A Big-Angle View of Small-Angle Measurements: SAXS Techniques
Welcome

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- A Tour of the Nano-Cosmos
  - Introduction to SAXS
  - 1D SAXS Instruments
  - 1D SAXS Application Examples
  - 2D Method - NanoSTAR
  - 2D Simultaneous SAXS/WAXS
  - NanoSTAR – Typical Applications
  - Summary
  - Q & A
Introduction to SAXS

Brian Jones
The SAXS Experiment

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

- Large \( \theta \) \( \rightarrow \) small \( d \)
- Small \( \theta \) \( \rightarrow \) large \( d \)

**SAXS**
scattering at particles or electron density changes
scattering angles: 0 - 4°

**XRD**
diffraction at crystal lattice
diffraction angles: 4 - 170°

\( \theta \) \( \rightarrow \) \( d \) \( \rightarrow \) 10 – 100nm
Nanostructural Parameters Obtained from SAXS

- Mean size, size distribution
- Shape (spheric, cylindric, platelet, cubic ...)
- Orientation, degree of orientation
- Mean distance between particles
Scattering Vector $q$

$q \equiv \frac{4\pi \sin \theta}{\lambda}$

d = \frac{2\pi}{q}

For isotropic systems (fluids, glasses, polycrystals):
→ no direction dependence of the scattered radiation
Example SAXS Scattering Curve log – log Scale

HDPE

$q [Å^{-1}]$

0.014° 0.14° 1.4°
Transmission SAXS

- X-rays are incident normal to the surface of the sample and transmission is sufficient to provide suitable SAXS scattering intensity.

Liquid dispersions, gels, powders, sheets, etc.
Grazing Incidence Small-Angle X-Ray Scattering (GI-SAXS)

GI – Incident angle close to the critical angle (0.1 to 1 degree)
SAXS - length scale, beam definition by multiple slits, and (usually) an area detector.

Nanoscale particles embedded in a matrix subsurface, supported on a substrate or buried in a thin layer on a substrate.

Examples:
Semiconductor quantum dots/islands
Porous films on substrates
Condensed powder
Nanoparticles embedded in polymers
1D SAXS Instruments

Brian Jones
SAXS Breakdown by Instrument Type

Point collimation
- NanoSTAR
  - Transmission SAXS
  - GI-SAXS
- D8 GADDS
  - Transmission SAXS
  - GI-SAXS

Line collimation
- D8 Advance
  - Transmission SAXS
  - GI-SAXS
- D8 Discover
  - Transmission SAXS
  - GI-SAXS
Bruker AXS Instruments
1D SAXS

D8 Advance

D8 Discover
1D SAXS Line Collimation Goals

- Monochromatic X-rays
- High intensity beam
- Well collimated beam
- Axial divergence minimal
- Beam width is narrow and adjustable for high-flux / high-resolution trade-off.
- Background to either side of direct beam is very low
- (optional) Can scan over the direct beam to determine sample transmission.
1D SAXS Line Collimation Goals

- Monochromatic X-rays
  Gobel Mirror
- High intensity beam
  Gobel Mirror
- Well collimated beam
  Gobel Mirror + aperture slits
- Axial divergence minimal
  Soller slits
- Beam width is narrow and adjustable for high-flux / high-resolution trade-off
  2 incident beam slits and 2 diffracted beam slits of various sizes
- Background to either side of direct beam is very low
  4 aperture slit system with narrow apertures and optional knife edge
- (optional) Can scan over the direct beam to determine sample transmission
  Rotary absorber
Gobel Mirror – Monochromatic, High Intensity, Collimated

- Parabolic, laterally graded, multilayer mirror
- Captures a large solid angle of divergent radiation and converts to collimated, monochromatic beam
Soller Slits
Reduce Axial Divergence

- Many closely spaced metal foil pieces stacked parallel to one another.
- Controls the angular acceptance angle along the axial direction.
Hardware Configuration
D8 Advance/Discover
Typical Experimental Setup

- Measurement diameter = 500mm – 600mm
- Cu tube (40kv, 40mA)
- 60mm 3rd generation Gobel mirror
- 0.2 mm mirror exit slit
- Rotary Absorber (RA)
- 0.1 mm slit after RA
- 0.1 deg anti-scatter slit
- 1.5 degree Soller slit
- 0.1 mm detector slit
- Scintillation counter
Please use your mouse to answer the question on your screen:

