Overview

• Why use neutrons?
• Neutron sources: A (very) brief history
• Theoretical concepts of neutron scattering (recap)
• Practical aspects of neutron scattering and refinements
• Where to perform experiments: Diffraction instruments at ORNL
Lots of references
How to get microscopic structural information

• 3 main choices → Diffraction has strong advantages

<table>
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<th>Advantages</th>
<th>Disadvantages</th>
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<td><strong>Microscopy</strong></td>
<td>Direct</td>
<td>Local information only</td>
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<tr>
<td>Optical, TEM, Field ion</td>
<td></td>
<td></td>
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<tr>
<td><strong>Scanning Probes</strong></td>
<td>Direct</td>
<td>Local information only</td>
</tr>
<tr>
<td>AFM, STM, SEM</td>
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<td>Surfaces only</td>
</tr>
<tr>
<td><strong>Diffraction Probes</strong></td>
<td>Quantitative data on correlations and distribution of structural features</td>
<td>Requires fitting</td>
</tr>
<tr>
<td>Electron X-ray Neutron</td>
<td>Probes entire sample</td>
<td></td>
</tr>
<tr>
<td>Neutrons → gives absolute values</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Why neutrons?

- **Wavelength**: Comparable to atomic distances (1-5 Å)
  - Strong nuclear interaction with nuclei

- **No charge**: Can travel through thick samples (cm) and equipment

- **Neutron spin** ($\mu_N$): dipole interaction with unpaired electrons $\Rightarrow \mu = -(L + 2S)\mu_B$
  - e.g. $3d^5 \text{Fe}^{3+}$ $L=0$ and $S=5/2 \Rightarrow 5\mu_B$
  - observed scattering of a similar magnitude to nuclear scattering (often smaller, sometimes larger)

- Magnetism can be investigated at a microscopic scale with a high precision
  - Magnetic structure
  - Quantitative moment size

$\Rightarrow$ **The best probe for magnetic structure determination**
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Neutron Scattering
Bertram Brockhouse and Clifford Shull - 1994 Nobel Laurate in Physics

“If the neutron did not exist, it would need to be invented”
How to get neutrons: A (very) brief history

- 1920: Rutherford predicted neutron
- 1932: Neutron discovered by James Chadwick

1942: First reactor, Chicago-Pile 1 (CP-1)

1943-1963 ORNL Graphite reactor

1965-present: HFIR

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**The Diffraction of Neutrons by Crystalline Powders**

E. O. Wollan and C. G. Shull
Oak Ridge National Laboratory, Oak Ridge, Tennessee

(Received January 5, 1946)
First determination of magnetic structure performed at ORNL

- Clifford G. Shull received 1994 Nobel prize in Physics.
- First direct evidence of antiferromagnetism in MnO.
- Neel model of ferrimagnetism confirmed in Fe$_3$O$_4$.
- First magnetic form-factor data obtained in Mn compounds.
- Production of polarized neutrons by Bragg reflection from ferromagnets demonstrated.
Neutron scattering: Reactor and Spallation source

**Reactor**

Fast neutrons are slowed by collisions in moderator (C,H\textsubscript{2}O, D\textsubscript{2}O) to produce thermal neutrons.

Small $\Delta \lambda$ used, but source on all the time.

**Spallation**

A pulse of protons impacts on a target (Ta, Hg) to produce a shower of fast neutrons. These are slowed down in a moderator (H, CH\textsubscript{4}).

Each pulse of neutrons contains a broad spectrum of energy ($\lambda$).

Pulse of neutrons ~30 times per second.
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• Practical aspects of neutron scattering and refinements
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Neutron Scattering

- Neutron source produces an incident beam of neutrons that scatters from a nucleus or unpaired electron [sample] into a defined cross-section \(d\Omega\) [detector].
Neutron Scattering Cross section

The scattering cross section can be measured in absolute units.

Flux: \[ \Phi = \frac{\text{Rate of neutrons through area}}{\text{area}} \]  
\[ 10^6-10^9 \text{ n/cm}^2/\text{s} \]
Neutron Scattering Cross section

The scattering cross section can be measured in absolute units.

