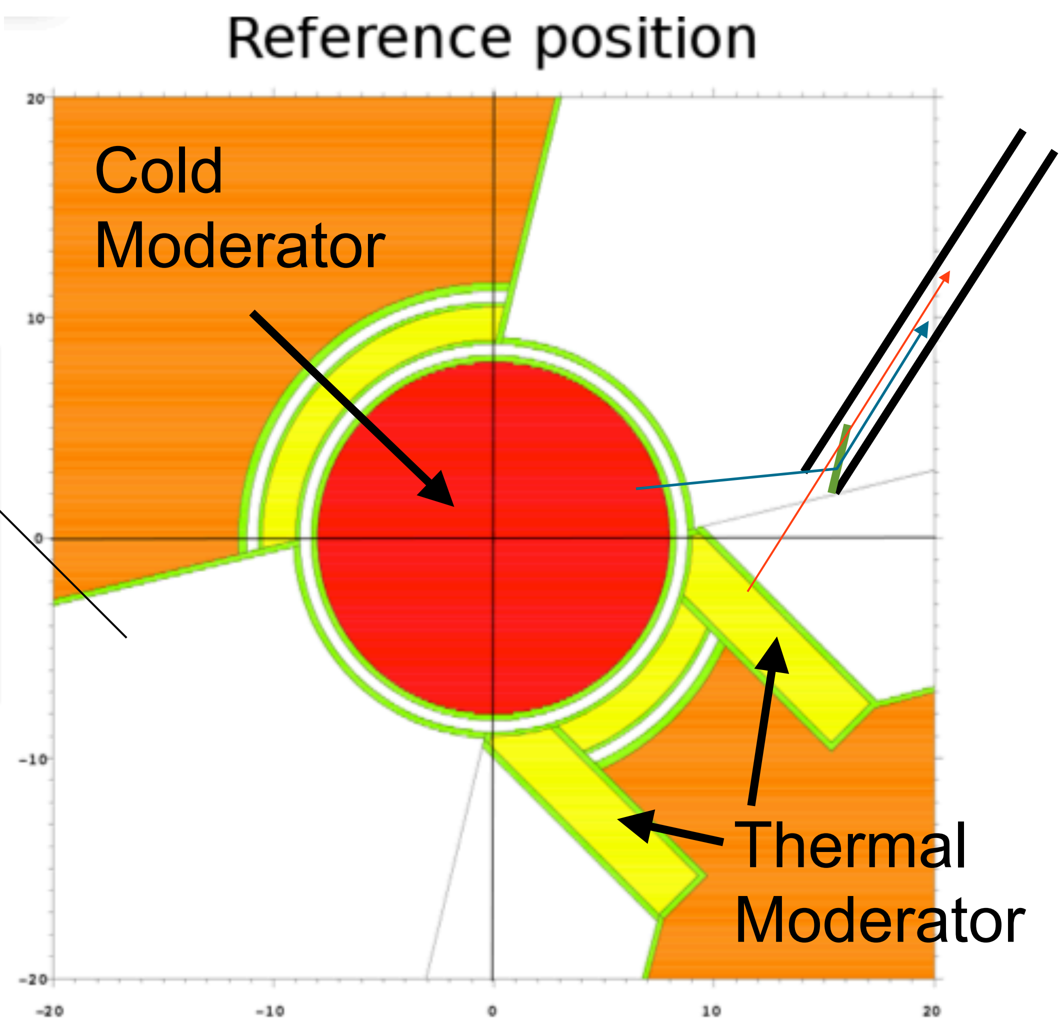


ESS reference suite of 22-Instruments

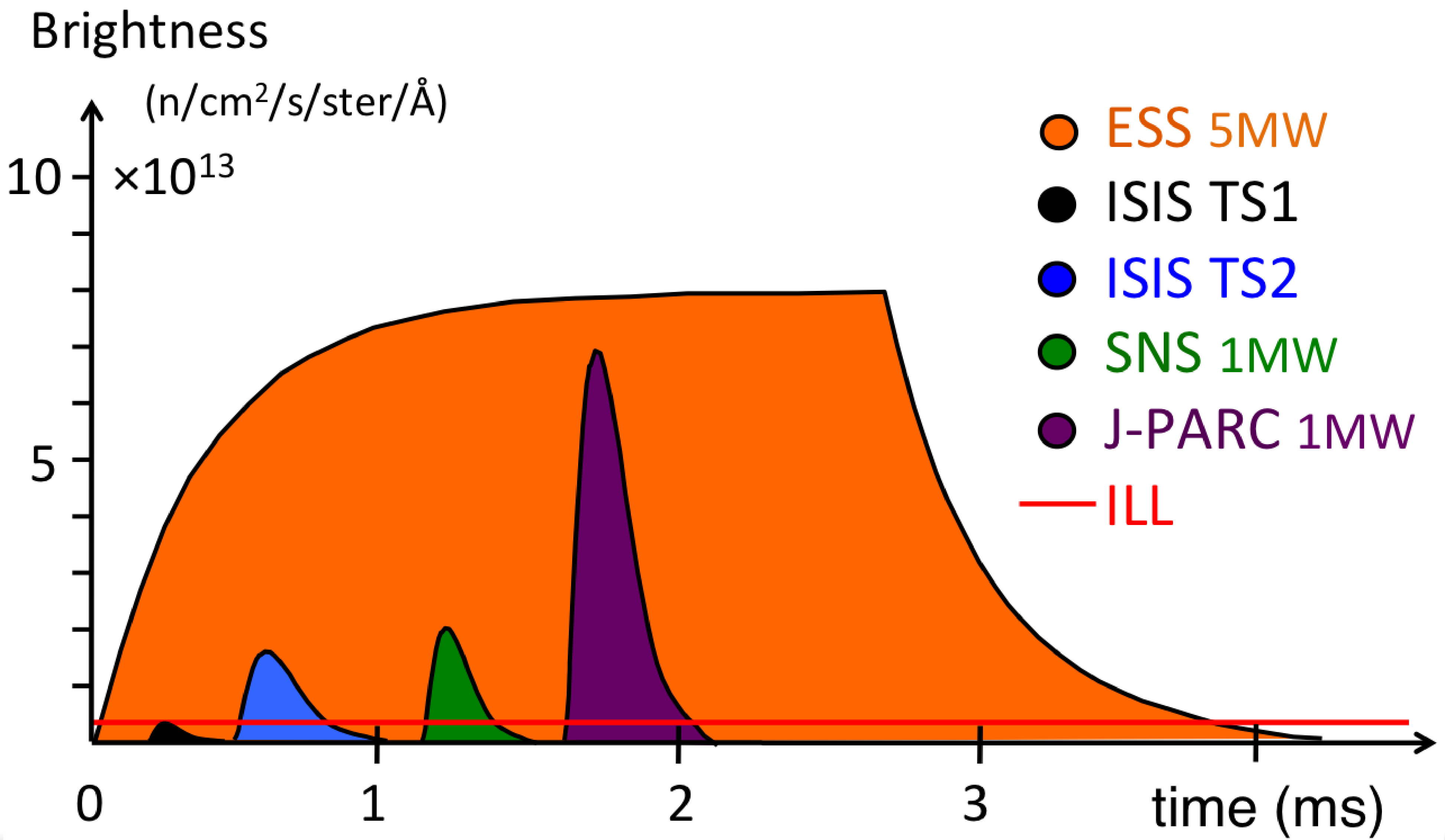
ESS Moderator



Advantages of cylindrical shape:
Better for serving many instruments – wide angle beam extraction
Highest neutronic performance

