Experiments with TOF and steady state (CW) neutrons: **Diffraction Experiments**

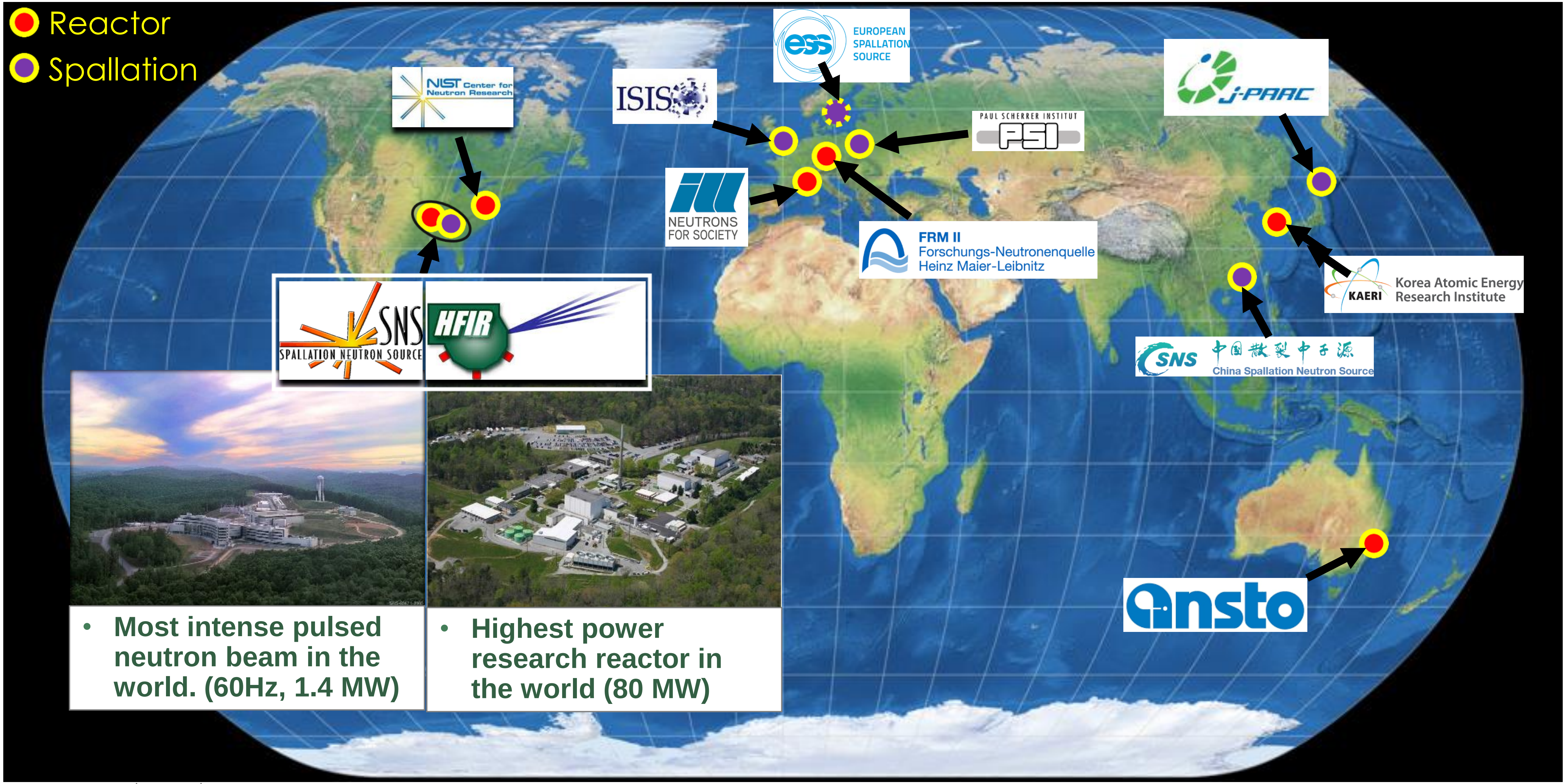
Stuart Calder

Neutron Scattering Division
Oak Ridge National Laboratory

Overview

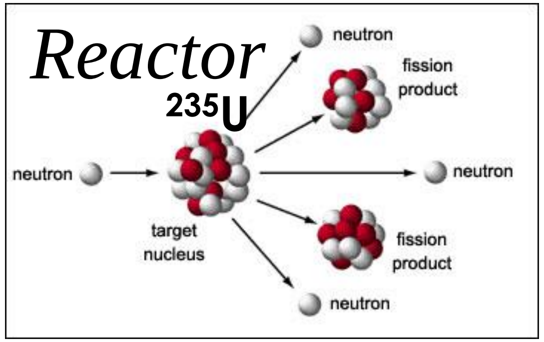
- General characteristics of Reactor and Spallation sources
  - ORNL has both!
- What this means for diffraction instruments
- Strengths for different experiments
- Consider which source is best suited to your science

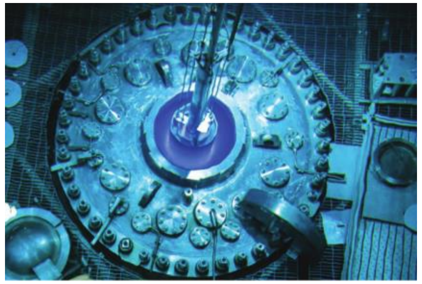
Neutron Sources around the world

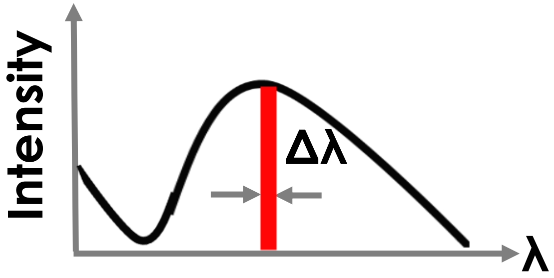


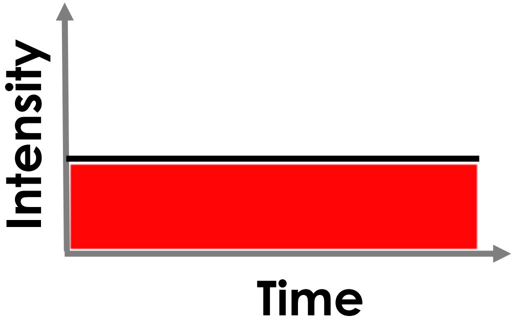
- Most intense pulsed neutron beam in the world. (60Hz, 1.4 MW)
- Highest power research reactor in the world (80 MW)