Flux: \[ \Phi = \frac{\text{Rate of neutrons through area}}{\text{area}} \]

Rate of scattering: [Cross section] \[ \sigma = \frac{\text{Rate of neutrons scattered}}{\Phi} \]

Atom → 1 barn = $10^{-24}$ cm$^2$.
Effective surface area of nucleus

10$^6$-10$^9$ n/cm$^2$/s
Neutron Scattering Cross section

The scattering cross section can be measured in absolute units.

**Flux:**
\[ \Phi = \frac{\text{Rate of neutrons through area}}{\text{area}} \]

10^6-10^9 n/cm²/s

**Rate of scattering:**
\[ \sigma = \frac{\text{Rate of neutrons scattered}}{\Phi} \]

Atom \( \rightarrow \) 1 barn = 10^{-24} cm².
Effective surface area of nucleus

**Rate of scattering into a specific solid angle:**
\[ \frac{d\sigma}{d\Omega} = \frac{\text{Rate of neutrons scattered into } d\Omega}{\Phi \times d\Omega} \]

Units of barn/steradian.
Neutron Scattering Cross section

The scattering cross section can be measured in absolute units.

Flux: \( \Phi = \frac{\text{Rate of neutrons through area}}{\text{area}} \)  \( 10^6-10^9 \text{ n/cm}^2/\text{s} \)

Rate of scattering: [Cross section] \( \sigma = \frac{\text{Rate of neutrons scattered}}{\Phi} \)  Atom \( \rightarrow \) 1 barn =\( 10^{-24} \text{ cm}^2 \).

Effective surface area of nucleus

Rate of scattering into a specific solid angle: [Differential cross section] \( \frac{d\sigma}{d\Omega} = \frac{\text{Rate of neutrons scattered into } d\Omega}{\Phi \times d\Omega} \)  Units of barn/steradian.

Rate of scattering into angle within energy range: [Partial differential cross section] \( \frac{d^2\sigma}{d\Omega dE_f} = \frac{\text{Rate of neutrons into } d\Omega \text{ and } dE_f}{\Phi \times d\Omega \times dE_f} \)  Units of barn/steradian/meV
**Neutron Scattering Cross section**

The scattering cross section can be measured in absolute units.

\[
\frac{d^2\sigma}{d\Omega dE_f} = \frac{k_f}{k_i} S(Q, \omega) \quad \text{Diffraction} \quad \frac{d\sigma}{d\Omega} = S(Q)
\]

<table>
<thead>
<tr>
<th>Flux:</th>
<th>( \Phi = \frac{\text{Rate of neutrons through area}}{\text{area}} )</th>
<th>10^6-10^9 n/cm²/s</th>
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<td>Units of barn/steradian/meV</td>
</tr>
</tbody>
</table>
Neutron Scattering

Scattering triangle:

\[
Q = k_f - k_i
\]

Momentum transfer:

\[
Q = \frac{4\pi \sin \theta}{\lambda} = \frac{2\pi}{d}
\]

Elastic scattering:

\[
|k_i| = |k_f|
\]

Diffraction:

\[
\frac{d^2\sigma}{d\Omega dE_f} = \frac{k_f}{k_i} S(Q, \omega) \quad \Rightarrow \quad \frac{d\sigma}{d\Omega} = S(Q)
\]
Neutron diffraction: Bragg scattering

**Diffraction from a crystal**

Bragg peaks when: \( \lambda = 2d \sin \theta \)

Braggs Law

**Diffraction in reciprocal space**

Bragg peaks when: \( Q = G_{hkl} \)
Scattering by a potential $V(r)$: Born approximation

- Neutron scattering can be treated as scattering from a central potential (nuclear or magnetic).
- This interaction potential with neutron and matter is weak.
  - Disregard multiple scattering
- This allows the use of the Born approximation.