What types of samples do you analyze? (Check all that apply):

- liquid
- powder
- gel
- sheet
- fiber
- thin film
- solid/bulk
SAXS Transmission Sample Holders

- Holder for sheets, powders, gels, etc.
SAXS Transmission Sample Holders

- Liquids, powders, deposited in capillary tube
D8 Advance Goniometer Head-Mount Stage

- Entire stage can be quickly removed and replaced
D8 Advance Goniometer Base Stage
Sample Holder Attached
D8 Advance
Primary Beamstop and Knife Edge

- Mounts to primary beam track.
- Beamstop behind the sample.
- Knife edge in front of sample.
- Compatible with many D8 Advance transmission sample holders
D8 Advance
Primary Beamstop and Knife Edge

- Knife edge used to reduce parasitic scatter
D8 Advance
Primary Beamstop and Knife Edge
D8 Discover

Vertical

Horizontal
D8 Discover
Centric Eulerian Cradle

- Mounted on diffractometer permanently
D8 Discover - Capillary Spinner for Eulerian Cradle Stage

- Beamstop and knife edge are adjustable or can be completely removed from beam path.

- Goniometer head base will accommodate numerous sample holders.
D8 Discover – Capillary Spinner Mounted to Eulerian Cradle

- Drawing of capillary spinner mounted to Eulerian cradle on a horizontal system
D8 Discover – Capillary Spinner Mounted to Eulerian Cradle

- Only knife edge is mounted
Knife edge and capillary holder are mounted to the capillary spinner for the Eulerian cradle.
1D SAXS – Application Examples

Brian Jones
Example 1: Glassy Carbon

Glassy Carbon is a porous material often used as a standard in SAXS. The pore size and shape have been previously determined with high accuracy.

- Ellipsoid pore shape
- Outer radius ~ 20-23 Å
- b/a (aspect ratio) = 0.3
Example 1: Glassy Carbon
1D SAXS Scattering Curve

Glassy carbon
Empty beam
Example 1: Glassy Carbon Analysis with DiffracPlus Nanofit

- Least-squares data analysis program for small angle scattering data by direct modeling
- Supports basic geometric models and polymer models, polydispersity, and concentration effects
Example 1: Glassy Carbon Direct Modeling with Nanofit

- The graph shows the experimental data (blue) and the fit (red).
- The measured scattering profile can be nicely described by using a model for ellipsoids.
- Previously determined Pore dimensions:
  - Outer radius: \( r = 20-23 \) Å
  - Aspect Ratio: \( b/a = 0.3 \)
- Results of D8 SAXS fit:
  - Outer radius: \( r = 19.7 \) Å
  - Aspect Ratio: \( b/a = 0.303 \)

Good Agreement!
Example 1: Glassy Carbon

Lower limit of fit \( \rightarrow q = 0.0106 \text{ A}^{-1} \)

\[ d = \frac{2\pi}{q} \approx 60 \text{ nm} \]
Example 1: Glassy Carbon Using Knife Edge
Example 1: Glassy Carbon
Improved Resolution with Knife Edge

Meaningful SAXS data as low as \( q = 0.0085 \text{ Å}^{-1} \)

\( d \sim 75\text{nm} \)
Example 2: NIST Reference Standard
Au Nanoparticles in Liquid Suspension

Reference Material 8011
Gold Nanoparticles, Nominal 10 nm Diameter

<table>
<thead>
<tr>
<th>Technique</th>
<th>Analyte Form</th>
<th>Particle Size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Force Microscopy</td>
<td>dry, deposited on substrate</td>
<td>8.5 ± 0.3</td>
</tr>
<tr>
<td>Scanning Electron Microscopy</td>
<td>dry, deposited on substrate</td>
<td>9.9 ± 0.1</td>
</tr>
<tr>
<td>Transmission Electron Microscopy</td>
<td>dry, deposited on substrate</td>
<td>8.9 ± 0.1</td>
</tr>
<tr>
<td>Differential Mobility Analysis</td>
<td>dry, aerosol</td>
<td>11.3 ± 0.1</td>
</tr>
<tr>
<td>Dynamic Light Scattering</td>
<td>liquid suspension</td>
<td>13.5 ± 0.1</td>
</tr>
<tr>
<td>Small-Angle X-ray Scattering</td>
<td>liquid suspension</td>
<td>9.1 ± 1.8</td>
</tr>
</tbody>
</table>