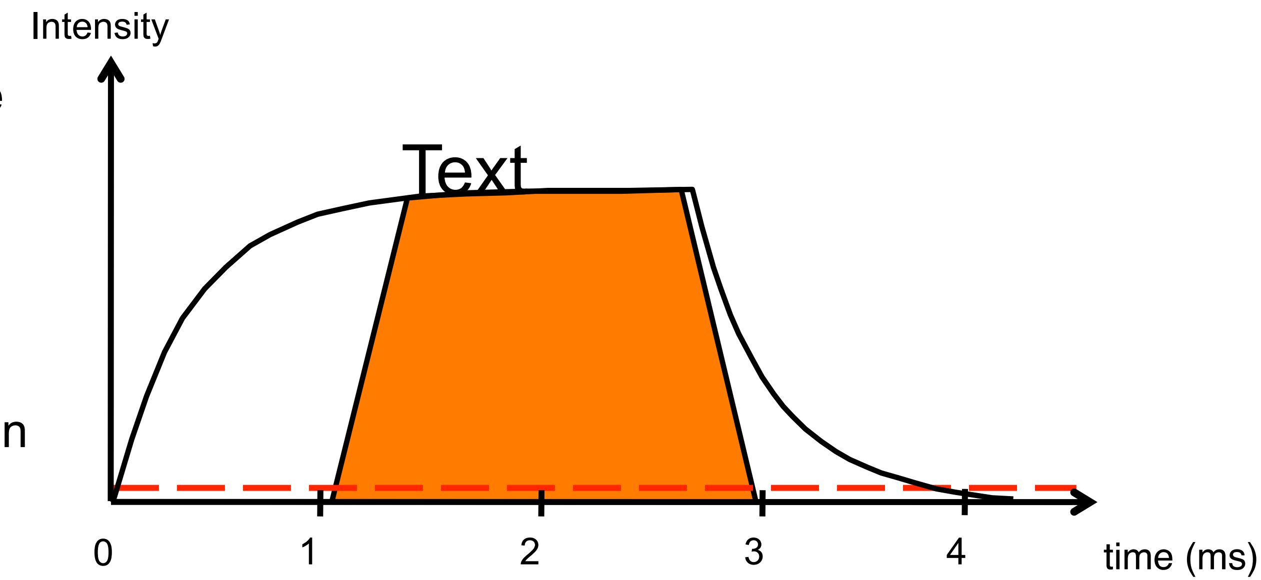


ESS Long Pulse Structure Compared to Other Sources

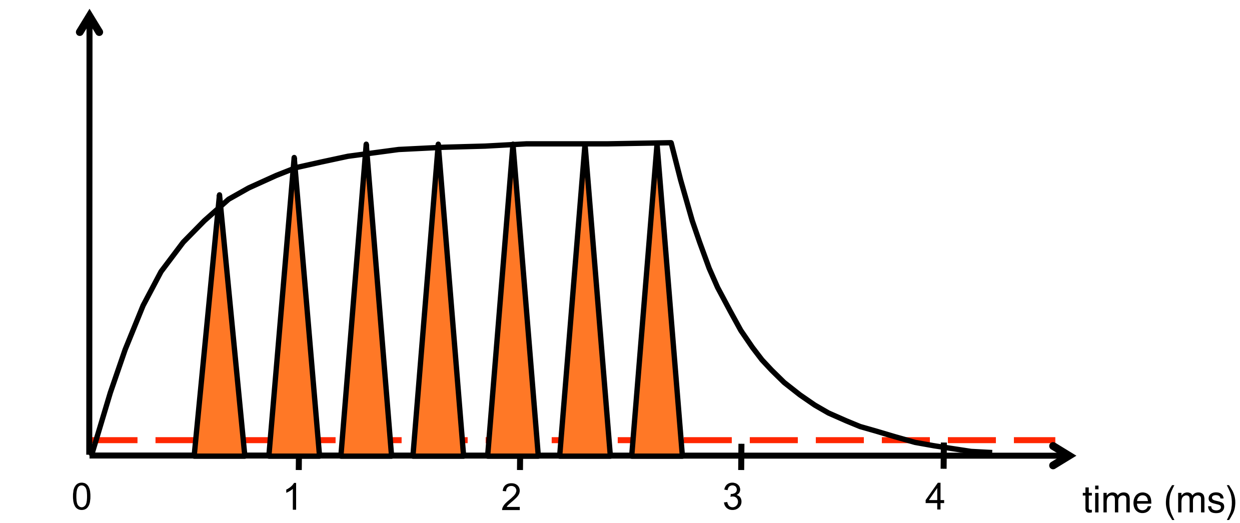


Two Strategies for Neutron Instrumentation at ESS

Use as much as possible of the whole pulse:
Good for low wavelength resolution instruments.
SANS, Reflectometry, single crystal diffraction.
Estimated gains 10-100 times than currently available.

