Small-angle neutron scattering at DNS-IV

Mikhail V. Avdeev

Frank Laboratory of Neutron Physics,
Joint Institute for Nuclear Research, Dubna, Russia
Outline

- SANS diffractometers: overview
- Tendencies of development at pulsed sources: ESS
- SANS at DNS-IV: first stage
SANS: areas of applications

- Complex fluids (surfactant solutions, polymers, liquid crystals, sols and suspensions)
- Biological macromolecules and membranes
- Amorphous substances (carbon, silicon, solid polymers, glasses, foams)
- Polycrystalline and composite materials
- Magnetic colloids
- Long-period and macromolecular structures
- Submicron and micron inhomogeneities (USANS, SESANS)

Fraction of SANS experiments within User Policies at neutron centers up to 50 %!
Measurements in absolute units:

\[ \frac{d\Sigma}{d\Omega} = F \left( \frac{I}{d_s T_s} \right) \left( \frac{I_w}{d_w T_w} \right) \]

Calibration factor
Optimal configuration

R_1 = 2R_2; L_1 = L_2

Δθ/θ ~ Δλ/λ

Typical characteristics

Q-resolution: 5 - 30%,
Q-range: 0.01 - 5 nm⁻¹,
Dynamic range: 5 - 100
Exposure time of one curve: 1 - 100 min
Polarizer (optional)

Extended sample environment system (T, p, H).
Automatic sample cartridge (5 – 30 samples)
PSD (50 × 50 - 100 × 100 cm, resolution 0.5 - 1 cm)
Steady-state SANS

KWS-1 (MLZ, Garching): Principal layout

https://www.mlz-garching.de/kws-1
Steady-state SANS

KWS-1 (MLZ, Garching): Technical data

Overall performance
• $Q = 0.0007 - 0.5 \text{ Å}^{-1}$
• Maximal flux: $1.5 \cdot 10^8 \text{ n cm}^{-2} \text{ s}^{-1}$
• Typical flux: $8 \cdot 10^6 \text{ n cm}^{-2} \text{ s}^{-1}$ (collimation 8 m, aperture 30 x 30 mm$^2$, $\lambda = 7 \text{ Å}$)

Velocity selector
• Dornier, FWHM 10%, $\lambda = 4.5 \text{ Å} - 12 \text{ Å}$, 20 Å

Chopper
• For TOF-wavelength analysis, FWHM 1%

Polariser
• Cavity with V-shaped supermirror, all wavelengths
• Polarisation > 90%, typical 95%

Spin-flipper
• Radio-Frequency (efficiency > 99.8%)

Neutron lenses
• MgF$_2$, diameter 50 mm, curvature 20 mm
• Packs with 4, 6, 16 lenses

Active apertures
• 2 m, 4 m, 8 m, 14 m, 20 m

Aperture sizes
• Rectangular 1 x 1 mm$^2$ – 50 x 50 mm$^2$

Sample aperture
• Rectangular 1 x 1 mm$^2$ – 50 x 50 mm$^2$

Sample stage
• Hexapod, resolution better than 0.01°, 0.01 mm

Detector
• Detection range: continuous 1.5 m – 20 m
• $^6$Li-Scintillator 1 mm thickness + photomultiplier
• Efficiency >95%
• Spatial resolution 5.3 x 5.3 mm$^2$, 128 x 128 channels
• Max. count rate 0.6 MHz ($T_{\text{dead}} = 0.64 \text{ µs}$)
Steady-state SANS

KWS-1 (MLZ, Garching): Sample environment

- Rheometer shear sandwich
- Rheowis-fluid rheometer (max. shear rate 10000 s⁻¹)
- Anton-Paar fluid rheometer
- Stopped flow cell
- Sample holders: 9 horizontal x 3 vertical (temperature controlled) for standard Hellma cells 404-QX and 110-QX
- Oil & water thermostats (range -40 – +250°C), electric thermostat (RT – 200°C)
- 8-positions thermostated (Peltier) sample holder (-40°C … +150°C)
- Magnet (horizontal, vertical)
- Cryostat with sapphire windows
- High temperature furnace
- Pressure cells (500 bar, 2000 bar, 5000 bar)
Steady-state SANS

KWS-1 (MLZ, Garching)

- Polarizer & spin-flipper
- PSD
- Hexapod
- Electromagnet
- Cryomagnet
Steady-state SANS

KWS-1 (MLZ, Garching)

Cobalt ferrite nanoparticles in quartz matrix
TOF-SANS at pulsed neutron sources

**ISIS (3)**
- LOQ – standard SANS (non-pol)
- SANS2d – extended SANS (non-pol)
- Larmor – SESANS

**ISIS (1)**
- ZOOM – VSANS (pol)

**SNS (2)**
- EQ-SANS – extended SANS (non-pol)
- USANS

**J-PARC (1)**
- TAIKAN – SANS and WANS (pol)

**IBR-2 (1)**
- YuMO – standard SANS (non-pol)

**ESS (2)**
- SKADI – General Purpose SANS (pol)
- LoKI – Broadband SANS (non-pol)

**LANSCE (0)**
ISIS TS2
\( \nu = 10 \, \text{Hz}, \Delta t = < 50 \, \mu\text{s} \)

Sans2d  
Time-of-flight Small-Angle Neutron Scattering instrument (TS2)

• Wide Q-range (0.02 < Q nm\(^{-1}\) < 20); most is accessible with one instrument configuration.