Mean particle size

Particle size distribution

*https://srmors.nist.gov/view_detail.cfm?srn=8011
Example 2: NIST SRM 8011 Au Nanoparticles – 1D SAXS Scattering Curve

- Scaled direct beam scattering
- NIST SRM 8011 scattering
Example 2: NIST SRM 8011 Au Direct Modeling with Nanofit

- Background corrected SAXS (blue) and fitted SAXS (red)
- Fitting results:
  - Mean sphere radius $= 45.54 \, \text{Å}$
  - Size distribution modeled by a Gaussian distribution with $\sigma = 3.65 \, \text{Å}$
Example 2: NIST SRM 8011 Au Nanoparticles – Comparison

Reference Material 8011
Gold Nanoparticles, Nominal 10 nm Diameter

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Results from D8 Advance SAXS modeled with Nanofit

Mean particle size = 9.108 nm

Figure 6. Particle size histogram generated by TEM analysis.
Example 3: Nano-Metallic Particles Deposited on Carbon Black Substrate

- For SAXS measurements, sample was deposited between 2 pieces of adhesive tape.

- For comparison, this sample was run on the 2D Bruker AXS dedicated SAXS instrument, the NanoSTAR
Example 3: Metallic Nanoparticles
1D SAXS Scattering Curve from NanoSTAR

Azimuthally averaged intensity vs. scattering vector $q$ and the scattering intensity of the holder that is used for correction

$$q = \frac{4\pi}{\lambda} \sin \theta$$

where $2\theta$ is the scattering angle and $\lambda$ is the used wavelength.
Example 3: Metallic Nanoparticles
1D SAXS Scattering Curve from D8 Advance

- **Detector scan:**
  - $2\theta = -2$ to $3^\circ$

- **Step size** = 0.02°

- **Count time** = 2.3 seconds/step

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**Powder in tape**

**Tape only (background)**

![Graph showing 1D SAXS scattering curve with indicated parameters](image-url)
Example 3: Metallic Nanoparticles
1D SAXS Scattering Comparison

- Comparison of background-corrected SAXS scattering shown on a double-log scale
- Intensity is given in arbitrary units and the profiles are separated by a scaling factor
- Note the similarity of the scattering profiles
Example 3: Metallic Nanoparticles Analysis - Comparison

- Scattering profiles after Carbon black is removed

**D8 Advance**

**NanoSTAR**
This profile is fitted assuming a model for spherical structures.

The best fit is obtained using size polydispersity (Schultz).

Fit results:
- Radius: 32.4 Å
- \( \sigma \) of Schultz size distribution: 11.9 Å.
Example 3: Metallic Nanoparticles
Direct Modeling with Nanofit – D8 Advance

- This profile is fitted assuming a model for spherical structures
- The best fit is obtained using size polydispersity (Schultz)

Fit results:
- Radius: 32.2 Å
- σ of Schultz size distribution: 9.7 Å.
Example 3: Metallic Nanoparticles
Nanofit Comparison

- **Fit results (NanoSTAR):**
  - Radius: 32.4 Å
  - $\sigma$ of Schultz size distribution: 11.9Å

- **Fit results (D8 Discover):**
  - Radius: 32.2 Å
  - $\sigma$ of Schultz size distribution: 9.7Å
GI-SAXS Examples
GI-SAXS Geometry Configuration

- Grazing incidence angle near the critical angle is set to make the configuration surface sensitive.
- Detector on secondary diffracted beam track is scanned along $q_y$
- Scattering geometry combining SAXS condition with conditions diffuse x-ray reflectivity
GI-SAXS Geometry
Modify Ultra GID Configuration

Replace Beam Compressor with Soller slit for GI-SAXS configuration
GI-SAXS Example 1 - Au Nanoparticles Embedded a Polymer Matrix

Fit result: 80 Å diameter spherical nanoparticles
GI-SAXS Example 2 – Quantum Dot Film

- Parallel beam
- 0.2 mm slit after mirror
- 0.12 degree thin film attachment (incident)
- 0.12 degree thin film (diffracted beam)

Secondary Detector Scan
- Incident angle = 0.5 deg.
- $2\theta = -5$ to 5 degrees
- 0.024 degree stepsize
- 0.4 s / step
- Total scan time < 3 minutes
Peaks are fit with TOPAS and positions are given in d-spacing.