$\rightarrow$ Neutron cross-section can be completely known and modelled

$\rightarrow$ Work in reciprocal space

**Born approximation**

- Wavefunction of scattering by a central potential:

$$
\Psi(r) = e^{ikr} + \int \frac{1}{4\pi} dr' e^{ik'r'} V(r') \Psi(r') \frac{e^{ikr}}{r}
$$

- Expand integral (Born series):

$$
- \frac{1}{4\pi} \int dr' e^{ik'r'} V(r') \Psi(r') \approx - \frac{1}{4\pi} \int dr' e^{ik'r'} V(r') \Psi(r') e^{ikr} + \left( \frac{1}{4\pi} \right)^2 \int dr dr' \frac{e^{ikr}}{|r-r'|} V(r) V(r') + ...
$$

- Take first term (Born approximation):

$$
\left( \frac{d\sigma}{d\Omega} \right)_{\text{Born}} = \left| \frac{1}{4\pi} \int dr e^{-iQ.r'} V(r) \right|^2
$$

where $Q = k_f - k_i$

- Cross section is proportional to the Fourier transform of the potential energy, $V(r)$. 
Scattering potential: Scattering from a single fixed nucleus

Incident plane wave
\[ \Psi_i \propto \exp(ik.x) \]
\[ [k=2\pi/\lambda] \]

Scattered circular wave
\[ \Psi_f \propto (-b/r) \exp(ik.r) \]

Nuclear interaction potential:
- Very short range (~10^{-15} m)
- Isotropic scattering
  Diffraction theory: If waves of any kind scatter from an object of a size << \( \lambda \) then the scattered waves are spherically symmetric (S-wave scattering).
- Scattering is elastic \( \rightarrow \) nucleus is fixed
- Details of the potential \( (V(r)) \) are unimportant.
  \( V(r) \) can be described by a scalar parameter \( b \) that depends only on the nucleus and isotope [Fermi Pseudopotential]
  \[ b \approx \text{scattering amplitude / length} \approx 10^{-12} \text{ cm} \]
Scattering potential: Nuclear neutron diffraction

• Nuclear interaction potential:
  \[ V_{\text{Nuclear}}(\mathbf{r}) = \frac{2\pi h^2}{m_n} b \delta(\mathbf{r}) \]

• Diffraction intensity:
  \[ S(\mathbf{Q}) = |\sum_j b_j \exp(i\mathbf{Q}.\mathbf{r}_j)|^2 \quad \text{(sum over all nuclei in sample)} \]

• Rigid crystal:
  \[ S(\mathbf{Q}) = N \frac{(2\pi)^3}{V_0} \sum_{hkl} |F_{hkl}(\mathbf{Q})|^2 \delta(\mathbf{Q} - \mathbf{G}_{hkl}) \]

  - Number of unit cells in crystal
  - Volume of unit cell
  - Structure factor

  \[ F_{hkl}(\mathbf{Q}) = \sum_j b_j \exp(i\mathbf{G}.\mathbf{r}_j)\exp(-W_d) \]

  - Scattering only when \( \mathbf{Q} = \mathbf{G} \), i.e. at allowed (H,K,L) positions
  - \( W_d \rightarrow \) Debye-Waller factor for atomic thermal motion
Scattering length (b)

- Measured scattering depends on $b$, the scattering length
- $b$ varies randomly with Z and isotope
  - offers advantages over x-rays
  - But need to check for absorption
- Coherent and incoherent nuclear scattering
- $b$ varies with isotope and with nuclear spin orientation
- Consider a sample with two isotopes $\rightarrow b_1$ and $b_2$

\[
\text{Structure factor (nuclear)}
\]

\[
F_{hkl}(\mathbf{Q}) = \sum_j b_j \exp(i\mathbf{G} \cdot \mathbf{r}_j) \exp(-W_d)
\]

---

Random $b_1, b_2$

- Mean $b_{coh}^{-}$
- Coherent scattering ($b \rightarrow b_{coh}$)
- Deviation from mean $\sigma_{inc}^{-}$
- Incoherent scattering

\[
S_{inc}(\mathbf{Q}) = \sum_j \left(\frac{\sigma_{inc}}{4\pi}\right)
\]

Values of $b_{coh}$ and $\sigma_{inc}$ tabulated: [https://www.ncnr.nist.gov/resources/n-lengths/](https://www.ncnr.nist.gov/resources/n-lengths/)
Scattering potential: Magnetic scattering

- Magnetic interaction potential:  \[ V_{\text{Magnetic}}(\mathbf{r}) = -\mu \cdot \mathbf{B}(\mathbf{r}) \]

