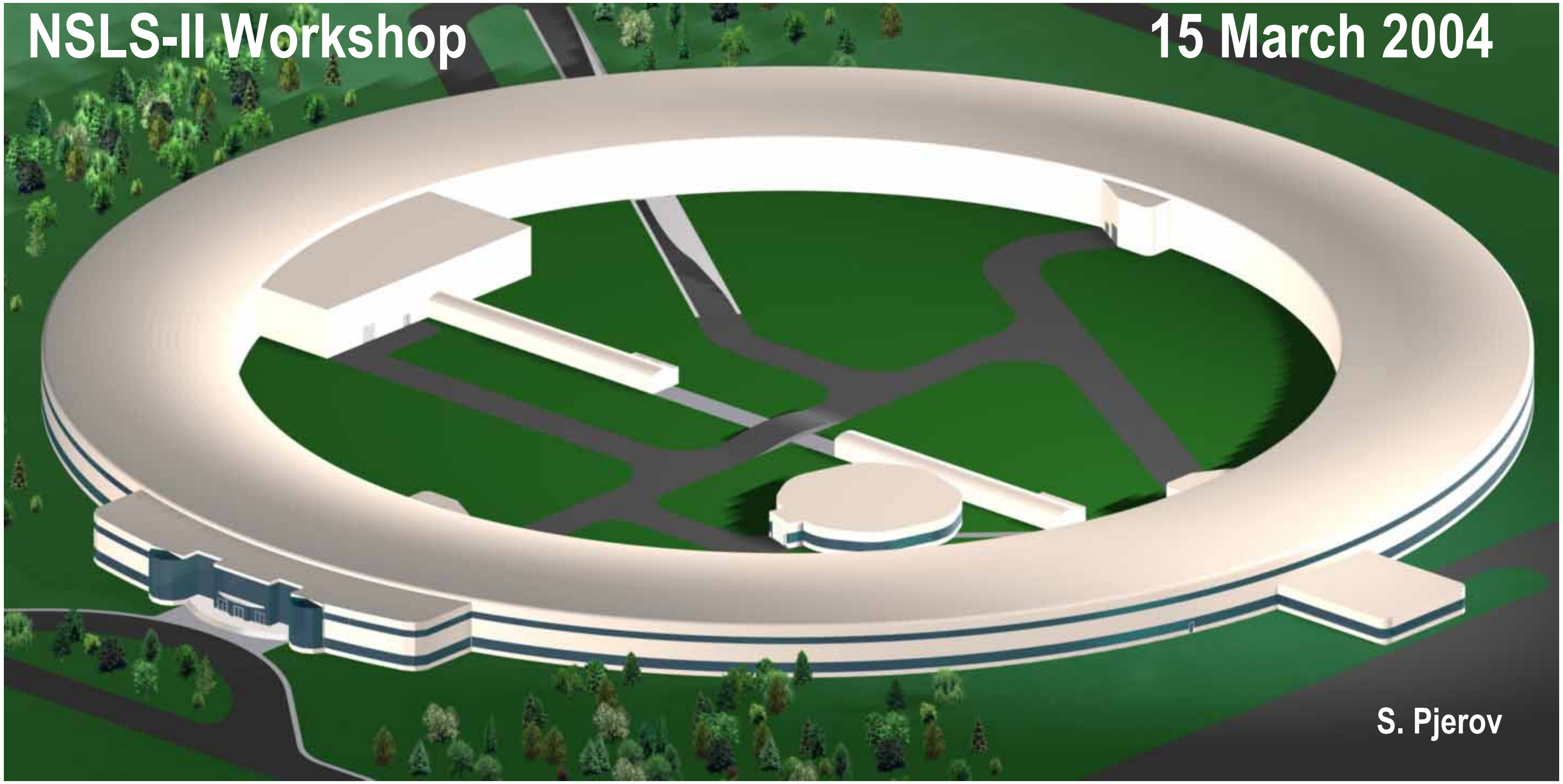
SAXS / XPCS Breakout Session



Workshop on Scientific Opportunities in Soft Matter and Biophysics at NSLS-II

September 5-6, 2003
Student Activity Center, Stony Brook University
Stony Brook, NY

Workshop Participants and Contributors

Participants:

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Contributors:

Ned Thomas (*M.I.T.*)
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Recommendation from the September 2003 Workshop

NSLS-II should be an *integrated project* involving a *parallel development* of all the key project components including:

- Insertion devices optimized for each technique
- Fast x-ray area detectors
- Optics for focusing hard and soft x-ray beams
- User friendly and more sophisticated data reduction and analysis software
- Manipulators for nano-samples
- Complementary in-situ probes
- Ancillary lab facilities for handling and characterizing samples including bio-hazard samples
- Quality user housing
- Training for new users