Neutron sources:
Reactor (CW) and Spallation (TOF)

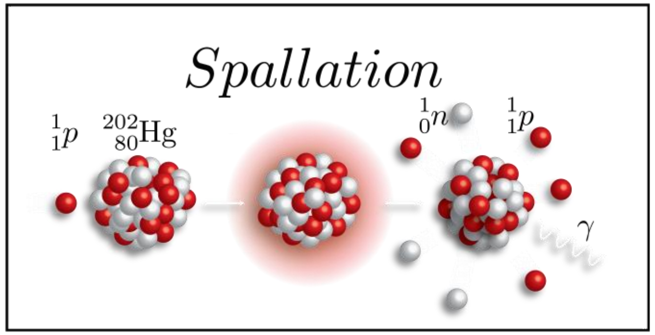


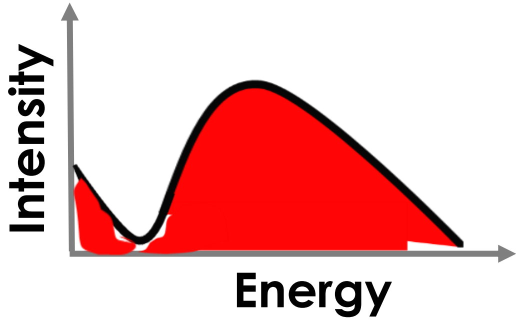




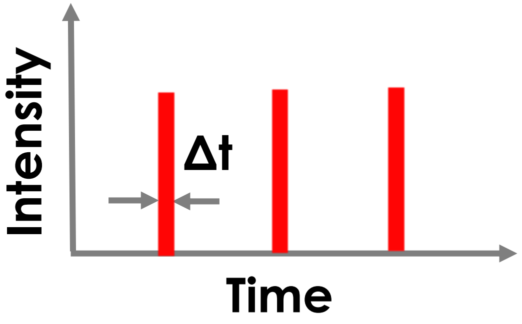


Small Δλ used, but source on all the time



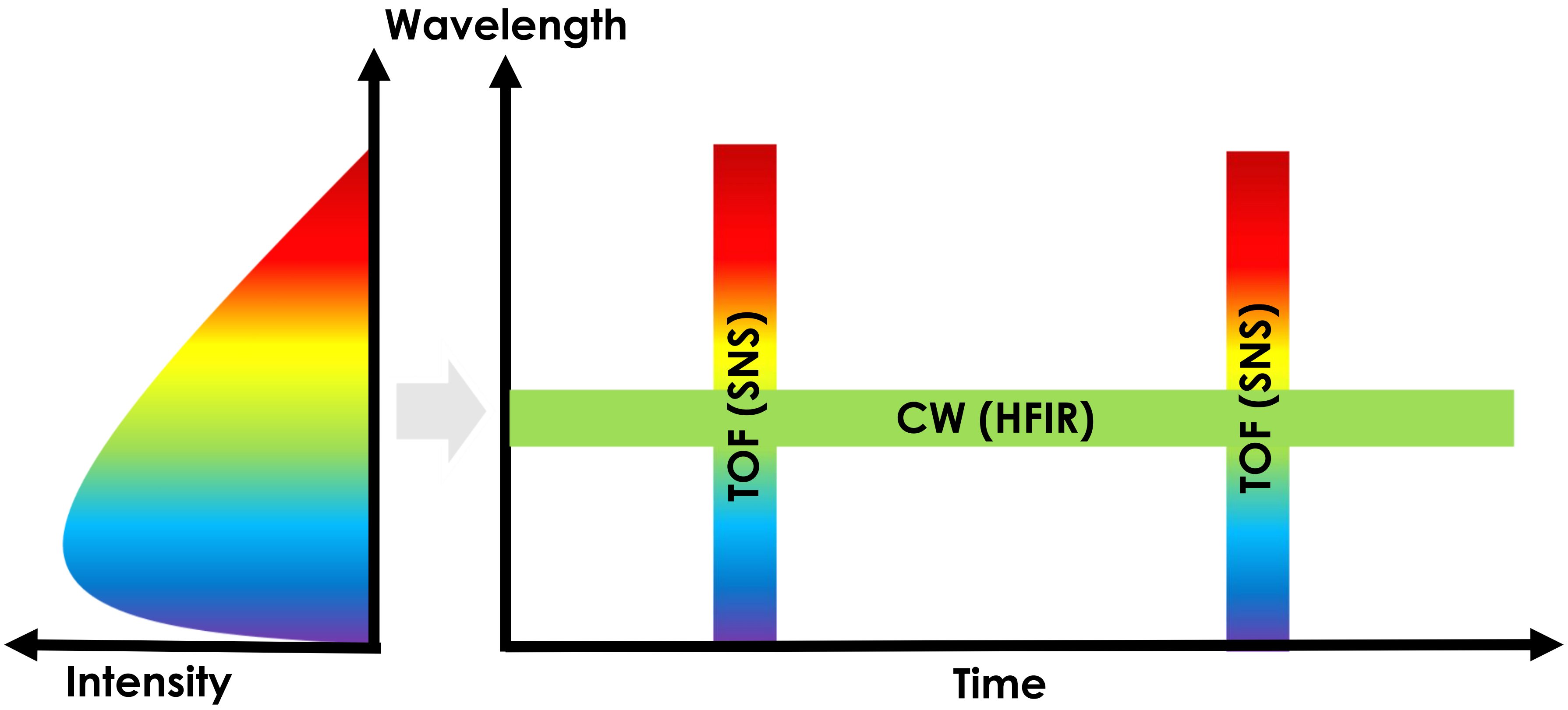


Each pulse of neutrons contains a broad spectrum of energy (λ)



Pulse of neutrons ~60 times per second

How do you like your neutrons?