- \( \mathbf{B}(\mathbf{r}) \) depends on electron spin and orbital currents
- Potential depends on direction of neutron spin \( \rightarrow \) vector interaction
- Neutrons are only sensitive to the component of the magnetic moment perpendicular to \( \mathbf{Q} \)
- Anisotropic scattering, unlike nuclear scattering.
- Depends on orientation of neutron spin \( \rightarrow \) polarization analysis can be powerful
- Depends on electronic states \( \rightarrow \) magnetic form factor is important

Magnetic scattering is due to interaction of the neutron spin \( \mu \) with the magnetic field of an unpaired electron, \( \mathbf{B}(\mathbf{r}) \).
Scattering potential: Magnetic scattering

- Magnetic scattering is due to the interaction of the neutron spin with the magnetic field of an unpaired electron

- Interaction described by a potential:

$$\mathbf{-\mu \cdot H} = -\gamma \mu_N \mathbf{\sigma \cdot H}$$

  Gyromagnetic ratio: $\gamma = -1.91$

  Nuclear magneton: $\mu_N = (m_e \mu_B)^2 / m_n$

  Pauli spin operator: $\mathbf{\sigma}$

- Magnetic scattering length proportional to the electron radius $e^2/m_e c^2$:

$$r_0 = -\gamma e^2 / m_e c^2 = -0.54 \times 10^{-12} \text{ cm}$$

Magnetic and nuclear scattering lengths are comparable
Magnetic diffraction intensity

\[ S_M(Q) = C \sum_{G_M} |F_M(G)|^2 \delta(Q - G_M) \]

(sum over all magnetic reciprocal lattice vectors \(G_M\))

Scattering only when \(Q = G_M\), i.e. at allowed (H,K,L) positions

Magnetic Structure factor:

\[ F_M(G) = \sum_j f_j(Q) m_{\perp j} \exp(iG \cdot r_j) \]

(sum over magnetically ordered nuclei in unit cell)

Form factor

Moment (perpendicular to Q)

H,K,L in reciprocal space

27
Neutrons Only Measure Moments Perpendicular to \( Q \)

- Scattering depends on Fourier transform of
  \[
  V_{\text{magnetic}}(\mathbf{r}) = -\mu_n \cdot \mathbf{B}(\mathbf{r})
  \]

- From Maxwell’s equation:
  \[
  \nabla \cdot \mathbf{B}(\mathbf{r}) = 0
  \]

  Fourier transform \( \rightarrow \)
  \[
  i \mathbf{Q} \cdot \mathbf{B}(\mathbf{Q}) = 0
  \]

  \( \rightarrow \) \( \mathbf{B}(\mathbf{Q}) \) is perpendicular to \( \mathbf{Q} \) to be non-zero

  \[
  M_\perp(\mathbf{Q}) = \mathbf{Q} \times (\mathbf{M} \times \mathbf{Q})
  \]

In experiments this can be a useful constraint:

\[ H=0 \text{ T} \]

\[ (001) \]

\[ I(001) = 0 \]

\[ H>H_c \]

\[ I(001) > 0 \]
Magnetic form factor

- Nuclear scattering of neutron is from a point charge → no form factor
- X-ray scattering is from charge cloud → form factor
- Magnetic scattering of neutron: $B(r)$ depends on the electron spin and orbital motion → form factor
- Scattering decreases with increasing Q due to intra-atomic interference.
- Analytical expressions are tabulated in P.J. Brown International Tables of Crystallography, Vol. C, section 4.4.5. for $j_1$ (spin only), $j_2$ (orbital), $j_3$ (orbital), etc

$$f(Q) = c_1<j_0(Q)> + c_2<j_2(Q)> + c_3<j_4(Q)> + \ldots$$

$$f(0) = 1$$

- Form factor depends on valance of ion.
- In general intensity drop-off more pronounced for higher Z
Neutron measurements: Nuclear and Magnetic scattering

- The scattered intensity $S(Q)$ is given by:

  $$ S(Q) = |F_N(Q)|^2 + P_z(F_{M\perp}(Q)F_N^*(Q) + F_{M\perp}^*(Q)F_N(Q)) + |F_{M\perp}(Q)|^2 $$

- Unpolarized measurements $\rightarrow$ no interference terms between nuclear and magnetic scattering

  $$ S(Q) = |F_N(Q)|^2 + |F_{M\perp}(Q)|^2 $$

- The scattered intensity is simply the two components added together.