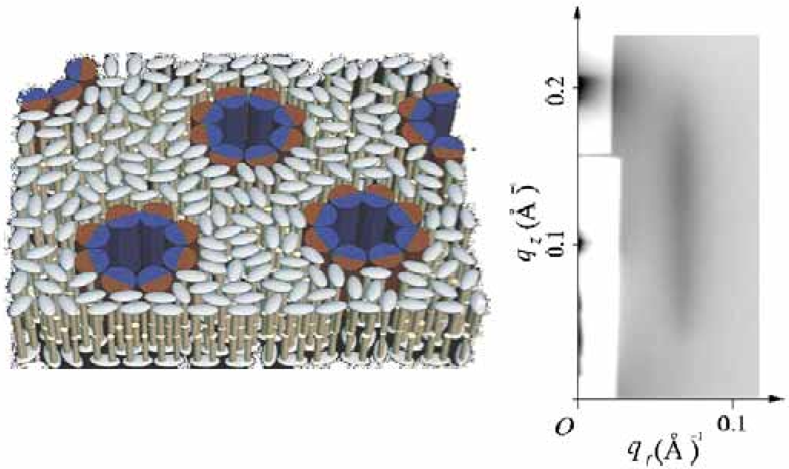
Soft Matter and Biophysics Research Opportunities and Challenges

Highlighted in *three sections* of the NSLS Proposal:

- Structure and Dynamics in Solutions and Membranes
- Nanoscience
- Soft Matter and Biomaterials

Structure and Dynamics in Solutions and Membranes

Challenge: characterize radiation sensitive systems with low X-ray contrast

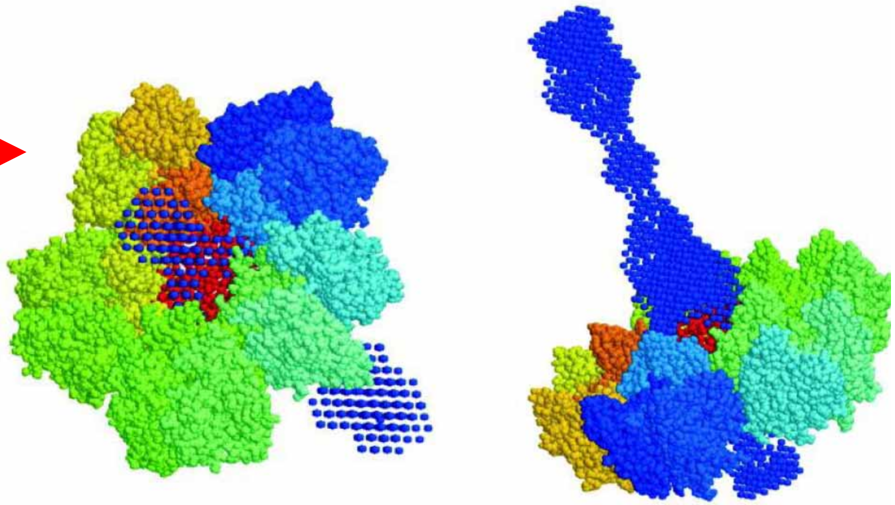


Right: scattering data from an ordered two-dimensional array of membrane pores formed by antimicrobial peptides

Left: model proposed to explain the scattering data

Requires the NSLS-II source combined with fast detectors and advances in data reduction

Low-resolution structural model of the *bacteriophage PRD1 vertex complex* restored from <u>SAXS solution scattering data</u>



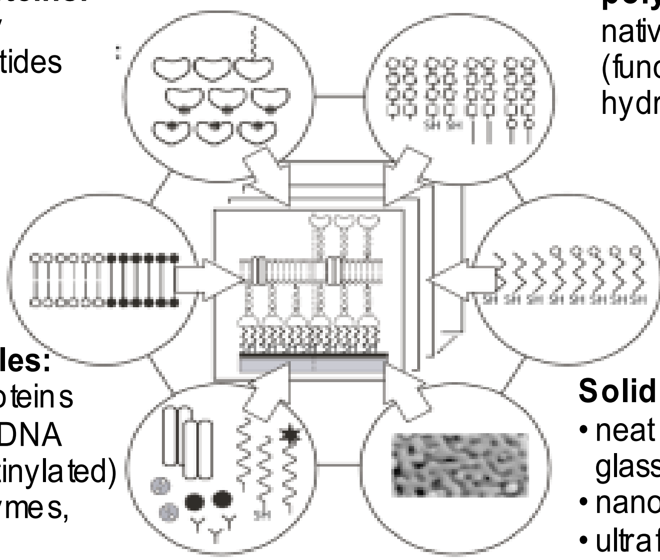
Nanoscience

***Challenge*: Characterize the bio-components that can comprise a supramolecular *construction set* used for interface functionalization**