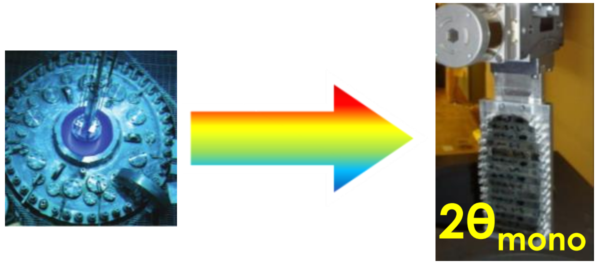


TOF (SNS):
All of the neutrons some of the time?

CW (HFIR):
Some of the neutrons all of the time?

What does this mean for diffraction experiments?

A typical CW diffractometer (HFIR)



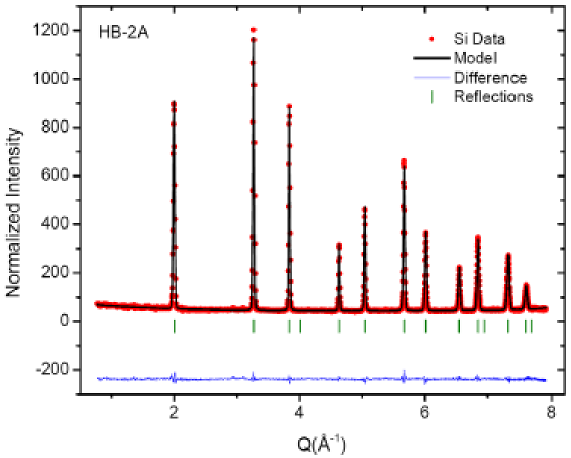
Monochromator:
- **selects λ.**
- **Control resolution.**

Single wavelength

Detector at 2θ measures single Q

$\lambda = 2d \sin\theta$

Q range limit is 4π/λ

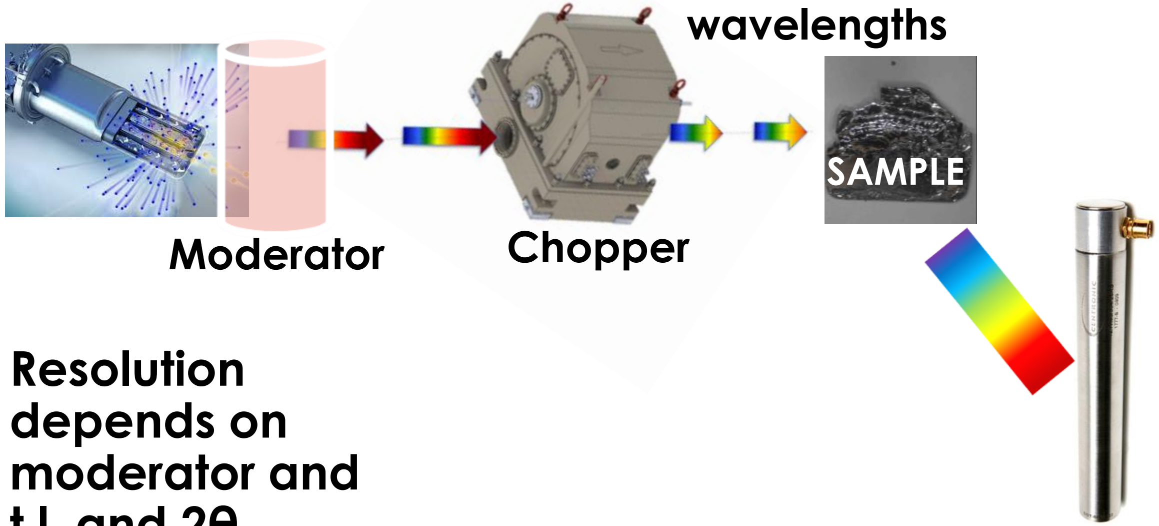


A typical TOF diffractometer (SNS)

De Broglie wavelength: $\lambda = h/mv = ht/mL$

Bragg's law: $\lambda = 2d \sin\theta = \text{constant} * t$

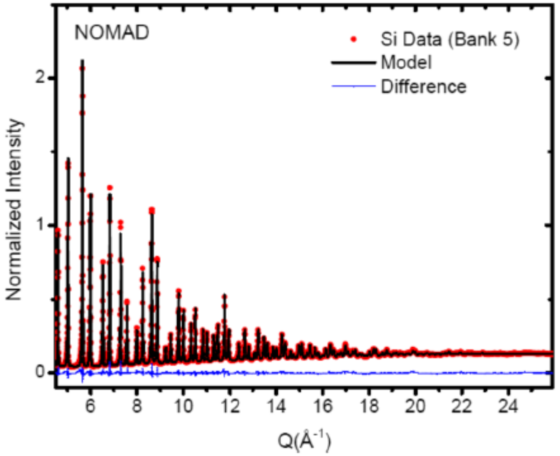
Multiple wavelengths



Moderator **Chopper**

Resolution depends on moderator and t,l, and 2θ

Q range determined by λ_{min}, λ_{max} and 2θ



Detector at 2θ measures multiple Q

Factors to consider for a diffraction experiment

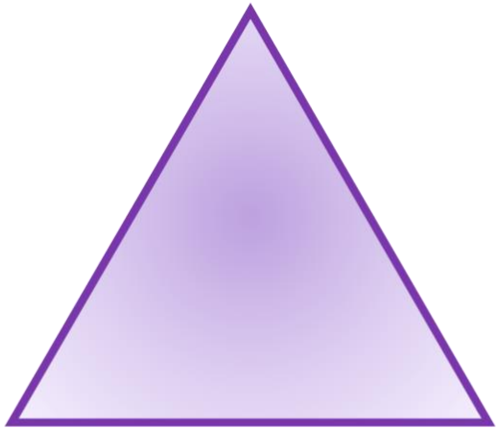
Intensity (CW)

- Higher flux
- Tune intensity with monochromator, collimators

Intensity (TOF)

- Higher peak brightness
- Balance of instrument distance, bandwidth, coverage

Intensity



Q-resolution

Q-range

Q-resolution (CW)

- Tunable over Q-range
- Simple, symmetric peak shape function

Q-resolution (TOF)

- High resolution
- Complex asymmetric peak shape related to moderator characteristics

Q-range (TOF)

- Wide Q coverage
- Q_{max} can be very high (>100 Å^{-1})
- Good coverage with even limited detectors

Q-range (CW)

- Q range limited, but tunable
- Highest Q_{max} (~20 Å^{-1})
- Need wide/continuous detector coverage

What are the strengths for diffraction experiments?