  - In refinements this means the nuclear and magnetic phases can be refined separately. (always case for Representational analysis)

  - Measure at low $Q$ to maximize scattered magnetic intensity.

  - Make use of the constraint that neutrons only measure perpendicular component.
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- Theoretical concepts of neutron scattering
- **Practical aspects of neutron scattering and refinements**
- Where to perform experiments: Diffraction instruments at ORNL and around the world?
<table>
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<tr>
<th>Powder</th>
<th>or</th>
<th>Single crystal</th>
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<tr>
<td><strong>Advantages</strong></td>
<td><strong>Advantages</strong></td>
<td><strong>Disadvantages</strong></td>
</tr>
<tr>
<td>• Often easier synthesis</td>
<td>• Propagation vector unambiguously determined.</td>
<td>• Synthesis can be hard</td>
</tr>
<tr>
<td>• See everything</td>
<td>• Low background so can see smaller moments</td>
<td>• Data correction: absorption, extinction, etc</td>
</tr>
<tr>
<td>• Propagation vector</td>
<td>• Directional dependence of field (or strain, etc)</td>
<td>• Need to search large reciprocal space (or have large detectors)</td>
</tr>
<tr>
<td>• If powders work then saved a lot of effort.</td>
<td>• Domain information</td>
<td>• Sample alignment considerations.</td>
</tr>
<tr>
<td>• Measurement more routine.</td>
<td>• Smaller mass (~mg)</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Disadvantages</strong></th>
<th><strong>Disadvantages</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>• Information is averaged and lost.</td>
<td>• Synthesis can be hard</td>
</tr>
<tr>
<td>• Hard to uniquely assign propagation vector.</td>
<td>• Data correction: absorption, extinction, etc</td>
</tr>
<tr>
<td>• No domain info</td>
<td>• Need to search large reciprocal space (or have large detectors)</td>
</tr>
<tr>
<td>• Field measurements hard to interpret quantitatively</td>
<td>• Sample alignment considerations.</td>
</tr>
</tbody>
</table>
Things to consider for an experiment

- Magnetic scattering is often weak and only observable at low Q due to magnetic form factor. Preparation is key to a successful experiment.

- Characterization in the lab is crucial (XRD, Magnetization, Heat capacity, etc) → know your sample and where to look with neutrons

- Powders
  - Typically the more mass the better → experiment and data analysis greatly improved.
  - Use of Al can reduce background compared to V. → Extra peaks, but for magnetic structure determination usually not an issue.

- Single crystal
  - Small masses often feasible (< mg), check with instrument teams.
  - Know crystal quality before experiment (alignment/mosaic/domains/twinning/etc)
Basics of fitting diffraction data

Measured peaks have position (Q or HKL), intensity and width

- **Peak positions**: determined by size and shape of unit cell
- **Peak intensities**: determined by the atomic number and position of the various atoms in the unit cell
- **Peak widths**: determined by instrument parameters as well as temperature, crystal size/quality, strain,

- Single crystal $\rightarrow$ integrated intensity of each peak is extracted. So in refinement only need to consider a few parameters (extinction, absorption)
- Powder $\rightarrow$ Overlapping peaks means modelling whole pattern. [Rietveld Refinement]
Fitting your data: Rietveld refinement (powder)

• Hugo Rietveld: “The method of using the total integrated intensities of the separate groups of overlapping peaks in the least-squares refinement of structures, leads to the loss of all the information contained in the often-detailed profile of these composite peaks. By the use of these profile intensities instead of the integrated quantities in the refinement procedure, however, this difficulty is overcome and it allows the extraction of the maximum amount of information contained in the powder diagram.”

• If pattern can be modelled, the fit between observed data and model can be optimized.

• In powder, unlike single crystal, need to model experiment dependent parameters
  - Background
  - Peak broadening (sample/instrument)
  - Lattice constant
  - Absorption and sample shape
  - Preferred orientation

• Refinement → need a good starting model

• Neutron data usually required for determining occupancy.
Peak shape varies with scattering angle

- Cagliotta formula: $\text{FWHM}^2 = U \tan^2 \theta + V \tan \theta + W$

- $U, V, W$ parameters are a function of instrument collimation and monochromator. G. Caglioti et al., Nucl. Instr. 3, 223-228 (1958)

- Does not take into account guides or focusing of monochromator.

