Neutron Instrumentation

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Canadian Neutron Beam Centre

12th International Neutron Scattering Summer School
Chalk River, Ontario, Canada
Topics (a.k.a. Zahra’s Challenge)

- Selection of neutron energy and its resolution (choppers, monochromators, spin-echo, backscattering, fast/thermal neutron filters, collimations)
- Neutron optics (guide tubes, supermirrors)
- Neutron detectors (gas, 3He, scintillation, semiconductors, 2D detectors, neutron beam monitors)
- Methods for enhancing flux and reducing measurement time (focusing monochromator/analyzer, multi-analyzers, position sensitive detectors, converging guides/collimators, etc...)
- Extreme sample environments (high pressure, high magnetic field, low temperatures)
- A few examples of new instruments around the world
Anatomy of a Neutron Scattering Experiment

Neutron scattering measurements are about determining how the interaction with the sample has affected the neutrons (neutron beams) to learn about the sample.

- What do we need to do?
  1. Source of neutrons of high enough flux
  2. A way of knowing the wavevector ($k_i$) of incident neutrons
  3. A sample (the motivation for the experiment)
  4. A way of knowing the wavevector ($k_f$) of scattered neutrons
  5. Neutron detectors (for 2 and 4)
Why Not Just Build a Universal Neutron Scattering Instrument That Can Do Everything We Need?

**Incident Beam** – know $k_i$ of the incident neutron beam

**Scattered Beam** – know $k_f$ of all scattered neutrons beams

Result: *Happy Camper*
Why Not Just Build a Universal Neutron Scattering Instrument That Can Do Everything We Need?

Incident Beam – know $k_i$ of the incident neutron beam

Scattered Beam – know $k_f$ of all scattered neutrons beams

Diffraction Quasi-Elastic Inelastic Reflectometry SANS Imaging $\mu$-Beam Stress Texture Holography H

If you remember only one theoretical equation from this Summer School, make it **Bragg’s Law**.

$$n\lambda = 2d \sin(\theta)$$

Triple Axis Spectrometer is the universal instrument.
Why Not Just Build a Universal Neutron Scattering Instrument That Can Do Everything We Need?

**Incident Beam** – know $k_i$ of the incident neutron beam

**Scattered Beam** – know $k_f$ of all scattered neutrons beams

The energy of the neutron is coupled to its wavelength and velocity:

$$\lambda^2 = \frac{81.81}{E} \quad \text{and} \quad v^2 = 191313 \cdot E$$

$S(Q, \omega)$ = scattering properties of the sample depend only on $Q$ and $\omega$, not on the neutron wavelength ($\lambda$)

**Message**: Many different types of neutron scattering instruments are needed because the accessible $Q$ and $\omega$ ranges depend on the neutron energy and because the resolution and detector coverage have to be tailored to the science for such a *signal-limited* technique.
Types of Neutron Scattering Instruments

- Elastic Scattering Instruments
  - Powder diffraction
  - Single Crystal diffraction
  - Stress & Texture
  - SANS (typical)
  - Reflectometry

Used to determine the average structure of materials (How the atoms are arranged).

*Based on diagram by Roger Pynn*
Making Choices
Continuous Source

- Often select \( k_i \) and measure \( k_f \)
  - Diffraction : \( |k_i| = |k_f| \)
  - Need to define/determine directions
  - Need to choose suitable \( \lambda (E) \)

\[
k = \frac{2\pi}{\lambda}
\]

\[
E = \frac{\hbar^2 k^2}{2m} \quad E = \frac{\hbar^2}{2m\lambda^2}
\]
Distance Collimation

Basically all collimators use distance collimation where the solid angle of neutrons that emerge is defined by the length of the collimator and dimensions of the front & back apertures. All other neutrons are lost.
Bragg’s Law, relates neutron wavelength (λ) to crystal lattice plane spacing (d) and scattering angle, 2θ:

\[ \lambda = 2d \sin \theta \]

or

\[ v = \frac{h}{2md \sin \theta} \]

From John Root (CNBC)
Defining the Initial Energy

- Choppers (TOF)
- Velocity selectors

Tuneable velocity and tuneable range of velocity (bandwidth) e.g. ~10%

*Only neutrons within the right velocity range can pass between the neutron-absorbing blades of a spinning turbine (velocity selector).*

From John Root (CNBC)
Making Choices (examples)

Continuous Source

- **Stress Mapping (known structure)**
  - **Want** $\lambda$ comparable to interatomic spacings, need separate peaks, need to detect shifts in peak position to determine strain to $10^{-4}$!