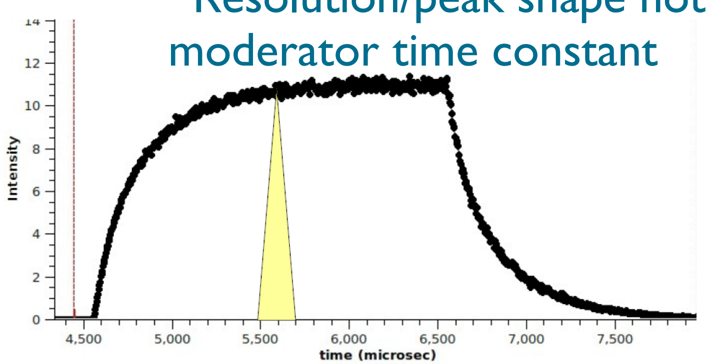


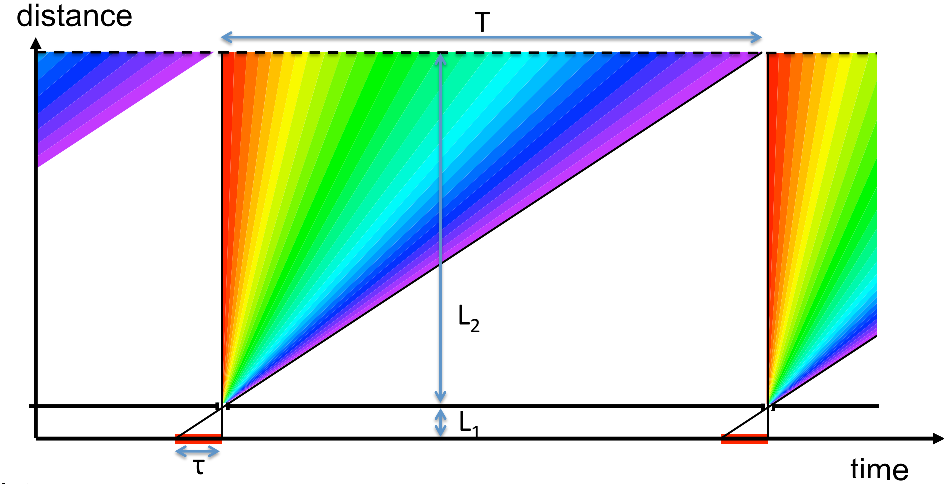
Cut the long pulse into smaller pulses:
Good for higher wavelength resolution instruments
Diffraction, cold/thermal spectrometers.
Long Instruments (80-100 m)
Estimated gains 10-30 times than currently available.
Thermal gains lower.

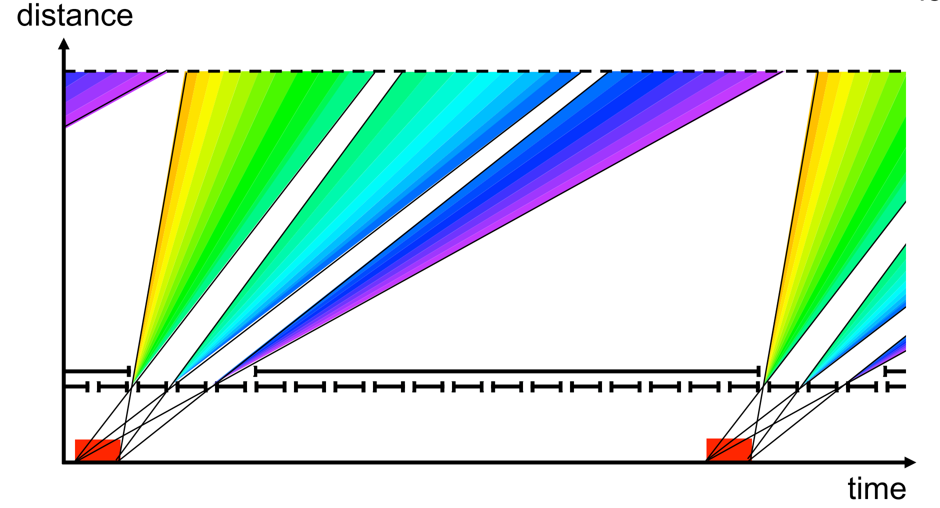


How to do it ?

