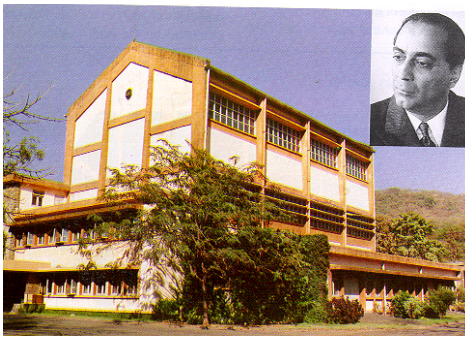
Small Angle Neutron Scattering Facility at BARC, India

SANS-I : Used for Studies of Soft Condensed Matter

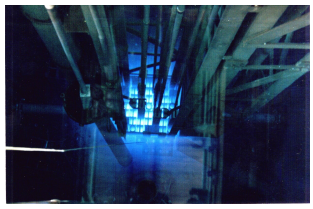
SANS-II: used to study Large inhomogeieties_

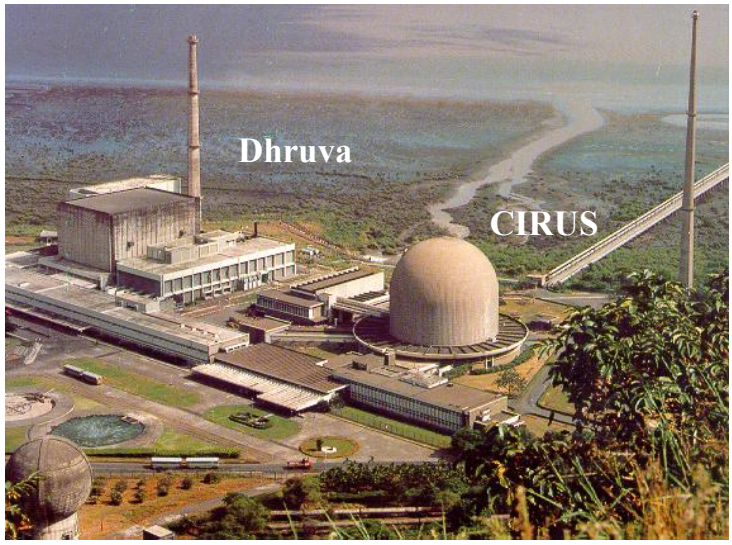
- **ceramics, metallurgical alloys, naturally occuring porous media like rock etc.**