S-layer (glyco) proteins:
native or genetically modified fusion peptides

Fluid lipid membrane:
- phospholipids
- tetraether lipids

Functional molecules:
- transmembrane proteins
- end-functionalized DNA (e.g., thiolated, biotinylated)
- biomolecules (enzymes, antibodies)
- nanoparticles (metallic, semiconductor)

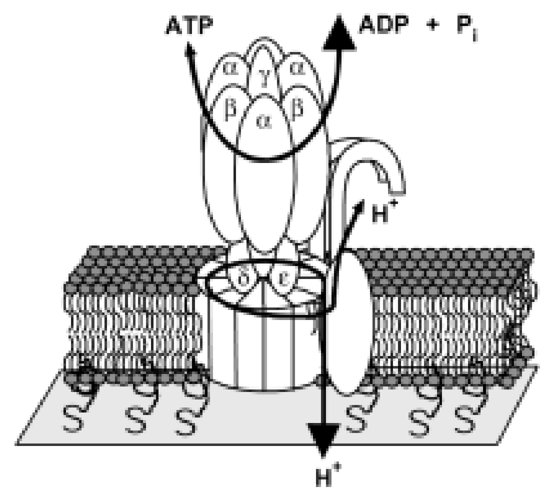


Secondary cell wall polymers (SCWP):
native and modified (functionalized with hydrophobic chains or lipids)

Coupling layers:
- functionalized SCWPs
- Thiolipids
- biotinylated PEGs

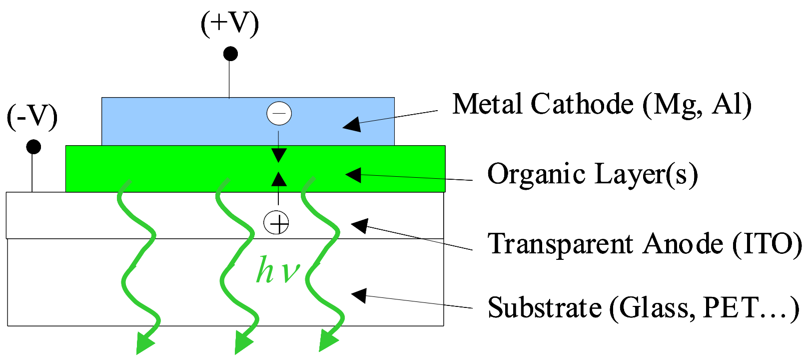
Solid supports:
- neat or gold-coated glass or silicon
- nanoporous gold films
- ultrafiltration membranes

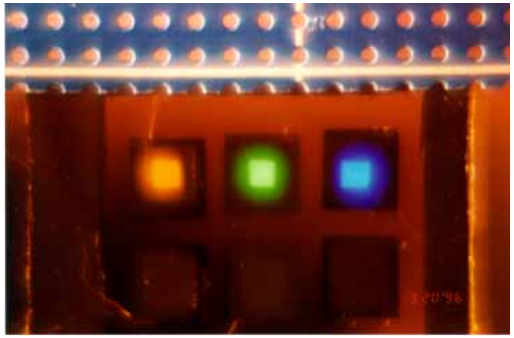
***Goal*: molecular machines, for example, one based on an ATPase reconstructed in an interfacial membrane mimic** →



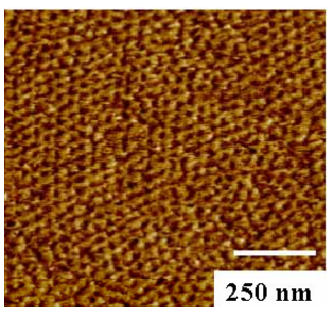
Nanoscience

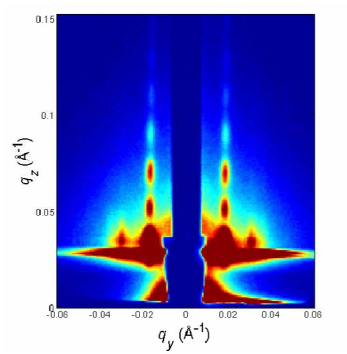
***Challenge*: characterize the structure of thin-film organic devices and nanopatterned organic films formed by directed self-assembly**