1st and 2nd order peaks are visible.

89.41(49) Å

45.2(10) Å
Summary
1D SAXS with D8 Advance/Discover

- 1D SAXS capability integrated into multipurpose instruments, D8 Advance and D8 Discover
- Sample stages for investigating liquids, powders, gels, sheets, fibers, thin-films, etc.
- Configuration can be easily modified to obtain maximum resolution or maximum intensity to accommodate the sample
- Powerful GI-SAXS capability using a modification of the Ultra GID configuration
- Dedicated software, Nanofit, for direct modelling of SAXS scattering resulting in a detailed analysis of particle shapes, sizes, size distributions, and concentration effects
2D Method - NanoSTAR

Kurt Erlacher
Audience Poll

Please use your mouse to answer the question on your screen:

What method do you need to analyze preferred-orientation samples?

- 1D SAXS
- 2D SAXS
- Not sure
NanoSTAR

Collimation systems for high flux, high resolution or Nanography

Automatic XY sample stage

Motorized reference sample holder

Multiple X-ray sources available

Sample can be investigated under vacuum or atmospheric condition

Simple alignment concept

D8 based electronics

Integrated radiation safety
IµS - Incoatec Microfocus Source
The Brightest Sealed Tube X-ray Source

- no moving parts,
- very long lifetime without maintenance
- extremely stable
- no water-cooling required
- easy to replace
- low cost of ownership - comparable to common sealed tubes
- significantly more intense than previous microfocus source designs
- operation power 30 W
- 2D parallel beam Montel mirror in an evacuated housing
Turbo X-Ray Source TXS

- Implementation of advanced technologies
  - Direct drive anode
  - Ceramic feed through for cathode powder supply
  - Alignment-free filament mounting
  - New shutter and safety concept, similar to sealed tube D8
Arrangement
- two identical mirrors in a side-by-side configuration

Benefits:
- more compact
- easy alignment
- symmetrical divergence spectrum

Montel mirror: two identical mirrors in a side-by-side configuration (W/C coating, deposit by magnetron sputtering)
Variable Source to Sample Distance

- Pin hole alignment better than 10µm, even under vacuum conditions
- Integration of primary beam path into the radiation safety system
- Easy exchange of pin holes for configuring high resolution set up (larger structures)
- Easy reconfiguration to scanning SAXS 2 pin hole collimation by removing the beam path tubes and pin hole pedestal and sliding the X-ray source along the track
Pinhole Collimation

- Distances between pinholes:
  1st pinhole - 2nd pinhole - 3rd pinhole - sample - detector:
    925 mm - 482 mm - 35 mm - variable
- Diameter of first / second / third pinhole:
  SAXS configuration: 0.75 / 0.4 / 1.0 [mm]
  HRSAXS configuration: 0.5 / 0.15 / 0.5 [mm]
- Available beamstop diameters: 2.0 - 4.2 mm
- SAXS configuration provides approximately 10 times higher flux
- Typical experimental q-range with e.g. 105 cm sample to detector distance:
  SAXS configuration: 0.009 Å⁻¹ to 0.21 Å⁻¹ (700 Å to 30 Å)
  HRSAXS configuration: <0.005 Å⁻¹ to 0.22 Å⁻¹ (>1250 Å to 30 Å)
Nanography
Scanning-SAXS

X-ray Nanography is the non-destructive investigation of nm structures of mm sized samples with µm resolution.

SAXS pattern at the beginning of the crack
SAXS pattern outside the crack
HI-STAR
2D Multi-Wire Detector

- Multi wire gas filled proportional counter
- Real time data collection and display
- High sensitivity and low background
- Dynamic range $> 10^6$
- Energy resolution $< 20\%$
- $>80\%$ single photon sensitivity for Cu-radiation

- The beamstop is made of low fluorescence material to ensure minimum background
- Mounted with Kapton strings for full 360 access to scattered photons
- Alignment accuracy better than $10\mu m$
VÅNTEC-2000
2D Mikro-Gap™ X-Ray Detector

- High Spatial Resolution
  unrivalled data accuracy in precision and accuracy
- High Local and Global Count Rate
- High Dynamic Range
- Radiation Hard
- Inert Counting Gas
  no maintenance required
- Large active area
  conveniently increases $q_{\text{max}}$
NanoFit is an interactive graphic-based, non-linear, least-squares data analysis program for small angle X-ray scattering (SAXS) data by direct modeling.