• Five 2 m guide sections with variable collimation apertures.

• Two moveable 1 m\(^2\) detectors giving the most detector area on any SANS instrument in the world and almost 77,000 pixels.

• High-flux at sample (3-10 times LOQ on TS1, depending on Q-range).

• Small sample size/volume (<15 mm diameter or only 0.3-3 ml).
Sans2d  Time-of-flight Small-Angle Neutron Scattering instrument (TS2)

PSD
Sans2d  Time-of-flight Small-Angle Neutron Scattering instrument (TS2)

Micelle – Vesicle transition in real time

$\tau \sim 10$ s
J-PARC/J-SNS pulsed neutron source
$v = 25 \text{ Hz}$

TAIKAN Small and Wide Angle Neutron Scattering Instrument

<table>
<thead>
<tr>
<th>Moderator</th>
<th>Coupled hydrogen moderator</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neutron wavelength band</td>
<td>0.05-0.8 nm (unpolarized neutron)</td>
</tr>
<tr>
<td>Q-range</td>
<td>$5 \times 10^{-2}$-$100 \text{ nm}^{-1}$ (unpolarized neutron)</td>
</tr>
<tr>
<td>Beam size</td>
<td>10 mm$\times$10 mm (Typical)</td>
</tr>
<tr>
<td>Auxiliary equipment and sample environment</td>
<td>Sample changer (10 samples, $T = -25 \ldots +125^\circ \text{ C}$), 4K cryostat, 1Tesla electromagnet, etc.</td>
</tr>
</tbody>
</table>
IBR-2 reactor

- Diffraction: 8
- SANS: 1
- Reflectometry: 3
- Inelastic: 2
- Imaging: 1

IBR-2 reactor

SKAT  EPSILON  HRFD  YuMO  DIN-2PI  KOLHIDA
NERA  DN-6  RTD
REMUR  REGATA  IZOMER
REFLEX-P  GRAINS  FSD  DN-12  NRT  FSS
YuMO small-angle diffractometer

1 – power modulator;
2 – reactor core with moderator;
3 – background chopper;
4 – first aperture (pin-hole);
5 – vacuum tube;
6 – second aperture (pin-hole);
7 – thermostate;
8 – sample table;
9 – goniometer;
10-11 – V-standards;
12 – ring-wire detector;
13 – position-sensitive detector;
14 – direct beam detector.
## YuMO small-angle diffractometer

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neutron flux at sample place</td>
<td>1-4×10^7 cm^{-2} s^{-1}</td>
</tr>
<tr>
<td>Neutron wavelength band</td>
<td>0.5 – 8 Å</td>
</tr>
<tr>
<td>q-range</td>
<td>0.007 – 0.5 Å^{-1}</td>
</tr>
<tr>
<td>q-resolution</td>
<td>5 – 20 %</td>
</tr>
<tr>
<td>Dynamic q-range (q_{max}/q_{min} in one measurement)</td>
<td>up to 100</td>
</tr>
<tr>
<td>Beam size at sample place</td>
<td>∅ 14 mm</td>
</tr>
<tr>
<td>Detectors</td>
<td>Two-detector system, He³, ring wire detectors, no-radial sensitivity</td>
</tr>
<tr>
<td>Detector of direct beam</td>
<td>^6Li-convertor</td>
</tr>
<tr>
<td>Detector PSD</td>
<td>PSD, ^3He, 60×60 cm², resolution 5×5 mm²</td>
</tr>
<tr>
<td>Number of samples in automatic cartridge</td>
<td>25</td>
</tr>
<tr>
<td>Temperature range</td>
<td>+4°C ÷ + 70°C</td>
</tr>
<tr>
<td>(standard quartz cells)</td>
<td>-20°C ÷ + 130°C</td>
</tr>
<tr>
<td>(requires special sample holder)</td>
<td></td>
</tr>
<tr>
<td>Sample environment</td>
<td>Electromagnet 2.5 T, (p, V, T)-cell</td>
</tr>
</tbody>
</table>
YuMO small-angle diffractometer