- **Typical steps**
  - Select sample $\{hkl\}$ or $d$ (guided by material)
  - Determine scattering geometry, e.g. $2\theta$
  - Bragg’s Law tells us $\lambda$
  - Use Ge or Si monochromator with moderate mosaic spread, high reflectivity, $(hkl)$ without higher order reflections e.g. $(113)$
Making Choices (examples)

Continuous Source

- **Powder Diffraction (structure determination)**
  - *Want* $\lambda$ comparable to interatomic spacings, need to resolve peaks, need enough peaks to determine structure, clean, may need speed to follow transients

- **Typical steps:**
  - Consider probable structures (low or high symmetry), phases, *i.e.* range of $d$-spacings
  - Short or long $\lambda$ to sufficiently separate peaks but have enough
  - Clean to avoid higher-order overlap
  - Use Ge or Si monochromator, *maybe filter*
Cleaning Up The Beam

Filters are used to select out unwanted neutrons

Beryllium & Sapphire filters transmit all neutrons above certain $\lambda$

$\lambda^2 = \frac{81.81}{E}$

Reduces background by removing fast & epithermal neutrons and $\gamma$
Cleaning Up The Beam

Filters are used to select out unwanted neutrons

Pyrolytic Graphite (PG) filters have systematic attenuation at various $\lambda$. Passes $\lambda$, but filters $\lambda/2$ and $\lambda/3$ @ 2.37, 1.55 and 1.49 Å

$\lambda^2 = 81.81/E$
Making Choices (examples)

Continuous Source

- **SANS**
  - **Want** $\lambda$ comparable typical spacings or structures, typically need a small beam so high intensity, need to resolve small angles
  - **Typical steps:**
    - Choose largest length scale you want to see, $L_{\text{max}} = 2\pi/Q_{\text{min}}$
    - Trade off between choices of $\lambda$ and $\min. \theta$ to get $Q_{\text{min}}$
    - Can sacrifice $E$ resolution to boost intensity while paying little price in $Q$ resolution, $\therefore$ use velocity selector

\[
Q = \frac{4\pi}{\lambda} \sin\left(\frac{\theta}{2}\right)
\]
Types of Neutron Scattering Instruments

- Inelastic Scattering Instruments
  - Direct Geometry TOF Spectrometers
  - Indirect Geometry TOF Spectrometers
  - Triple-Axis Spectrometers
  - Backscattering Spectrometers
  - Neutron Spin-Echo Spectrometers

Used to study dynamics (phonons, magnons, diffusion, …)

Based on diagram by Rex Hjelm
Making Choices
Continuous Source

• Often select $k_i$ and measure $k_f$
  
  • Inelastic: $|k_i| \neq |k_f|$

  \[ k = \frac{2\pi}{\lambda} \]

  \[ E = \frac{\hbar^2 k^2}{2m} \quad E = \frac{\hbar^2}{2m\lambda^2} \]

  • Need to define/determine directions (4 of them on triple axis)
  
  • Need to choose suitable $\lambda (E)$
Bragg’s Law, relates neutron wavelength ($\lambda$) to crystal lattice plane spacing ($d$) and scattering angle, $2\theta$: 

\[ \lambda = 2d\sin\theta, \text{ or } \nu = \frac{h}{2md\sin\theta} \]

Adapted From John Root (CNBC)
Making Choices (example)

Continuous Source

- Phonons
  - Want $\omega$ comparable to excitations, to separate phonon modes, to span a broad range of $Q$ and $\omega$

- Typical steps
  - Consider the $q$ and $\omega$ of interest and $Q$, the implications due to:
    - Resolution ellipse
    - Incident energy
    - Scanning modes
    - Nuclear versus magnetic scattering
Brockhouse’s triple-axis spectrometer allows one to vary $q$ and $\omega$ independently.