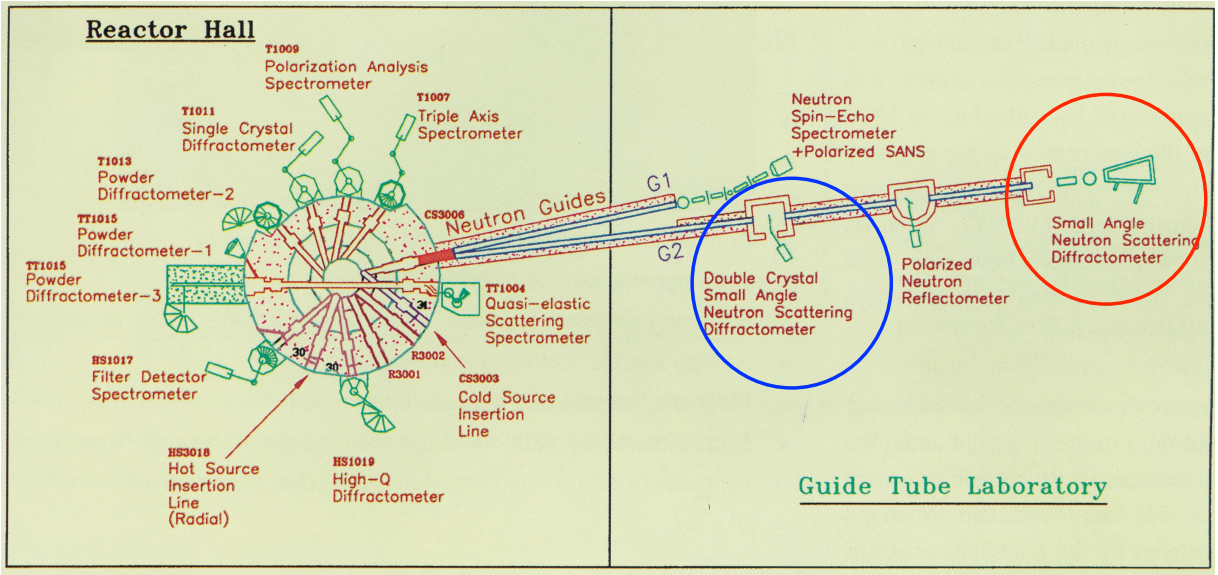
Research Reactors at BARC, Trombay



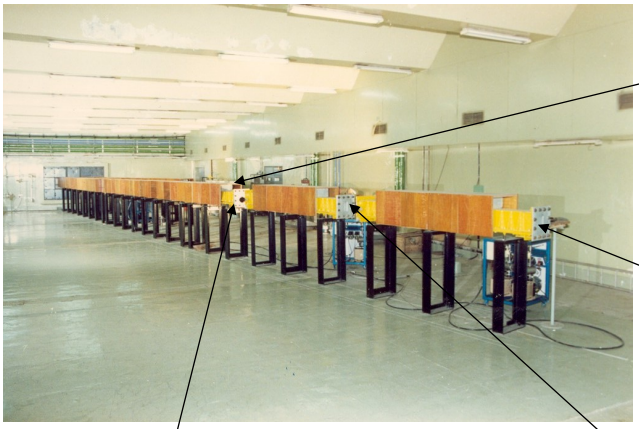
Apsara

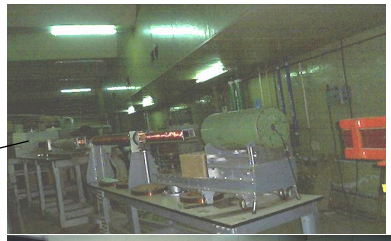






Layout of the NBR facility at Dhruva Reactor, BARC, INDIA





Spin Echo
+
POLSANS

SANS

MSANS

PNR

SMALL ANGLE NEUTRON SCATTERING-I

Beam port	Guide G1
λ^* (guide cut-off)	2.2 Å
Monochromator	BeO Filter at 77 K
$\lambda_{cut\text{-}off}$	4.7 Å
$\lambda_{average}$	5.2 Å
$(\Delta\lambda/\lambda)$	~15 %
Flux at sample	2.2×10^5 n/cm^2/sec
Source slit (S1)	3 cm × 2 cm
Sample slit (S2)	1.5 cm × 1 cm
Distance S1 & S2	2 m
Angular divergence	± 0.5°
Detector (D)	linear He^3-PSD
Distance S2 & D	1.85 m
Q range	0.017 – 0.35 Å^{-1}

The Instrument is suitable for studies of inhomogeneities of the sizes in the range 10 to 150 Å.
Widely used to investigate the structure (shape and size) of different kinds of mesoscopic systems such as micellar solutions, magnetic fluids, protein solutions and colloidal suspensions.

V.K. Aswal and P.S. Goyal, Current Science, 79, 947 (2000)

Small-Angle Neutron Scattering and Soft Condensed Matter

Self-assembly to supramolecular structures for controlling macroscopic properties

Recent publications

- Langmuir 24, 683 (2008).
- *J. Macromol. Sci.: Physics 47, 338 (2008)*
- J. Phys. Chem. B 112, 4594 (2008).
- J. Mol. Liquids 138, 155 (2008)
- *J. Colloid Interface Sci. 320, 452 (2008)*
- *Soft Matter 5, 2919 (2009)*
- *J. Nanosci. Nanotechnol. 10, 6356 (2010)*
- *J. Phys. Chem. B 114, 10986 (2010)*
- *Nanomedicine 5, 575 (2010)*
- Langmuir 26, 17882 (2010)

