High Flux Diffractometers on Reactors
Alan Hewat, ILL Grenoble
Neutron Reactors aren’t getting any brighter
But we can do a lot more with available neutrons
Classical Neutron Diffraction from a single crystal

Monochromator → Source

Detector ← Crystal

$\Delta \lambda = 1\%$ (monochromatic beam)

$\Delta \tau = 100\%$ (continuous source)
TOF Neutron Diffraction from a single crystal

Multiple reflections sorted by Time-Of-Flight

\[ \Delta \lambda = 100\% \text{ (white beam)} \]
\[ \Delta \tau = 1\% \text{ (pulsed source)} \]
White Beam Neutron Diffraction from a single crystal

Multiple reflections sorted by the Crystal itself

Source

$\Delta \lambda = 100\%$ (white beam)
$\Delta \tau = 100\%$ (continuous source)

Detector

Crystal
Multiple reflections sorted by the Crystal itself

“Niimura Special” Neutron Image Plates (NIP)

NIPs inside cylinder, Laser readout
Australian Vivaldi constructed & tested at ILL Grenoble
May 2005
KOAL - Australian VIVALDI

Flux from thermal neutron guide
High res. ($l = 1.03\text{Å}$) $1.29 \times 10^9 \text{n.cm}^{-2}\text{s}^{-1}$

Flux comparable to that on Vivaldi
KOALA also uses Niimura Special IPs
Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focussing)
- A wide band of wavelengths
- A very high flux on the sample
- A very large area detector
What about Classical Neutron CW Diffractometers

Δλ = 1% (monochromatic beam)
Δτ = 100% (continuous source)
Large detectors + high flux on the sample

Efficiency for a given resolution = * time averaged flux on the sample
* sample volume
* detector solid angle
* detector efficiency

Jorgensen, J.D., Cox, D.E., Hewat, A.W., Yelon, W.B.

"Scientific opportunities with advanced facilities for neutron scattering"
Shelter Island Workshop, 1984
Comparison of TOF & CW Diffractometers

**The time-averaged Flux*Detector criterium**

<table>
<thead>
<tr>
<th></th>
<th>D20</th>
<th>GEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux average on sample</td>
<td>5x10⁷</td>
<td>~2x10⁶</td>
</tr>
<tr>
<td>Detector solid angle</td>
<td>0.27 sr</td>
<td>4.0 sr</td>
</tr>
<tr>
<td>Efficiency=Flux*Detector</td>
<td>1.7</td>
<td>1</td>
</tr>
</tbody>
</table>

The CW and TOF machines have similar efficiencies

The time-averaged flux is very high on a CW machine (D20)
The detector is very large on a TOF machine (GEM)
Why is sample flux so high from a reactor?

A: Large vertically focusing monochromators?

No! Focusing in real space only gives a factor of $x^2$ to $x^4$.

Sample

Focussing monochromator

Source

cf use of convergent guide with TOF

ICNS Sydney Nov 2005 http://www.ill.fr/dif/AlanHewat/
A: Large wavelength-band focusing monochromators?
Yes! Focusing in reciprocal space can give a factor of x10

\[ \Delta d/d \approx 0.1\% \text{ for } \Delta \lambda/\lambda \approx 1\% \]

Why is sample flux so high from a reactor?
Before and After (data in 2 min.)

High flux compatible with good Resolution
High take-off option on D20

Higher D20 resolution since 2003

ICNS Sydney Nov 2005 http://www.ill.fr/dif/AlanHewat/
Very fast chemical and electrochemical kinetics

(Ti, Si, C) reaction $\rightarrow$ Ti$_3$SiC$_2$
University of Newcastle, Australia

- The explosive SHS reaction was studied in real time with neutrons
- The reaction is exothermic, & heats the sample to 2200°C in <1 sec
- The complete diffraction pattern (left) is collected at 300 ms intervals - A World Record

Super-D2B – a new High Resolution Powder Diffractometer

128 x 300 mm high resistive wire detectors, high resolution collimators

ICNS Sydney Nov 2005 http://www.ill.fr/dif/AlanHewat/
2D detectors for CW Powder Diffraction
E. Suard, C. Ritter, A. Hewat, P. Attfield et al.
Phase co-existence in the charge ordering transition in CaMn$_7$O$_{12}$

J. Phys. Condens. Matter 14, 5747-5753

Neutrons measure small changes in M-O distances - charge transfer
ANSTO-ECHIDNA HRPD diffractometer
Klaus-Dieter Liss with EuroCollimators & AZ-Systèmes
Only 1 ILL Thermal Beam for Diffraction (H11)
A New ILL Thermal Beam for Diffraction
A New ILL Thermal Beam for Diffraction
Alan Hewat & Henry Fischer, ILL

First presented at the ILL “Instrument Day” 26 Feb 2002
Highest priority for Instrument Committee 17 Oct 2003
DRACULA construction scheduled for 2006 Millennium Programme
Based on new $120^0 \times 30^0$ 2D-detector for D19