- The displayed data (blue dots) are the calculated scattering data for a model of Polydisperse Spherical Block Copolymer Micelles with a smooth interface and a Hard Sphere Structure Factor with statistical noise added.
- The red line is the fitted scattering profile using the same model.
DIFFRAC\textsuperscript{plus} NanoFit

- Set of several built-in nano particle models
  - Basic geometrical models (spherical, ellipsoidal, cylindrical).
  - Selected polymer models (flexible and semi-flexible chains, Gaussian star, spherical block copolymer micelle)
  - Polydispersity (Gaussian or Schultz size distribution)
  - Concentration effects (Hard-Sphere or RPA structure factor)

- Automatic Fitting
  - Different refinement methods for automatic evaluation:
    - Levenberg-Marquardt
    - Simplex
  - Online display of intermediate results and changes of the chi\textsuperscript{2} cost function.
  - Selectable fit region.

- Graphical evaluation of one-dimensional data sets
  - Display and comparison of measured and simulated data.
  - Simple, interactive evaluation of SAXS measurements:
    - Easy interactive adjustment of all available model parameters.
    - Wide selection of commonly used axis scaling.
2D Simultaneous SAXS/WAXS

Kurt Erlacher
Simultaneous SAXS/WAXS Experimental Setup

- Turbo X-ray Source, focal spot = 0.1 mm x 1 mm
- Cu-Kα 50 kV / 24mA, from point focus (0.1 mm x 0.1 mm)
- Montel-P multilayer optics
- Diameter of first / second / third pinhole = 750 mm / 400 mm / 1000 mm
- Diameter of beamstop: 4.3 mm
- SAXS:
  sample – detector distance: 1063.5 mm
  Bruker AXS HI-STAR position sensitive area detector
- WAXS:
  sample – detector distance: 51.8 mm
  FUJIFILM FLA-7000 Imaging Plate reader system
- Software: SAXS for Windows™ NT
  SigmaPlot™
Simultaneous SAXS/WAXS

- **FUJI FLA-7000 IP WAXS DETECTOR**
- Image Plate detector system for recording WAXS (wide angle x-ray scattering)
- About 20 x 25 cm large IP is mounted into the NanoSTAR sample chamber
- Read-out of the signal is executed off-line using a FLA-7000 scanner (Fuji)
- Obtained SAXS/WAXS data are read by Bruker AXS 2D software for further data evaluation
Simultaneous SAXS/WAXS Samples

- Reference Materials: Silver Behenate
  Corundum (NBS SRM 674)

- Ordered Mesoporous silica: Meso-SiO2

- Samples were measured at room temperature

- SAXS and WAXS signals were collected simultaneously!

1) samples were kindly provided by M.-O. Coppens, Delft University of Technology, and Rensselaer Polytechnic Institute, Troy NY
System Calibration
Silver Behenate (AgBh)

- AgBh is ideal because it can be used as a calibrant for both, the SAXS (top) as well as the WAXS (bottom) signal
- [http://srs.dl.ac.uk/NCD/station82/silver_behenate.html](http://srs.dl.ac.uk/NCD/station82/silver_behenate.html)
- Debye-Scherrer rings are used for determination of:
  - exact sample-detector distance
  - center position of primary beam
- Measurement time was 120s.
NBS SRM 674
\( \alpha \)-Al\(_2\)O\(_3\)

- \( \alpha \)-Al\(_2\)O\(_3\) is a good standard for the WAXS region only (and was used as a cross reference)
- Notice the sharp peak profile
- Measurement time was 300s
- Incident beam was attenuated by a factor of 10
MESO-SiO2  
2D SAXS / WAXS Pattern

- Individual SAXS (left) and WAXS (right) pattern that were measured simultaneously
- Measurement time was 318 s
MESO-SiO2

- Individual SAXS (left) and WAXS (right) profiles that were measured simultaneously
- Measurement time was 318 s
**MESO-SiO2**

Combined SAXS/WAXS Profile

- SAXS/WAXS profiles that were measured simultaneously
- Intensities are plotted vs. reciprocal lattice vector $q$ (left) and vs. scattering angle $2\Theta$ (right)
- Measurement time was 318 s
Simultaneous SAXS/WAXS experiments were performed on the standards Silver Behenate, $\alpha\text{-Al}_2\text{O}_3$ as well as on the mesoporous silica samples MESO-SiO2.