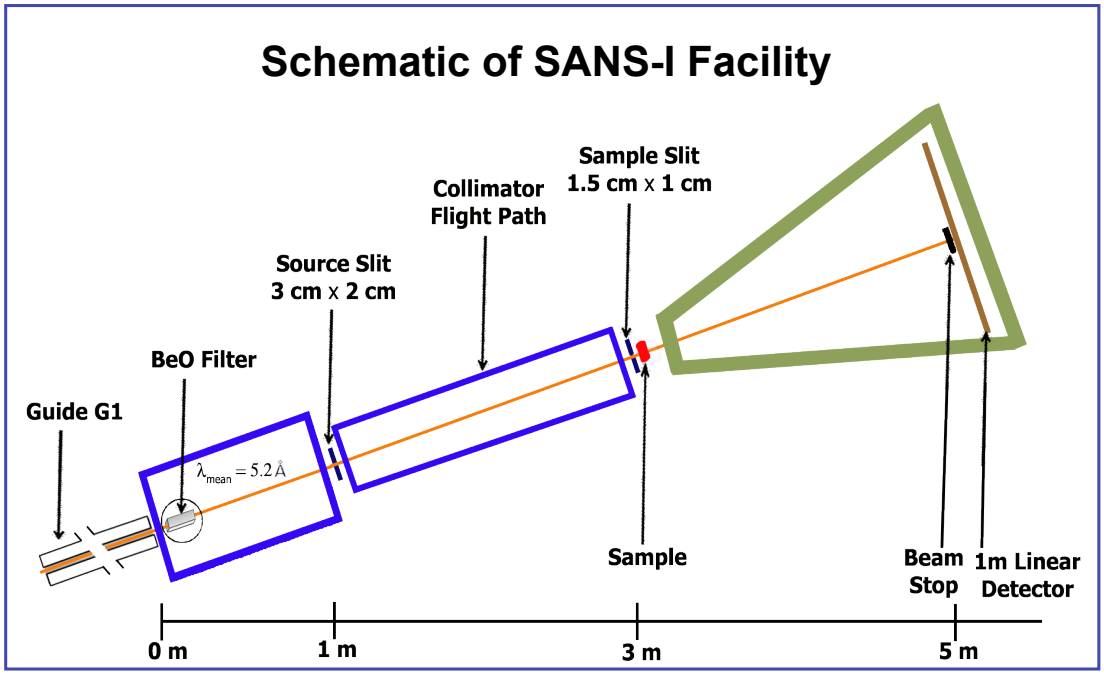
Structural evolution of different phases in protein solution

Recent Publications

- J. Phys.: Condens. Matt.19, 326102 (2007).
- Physica B 398,112 (2007).
- Int. J. Biol. Macromol. 41, 301 (2007).
- J. Phys. Chem. B 111, 10137 (2007).
- J. Pharmaceutical Sci. 96, 2436 (2007).
- Phys. Rev. E 77, 031901 (2008).
- many others

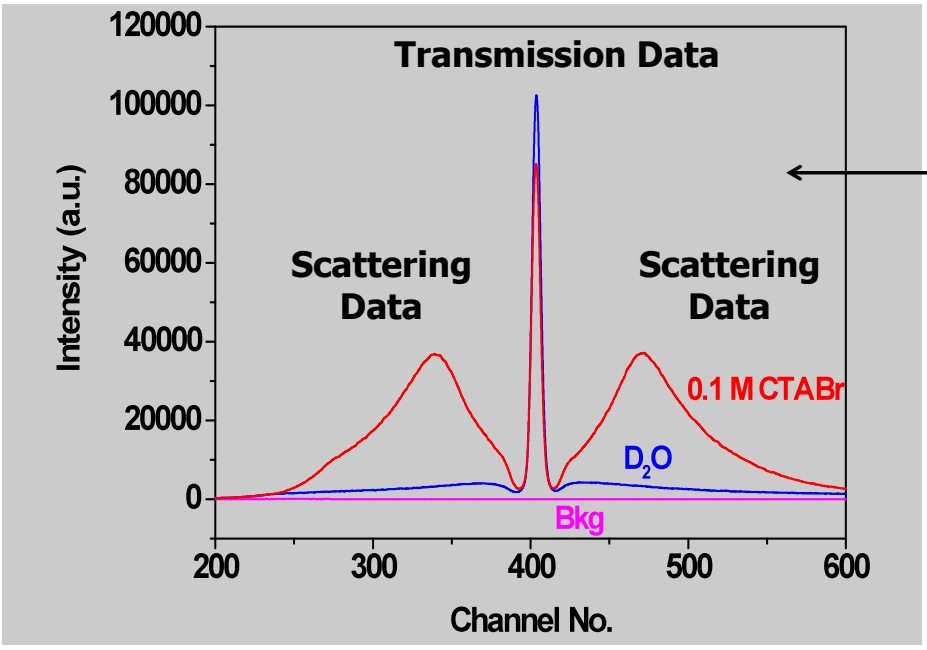
Scattering techniques using neutrons or x-rays provide unique advantages of possibility of varying contrast for soft matter systems and study them under native conditions.

SANS-I Facility at Dhruva Reactors, BARC, India



Fixed Geometry Instrument covers a Q range 0.015 – 0.35 Å^{-1}

Typical SANS Data at Dhruva Reactor



Measuring Time ~ 8 Hrs

Most of the samples are measured in time range of 4 to 12 Hrs.