A typical CW diffractometer

Strengths

- High flux beam
- Simple beam profile corrections
  - Absorbing, attenuating samples and holders
- Open instruments with variable sample environment
- Tunable resolution and range
- Beam is always on

A typical TOF diffractometer

Strengths

- Widest Q-range
- Resolution high over wide range
- High peak brightness
- Pump probe measurements
- Count rate
  - SNS instruments historically have more detector coverage

Sample Environment considerations

Other factors

- An instrument is only useful if the sample is measured under the desired conditions.

https://neutrons.ornl.gov/sample

Equipment

- Low Temperature & Magnetic Fields
- High Temperature & Engineering Materials
- High Pressure & Gas Handling
- Special Environments

To only show available items for specific beamline, use this filter.

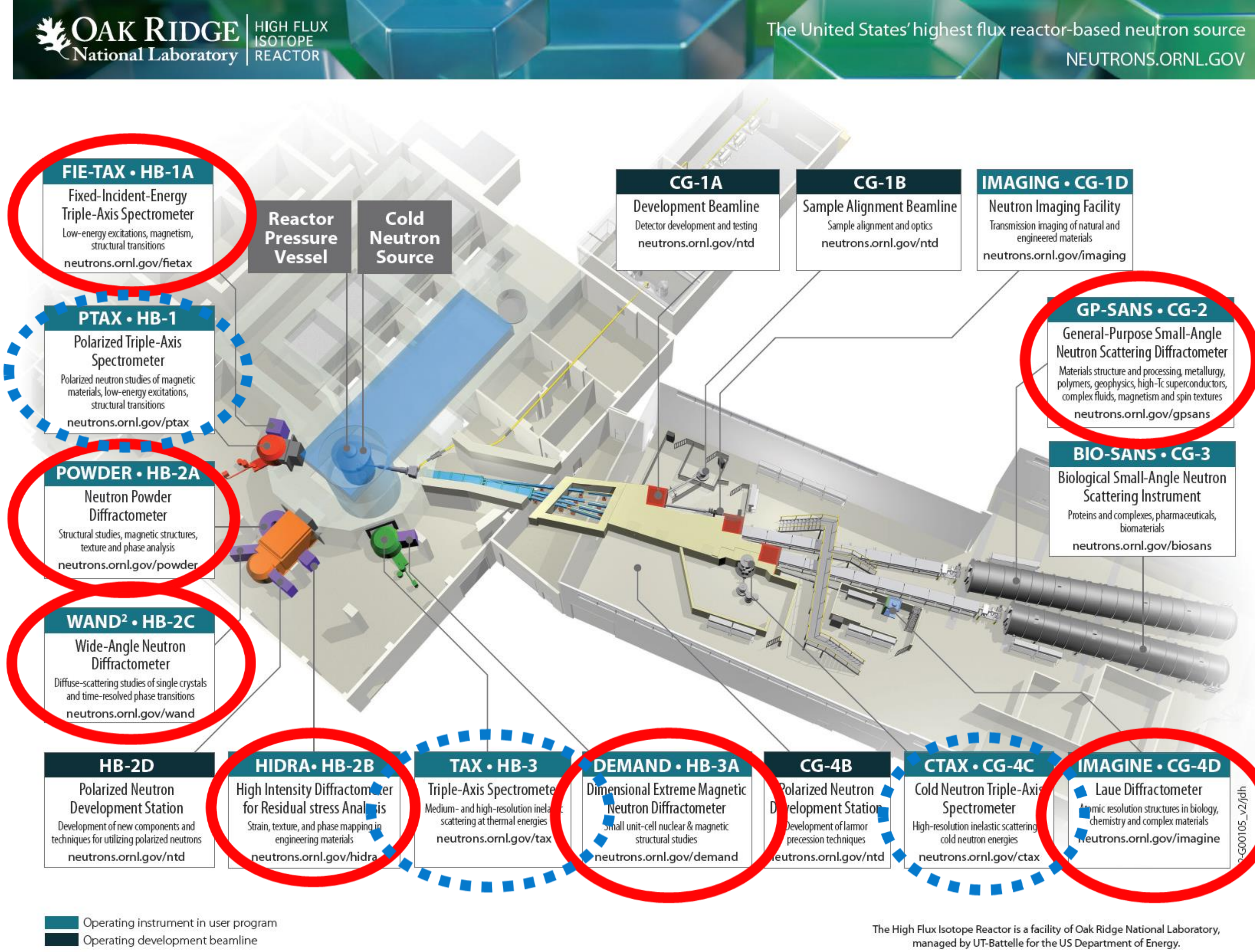
HB-2A

ID	Sample Space Dia.	Temp Range	Instruments	Associated Resources	Subcategory	Description
ULT-E	40mm	0.03 to 300 K	CG-2, CG-3A, CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3	CRYO-A, CRYO-C, CRYO-O, MAG-B, MAG-E, MAG-G	Insert	Dilution Refrigeration Insert
ULT-G	40mm	0.3 to 300 K	CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3, HB-3A	CRYO-A, CRYO-C, CRYO-N, CRYO-O, MAG-B, MAG-I	Insert	Helium-3 Insert
ULT-H	40mm	0.03 to 300 K	CG-2, CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3, HB-3A	CRYO-A, CRYO-C, CRYO-N, CRYO-O, MAG-B, MAG-E, MAG-G, MAG-I	Insert	Dilution Insert
ULT-J	60mm	0.03 to 300 K	CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3	CRYO-D, CRYO-J, CRYO-K, CRYO-L, CRYO-M	Insert	Dilution Insert
ULT-K	40mm	0.3 to 300 K	CG-2, CG-4C, HB-1, HB-1A, HB-2A	CRYO-A, CRYO-N, CRYO-O, MAG-E, MAG-G, MAG-I	Insert	Helium-3 Insert
ULT-M		0.3 to 300 K	HB-2A, HB-2C, HB-3, HB-3A		Bottom Loading	

Variety of optimized diffraction instruments at SNS/HFIR

HFIR (CW)