All samples show distinct scattering characteristic in the wide angle regime.

In order to get a fully continuous profile from the SAXS towards the WAXS region, it is possible to asymmetrically align the Image Plate for WAXS experiments.

In addition, the sample to Image Plate position can be varied such that the max. 2 Theta angle is either around 50°, 70° (current data) or 82°.
NanoSTAR U
Typical Applications

Kurt Erlacher
Typical Applications

- **Gold Nanoparticles**
  - Size dependence on preparation temperature
- **Biological Macromolecules**
  - Dimension of viruses and its monodispersity
  - Conformation state
- **Block Copolymer Micelles**
  - Shape and dimension of micelles
  - Radial excess electron density profile
- **Liquid Crystals**
  - Microdomain structure like lamellar, cylinder or hexagonal array
- **HDPE**
  - Lamellar thickness
- **Nanography**
  - Distribution of mineral particles in trabecular bone
- **Superalloys**
  - Size of precipitates as a function of temperature treatment
Application Gold Nano-Particles

- Preparation according Schiffrin Procedure\(^1\) for series 1 and 8
- Modified procedure to avoid water in the synthesis for an extended temperature range
- All samples were prepared with the same relative amount of gold/thiol (4:1) and gold/NaBH\(_4\) (1:10)

SAXS Profiles and Results

\[ P(q, R) = \left( \frac{3 \sin(qR) - qR \cos(qR)}{(qR)^3} \right)^2 \]

\[ q = \frac{4\pi}{\lambda} \sin(\theta) \]

\[ I(q) \propto \int D(R) V(R)^2 P(q, R) \, dR \]
The Radius of Gyration $R_g$ can be calculated from the particle size distribution $D(R)$ by

$$R_g^2 = \frac{3}{5} \frac{M_8}{M_6}$$

with

$$M_n = \int D(R) R^n dR$$
Comparison with TEM

T = -17.0°C

T = 31.5°C

T = 81.4°C
A clear relation between the average size of the colloids and their preparation temperature is observed.

The size of the colloids is not only controlled by the gold to thiol ratio but also by the temperature.

By means of a non aqueous approach it is possible to expand the temperature interval in which the gold colloids are prepared.

A trend towards a similar temperature dependence was found.

Application TBSV
Tomato Bushy Stunt Virus

Physical and biochemical properties

Particle morphology
Virions isometric; 30 nm in diameter.

Physical properties
One sedimenting component in purified preparations; sedimentation coefficient 135 S. Density 1.36 g cm\(^{-3}\) in CsCl (unfixed).

Biochemical properties
Genome consists of RNA; single-stranded. Total genome size 4.7 kb. Genome unipartite; largest (or only) genome part 4.7 kb.

Features of proteins
Virion protein(s) one; M, 37000; coat protein.

Cytopathology
Virions found in cytoplasm, in nuclei, and in mitochondria

SAXS Data
2D Pattern of TBSV 20 mg/mL

- The sample shows an isotropic scattering behavior
- Measurement time is 5400 s
Background corrected data of the azimuthally averaged scattering intensities of the TBSV sample. The red line gives the fit of the Fourier Transform of the pair distance distribution function $p(r)$ to the experimental data.
The shape of the $p(r)$ functions indicates spherical particles.

- $R_g = 123.1 \pm 0.1 \text{ Å}$
- $I(0) = 121.2 \pm 0.4 \text{ cm}^{-1}$
- $D_{\text{max}} = 320 \text{ Å}$

Note that the error bars of the $p(r)$-function are smaller than the thickness of the drawn red line.

The data were fitted using a program written by Jan Skov Pedersen.
Background corrected data of the azimuthally averaged scattering intensities of the TBSV sample.

Measurement time was 5400s.