Data Reduction & Treatment

1. Correction for solvent & background

$$I_{corrected}(Q) = \left[\frac{\{I_s(Q) - I_{bkg}(Q)\}}{T_s} - \frac{\{I_{sol}(Q) - I_{bkg}(Q)\}}{T_{sol}}\right] T_s$$

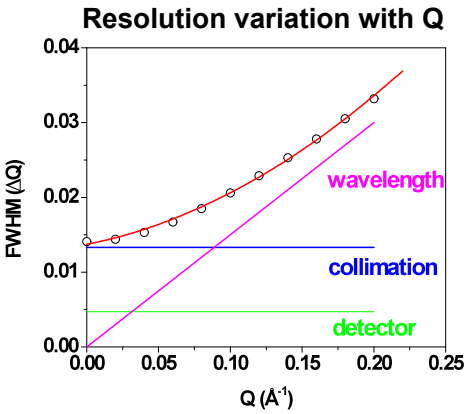
2. Normalization to cross-sectional unit

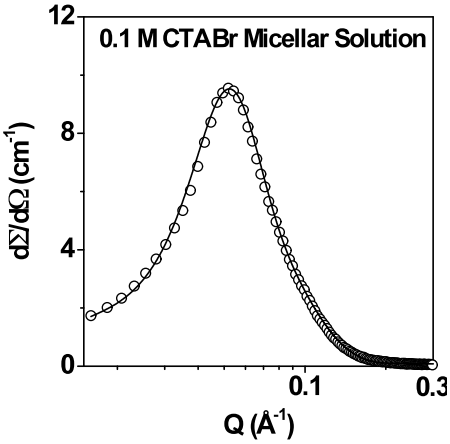
$$I_{corrected}(Q) = K T_s t \frac{d\Sigma}{d\Omega}(Q)$$

$$\left[\frac{d\Sigma}{d\Omega}(Q)\right]_{expt} = \frac{1}{Kt}\left[\frac{\{I_s(Q) - I_{bkg}(Q)\}}{T_s} - \frac{\{I_{sol}(Q) - I_{bkg}(Q)\}}{T_{sol}}\right]$$

3. Resolution Effect

$$\left[\frac{d\Sigma}{d\Omega}(Q)\right]_{expt} = \int R(Q,\bar{Q})\left[\frac{d\Sigma}{d\Omega}(\bar{Q})\right]_{theory} d\bar{Q}$$





Typical SANS Data and Model Fitting

Beam Time Availability on SANS-I

Total reactor working days per year ~ 250

Average days for one beam time ~ 10

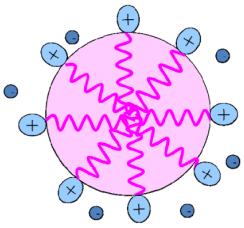
Number of users per year ~ 25

- In-house research
- UGC-DAE CSR Collaborative Research Schemes
- BRNS Projects
- Student Research Projects

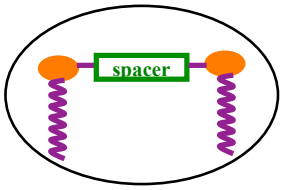
Field of Research: Soft Matter and Nanomaterials

Structure and Interaction of Micelles

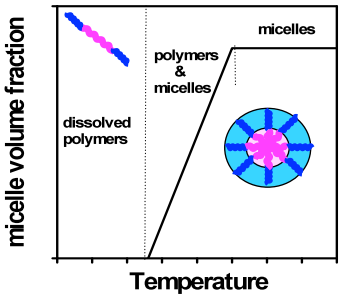
Counterion Condensation on Charged Micelles



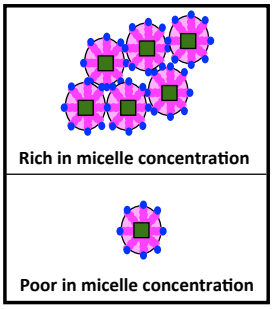
Micellar Structure of Novel Surfactants



Self-assembly of Block Copolymers

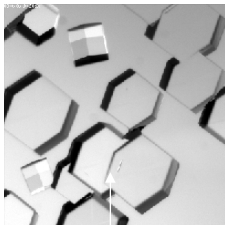


Phase Separation in Micellar Solutions

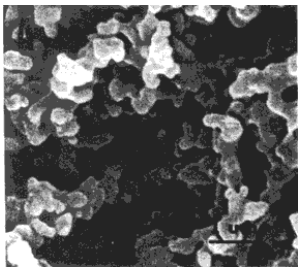


Structural Evolution in Protein Solutions

Coacervation
Characterization of its structures



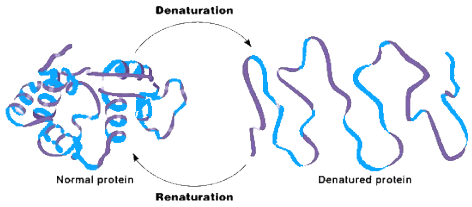
Crystallization
Understanding of Tuning of Crystallization Rate



Different phases in protein solution are followed by structural evolution among the molecules.