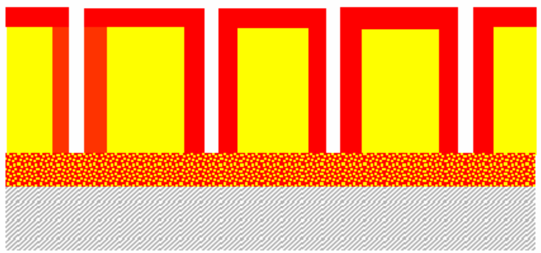




***Left*: schematic of an organic electroluminescent device, where the thickness of the active organic layer is ~100 nm. *Right*: photo of an integrated three-color organic device based on a dye-doped polymer.**







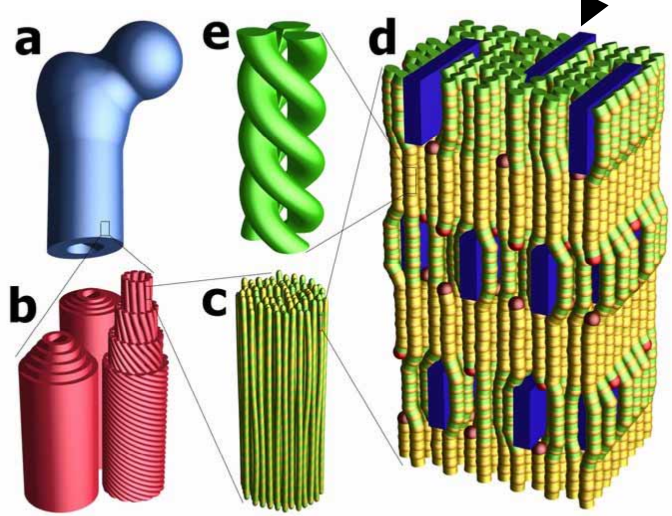
***Left*: AFM image of a hexagonal nanoscale pattern self-assembled in a solution-processed copolymer film. *Middle*: GISAXS diffraction from patterned film. *Right*: model of through-pores supported by X-ray data.**

Soft Matter and Biomaterials

Challenge: characterize systems that order over broad range of length scales from 0.1 nm to 10μm

Hierarchical structure of bone

~10nm embedded mineral crystals



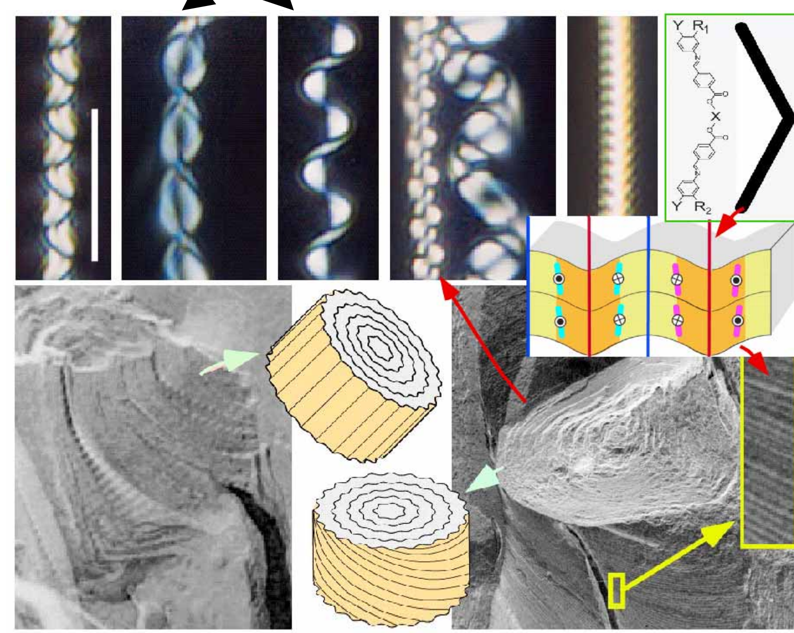
5 μm dia. bundles of 500 nm dia. collagen fibers