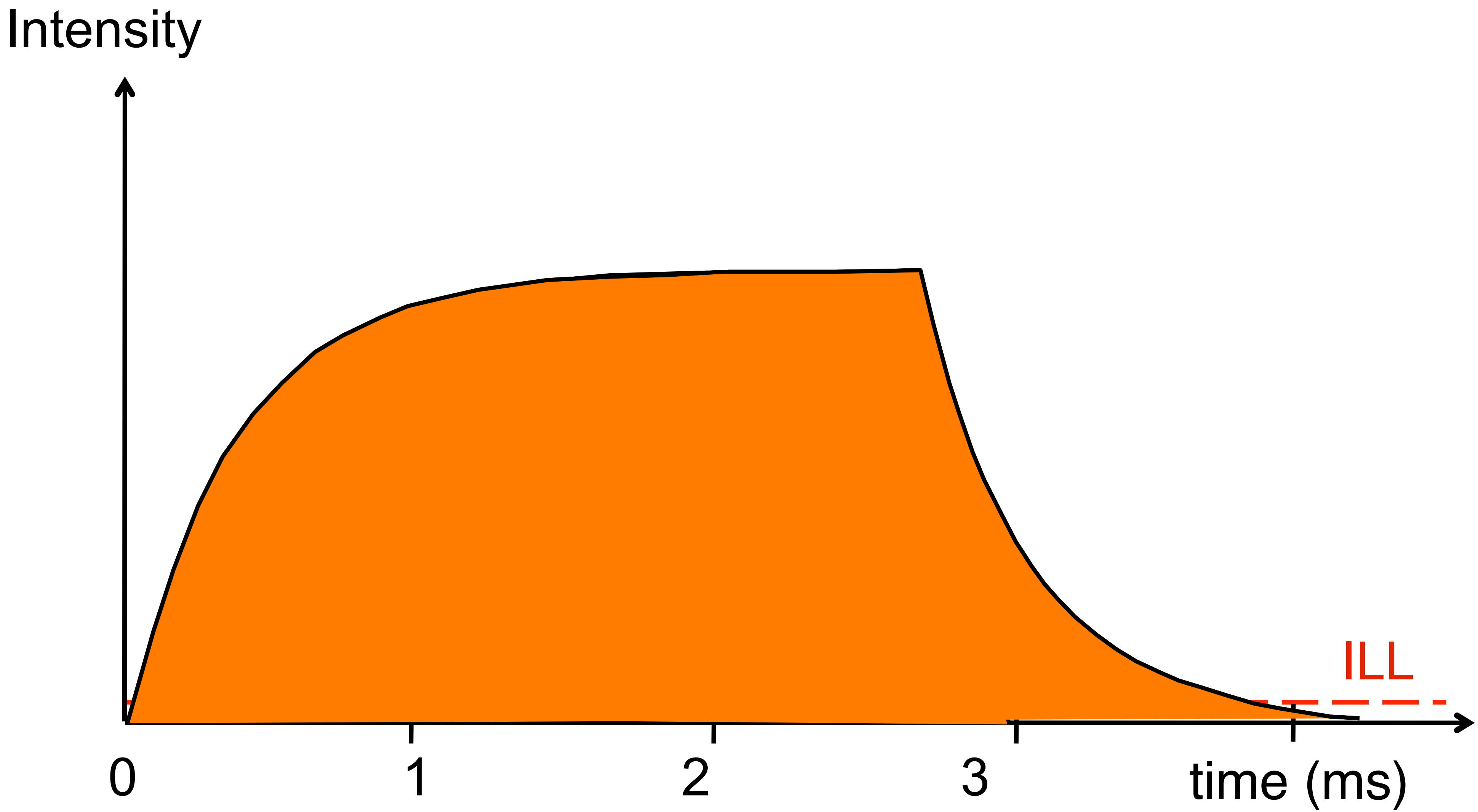
- The long pulse is too broad to use for diffraction studies.
  - Choppers will play the role of the moderator-response time in a conventional short pulse source.
  - Important to get the first pulse shaping chopper as close as possible to moderator.
  - Tunable wavelength range (1.9 Å band up to a maximum of 6 Å)
  - Can ‘slew’ choppers to cover complete wavelength band in several pulses
  - Tunable $\Delta\lambda/\lambda$ (from < 0.02% - 5% at λ = 1.45 Å) with PSC
  - Resolution/peak shape not determined by moderator time constant