- Spallation sources need extra terms to model resolution from pulse shape.

- Debye-Scherrer cone scattering causes asymmetric peak shapes at low/high angle in $I(Q)$ 1d plots.

If converted to 1d $\rightarrow$ asymmetric at low $2\theta$, symmetric at $2\theta=90^\circ$
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• Practical aspects of neutron scattering: performing a successful experiment
• Where to perform experiments: Diffraction instruments at ORNL
## Oak Ridge National Laboratory (ORNL)

<table>
<thead>
<tr>
<th>HFIR</th>
<th>SNS-FTS</th>
<th>SNS-STS</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Neutrons produced from a reactor core.</td>
<td>- Neutrons produced from an accelerator/target.</td>
<td>- Planned Second Target Station in 202X</td>
</tr>
<tr>
<td>- Highest flux reactor based source in the U.S. (80 MW)</td>
<td>- Most intense pulsed neutron beam. (60Hz, 1.4 MW)</td>
<td>- Most intense pulsed source of cold neutrons</td>
</tr>
</tbody>
</table>

- Several complimentary diffraction beamlines at ORNL.
- Science of the material will dictate choice of instrument(s).
- Second target station will add further capabilities.
HFIR Magnetism:

- **HB-2A**
- **WAND**²
- **HB-3A Four-Circle (DEMAND)**

- Additional options:
  - **HB-1A** (low background)
  - **GP-SANS** (low Q)
  - **HB-1** (polarization)
SNS
Magnetism:

- **POWGEN**
- **CORELLI**
- **SNAP**

- Additional options:
  - **TOPAZ** (large unit cells)
  - **NOMAD** (PDF)
  - **HYSPEC** (polarization)
Powder diffraction (ORNL)

- POWGEN (SNS) → Highest resolution for detailed crystal structure. Large Q available.
- WAND² (HFIR) → High flux, medium/low resolution
HB-2A: Instrument aims and layout

<table>
<thead>
<tr>
<th>Applications</th>
<th>Neutron Beam</th>
<th>Resolution (Δd/d)</th>
<th>Q-range</th>
<th>Beam size at sample</th>
<th>Measurement times</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetic structure determination under extreme environments. Polarized capabilities.</td>
<td>Constant wavelength (vertically focused Ge monochromator)</td>
<td>2.2e³ (variable) Balance between high flux/resolution</td>
<td>0.2-5.1 Å⁻¹ (λ=2.41 Å) 0.35-8 Å⁻¹ (λ=1.54 Å)</td>
<td>60 x 30 mm Typical can sizes are 6-15 mm diameter and 50 mm height.</td>
<td>Variable. Refinable data in minutes. Magnetic structure in 0.5 - 8 hours. Typically need gram sized sample for magnetic structures.</td>
</tr>
</tbody>
</table>

Flexible open design to allow for variety of complex and easily changeable sample environments.

Temperature: 30mK – 1700K
Magnetic field: 8T
Pressure: 2 GPa (BeCu clamp cell) 6kbar (Al helium gas pressure cell)
HB2A instrument

Detector bank:
44 $^3$He tubes (11 cm x 3 cm) spaced at ~ 2.7° intervals
Scattering range (2θ): 2-155°

Beam size: 
2 x 1 inches

Monochromator:
Vertically focused: 15 Ge [HHL]
Take-off angle: 90°

Collimation:
pre-monochromator ($\alpha_1$): 12' or open (35') [Fixed]
pre-sample ($\alpha_2$): 6', 16', 21', 31', open
pre-detector ($\alpha_3$) (x 44): 12' [Fixed]

<table>
<thead>
<tr>
<th>Ge(hkl)</th>
<th>λ (Å)</th>
<th>$d_{max}$ (Å)</th>
<th>$4\pi\sin\theta/\lambda$</th>
<th>Flux (n/cm²s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(113)</td>
<td>2.41</td>
<td>27.6</td>
<td>0.2-5.1</td>
<td>5 x 10⁶</td>
</tr>
<tr>
<td>(115)</td>
<td>1.54</td>
<td>17.6</td>
<td>0.35-7.9</td>
<td>1 x 10⁷</td>
</tr>
</tbody>
</table>

Resolution:

Residual: 12'-31'-6'

- Ge (228) $\lambda = 0.94$ Å
- Ge (117) $\lambda = 1.12$ Å
- Ge (115) $\lambda = 1.54$ Å
- Ge (113) $\lambda = 2.41$ Å

+$q = 4\pi \sin \theta / \lambda$ (Å⁻¹)
HB2A: What science is possible?