1.5 nm dia. collagen molecules

Hierarchical filamentary liquid crystal structures

Few μm dia. Helical filaments

0.3 nm long bent-core molecules



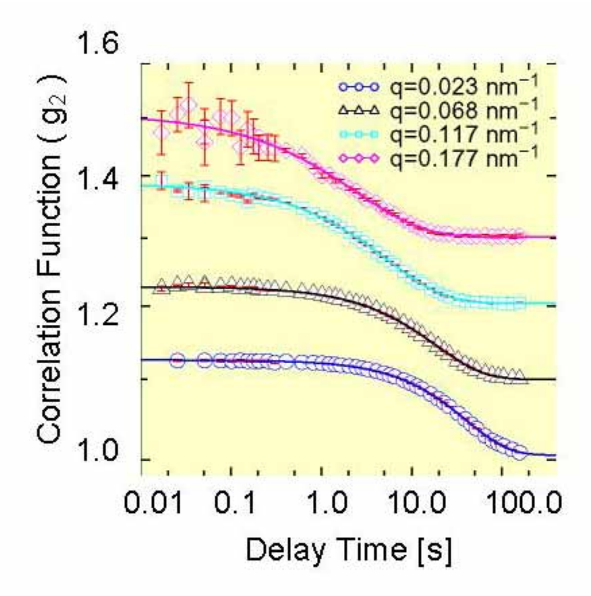
Undulated layers with a 100 nm period

Impact of *NSLS-II* on Soft Matter and Biophysics Synchrotron Research Techniques

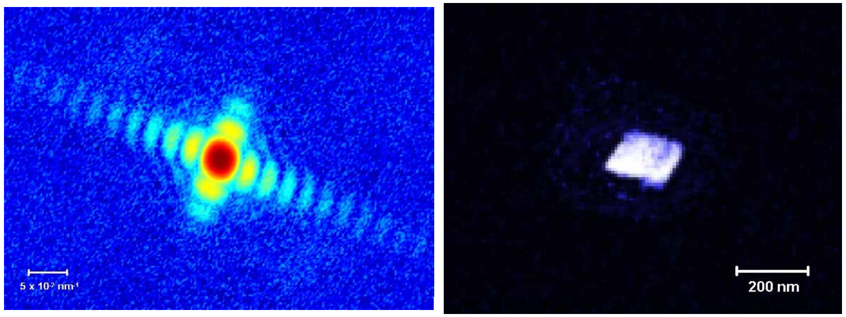
- The high *brightness and coherence* will dramatically enhance X-ray techniques as
  - XPCS
  - X-ray focused-beam techniques as SAXS and GISAXS applied to small samples or used to probe spatial inhomogeneities
  - Flow experiments where spatial-resolution translates into time-resolution
- The high X-ray *flux* will enhance flux-limited techniques as time-resolved SAXS and time-resolved GISAXS
- High brightness and flux at lower energies (1-4 keV) will enhance resonant X-ray techniques at the Si, S, P, and Cl K-edges that are of interest in the study of soft matter and biophysics systems

Impact of *NSLS-II* on XPCS and Coherent Imaging

State-of-the-art XPCS measurements on a block copolymer: measurement time-resolution ~ 10 msec.



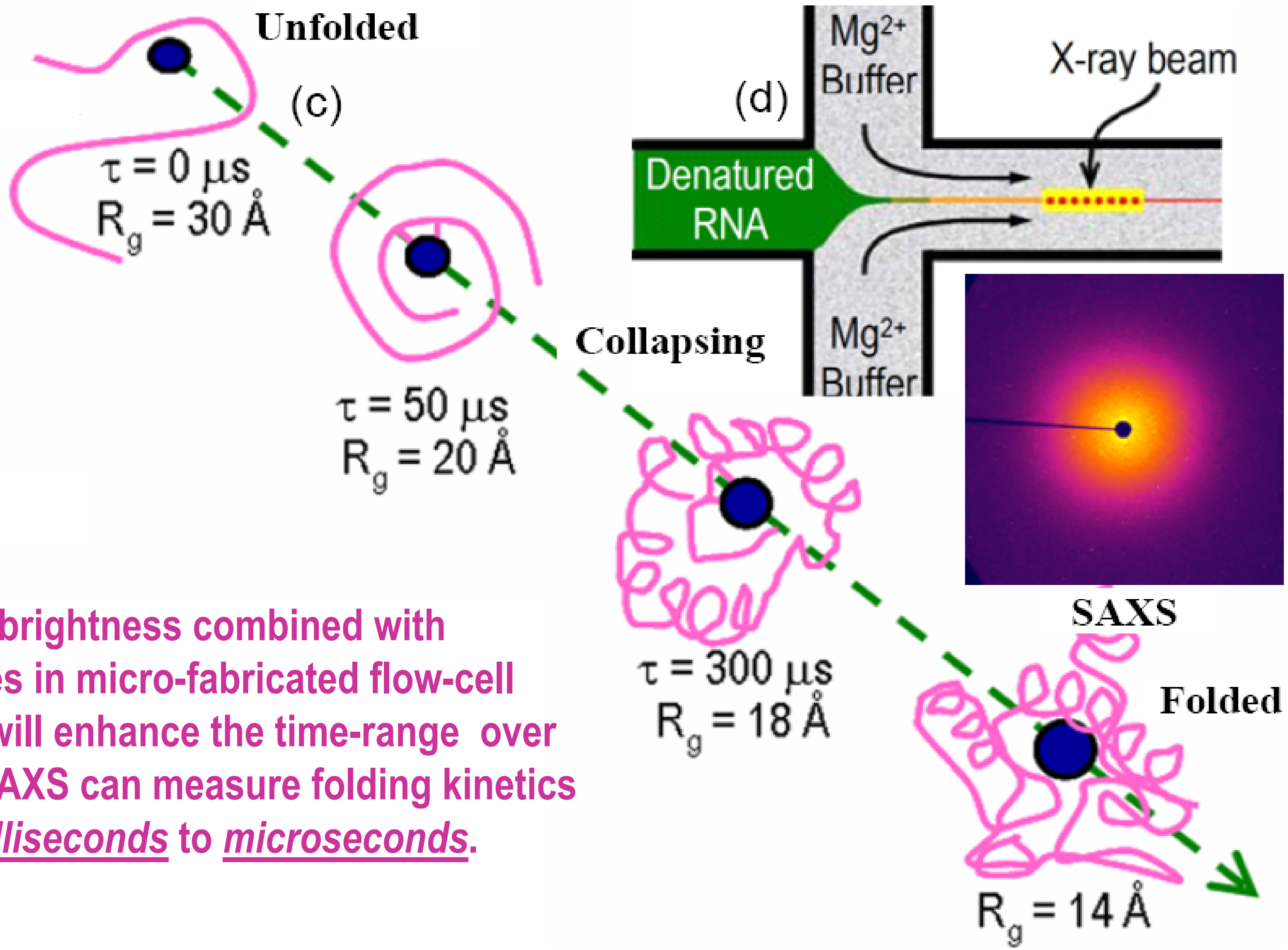
NSLS-II combined with faster detector will extend the time-resolution of XPCS to ~ 100 nsec.