- Constant-Q allows a scan in energy at fixed wave vector.
- Constant-$E$ allows a scan in wave vector at fixed energy.
- Can vary $E_i$ or $E_f$ or both.

As the instrument’s resolution function passes through the phonon dispersion curve, a phonon peak is observed.
Coherent Inelastic Scattering

- Energy transfer between neutron and crystal (e.g. phonons)
- Must conserve energy and momentum
  - $E_i - E_f = \hbar \omega$
  - $k_i - k_f = \tau + q$ (q wave vector of the vibration)

\[
\left(\frac{d\sigma}{d\Omega d\omega}\right)_{coh+1} \propto \frac{k_f}{k_i} \sum_s \sum_{\tau} A_s \langle n_s + 1 \rangle \delta(\omega - \omega_s) \delta(Q + q - \tau)
\]

\[
\left(\frac{d\sigma}{d\Omega d\omega}\right)_{coh-1} \propto \frac{k_f}{k_i} \sum_s \sum_{\tau} A_s \langle n_s \rangle \delta(\omega + \omega_s) \delta(Q - q - \tau)
\]

- More signal when you create phonons
- Fix $k_f$ and scan $k_i$ in Constant Q
- $\delta$-functions indicate you get coherent scattering only when:
  - $\omega = \omega_s$ and $Q = \tau + q$
Methods for Inelastic Scattering

xtal – xtal (triple-axis)

source

2θ

sample

 detector

plane spacing $d_{c2}$

$θ_{B2}$

plane spacing $d_{c1}$

TOF – TOF (direct-geometry)

chopper (open briefly at time $t_c$)

detected at time $t_d$

pulsed source

2θ

sample

detector

$pulse at time t_0$

$L_1$

$L_3$

$L_2$

xtal – TOF

chopper (open briefly at time $t_c$)

sample

detector

$pulse at time t_0$

$L_1$

$L_3$

$L_2$

source

$θ_B$

plane spacing $d_c$

TOF – xtal (indirect geometry)

pulsed source

$L_1$

$L_3$

$L_2$

sample

detected at time $t_d$

Each has their advantages and disadvantages

From Kent Crawford (ORNL)
What About That Universal Instrument?

**Message:** Many different types of neutron scattering instruments are needed because the accessible $Q$ and $\omega$ ranges depend on the neutron energy and because the resolution and detector coverage have to be tailored to the science for such a **signal-limited** technique.

Many of the previous examples mentioned high intensity. Indeed all experiments want high intensity. It allows for smaller samples, faster data collection, better statistics...

**Why are we signal limited, can we do better?**
Liouville's Theorem

- In the geometrical-optics the propagation of neutrons can be represented as trajectories in a six-dimensional phase space \((p, q)\), where the components of \(q\) are the generalized coordinates and the components of \(p\) are the conjugate momenta. Uh?

- Simply stated: Liouville's Theorem says that phase space volume is conserved. Oh...

- Translation: It costs flux to increase resolution and it costs resolution to increase flux. Ah... crap.
Neutron Guides

Guides transport neutrons from the source to distant instruments. Reduces background \((n^0 \& \gamma)\).

\[ m = 0.1 \text{ deg/Å} \]

- Straight / Curved
- Converging
- Elliptical
- Anti-Trumpet
- Multichannel
- Beam Splitting

From Lee Robertson (ORNL)

Guide Installation at ISIS

Multichannel Curved Guide
*Fabricated by Swiss Neutronics*
Polarizers and Spin Manipulators

Heussler Monochromator, AlCuMn

Larmor Precession Flipper

Polarizing Supermirrors

\[ \text{\^{3}He Cell} \]

Unpolarized Neutron Beam

Polarized Neutron Beam

\[ \text{\^{3}He Spin Filters} \]

Spherical Neutron Polarimetry

POLI-HEiDi at FRMII

From Lee Robertson (ORNL)
Neutron Optics: Focusing

The radius of the monochromator has to be changed for different wavelength since the distances are fixed:

\[ R_v = \frac{2L_0 L_1 \sin \theta}{L_0 + L_1} \]

From Lee Robertson (ORNL)

Double focusing “Popovici” monochromator. The vertical curvature is fixed while the horizontal curvature is variable by bending stacks of thin silicon wafers. The gain is achieved both by spatial focusing and ‘wavelength focusing’.