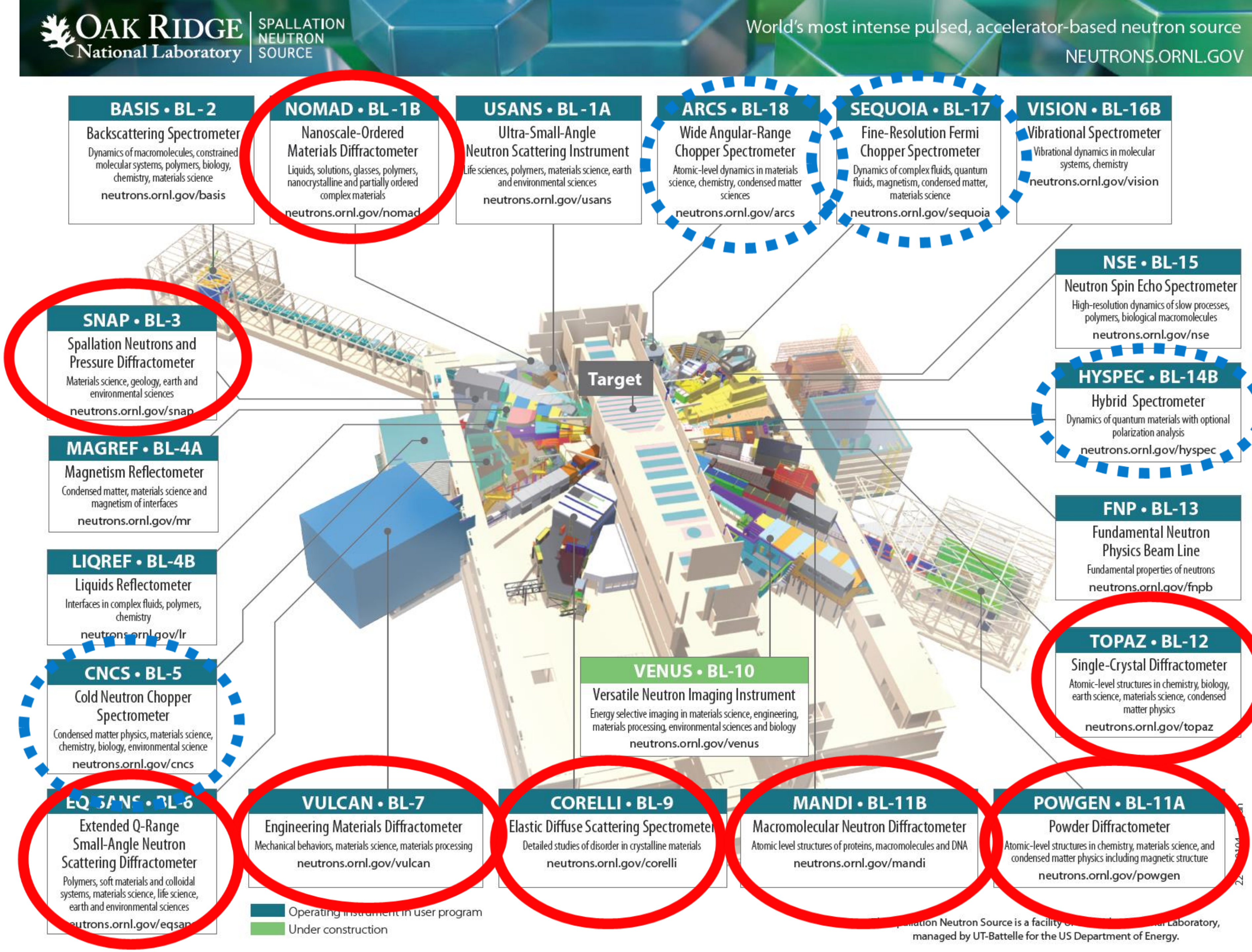
- HB-2A
- DEMAND
- IMAGINE
- WAND^2
- HB-1A
- HIDRA
- GP-SANS
  - HB-1
  - HB-3
  - CTAX



SNS (TOF)

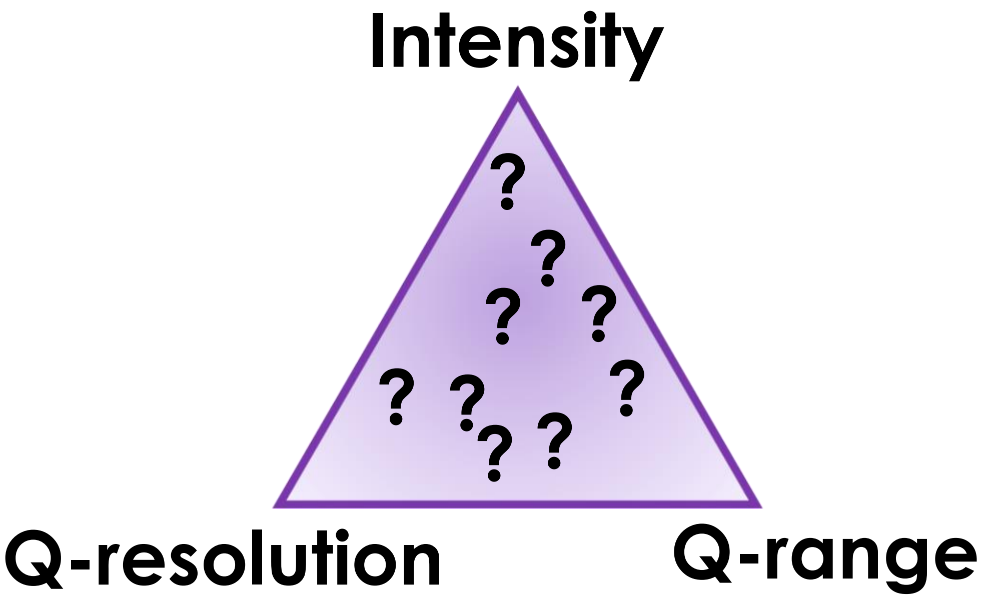
- POWGEN
- NOMAD
- TOPAZ
- CORELLI
- MANDI
- SNAP
- VULCAN
- EQ-SANS

  - HYSPEC
  - ARCS
  - SEQUOIA
  - CNCS



Is diffraction best at CW or TOF instruments?

- It depends on the question to be answered in your science!



Other factors
Sample environment
Polarization
Pump-probe
Source stability
Sample absorption
…

Consider some common questions diffraction can answer and match to strengths of sources

- Measure nuclear structure
- Measure magnetic structure
- Measure disorder in structure
- … Lots more cases for you to consider!