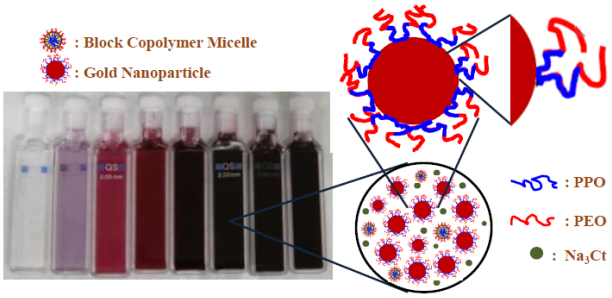
Gellation
Correlation of Rheology with Gel Structure

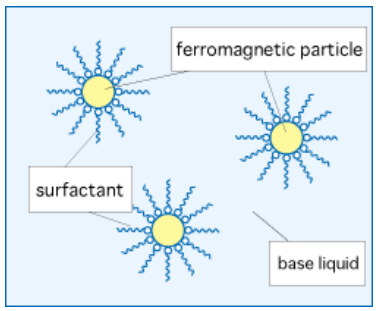
Denaturation
Understanding of Mechanism of Denaturation by Different Means



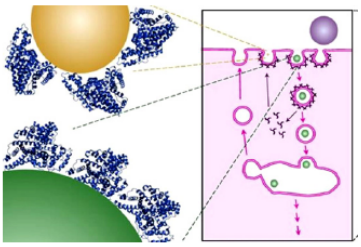
Nanoparticles

Synthesis and Characterization of Gold Nanoparticles



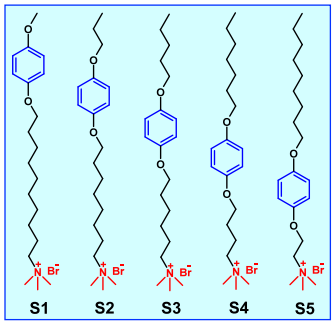


Ferrofluids and Magnetorheological Fluids



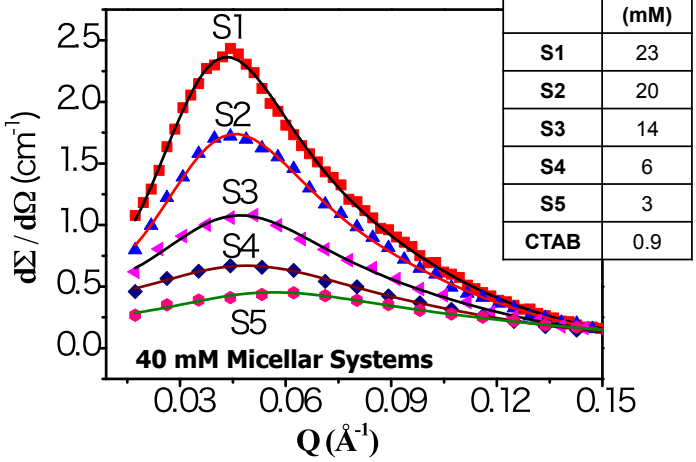
Nanoparticle and Macromolecule Interactions

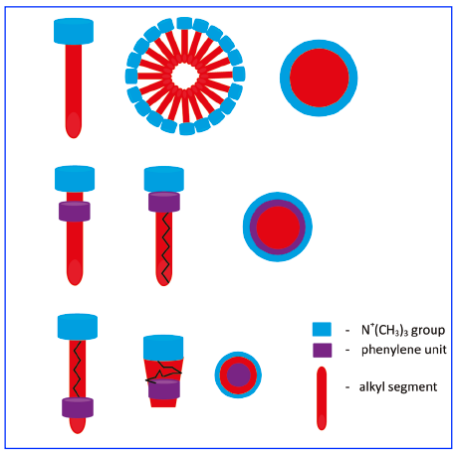
Effect of Chemical Architect on Micellar Structure



Chemical architect of surfactants

S	CMC (mM)
S1	23
S2	20
S3	14
S4	6
S5	3
CTAB	0.9





Micellar structures

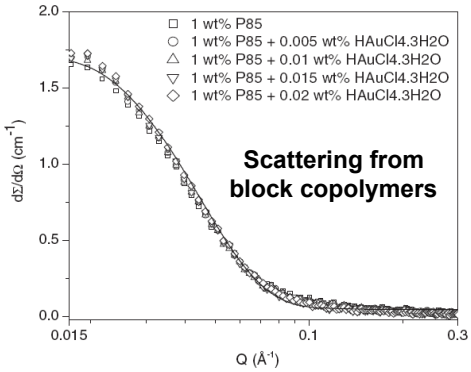
S	CMC (mM)	*N*	Semi-major axis (a) *Å*	Semi-minor axis (b) *Å*	*a/b*	Fractional charge
S1	23	34	24.0	14.0	1.71	0.60
S2	20	58	29.8	16.4	1.81	0.33
S3	14	78	34.7	17.7	1.96	0.29
S4	6	100	38.8	18.9	2.05	0.18
S5	3	120	44.4	19.4	2.28	0.14