Virtual Source

![Diagram of Virtual Source with Small Aperture, Mono, and Sample]
Neutron Optics: Focusing

- Focusing Mirrors:
  - Develop a nested advanced KB mirror system to make a more compact assembly and to achieve the highest performance.
  - Identify applications where focusing optics can replace neutron guides and offer better performance.

From Lee Robertson (ORNL)
Neutron Optics: Focusing

Wolter Optics for focusing beams with large cross-sections
(Boris Khaykovich at MIT and Michael Gubarev at NASA)

Optical path for Wolter optics

An X-ray optic module with 12 nested mirrors

Image of a calibration Grating using the X-ray optic

From Lee Robertson (ORNL)
Neutron Detectors: $^3$He

- Approximately 75% of the detectors for neutron scattering use $^3$He
- These detectors are efficient, stable, low noise, have excellent gamma discrimination, and good timing
- Unfortunately they are becoming rare

ARCS Detector Array
930 $^3$He LPSD’s

Chalk River
32-wire $^3$He

From Lee Robertson (ORNL)
Neutron Detectors: Scintillators

- Wavelength-shifting fiber detector developed for powder diffraction applications
- Blue scintillation light is shifted to green and captured in the fibers
- Uses a $^6\text{LiF}/\text{ZnS}:\text{Ag}$ corrugated scintillator pressed to increase density
- $308 \times 152$ pixels – 5mm wide and 50mm tall

From Lee Robertson (ORNL)
Neutron Detectors: Anger Cameras

- The Anger camera optics package maps the scintillator (2mm GS20 Li glass) area to the 6mm x 6mm PMT anodes
- Fit measured light cone with a 2-gaussian function to determine neutron position (current resolution is 0.8mm)
- Developed for single crystal instruments

From Lee Robertson (ORNL)
Detectors: High Count Rates

- A new detector based on inclined plates of $^{10}$B. The neutron is captured and the ionization event is multiplied by the Gas Electron Multiplier (GEM).

- A new two-dimensional thermal neutron detector capable of very high rates is being developed (ORNL-BNL collaboration). It is based on neutron conversion in $^3$He in an ionization chamber that uses only a cathode and anode plane.

From Lee Robertson (ORNL)
Getting Better Energy Resolution

Back Scattering

Neutron Spin Echo

Concept is that only the energy transfer, $\omega$, is needed. A large range of incident neutron energies (e.g. 20%) can be used provided that the neutrons carry individual information of their energy. NSE use the neutron spin's Larmor precession as an internal clock to achieve that objective.

\[
\frac{\delta \lambda}{\lambda} = \frac{\delta d}{d} + \frac{\delta \theta}{\tan(\theta)}
\]
Topics (a.k.a. Zahra’s Challenge)

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- Extreme sample environments (high pressure, high magnetic field, low temperatures)
- A few examples of new instruments around the world

Highlights of recent CNBC developments...
**ψXYZ Sample Stage**

A new sample stage has been designed that will provide many improvements over the current system within the same footprint.

<table>
<thead>
<tr>
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<th>Old</th>
<th>New</th>
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<tbody>
<tr>
<td>ψ Load Capacity (tonne)</td>
<td>≥ 0.91</td>
<td>1.82</td>
</tr>
<tr>
<td>XY Load Capacity (tonne)</td>
<td>0.68</td>
<td>1.82</td>
</tr>
<tr>
<td>XY Travel (mm)</td>
<td>180 × 180</td>
<td>203 × 406</td>
</tr>
<tr>
<td>Z Travel (mm)</td>
<td>None *</td>
<td>100</td>
</tr>
<tr>
<td>Height below neutron beam (mm)</td>
<td>324</td>
<td>437</td>
</tr>
</tbody>
</table>

* Vertical lift was done with various linear translators ranging in travel from 100 to 350 mm, but all with a capacity of 50 kg or less so counter balancing of the load was often required.

A 2nd ψ-Z will be built for E3.
Our 32-wire $^3$He detector is now also available with a 300 mm active height. This will enable a 2.4-fold increase in count rate.

High $^{10}$B poly shielding to reduce background while allowing for tall beam channels.