Measure a crystalline structure

Need

- To collect lots of reflections

Instrument should have

- High Q-resolution **(TOF/CW)**
- Large Q-range **(TOF)**
- High Q **(TOF)**

- **TOF is typically best, especially for powders.**
- **Single crystal: TOF for largest unit cells, but CW can work well.**

Other factors?

Specialized SE

TOF/CW

Absorbing, H

- **CW easier**
- **TOF feasible**

Specific Q region

TOF/CW

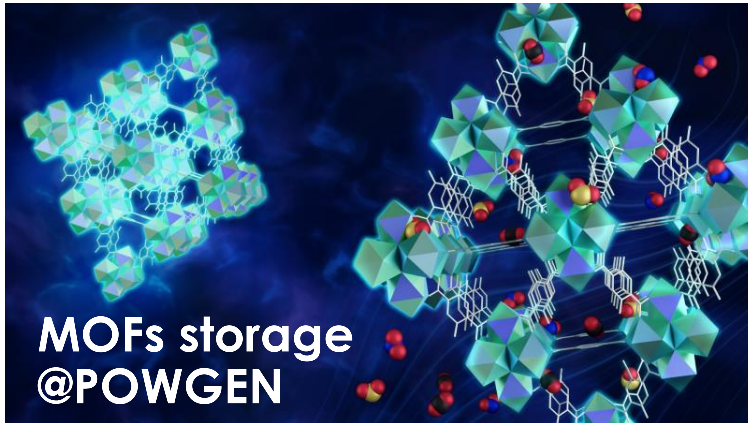
Small sample

TOF/CW

Measure the crystal structure

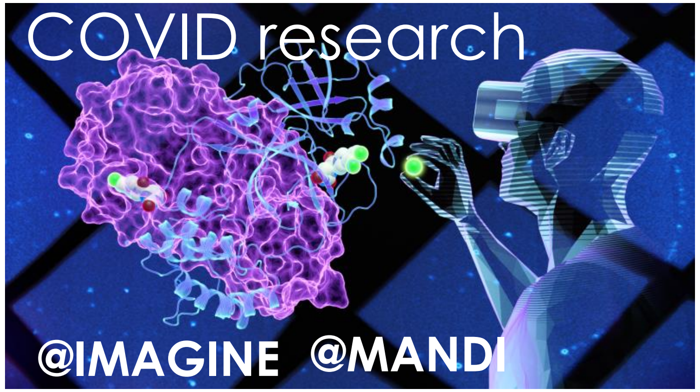
Instruments at HFIR/SNS

- Powder
  - **POWGEN (TOF):** High resolution and Q coverage
  - **NOMAD (TOF):** High flux and Q coverage
  - **HB-2A (CW):** Smaller unit cell, complex SE, absorbing samples.



- Single crystal
  - **DEMAND (CW):** Smaller unit cell inorganic materials
  - **TOPAZ (TOF):** High resolution and coverage for inorganic/organic and larger structures
  - **IMAGINE (CW):** Quasi-Laue for macromolecules
  - **MANDI (TOF):** Protein crystallography

DEMAND (10^4 Å^3)
TOPAZ (10^5 Å^3)
IMAGINE (10^6 Å^3)
MaNDi (10^7 Å^3)



Measure the Magnetic structure

Need
- To measure where magnetic scattering is strongest.
- Good signal to noise.
- Variety of sample conditions.

Instrument should have
- Low Q **(CW/TOF)**
- High intensity **(CW)**
- Low background **(CW/TOF)**
- Low T and magnets **(CW)**

- **CW is typically best, but TOF has ever increasing options.**

Other factors?

Short-range order

TOF/CW Consider low Q or mPDF options

Specialized SE

CW/ TOF

Polarization

CW

Absorbing, H

- **CW easier.**
- **Consider TAS.**
- **TOF feasible**

Specific Q region

TOF/CW
- **TOF: If need both high/low**

CW:
- **One region**

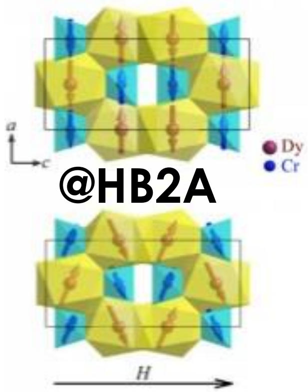
Small sample

TOF/CW

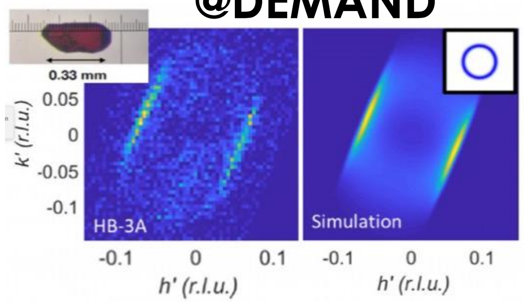
Measure the magnetic structure

Instruments at HFIR/SNS

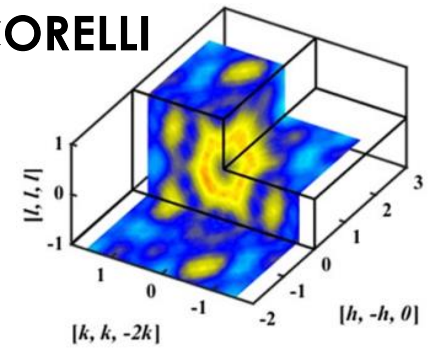
- Powder
  - **HB-2A (CW):** Access low Q. Low Temperature and magnets. Polarization.
  - **POWGEN (TOF):** High resolution and Q coverage.
- Single crystal
  - **DEMAND (CW):** Low Q coverage and variety of sample environments. Polarization.
  - **TOPAZ (TOF):** Wide coverage in Q. New 5K option.
  - **CORELLI (TOF):** Diffuse scattering. Variety of sample environments.
  - **GP-SANS (CW):** Very low Q for large spin textures (e.g. Skyrmions).
- Both Powder and single crystal
  - **WAND² (CW):** High flux → Long range and Diffuse signals
  - **HB-1A (CW):** Excellent signal-to-noise for weak signal



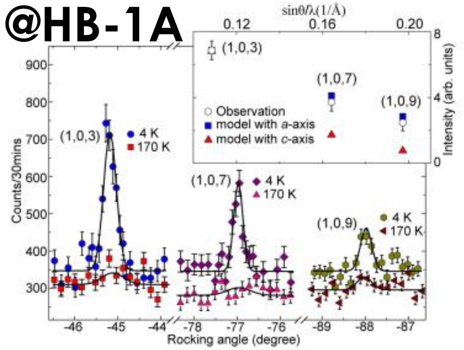
@DEMAND



@CORELLI



@HB-1A



Disordered materials (PDF)

Need

- To access high Q and broad scattering

Instrument should have

- Wide Q-range **(TOF)**
- High Q_{max} **(TOF)**
- High intensity **(TOF/CW)**

- **TOF is typically best, especially for powders.**
- **But CW can work well.**

Other factors?