[Langmuir 26, 17882 (2010)]

Enhanced High Yield Synthesis of Gold Nanoparticles

Low Yield Synthesis

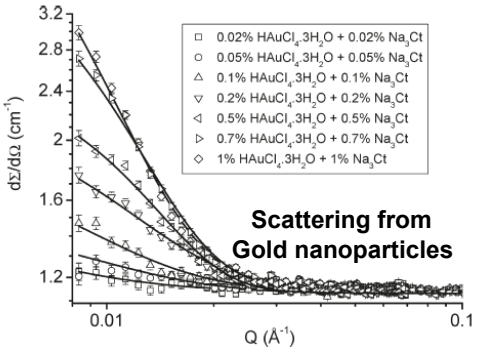
1 wt% P85 with varying gold salt concentration



Most of the block copolymers are unused

High Yield Synthesis

1 wt% P85 with varying gold salt concentration

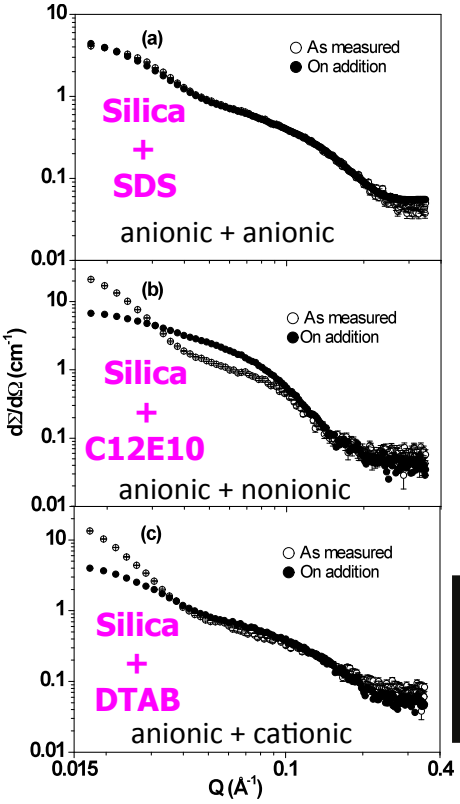


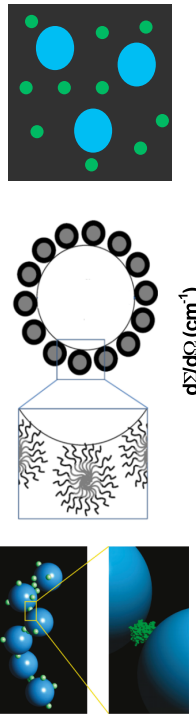
Use of most of block copolymers

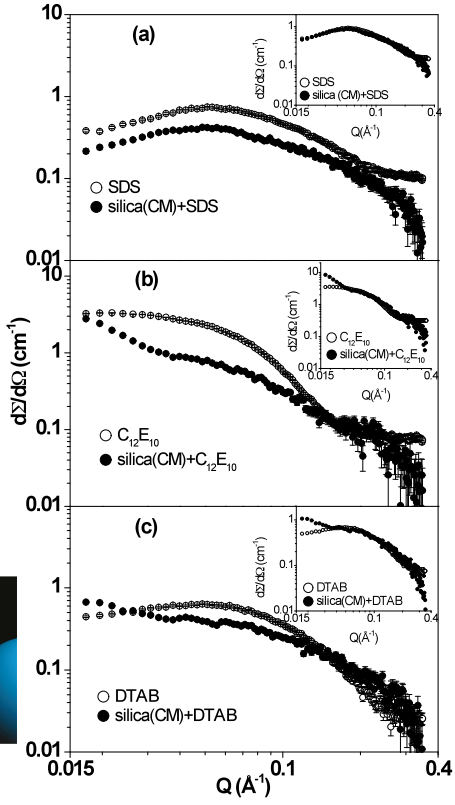
[JNN 10, 6356 (2010) & Langmuir 27, 4048 (2011)]