Pulse Shaping Chopper



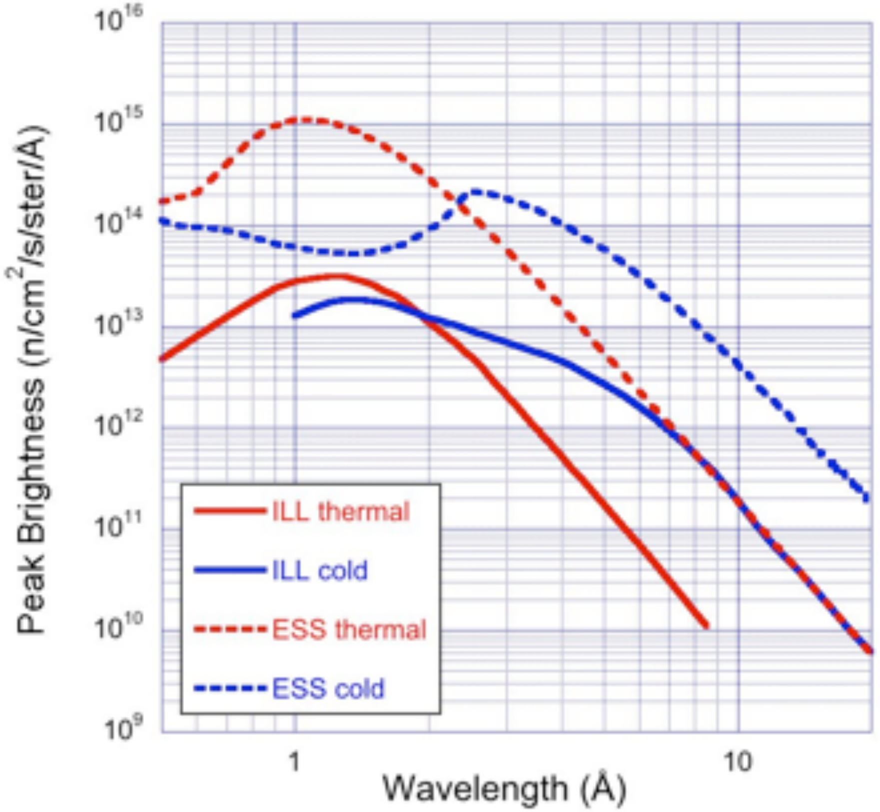
Instrument Workshop Summary

- Recommended Phase 1 Diffractometers
  - Single-crystal diffractometer for macromolecular crystallography
  - Single-crystal diffractometer for magnetism
  - Narrow-bandwidth, high-resolution tunable powder diffractometer

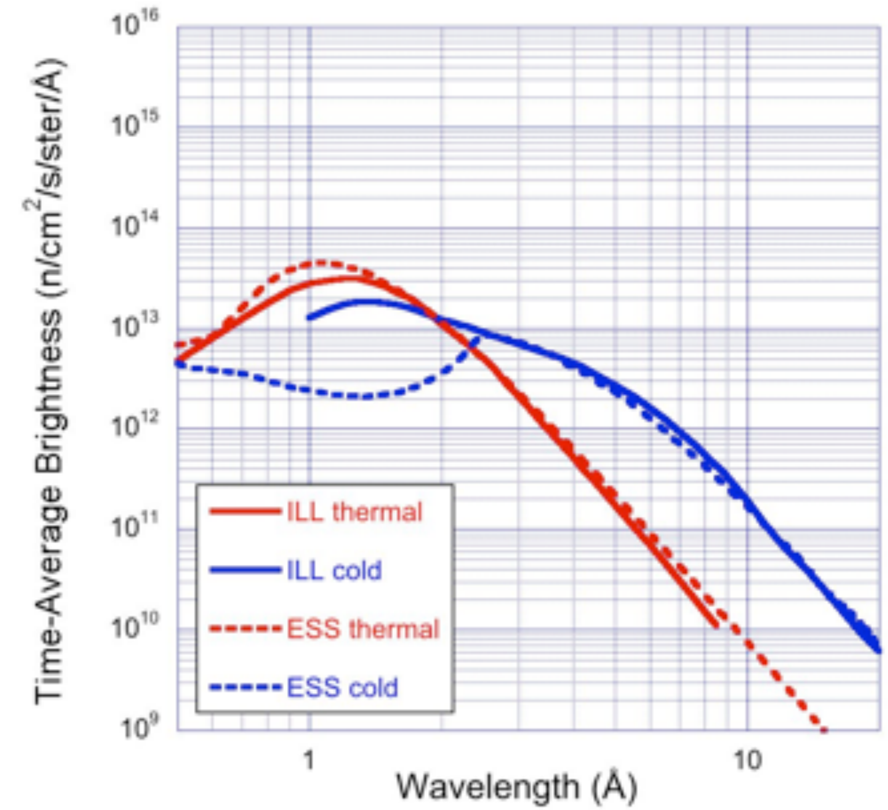
- Suggested for further consideration
  - Hybrid diffractometer
  - Structured pulse engineering spectrometer
  - Single-crystal (and/or powder) diffractometer for extreme conditions

Concepts for powder diffractometers at ESS

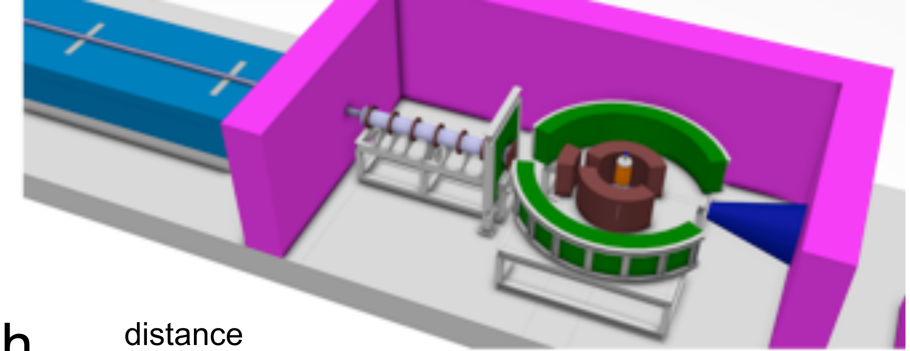
Use source peak brightness
➔TOF wavelength band instrument (conventional spallation source instrument)