Specialized SE

TOF/CW

Absorbing, H

- **Hard for powders**

Small sample

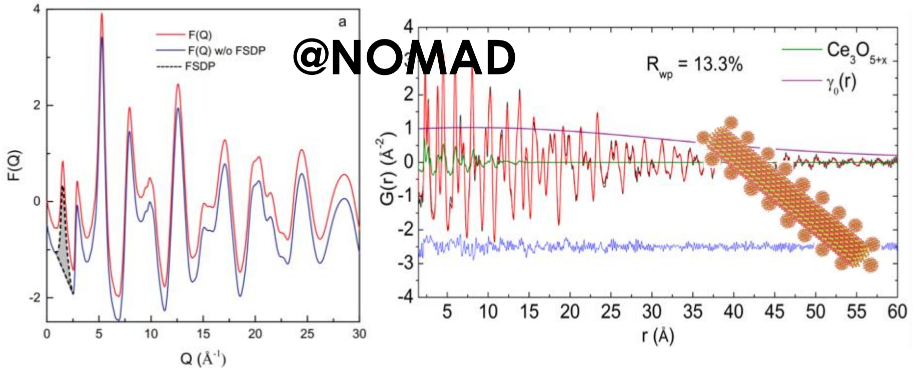
TOF/CW

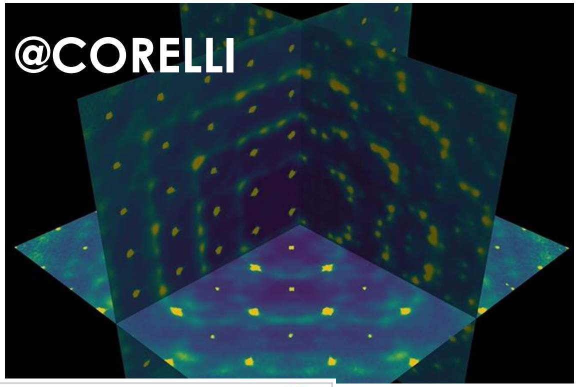
Disordered material (PDF)

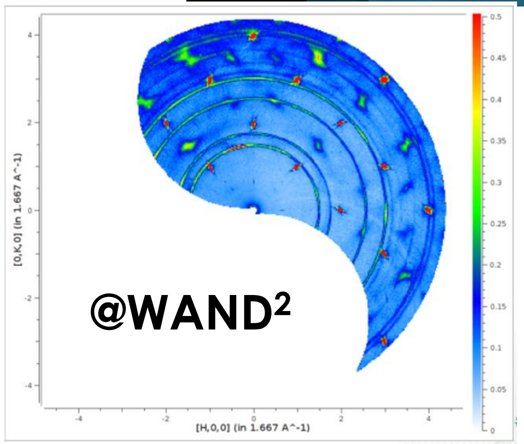
<u>Instruments at HFIR/SNS</u>

- Powder
  - **NOMAD (TOF):** Dedicated total scattering beamline.
  - **POWGEN (TOF):** Longer counting, but better resolution if needed.
  - **SNAP (TOF):** Pressure PDF
  - **WAND² (CW):** Potential for PDF and mPDF

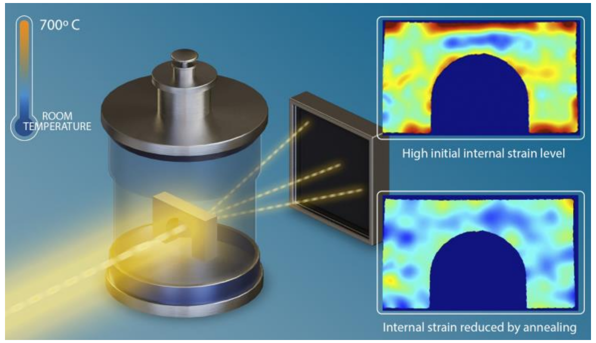
- Single crystal
  - **CORELLI (TOF):** Dedicated diffuse scattering beamline
  - **TOPAZ (TOF):** Large reciprocal space coverage
  - **WAND² (CW):** High flux and variety of sample environments



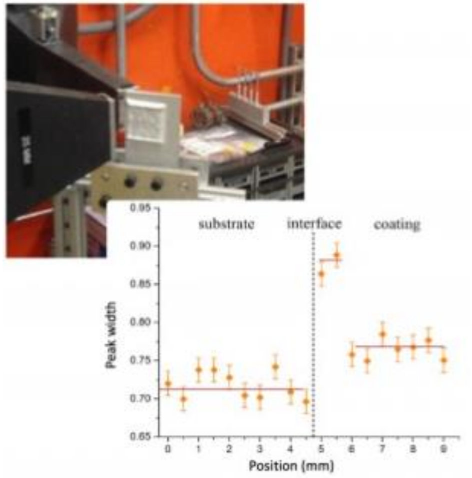




Many more diffraction experiments



Engineering diffraction
Measure strain/stress



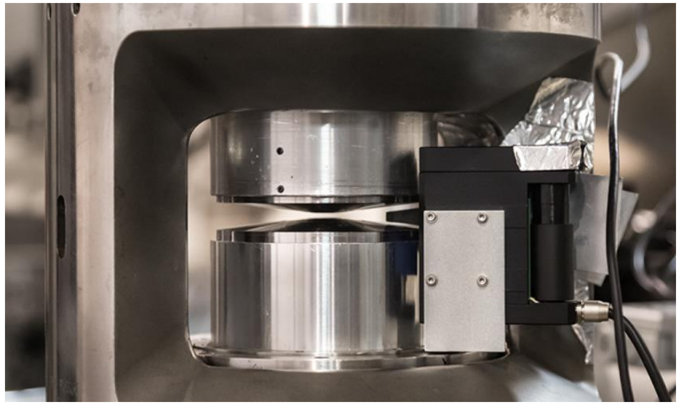
VULCAN (TOF)

- **In situ loading studies of crystalline/amorphous materials at high temperatures: phase transformation, fatigue damage, creep behaviors, and other deformation mechanisms in nanostructured materials, piezoelectric and shape-memory alloys.**

HIDRA (CW)

- **Optimized for strain measurement and determination of residual stress in engineering materials.**
- **Spatial resolution at a fraction of a millimeter is possible**

Many more diffraction experiments



High pressure diffraction

SNAP (TOF)

- Dedicated instrument for high pressure >100GPa.
- Powders and single crystals.
- Wide Q-coverage
- PDF options

WAND^2, DEMAND, HB-2A, TAS (CW)

- Variety of options for pressure measurements.