✓ **Temperature dependence of crystal and magnetic structures (0.03 K<T < 700 K)**

Magnetic structure and spin-orbit-driven excitation points to potential Weyl fermions

Stuart Calder (ORNL) et al.

✓ **Pressure studies with He gas cells up to 6 kbar**

Negative Thermal Expansion (NTE) in Cubic ScF$_3$

B. Greve and A. Wilkinson (Georgia Tech)

✓ **Magnetic field induced phenomena, up to 6 Tesla**

Magnetic pole reversal in La$_{1-x}$Pr$_x$Co$_2$P$_2$

M. Shatruk (FSU) et al.

✓ **High-temperature studies, up to 1200 °C**

Probing the Long term stability of Solid Oxide Fuel Cells

Y. Liu and J.W. Fergus (Auburn University)

✓ **Development of High pressure cells to study magnetic materials at low T**

C. dela Cruz (ORNL) and Y. Uwatoko (Univ. of Tokyo)

NiCrAl “Russian” pressure cell: 2.5 GPa

BeCu clamp cell: 2 GPa

3-Sample changer available for measurements from 1.5 to 300 K. This allowed rare magnetic order of U(IV) ions to be investigated in a series of compounds.

V. O. Garlea et al., PRX 9, 011038 (2019)
Recent developments

**Polarized neutron diffraction at HB2A**
- V-cavity polarizer commissioned.
- Measurements of weak ferro- and ferri-magnets. <0.01 μB
- Almost perfect transmission of 50% and flipping ratio of 14.
- The difference scattering from neutron beam polarized parallel and antiparallel to the applied magnetic field gives an **improvement of nearly one order of magnitude in moment sensitivity.**

**Design and development of a High Pressure Cell**
- Collaboration with the Uwatoko group, ISSP, Univ. of Tokyo.
- Optimized for low-Q scattering
- Designed for pelletized samples to be pressurized up to **2 GPa**
- Compatible with low temperature liquid Helium cryostats to reach base temperatures of **1.5K**

**NiCrAl “Russian” pressure cell: 2.5 GPa**
- Multigram sample in BeCu clamp cell: 1.5 GPa
- **In-situ** Al-He gas cell: 6 kbar
POWGEN
POWGEN (SNS)

- General purpose diffractometer that encompasses magnetic scattering.
- Alternative design to previous spallation diffractometers → data reduced to single pattern.
- Recently upgraded detector layout.
- Different wavelengths (Frames) available.

Recent detector upgrade: https://doi.org/10.1107/S160057671901121X
Science: magnetism plus structure

- Large Q range for doing simultaneous structural and magnetic refinements:
- magnetic structure, magnetoelastic coupling, etc.

(a) Crystallographic structure of CeNiAsO, (b) incommensurate spin density waves for $T_{N2} < T < T_{N1}$, and (c). coplanar commensurate order for $T < T_{N2}$.


Zhang et al., Physical Review B, 93, 094413 (2016)
POWGEN Mail-in program

24-sample PAC changer available for measurements from 10 to 300 K:
mail-in / rapid access proposals

https://neutrons.ornl.gov/powgen/mail-in
Magnetite (Fe3O4)

- Sample cooled down to 10 K at a nominal 1 K/minute
- Temperature controlled for a near constant DT/D(pcharge) = constant counting statistics
- Ramping accelerates with increasing beam power & halts when it trips

Plot of proton charge and controller set-point during data collection on Fe3O4.

Magnetic structure refinement taken from a slice of temperature ramping data at 295 K
SNAP (SNS)

- Dedicated pressure beamline.
- Powders and single crystals possible.
- Accessible Q as low as 0.78 Å⁻¹.
- Pressure 0-80 GPa at room temperature.
- 0-10 GPa between 85 and 300 K. Furnace also available.