Detonation nanodiamonds in aqueous dispersions

Lipid vesicles in water

FTNS SANS

(b)
CONCEPT OF SMALL-ANGLE DIFFRACTOMETER IN CLASSICAL CONFIGURATION AT THE CRYOGENIC MODERATOR OF IBR-2 REACTOR

Beamline 10A

$T = 100$ K

Spectrum calculations

Total flux calculations
(flux density on moderator $10^{12}$ cm$^{-2}$ s$^{-1}$)

<table>
<thead>
<tr>
<th>Temperature of moderator</th>
<th>30 K</th>
<th>100 K</th>
<th>300 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before bender</td>
<td>1.0e9</td>
<td>4.3e8</td>
<td>1.8e8</td>
</tr>
<tr>
<td>After bender</td>
<td>3.9e8</td>
<td>8.5e7</td>
<td>1.4e7</td>
</tr>
<tr>
<td>Sample position (collimation length 1 m)</td>
<td>2.3e8</td>
<td>5.6e7</td>
<td>1.0e7</td>
</tr>
<tr>
<td>Sample position (collimation length 10 m)</td>
<td>7.4e6</td>
<td>2.7e6</td>
<td>7.2e5</td>
</tr>
</tbody>
</table>

30 K – working mode (flux at sample $> 10^6$ cm$^{-2}$ s$^{-1}$)
300 K – mode for high-scattering systems (flux at sample $> 10^5$ cm$^{-2}$ s$^{-1}$)

Polarized neutrons

Transmission polarizer: V-shaped

Transmission polarizer: S-shaped

Transmission $^3$He analyzer

unpolarized incoming neutrons

polarized $^3$He

polarized outgoing neutrons
Concepts of SANS instrumentation at neutron sources

MLZ, Garching

KWS-1 high resolution SANS diffractometer with full polarization analysis

KWS-2 high flux SANS diffractometer (non-polarized beam)

KWS-3 is a very small angle neutron scattering (VSANS) instrument
Concepts of SANS instrumentation at neutron sources

**ORNLS, Oak-Ridge**

**GP-SANS** General-Purpose Small-Angle Neutron Scattering Diffractometer

**BIO-SANS** Biological Small-Angle Neutron Scattering Instrument

**EQ-SANS** Extended Q-Range Small-Angle Neutron Scattering Diffractometer

**ANSTO, Sydney**

**Quokka** Small-angle neutron-scattering instrument

**Bilby** Small-angle neutron-scattering instrument (TOF option) (built due to strong excess of proposals)
ESS pulsed neutron sources, \( v = 14 \text{ Hz}, \Delta t_0 = 2860 \mu \text{s} \)

SKADI – General Purpose, polarized

\( L_1 = L_2 \approx 25 \text{ m} \)

LoKI – Broadband SANS, non-polarized

\( L_1 = L_2 \approx 10 \text{ m} \)

ESS parameters:

- Average beam power, MW \( 5 \)
- Peak beam power, MW \( 125 \)
- Proton kinetic energy, GeV \( 2.0 \)
- Pulse repetition rate, Hz \( 14 \)
- Average pulse current, mA \( 62.5 \)
- Macro-pulse length, \( \mu \text{s} \) \( 2860 \)
- Number of target stations \( 1 \)
- Number of moderators \( 2 \)
- Number of instruments \( 16 \) (22)
- Number of neutron beam ports \( 42 \)
- Separation between ports degrees \( 6 \)
SKADI SANS diffractometer, ESS

- **In Situ** Chemical Reaction
- Stroboscopic Investigations of Magnetic Liquids
- High-Resolution Structure Formation (Quasi-crystals)

<table>
<thead>
<tr>
<th>Flux at sample</th>
<th>&gt; $10^8$ cm$^{-2}$ s$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>q-range</td>
<td>0.0001 – 1 Å$^{-1}$</td>
</tr>
<tr>
<td>q-resolution</td>
<td>&lt; 5 %</td>
</tr>
<tr>
<td><strong>Dynamic q-range</strong></td>
<td>~ 1000 (Two detector system; Dubna-type, size ~ 1 × 1 m)</td>
</tr>
</tbody>
</table>

Total costs > 15 MEu
SoNDE Detector, ESS

Position reconstruction by Anger method based on photomultiplier light sensors

Hamamatsu H8500 multianode photomultiplier with high voltage cable (picture from Hamamatsu). The device has got a sensitive area of 89% and pixel sizes of about 6 mm x 6 mm

Project (No. 654124) is funded by the Horizon 2020 Framework Programme of the European Union.
Sample Environment Systems for Fluids Including Gases, Liquids and Complex Fluids (FLUCO)

- Temperature, spanning the approximate range of 223 - 473K;
- Relative humidity, using H$_2$O, D$_2$O or solvents including organic solvent;
- Physical forces, including shear, torque, and stretch viscosity, including dynamic and kinematic, and fluidity friction;
- Small magnetic fields, up to 1T. For high magnetic fields, please see the Temperatures and Fields platform;
- Electrical properties, including potentiostat measurements.
LoKI SANS diffractometer, ESS