Review articles for the Diffraction Suite

REVIEW OF SCIENTIFIC INSTRUMENTS **89**, 092802 (2018)

A suite-level review of the neutron single-crystal diffraction instruments at Oak Ridge National Laboratory

L. Coates,[1,a)] H. B. Cao,[1] B. C. Chakoumakos,[1] M. D. Frontzek,[1] C. Hoffmann,[1] A. Y. Kovalevsky,[1] Y. Liu,[1] F. Meilleur,[1,2] A. M. dos Santos,[1] D. A. A. Myles,[1] X. P. Wang,[1] and F. Ye[1]

[1]*Neutron Scattering Division, Oak Ridge National Laboratory, 1 Bethel Valley Road, Oak Ridge, Tennessee 37831, USA*
[2]*Department of Molecular and Structural Biochemistry, North Carolina State University, Raleigh, North Carolina 27695, USA*

(Received 26 March 2018; accepted 9 July 2018; published online 24 September 2018)

The nascent suite of single-crystal neutron diffractometers at the Oak Ridge National Laboratory has no equal at any other neutron scattering facility worldwide and offers the potential to re-assert single-crystal diffraction using neutrons as a significant tool to study nuclear and magnetic structures of small unit cell crystals, nuclear structures of macromolecules, and diffuse scattering. Signature applications and features of single-crystal neutron diffraction are high resolution nuclear structure analysis, magnetic structure and spin density determinations, contrast variation (particularly D_2O/H_2O) for nuclear structural studies, lack of radiation damage when using crystals of biological molecules such as proteins, and the fidelity to measure nuclear and magnetic diffuse scattering with elastic discrimination.
https://doi.org/10.1063/1.5030896

REVIEW OF SCIENTIFIC INSTRUMENTS **89**, 092701 (2018)

A suite-level review of the neutron powder diffraction instruments at Oak Ridge National Laboratory

S. Calder,[1,a)] K. An,[1] R. Boehler,[1,2] C. R. Dela Cruz,[1] M. D. Frontzek,[1] M. Guthrie,[3,4] B. Haberl,[1] A. Huq,[1] S. A. J. Kimber,[1] J. Liu,[1] J. J. Molaison,[1] J. Neuefeind,[1] K. Page,[1] A. M. dos Santos,[1] K. M. Taddei,[1] C. Tulk,[1] and M. G. Tucker[1]

[1]*Neutron Scattering Division, Oak Ridge National Laboratory, 1 Bethel Valley Rd., Oak Ridge, Tennessee 37831, USA*
[2]*Geophysical Laboratory, Carnegie Institution of Washington, Washington, District of Columbia 20015, USA*
[3]*European Spallation Source, Lund 221 00, Sweden*
[4]*University of Edinburgh, Edinburgh EH8 9YL, United Kingdom*

(Received 6 April 2018; accepted 19 July 2018; published online 28 September 2018)

The suite of neutron powder diffractometers at Oak Ridge National Laboratory (ORNL) utilizes the distinct characteristics of the Spallation Neutron Source and High Flux Isotope Reactor to enable the measurements of powder samples over an unparalleled regime at a single laboratory. Full refinements over large Q ranges, total scattering methods, fast measurements under changing conditions, and a wide array of sample environments are available. This article provides a brief overview of each powder instrument at ORNL and details the complementarity across the suite. Future directions for the powder suite, including upgrades and new instruments, are also discussed. https://doi.org/10.1063/1.5033906

Conclusions

- ORNL is unique in having world class CW and TOF sources.
- HFIR and SNS diffraction instruments have different strengths.
- Choose the best instrument for your experiment based on your science.

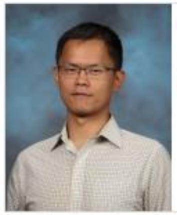
Lots of good options for diffraction at HFIR/SNS!

ASK AN INSTRUMENT SCIENTIST!!!

https://neutrons.ornl.gov/suites/diffraction


Stuart Calder
POWDER


Andrey Kovalevsky
IMAGINE, MANDI


Cheng Li
NOMAD, POWGEN


Clarina dela Cruz
POWDER


Keith Taddei
POWDER, WAND^2


Joerg C. Neuefeind
NOMAD


Dean Myles
IMAGINE, MANDI


Yan Chen
VULCAN


Thomas Proffen
POWGEN


Jue Liu
NOMAD

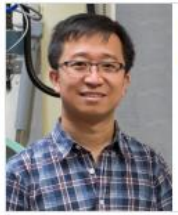

Qiang Zhang
POWGEN


Alicia Manjón Sanz
POWGEN


Ke An
VULCAN

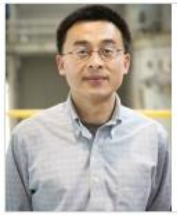

Xiaoping Wang
TOPAZ


Matthias Frontzek
WAND^2


Huibo Cao
DEMAND


Yan Wu
DEMAND, WAND^2


Bryan Chakoumakos
DEMAND


Feng Ye
CORELLI


Flora Meilleur
IMAGINE, MANDI


Chris Tulk
SNAP


Jeffrey Bunn
HIDRA


Andrew Payzant
HIDRA


Chris Fancher
HIDRA


António M. dos Santos
SNAP


Christina Hoffmann
CORELLI, TOPAZ

Questions?

Lots of good options for diffraction at HFIR/SNS!

Experiments with TOF & steady-state neutrons - Stuart Calder and Garrett Granroth

https://forms.office.com/g/zydFT6CTjU