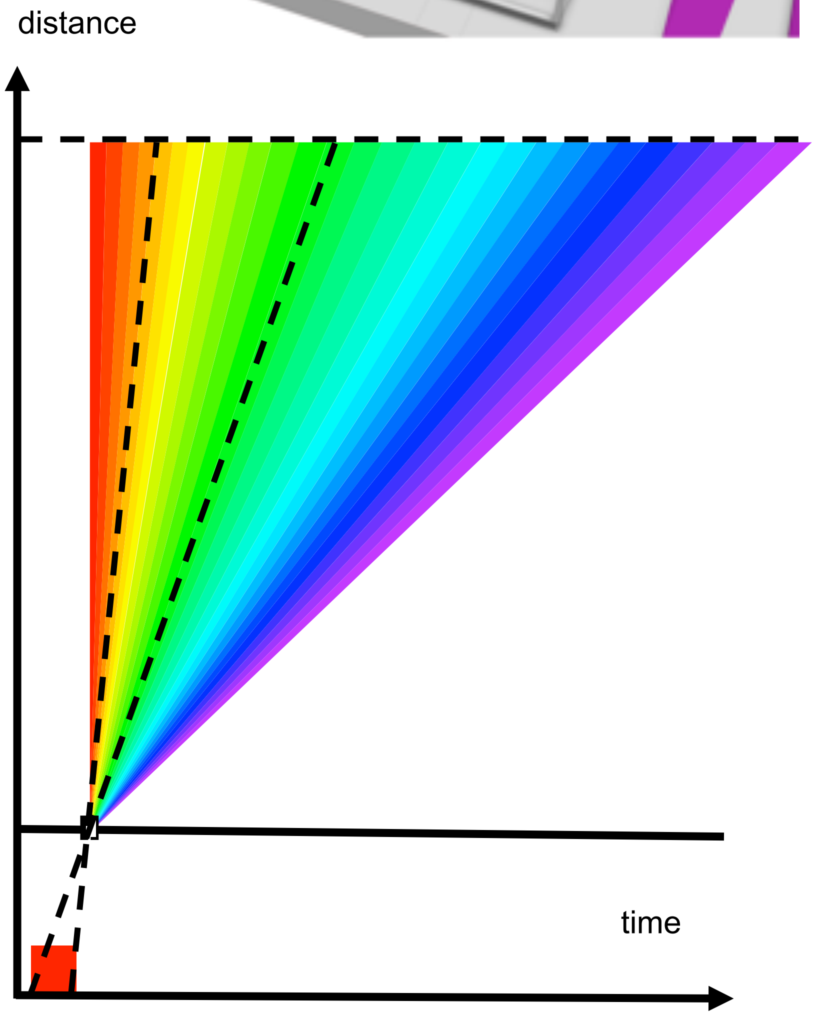
Use source time-average brightness
➔Monochromator instrument with TOF detector (reactor-like instrument with enhanced capabilities)



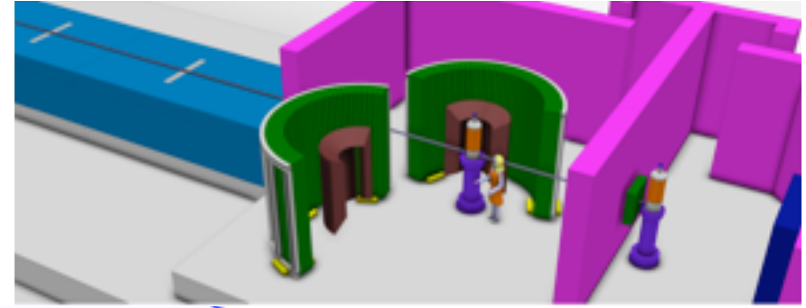
Thermal powder TOF diffractometer



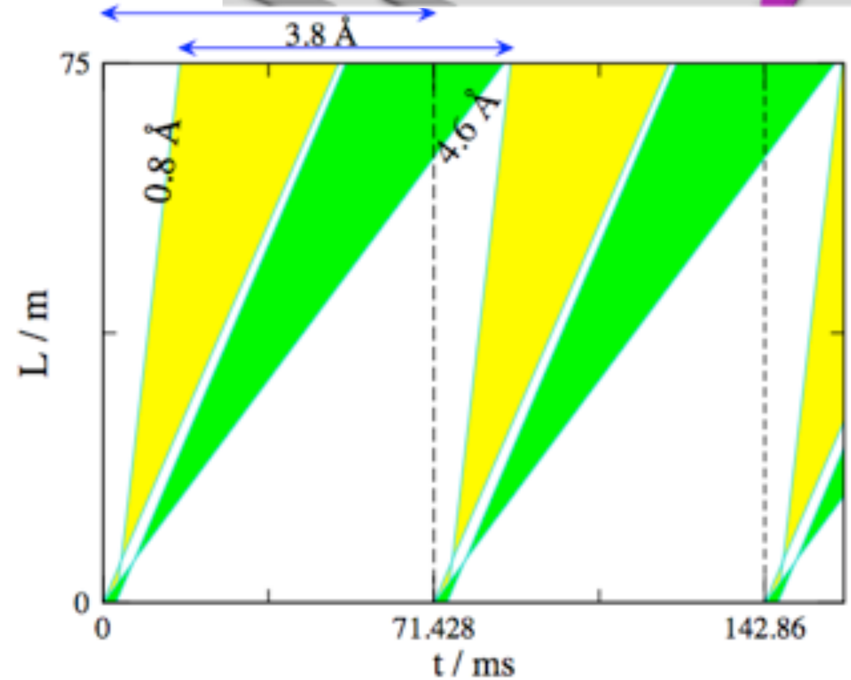
- Up to 1.9 Å single frame wavelength band (normal mode 0.5 – 2.4 Å)
- High flexibility cf. SPSS instrument
  - Tuneable wavelength range (1.9 Å band up to a maximum of 6 Å)
  - Can ‘slew’ choppers to cover complete wavelength band in several pulses
  - Tuneable Δλ/λ (from < 0.02% - 5% at λ = 1.45 Å) with PSC
  - Tuneable flux with PSC
  - Resolution/peak shape not determined by moderator time constant
- Long instrument (156 m)
  - low background