***Left*: coherent X-ray diffraction from a 160 nm silver nanocube. *Right:* reconstructed image**

NSLS-II will enable the measurement of objects ~ 10 nm in size.

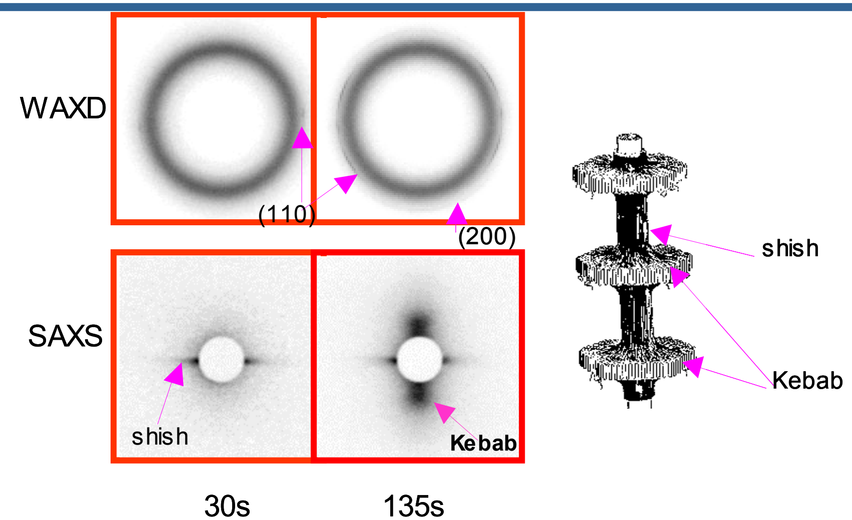
Impact of *NSLS-II* on Solution Scattering



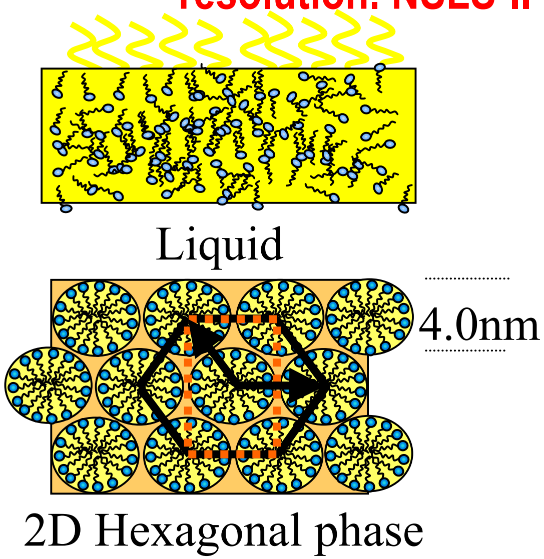
NSLS-II brightness combined with advances in micro-fabricated flow-cell mixers will enhance the time-range over which SAXS can measure folding kinetics from <u>*milliseconds*</u> to <u>*microseconds*</u>.