Nanoparticle-Surfactant Interaction

Both components visible







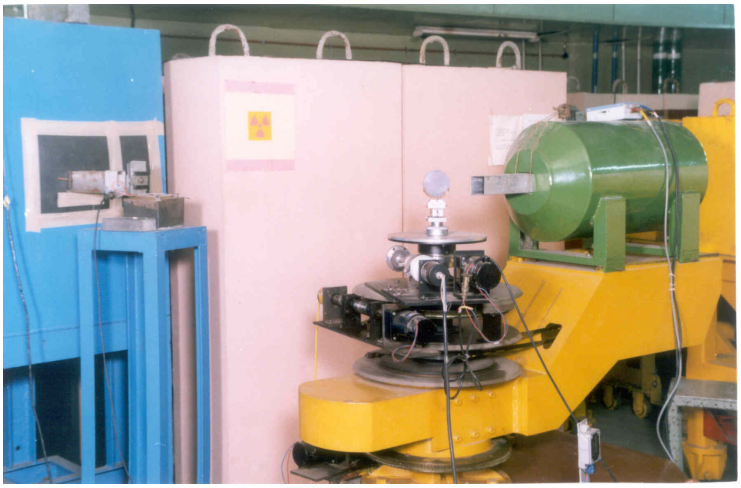
Silica is contrast matched

As measured vs. on addition of components

Surfactant as seen in the Complex

[J. Phys.: Condensed Matt. 23, 035101 (2011)]

SMALL ANGLE NEUTRON SCATTERING-II

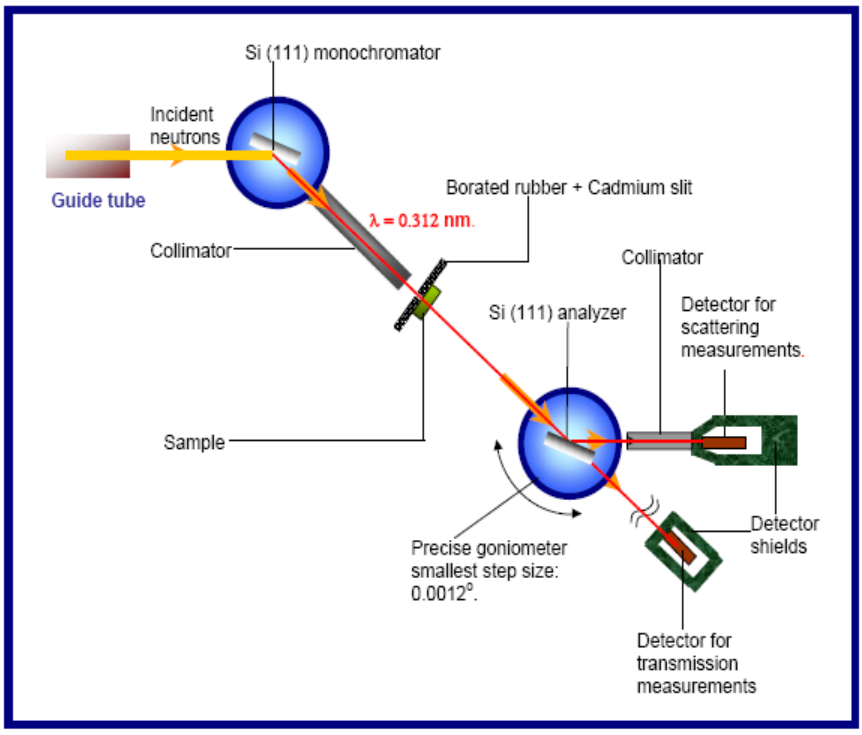


Beam port	Guide G1
Monochromator	Si(111)
Wavelength (Å)	3.12 Å
($\Delta\lambda/\lambda$)	~1 %
Flux at sample	500 n/ cm^2/sec
Analyser	Si(111)
Q range	0.0003 -0.0173 $Å^{-1}$
Real space resolution	200 - 10000 Å
Detector	BF_3 Counter

This is a double crystal based medium resolution small-angle neutron scattering instrument.
The instrument consists of a non-dispersive (1, -1) setting of (111) reflections of silicon single crystals with sample between the two crystals.