Dynamic q-range > 1000

$L_{1\text{max}} = 10\, \text{m}$
$L_{2\text{max}} = 10\, \text{m}$
Repetition rate = 14Hz or 7Hz
$\delta\lambda_{\text{max}} = 10\, \text{Å} \text{ at } 14\text{Hz}$

Max flux on sample $\sim 1 \times 10^9 \, \text{n/cm}^2/\text{s}$

2x line-of-sight closure
Boron-10 "Lined tube" detector system

Costs 12 MEu
"Window frame" detector system

Simulation for scattering from 1 mm thick H$_2$O

Costs 15 MEu
Expected parameters of DNS-IV compared to SNS and ESS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>DNS-IV</th>
<th>SNS</th>
<th>ESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Time-average flux density:</td>
<td>$(0.5 \div 12) \cdot 10^{14}$</td>
<td>$0.1 \cdot 10^{14}$</td>
<td>$3 \cdot 10^{14}$</td>
</tr>
<tr>
<td>2. Half-width of fast neutrons:</td>
<td>$(20 \div 200) , \mu s$</td>
<td>$(20 \div 50) , \mu s$</td>
<td>$2860 , \mu s$</td>
</tr>
<tr>
<td>3. Pulse repetition rate:</td>
<td>$(10 \div 30) , Hz$</td>
<td>$60 , Hz$</td>
<td>$14 , Hz$</td>
</tr>
<tr>
<td>4. Time-average power:</td>
<td>$(5 \div 10) , MW$</td>
<td>$1 , MW$</td>
<td>$5 , MW$</td>
</tr>
<tr>
<td>5. Background power:</td>
<td>$3.2 %$</td>
<td>$&lt;1%$</td>
<td>$&lt;1%$</td>
</tr>
<tr>
<td>6. Number of beam ports:</td>
<td>$20 - 32$</td>
<td>$22$</td>
<td>$42$</td>
</tr>
</tbody>
</table>
SANS instruments for DNS-IV.
First stage

<table>
<thead>
<tr>
<th>No.</th>
<th>Instrument</th>
<th>Main issue</th>
<th>Moderator</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>General purpose</td>
<td>high resolution, $q_{\text{min}} = 10^{-4}$ Å$^{-1}$ polarized neutrons, wide angle analyzer, two PSD $1 \times 1$ m, $5 \times 5$ mm, extended sample environment (combinations with other techniques, operando studies)</td>
<td>30 K</td>
</tr>
<tr>
<td>2</td>
<td>Real time</td>
<td>medium resolution, $q_{\text{min}} = 10^{-3}$ Å$^{-1}$ non-polarized PSD $0.64 \times 0.64$ m, $5 \times 5$ mm</td>
<td>30 K</td>
</tr>
<tr>
<td>3</td>
<td>Micro-samples</td>
<td>medium resolution, $q_{\text{min}} = 10^{-3}$ Å$^{-1}$ focusing devices, non-polarized PSD $0.64 \times 0.64$ m, $5 \times 5$ mm</td>
<td>30 K</td>
</tr>
</tbody>
</table>
Requirements to DNS-IV

1. Time-average flux density: \( (0.5 \div 12) \times 10^{14} \) \( \rightarrow \) \( \Phi_0 = 10 \times 10^{14} \text{ n/cm}^2/\text{s} \)

2. Half-width of fast neutrons: \( (20 \div 200) \mu\text{s} \) \( \rightarrow \) \( \Delta t_0 = 200 \mu\text{s} \)

3. Pulse repetition rate: \( (10 \div 30) \text{ Hz} \) \( \rightarrow \) \( \nu = 10 \text{ Hz} \)

4. Moderators (at least three): VC, C, Th \( \rightarrow \) Very Cold (~30 K)

5. Background power: 3-7 % \( \rightarrow \) < 5 %, restriction for high resolution in direct space (large q)

6. Size of moderator: 10 – 20 cm \( \rightarrow \) 20 cm
Conclusions

- Current and future trend in design of SANS instruments is determined by high user demand and users’ interest to combination of instruments of a wide range of purposes (with fairly good characteristics) with specialized instruments (in situ, wide dynamic range, microsamples, special tasks).

- To date, vast experience in design of SANS instruments has been accumulated. Further improvement of this type of instruments, including detector systems, seems extremely costly.

- A “standard” set of SANS instruments can be implemented at DNS-IV based on a combination of basic characteristics (intensity, resolution, q-range) comparable to ISIS, SNS, J-SNS and ESS.

- The main line of improvement of future SANS instruments is the development and design of the sample environment system of the new generation:
  - Combination with complementary methods
  - Specialized systems for practical tasks (catalysis, electrochemistry, food products, materials science, radioactive materials, industrial processing, etc.)