S. Hirai, et al. “Giant atomic displacement at a magnetic phase transition in metastable Mn₃O₄” PRB 87 014417 (2013)
Single crystal diffraction (ORNL)

- HB-3A (DEMAND) $\rightarrow$ magnetic structure determination $\rightarrow$ new detector for larger Q coverage and use with extreme sample environments
- Corelli $\rightarrow$ Diffuse and large access to full reciprocal space.
- WAND$^2$ $\rightarrow$ Diffuse and standard diffraction. Very fast!
Current capabilities at DEMAND

Mission:
Nuclear and magnetic structures as a function of T, P, B, and E, e.g., magnetic structures, phase transitions and possible accompanied structural changes, order parameters and exploring structural phase diagrams.

Users have studied problems in physics, materials science, chemistry, and mineralogy.

Magnetism use 80% beam time

Monochromator

Wavelengths: 1.005 Å, 1.546 Å, 2.541 Å

$2\theta$ ($^\circ$)
DEMAND – extreme sample environment and polarized neutron diffraction


T = 50 mK - 800 K
H = 0 - 5 T
P = 0 - 10 GPa
E = 0 - 1100 Volts

Unpolarized/polarized neutron diffraction
The circular distribution of $k$-vectors definitively confirms $\text{Fe}_3\text{PO}_7$ to have a “partially ordered” antiferromagnetic (AFM) helical state. One candidate for such a state is a short-range ordered AFM Skyrmion phase.

Kathryn Ross, Colin Sarkis, James Nielson (Colorado State University)
Huibo Cao (ORNL)
Capabilities at DEMAND - high pressure

Hydrostatic pressure to 7.4 Gpa
~0.1 mm$^3$ crystal

Zhai K., Wu Y., Shen S., Tian W.,
Cao H.B., Chai Y.S., Chakoumakos B.C.,
Shang D., Yan L., Wang F., Sun Y.,
Nature Communications, 8, 519 (2017).
Capabilities at DEMAND - high q-resolution

DEMAND ==============Cold neutron================

➔ SANS
WAND$^2$

• Recently upgraded detector.
• 120 degree coverage in-plane.
• Plus/minus 7.5 degree out of plane.
• High flux, medium to low resolution.
• Array of complex sample environment with easy access to install.
• 0.05 K, 1000 K, 0-8 T, Pressure
WAND$^2$ - Science

- LaSrCuO$_4$
- Igor Zaliznyak
- LSCO
- $T = 300$ K; HK2
- 3D mapping of diffuse scattering
WAND$^2$ - Science

- LaSrCuO$_4$

Igor Zaliznyak

LSCO

$T = 4.5$ K; HK2

- 3D mapping of diffuse scattering
WAND$^2$

- Shorter data collection time → In-situ synthesis
- Pump-Probe experiments
- Stroboscopic measurement for reversible processes
WAND$^2$

- Shorter data collection time $\rightarrow$ In-situ synthesis
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WAND$^2$

- Shorter data collection time → In-situ synthesis
- Pump-Probe experiments
- Stroboscopic measurement for reversible processes
Corelli (SNS)

- Large detector coverage
- Most/all HKL covered for crystals
- Diffuse scattering.
- Magnets, pressure, dilution temperatures available.
Magnetic diffuse scattering with alternating kagome/triangular lattices—LuBaCo$_4$O$_7$
Simultaneous occurrence of multiferroism and short-range magnetic order in DyFeO$_3$

Jinchen Wang, Juanjuan Liu, Jieming Sheng, Wei Luo, Feng Ye, Zhiying Zhao, Xuefeng Sun, Sergey A. Danilkin, Guochu Deng, and Wei Bao

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Conclusion: Determining a magnetic structure with neutron scattering

- Find a good problem and grow sample (powder or crystal)
- Do lots of characterization measurements in laboratory
- Understand background/theory of sample and neutron
- Apply for beamtime (speak to instrument scientist)
- Sample and experiment preparation are crucial (speak to instrument scientist)
- Perform measurement
- Analyze crystal structure (maybe need more measurements)
- **Analyze magnetic structure**: Starting model (magnetic symmetry) → compare to data → repeat
- If lucky write up paper
- Otherwise more data → Powder → single crystal → polarization → inelastic → etc
Learn about the STS and have input on the development

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