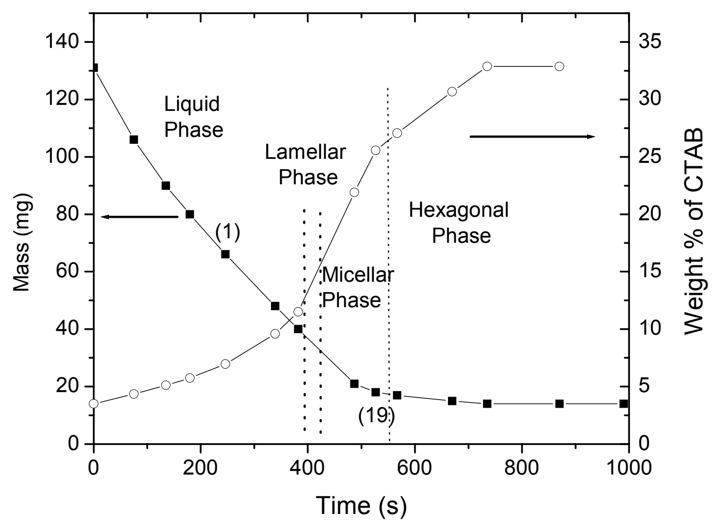
Impact of *NSLS-II* on Time-Resolved SAXS and GISAXS

In-situ SAXS / WAXS measurements during polymer processing: first extended-chain "shish" form followed by crystalline folded-chain "kebabs".



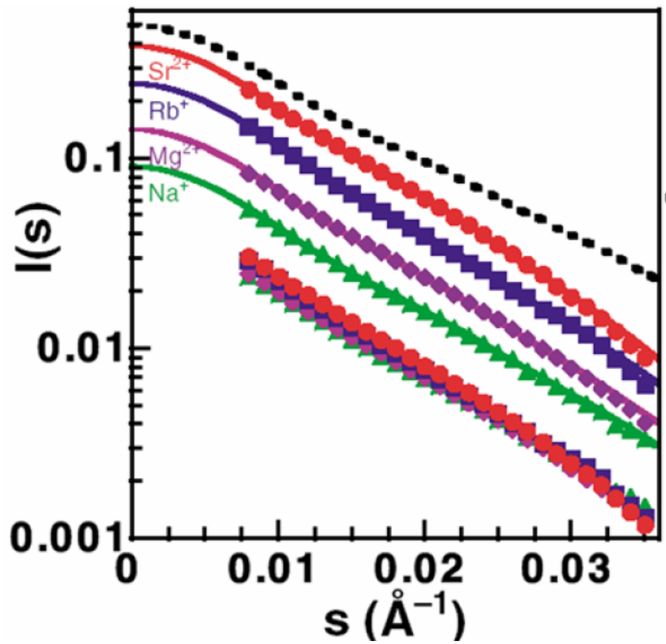
Weakly scattering systems can currently be observed with a ~ 1 second time-resolution. NSLS-II will improve time-resolution to sub-millisecond times.

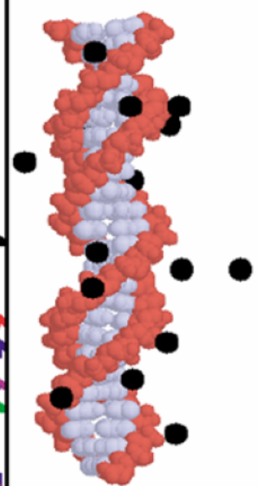




Liquid crystalline phase formation tracked during solvent evaporation of a silica surfactant solution.

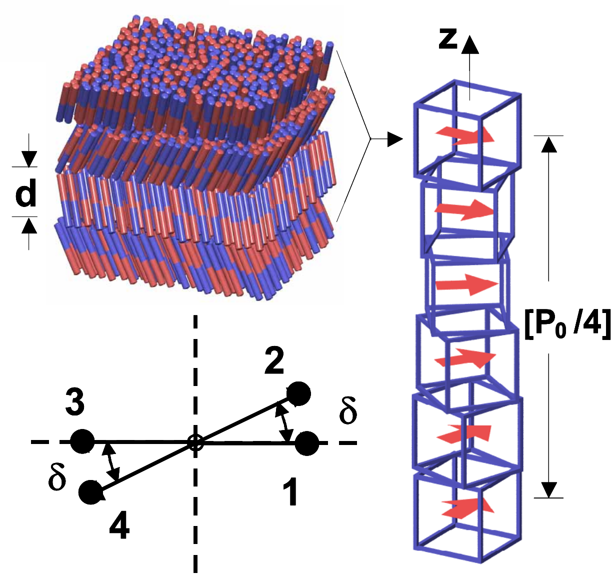
Impact of *NSLS-II* on Anomalous SAXS and Resonant Scattering