Bispectral Powder Diffractometer

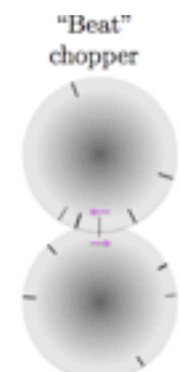


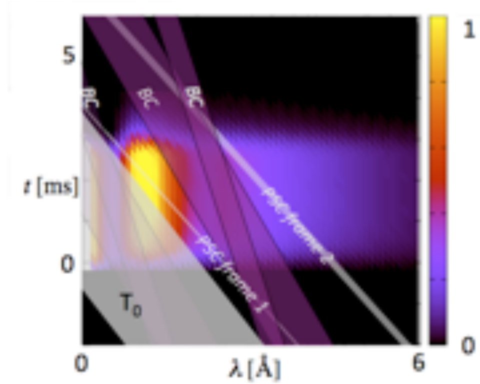
- Bispectral extraction (0.8 – 10 Å)
- Wavelength frame multiplication gives 3.8 Å wavelength band
  - Normal mode (0.8 – 4.6 Å)
  - Tuneable wavelength range
- Beat chopper
  - Flexible resolution
  - Flexible flux
- Shorter instrument (75m cf. 150m)
- Complementary with thermal powder diffractometer



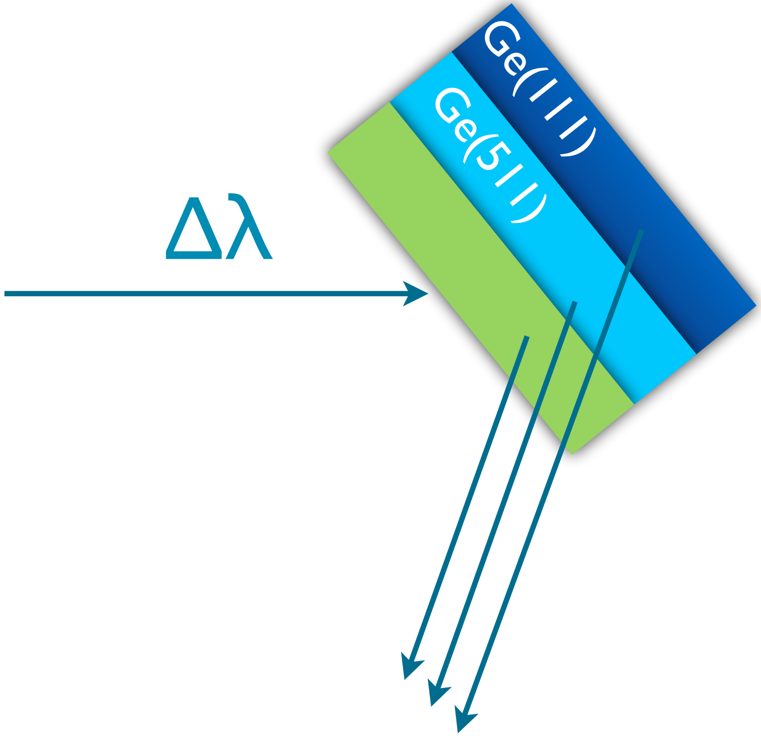
Time resolution options

$\delta t(\lambda_1)$	$\delta t(\lambda_1 + 1.9Å)$	(Hz) : (Hz)
27.5 μs	82.5 μs	84 : 70
60.6 μs	181.8 μs	45 : 28
151.5 μs	353.5 μs	28 : 14
227.4 μs	606.3 μs	14 : 14
378.8 μs	681.8 μs	0 : 14