S. Mazumder et al., J. Neutron. Res.,9,39 (2001)

SMALL ANGLE NEUTRON SCATTERING-II



S. Mazumder et al., J. Neutron. Res.,9,39 (2001)

SANS from large inhomogeieties

- cements, ceramics, magnet domains etc.
- Studies :

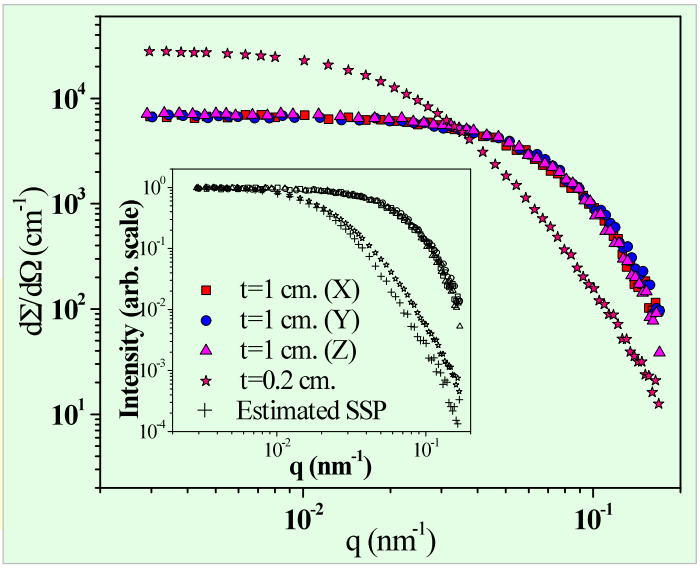
Pore morphology and pore-matrix interface roughening of metamorphosed sedimentary rocks, sandstones, igneous rock and Ceramic sintering samples.

Importance of precipitates in alloys

Precipitate hardening: hinders dislocation movement

Carbide Precipitates in PH 13-8 Mo stainless steel

SANS Study

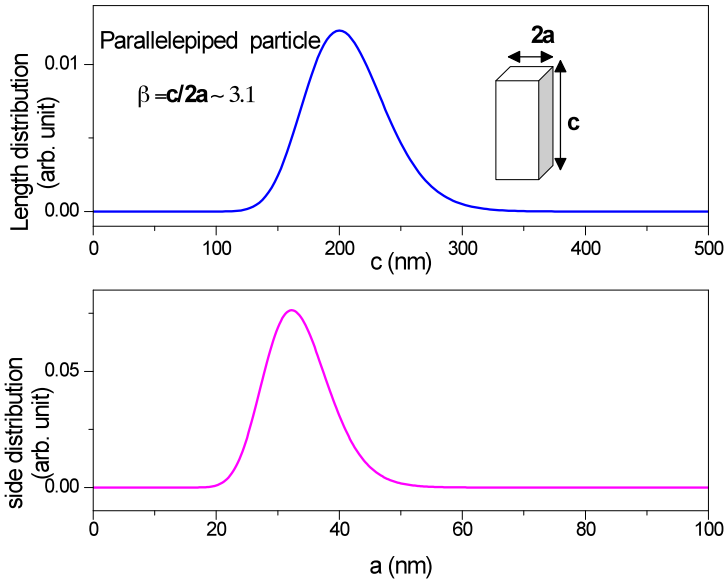


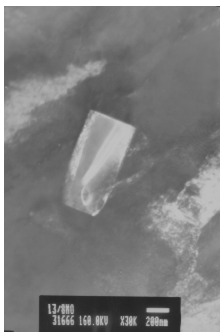
Sample:
Solution quenched from 1000°C

Sample thickness
t= 1 and 0.2 cm.

Inset: **Estimated Single Scattering Profile**

Carbide precipitates in solution quenched PH 13-8 Mo stainless steel





TEM image shows block-like precipitate

Optimum size & size distribution are obtained from SANS data

D. Sen, A.K. Patra, S. Mazumder, J. Mittra, G.K. Dey and P.K. De, Mat. Sci & Eng. A 397, 370 (2005); J. Mittra, G.K. Dey, D. Sen, A. Patra, S. Mazumder and P.K. De, Scripta. Materilia 51 349 (2004).

•SANS-I facility covers a Q range 0.015 – 0.35 $Å^{-1}$ and used to study length scale in the range 1 to 20 nm.

• The facility is used for in-house research and also it has a strong user program. A variety of systems of micelles, protein solutions and nanoparticles have been investigated.

• The number of publications from the SANS-I facility are more than 100 in last 5 years including published in Langmuir, J. Phys. Chem. B, Macromolecules, Phys. Rev. E, Soft Matter, J. Colloid Interface Sci., J. Phys. Condense Matt. etc.

• There are plans to upgrade the facility with neutron velocity selector and 2-D detector.

• SANS-II facility cover a Q range 0.0003 -0.0173 $Å^{-1}$ $Å^{-1}$ and used to study length scale in the range 20 - 1000 nm.

• The facility is widely used to investigate the mesoscopic inhomogeneties in ceramics, metallurgical alloys, naturally occuring porous media like rock etc.