- 32 wires
- 3 switchable vertical segments for software-controlled detector height

3 detector distances: 1016, 1397 & 1778 mm

Pitch, roll, & elevation adjustments, plus motorized radial & transverse positioning.

Tapered snout and channel allow for full performance increase.

Mounted laser dial gauge.

Incorporated Pb shields for studying active samples.
A new detector shielding drum for N5

Lower BG, high scattering angles (115° compared to 104°, more efficiency during change over between experiments.
Vertically focusing Heusler monochromator

Installed a new monochromator table needed for larger weight and shielding purposes

Preliminary FR tests: 15-20 for small beam (~1 cm²) & 10-15 for larger beams
A new 5-coil assembly for polarized NS at C5

Provides a vertical field and a horizontal field along any direction (parallel to Q or perpendicular to Q) automatically using spectrometer control program.
A top loading cryogen free variable temperature system, Lemon, is capable of reaching a continuous base temperature of 1.5 K. Top loading is into static exchange gas. The system uses a simple gas handling system to circulate and liquefy a volume of $^4$He gas in situ. Using the same probe and without manual interference temperatures as high as 800 K are also accessible. All this with a sample diameter as large as 70 mm.
Solidification Cell

Precipitate peaks appear
Servo-Hydraulic Load Frame & Accessories

Project* was funded to purchase an MTS servo-hydraulic load frame to enable measurements on samples under tensile, compressive, and cyclic loading.

Features:
- ±100 kN capacity
- 10 Hz operation (fatigue testing)
- 250 mm stroke
- Grip alignment system
- Hydraulic collet grips
- Acoustic emission system
- Strain gauge conditioning
- Extensometers (including high T)
- Control system

* Project received financial and written support from: NRC, other NRC institutes, UBC, McMaster, Queen’s, Ryerson, DRDC, AECL, and Cornell
IR Furnace
Heat treatment, solidification, quenching

- Fast heating / cooling
- Vacuum or controlled gas atmosphere
- Only quartz tube, crucible, and sample in the beam

15 mm 1018 steel slug Ar
Furnace cooling, forced Ar

IR Heaters used in conjunction with new high-temperature compression set up
Electronics Upgrade

- More user-friendly to install or interchange motors/encoders
- Faster configuration
- Reduced experimental set-up time
- More tidy

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<th><strong>Old</strong></th>
<th><strong>New</strong></th>
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<tbody>
<tr>
<td>Motors/Encoders</td>
<td>16, limited hardware control of parameters, required expert knowledge</td>
<td>32, user-friendly control of parameters</td>
</tr>
<tr>
<td>Remote Control</td>
<td>16 switches &amp; direction switch, priority set at rack</td>
<td>Selection knob, speed control, live display, print button, priority set on remote with automatic return to software control</td>
</tr>
<tr>
<td>Sample Environments</td>
<td>Limited to one environment control</td>
<td>Multiple environment controls will be possible</td>
</tr>
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</table>
Laser Scanner and SScanSS

1. Laser profile actual sample
2. Add fiducial points
3. Plan measurements in SSscanSS
4. Align instrument and sample using metrology system
5. Use Tracking feature to monitor
Control System: Current Version

- Single mainframe computer.
- Custom hardware for device control
- Custom software
- Use of unsupported and obsolete hardware/software
- Single, command line client
- Limited device expansion

Single Client

VAX computer

Devices
Control System: Next Generation

- Distributed client/server
- Modular: Clients, devices, servers can be added, removed, changed
- Off-the-shelf hardware: cheaper, easier to maintain
- Open-source software: make use of other smart peoples’ work
- Multiple clients: GUI, Web based, ...
- Easier to add new devices and device types
Key Messages

- The Brockhouse Triple Axis is nearly the “universal instrument” exists, but it cannot do everything well enough to be the only instrument.
  - Good place to try something novel.
- Neutron instruments worldwide are more and more specialized tuned to do certain things very well.
  - Pick the right instrument for your science.
- There are many kinds of instruments and too many details to know.
  - Unless your thesis advisor is a neutron scattering expert, interact with the facility experts. We are all happy to help you.
Acknowledgements

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  - Kent Crawford, ORNL
  - Lee Roberston, ORNL