S. Mason, T. Forsyth, J. Howard et al.
Dracula Focussing Geometry
Doubly focussing monochromator, l-focussing

Si[hhhl] $2T_m = 90^\circ$ monochromator
Doubly focusing bent wafers (SALSA)

D-spacings in the range $2T = 60^\circ - 120^\circ$

- $[115] \ l = 1.54 \ \text{Å} \ d = 0.89 \ \text{Å} \ to \ 1.54 \ \text{Å}$
- $[113] \ l = 2.44 \ \text{Å} \ d = 1.39 \ \text{Å} \ to \ 2.44 \ \text{Å}$
- $[111] \ l = 4.61 \ \text{Å} \ d = 2.66 \ \text{Å} \ to \ 4.61 \ \text{Å}$
**Comparison of TOF & CW Diffractometers**

The time-averaged Flux*Detector criterium

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<thead>
<tr>
<th></th>
<th>D20</th>
<th>GEM</th>
<th>DRACULA</th>
<th>SNS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux average on sample</td>
<td>$5 \times 10^7$</td>
<td>$\sim 2 \times 10^6$</td>
<td>$\sim 10^8$</td>
<td>$\sim 2.5 \times 10^7$</td>
</tr>
<tr>
<td>Detector solid angle</td>
<td>0.27 sr</td>
<td>4.0 sr</td>
<td>1.5 sr*</td>
<td>3.0 sr</td>
</tr>
<tr>
<td>Efficiency=Flux*Detector</td>
<td>1.7</td>
<td>1</td>
<td>18</td>
<td>9</td>
</tr>
</tbody>
</table>

* Based on new D19 detector: $R=760$ mm, $h=400$ mm, 800 linear resistive wires $30^\circ \times 160^\circ$

TOF machines already have big detectors
With big detectors ILL can compete with SNS intensity
Dracula Sample Environment
Radial Collimator, Cryomagnet & Pressure

- 100Kbar ILL Paris-Edinburgh cell
  (N. Kernavanois et al.)
- D20 Radial Oscillating Collimator
  (H. Fischer, Th. Hansen, P. Henry)
Radial Collimator = 95% transmission, 3% background

- No collimator
- With collimator
- No cryomagnet

Henry Fischer Aug'05
New Science on Dracula

New science with a very high intensity neutron diffractometer

- High Pressure and Extreme Sample Environments
- New Materials from High Pressure Synthesis
- Isotope replacement contrast
- Strongly absorbing elements
- In-situ chemical kinetics
- Very fast reactions
- Very weak magnetic order and polarised neutrons
- Texture
- Even Single crystal diffraction
2D Electronic Detectors & Image Plates
8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

LADI image plate vs D19 proto detector
2D Electronic Detectors & Image Plates
8mm Lysozyme crystal, Bruno Guerard, ILL Grenoble

LADI image plate (12 hours above) vs D19 proto detector (1 hour below)
ORIENT EXPRESS, a new CCD detector
Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble

Orient Express
On H24 thermal neutron guide
Dual lens-coupled chilled ICCD’s

CCD detectors in backscattering
Neutron beam
TV camera for crystal alignment
Crystal on goniometer

ICNS Sydney Nov 2005 http://www.ill.fr/dif/AlanHewat/
ORIENT EXPRESS, a new CCD detector
Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble

Ruby test crystal
10 minutes with photographic film
10 seconds with neutron CCD camera
ORIENT EXPRESS, a new CCD detector
Bachir Ouladdiaf (ICNS-Sydney), ILL Grenoble
Recipe for a High Flux Neutron Diffractometer

- A large incident solid angle (focussing)
- A wide band of wavelengths
- A very high flux on the sample
- A very large area detector
Next Generation Neutron Diffractometers
A large incident solid angle (focussing)

Focusing neutrons to study small samples

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Kirkpatrick–Baez microfocusing optics for thermal neutrons

Doubly-focussing thermal guides super-mirrors (M≈3) 0.8Å<l<3Å
Next Generation Neutron Diffractometers
A very large area detector

"Hedgehog" ILL diffractometer project (1975)
Next Generation Neutron Diffractometers
A very large area detector

“SXD” ISIS single crystal diffractometer (1995)

11 Position-sensitive scintillator detectors
192 × 192 mm² active area; 3 mm resolution
Next Generation Neutron Diffractometers

CYlindrical CCD Laue Optics Photo Scintillator

CYCLOPS
Double octagonal array of CCD neutron cameras
Recipe for a High Flux Neutron Diffractometer

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CW diffractometers finally have what it takes
High Flux Diffractometers on Reactors
Alan Hewat, ILL Grenoble

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Sax Mason (D19)
Garry McIntyre (D10, Vivaldi)
Bachir Ouladdiaf (D10, Orient Exp.)
Thilo Pirling (SALSA)
Clemens Ritter (D2B)
Anne Stunault (D3)
Emmanuelle Suard (D2B)
Clive Wilkinson (Vivaldi)

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