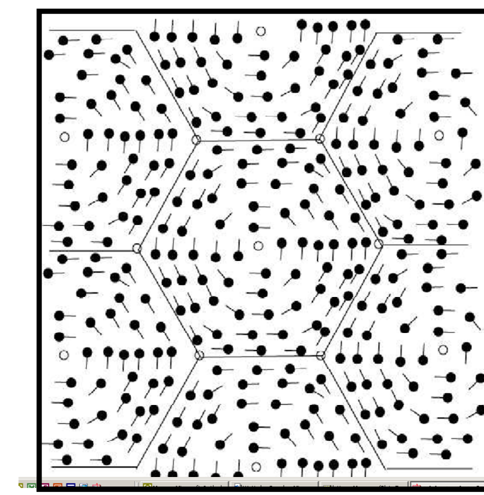




Counterion clouds surround negatively charged DNA in solution - anomalous SAXS enables a measurement of counterion correlations

Inter-layer and intra-layer orientational ordering with nanoscale periodicities can be measured with low-energy resonant X-ray scattering at S, Cl, P, Si edges - common atoms in organic molecules





Scattering Beamlines at the NSLS Utilized for Research on Soft Matter and Biophysics

BEAMLINE	TECHNIQUE	USAGE(%)
X1A1	STXM	25
X1A2	STXM	25
X3A2	SAXS	100
X10A	SAXS, XRD	100
X10B	XRD	100
X13B -ID	Microprobe	33
X16C	XRD	10
X18A	XRD,XRR	33
X19A	RXRD	10
X19C	GISAXS, Liq Surf	50
X20A	Micr oprobe, XRD	33
X21 -ID	SAXS	33
X22A	XRD	50
X22B	GISAXS,Liq Surf	100
X27C	SAXS,WAXS	100

SUMMARY OF CURRENT FACILITY USAGE

1.33 (2.0)	ID -Beamlines (when X9 -ID replaces SAXS on X21)
7.35	Bend Magnet Beamlines

~ 9 Full-Time Beamlines Total

Suite of Insertion Device Beamlines for Soft Matter and Biophysics Research

ID Beamline	Source feature emphasized	Techniques enabled
SAXS	Small (μm) mirror-focused beam	SAXS, WAXS
GISAXS	Small (μm) mirror-focused beam, downward-deflection	GISAXS, GID, Liquid Spectrometer
Nanoprobe 1	Ultra-small (~10nm) optic-focused beam of hard x-rays (3-20keV) Complementary in-situ probes (microscopy, spectroscopy, rheology)	High-res tomography, High-res anomalous and fluorescence imaging, Diffraction-imaging
Nanoprobe 2	Ultra-small (~10nm) optic-focused beam of soft x-rays (0.5-3keV)	Spectromicroscopy, Diffraction-imaging
ASAXS	Tune-ability (1-15 keV)	ASAXS, AWAXS, Resonant Scattering
USAXS	High-brightness (long length, optics free)	XPCS, USAXS, Imaging
IXS	Energy resolution	Inelastic X-ray Scattering

Breakout Session Agenda

2:00 - 2:15 pm	Ron Pindak *BNL-NSLS*	Summary: Workshop on "Scientific Opportunities in Soft Matter and Biophysics at the NSLS-II"
2:15 - 2:45 pm	Tobin Sosnick *University of Chicago*	"SAXS and Collapse in Protein Folding"
2:45 - 2:55 pm	Lin Yang *BNL-NSLS*	"Focused Discussion on Optimizing Solution Small Angle Scattering:Fast Detectors, High-Throughput, Time-Resolved"
2:55 - 3:25 pm	Robert Leheny *Johns Hopkins University*	"XPCS and the Slow Dynamics in Clay Gels"
3:25 - 3:35 pm	Simon Mochrie *Yale University*	"Focused Discussion on Optimizing a Beamline for X-Ray Photon Correlation Spectroscopy"
3:35 - 3:50 pm	Group Photo and Coffee Break	
3:50 - 4:20 pm	Detlef Smilgies *Cornell University*	"GISAXS: A Tool for the Study of Structure and Kinetics of Organic Thin Films on Nanoscopic Length Scales"
4:20 - 4:30 pm	Ben Ocko *BNL-Physics*	"Focused Discussion on GISAXS and GID: Microfocused Coherent Beams and Parallel Detection"
4:30 - 5:00 pm	Open Discussion	