The red line gives the fit of the Fourier Transform of the pair distance distribution function $p(r)$ to the experimental data.
The shape of the $p(r)$ function indicates spherical particles.
- $R_g = 120.7 \pm 0.3 \text{ Å}$
- $I(0) = 4.77 \pm 0.03 \text{ cm}^{-1}$
- $D_{max} = 320 \text{ Å}$

The data were fitted using a program written by Jan Skov Pedersen.
TBSV Compact Experiment and Fit

- Linear plots of the low-q-range of the TBSV samples
- Intensity ratio between first peak and the previous minimum is around 10 for the high concentration sample
Application Lysozyme

- Lysozyme in aqueous solution
- The red lines give the fit of the Fourier Transform of the pair distance distribution function $p(r)$ to the experimental data
- The sample was measured in a quartz capillary at $T=4^\circ C$
- Measurement time was 5400s for both, the sample and the solvent

**Resultant pair distance distribution function $p(r)$ normalized by the concentration.**

**6.6 mg/mL:**
- $R_g = 14.8 \pm 0.1 \text{ Å}$
- $I(0) = 0.060 \pm 0.004 \text{ cm}^{-1}$
- $D_{max} = 45 \text{ Å}$

**2.6 mg/mL:**
- $R_g = 14.3 \pm 0.2 \text{ Å}$
- $I(0) = 0.024 \pm 0.003 \text{ cm}^{-1}$
- $D_{max} = 42 \text{ Å}$

Visualization of hen egg white lysozyme as provided by the Protein Data Bank (PDB 2LYZ).
The concentration is about 1.7 wt%.

The red lines give the fit of the Fourier Transform of the pair distance distribution function \( p(r) \) to the experimental data.

The sample was measured in a quartz capillary at \( T = 4^\circ C \)

Measurement time was 5400s for both, the sample and the solvent.

- Resultant pair distance distribution function \( p(r) \) normalized by the concentration.
  - \( R_g = 31.28 \pm 0.03 \text{ Å} \)
  - \( R = 40.4 \text{ Å} \)
  - \( I(0) = 1.230 \pm 0.003 \text{ cm}^{-1} \)
  - \( D_{\text{max}} = 82 \text{ Å} \)
The experimental data can be described very well with a model for spherical particles with a smooth interface and a Hard Sphere structure factor.

The obtained particle size is 40.0 Å with a moderate Gaussian size distribution ($\sigma$ of 2.5 Å).
Application Brij 700 in Water

- Concentration is 1wt%.
- Measurement time was 7200s.
- Fit using advanced model with compact core and highly solvated corona of PEO

- Excess electron density distribution
Liquid crystal sample is a mixture of Pluronic P84 (41 wt%), water (33 wt%) and p-xylene (26 wt%).

The 2D pattern show a weak anisotropy. The anisotropy was maybe caused by slightly squeezing the gel-like sample within the Paste Sample holder.

Measurement time from left to right was 1 min., 5 min., 10 min.

Images show 2D pattern of the scattering intensity.
Background corrected scattering intensity of the sample.

4 Peaks are identified:

<table>
<thead>
<tr>
<th>d [Å]</th>
<th>q [Å⁻¹]</th>
</tr>
</thead>
<tbody>
<tr>
<td>127.9</td>
<td>0.0491</td>
</tr>
<tr>
<td>63.9</td>
<td>0.0982</td>
</tr>
<tr>
<td>42.6</td>
<td>0.1475</td>
</tr>
<tr>
<td>31.7</td>
<td>0.198</td>
</tr>
</tbody>
</table>

Peak positions (1:2:3:4) indicate lamellar microstructure!
Application Liquid Crystal II

- Polymer (34EO) content 60%
- Measurement time was 20 min
Azimuthaly averaged scattering intensity
Transmission of the sample was 0.2894
Peak positions (1:√3:2) indicate **hexagonal** microstructure
Application HDPE
Using the High Resolution Setup

- diameter of first / second / third pinhole = 500 mm / 150 mm / 500 mm
- diameter of beamstop: 2.0 mm
High Resolution SAXS Data
High Density Polyethylene

Azimuthally averaged scattering intensity of the HDPE sample (background corrected)

Intensities are given in absolute units

Measurement time was 600s
Application X-ray Nanography
Investigation of a Bone Section

macrostructure  microstructure  nanostructure

corticalis

spongiosa

~ cm  ~ mm

1.5 nm

67 nm

collagen-mineral fibre composite

thickness of calcium-phosphate platelets: 2-4 nm
Application Nanography
Investigation of a Bone Section

orientation distribution map
of mineral crystals
in human bone

P. Fratzl, H.F. et. Al.
Nanography
Two Scales in One

X-ray Nanography is the non-destructive investigation of nm structures of mm sized samples with μm resolution.