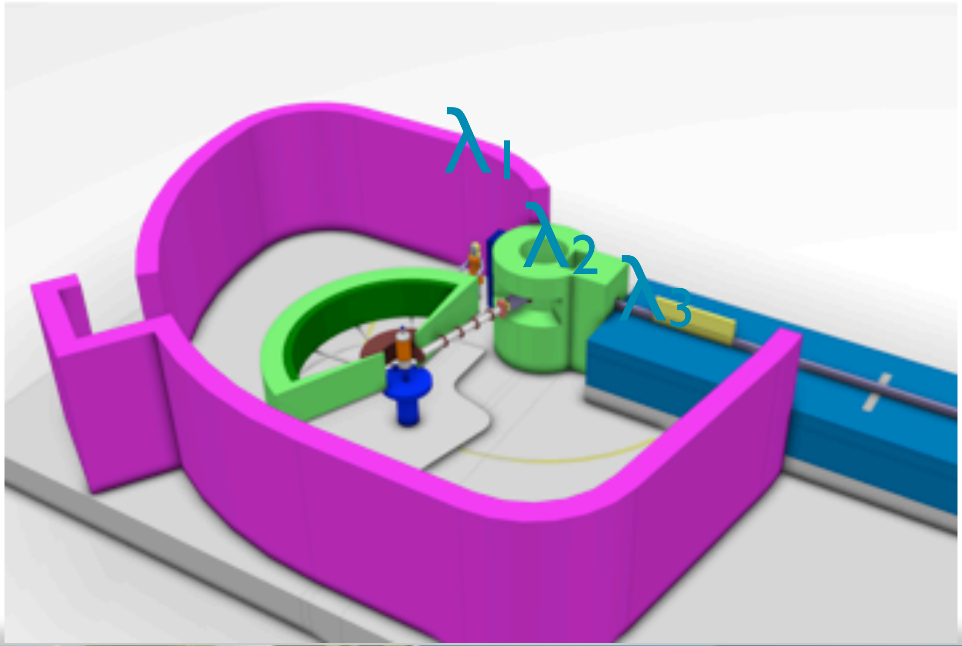




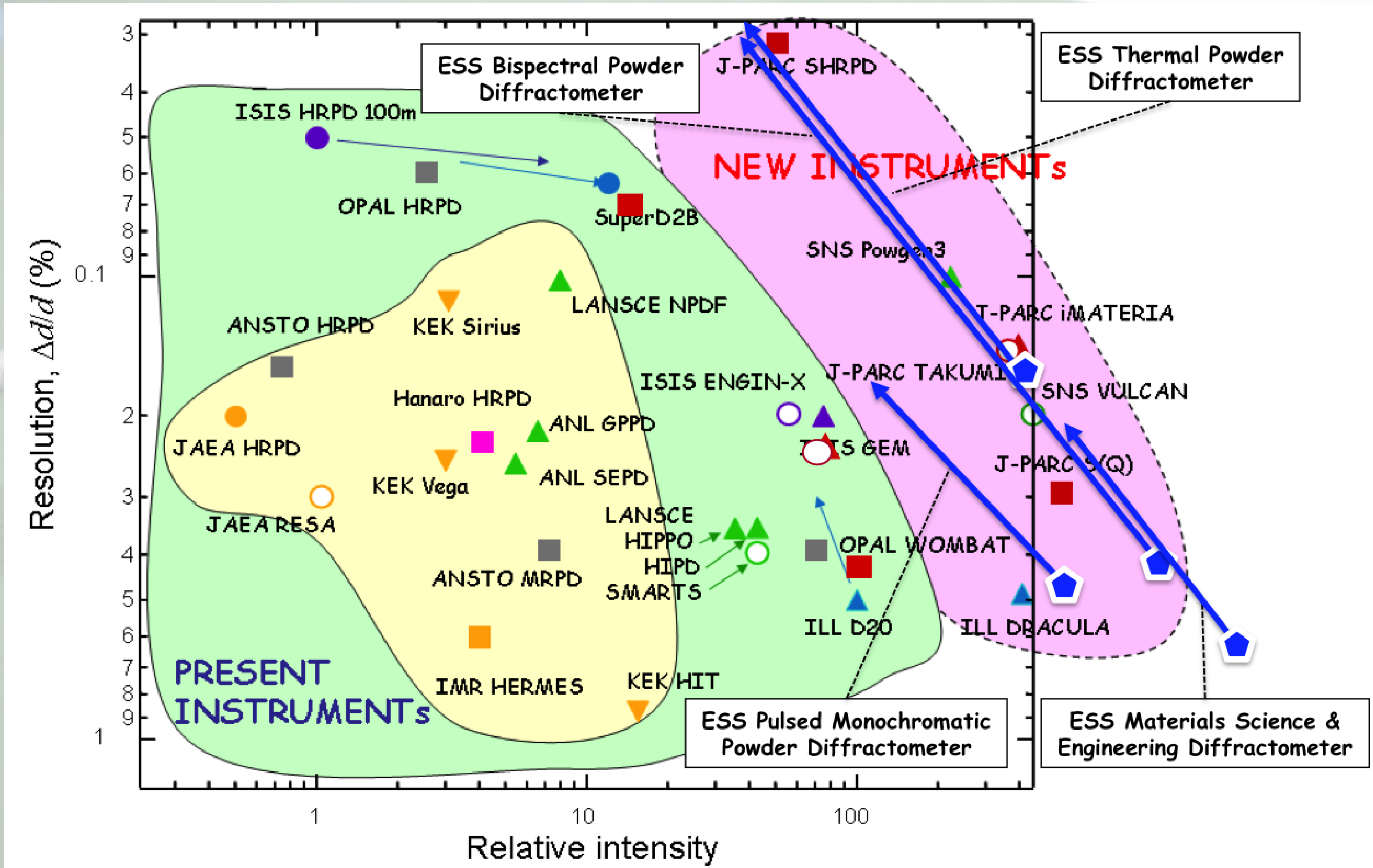
Pulsed monochromatic powder diffractometer



- Like a reactor instrument in appearance
  - Q_{max} ~ 12.5 Å^{-1}
  - Variable resolution (takeoff angle)
  - Trade flux for resolution (takeoff angle)
  - But longer instrument (total flight path 50 m cf. 22m at D20)
  - And optimised beam transport
- Background suppression
  - Only integrate around elastic line
- New capabilities cf. reactor instrument
  - Multi-wavelength data collection
  - Separate coherent/incoherent scattering
  - Elastic/inelastic measurements
  - Fast kinetics



Neutron Diffraction and ESS



Diffraction at ESS

- ESS will offer levels of versatility not available in any type of diffractometer either at reactor or short-pulse spallation sources.
- Mechanical chopper systems provide for the
  - Wavelength resolution
  - Wavelength band
  - Peak shape
- ESS diffractometers will require significant software developments for data reduction and analysis
- Emphasis in instruments will be towards sample environments, following current trends
- To understand results scientific computing is becoming increasingly important