Diameter of X-ray beam (μm-range)

Specimen

Detector

X-ray scattering (nm-range)
Application Superalloys
Precipitates in Inconel 718

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Cr</th>
<th>Fe</th>
<th>Nb</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Mn</th>
<th>Si</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt %</td>
<td>52.67</td>
<td>18.37</td>
<td>18.06</td>
<td>6.00</td>
<td>2.91</td>
<td>1.00</td>
<td>0.45</td>
<td>0.21</td>
<td>0.29</td>
<td>0.04</td>
</tr>
<tr>
<td>at %</td>
<td>51.79</td>
<td>20.39</td>
<td>18.66</td>
<td>3.73</td>
<td>1.75</td>
<td>2.14</td>
<td>0.54</td>
<td>0.22</td>
<td>0.60</td>
<td>0.19</td>
</tr>
</tbody>
</table>

- **Temperature treatment**
  - 2h homogenized at 960°C (Serie A)
  - at 1060°C (Serie B)
  - annealed at 720°C for (30, 60, 120, 240, 480 and 960) min

- **Precipitates:**
  - Ni₃M-Type: Ni₃(Nb, Al, Ti)
  - γ', fcc
  - (γ'', bcc)

sample preparation for SAXS
Inconel Data Treatment

- **Background correction**
  \[ I_c = \frac{I_{\text{raw}} - \tau I_{\text{bg}}}{-\ln \tau} \]

- **Subtraction of „large“ particles**
  \[ I = I_c - a q^{-4} \]
Inconel Scaling Behavior

\[ I(q,t) = \beta_m(t) G(q/\alpha_m(t)) \]

- 2 main parameters

- Mean particle distance

\[ D = 2\pi/(q\alpha_m(t))_{\text{max}} \]

- Radius of Gyration

\[ I_S(q) = \frac{1}{\beta_m} \lambda e^{-\frac{1}{3}(q/\alpha_m)^2 R_g^2} \]
Comparison
SAXS / SANS

**SAXS**
Co-NanoSTAR
- Co-Kα (λ = 1.79 Å) 35 kV / 34 mA
- cross coupled Göbel mirrors
- two pinhole system (100 μm, 300 μm)
- sample to detector distance: 64 cm

**SANS**
D11 facility at the Institute Laue-Langevin (ILL), Grenoble, France
- λ = 8 Å
- Δλ/λ = 9%
- Sample to detector distance: 1.1 m and 4.0 m
Inconel SANS Data

- $q^{-4}$ at $q<0.02\text{Å}$, „large“ particles

- 2 main parameters

![Graphs showing mean particle distance and radius of gyration over annealing time.](image)

Bruker AXS
Inconel TEM Dark Field Micrographs

- **disc like shape**

\[
R_g^2 = \frac{d^2}{8} + \frac{h^2}{12}
\]

radius of gyration

- **annealing time [h]**

- **radius of gyration [Å]**

Bruker AXS
Summary
2D SAXS with NanoSTAR

- Nearly synchrotron like performance on weakly scattering systems
- Fast and automated measurements on virtually any application (analysis of polymers, biological materials, fibres, metals, nanopowders, complex fluids, proteins, etc)
- Analysis of sizes, size distributions, shapes and orientation distributions
- Efficient solution for scientists as well as researchers requiring fully automated measurements
- All in one instrument
  - nanostructure analysis by means of Small Angle X-ray Scattering (SAXS)
  - nanostructure mapping with scanning SAXS / X-ray Nanography
  - molecular structure determination by Wide Angle X-ray Scattering (WAXS)
SAXS – The Big-Angle View

Brian Jones
Thank you for attending!

Please type any questions you may have in the Q&A panel.

Copies of this presentation and related resource materials will be emailed to you.