Argonne NATIONAL LABORATORY

SAXS at APS

Presented by

Pete R. Jemian

group leader, APS beam line controls and data acquisition

2010-01-12

HDF5 as hyperspectral data analysis format

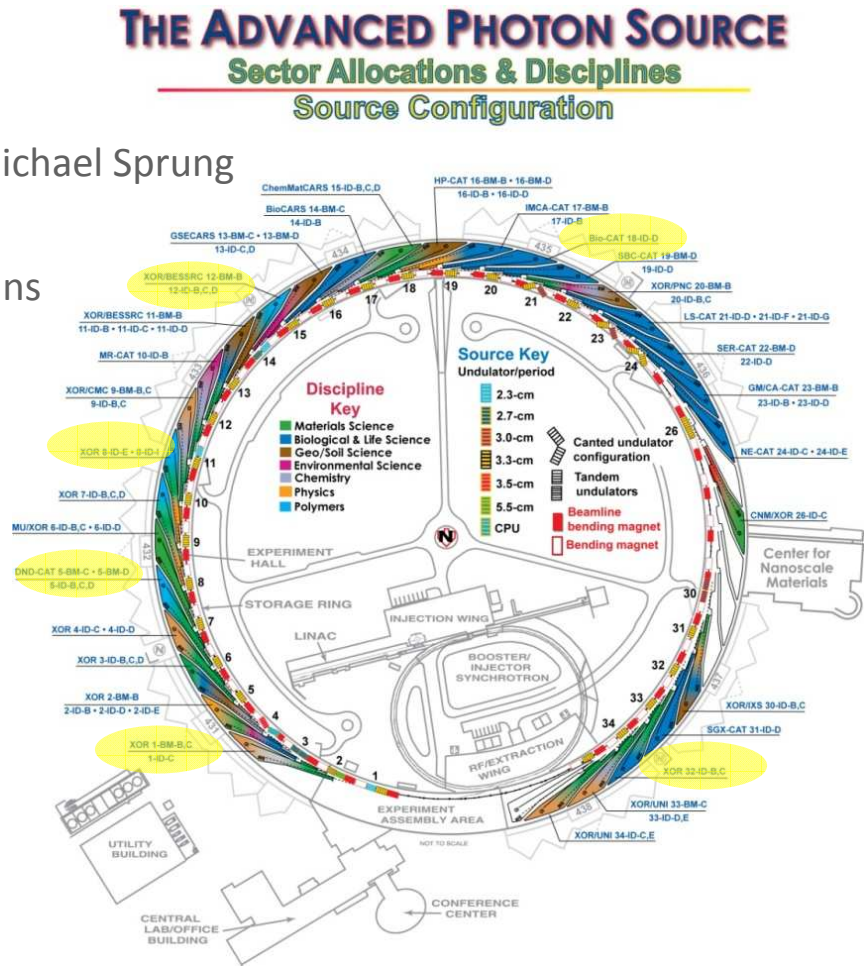
ESRF, Grenoble, France

Big thanks to the organizers (Andy Götz and V. Armando Solé) for hosting this conference!

Overview

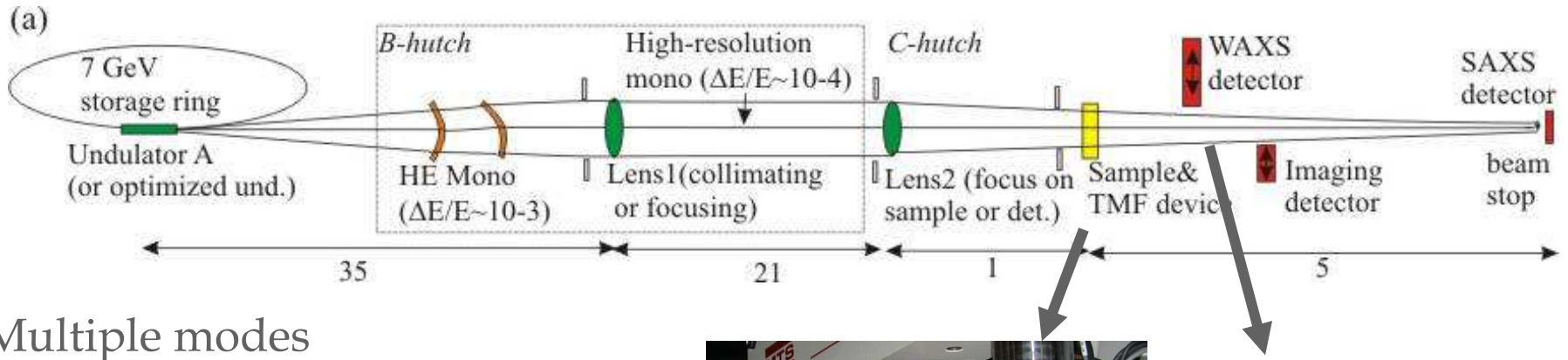
- SAXS Beam Lines at the APS
  - 1ID High-Energy SAXS/WAXS, Jon Almer
  - **5ID SAXS-WAXS**, Stephen Weigand
  - **8-ID XPCS**, Alec Sandy, Suresh Narayanan, Michael Sprung
  - 8-ID GISAXS, Jin Wang
  - 12-BM SAXS, Nadia Leyarovska , Randy Winans
  - **12-ID SAXS**, Sönke Seifert, Randy Winans
  - 12-ID GISAXS, Byeongdu Lee , Randy Winans
  - **18-ID SAXS**, Tom Irving, Liang Guo
  - **32-ID USAXS**, Jan Ilavsky
- Current status and parameters
- Software in use by some instruments

- canSAS 1-D v1.0 XML Standard for SAS Data



Slide(s) courtesy of Jon Almer

1-ID: HE-SAXS/WAXS

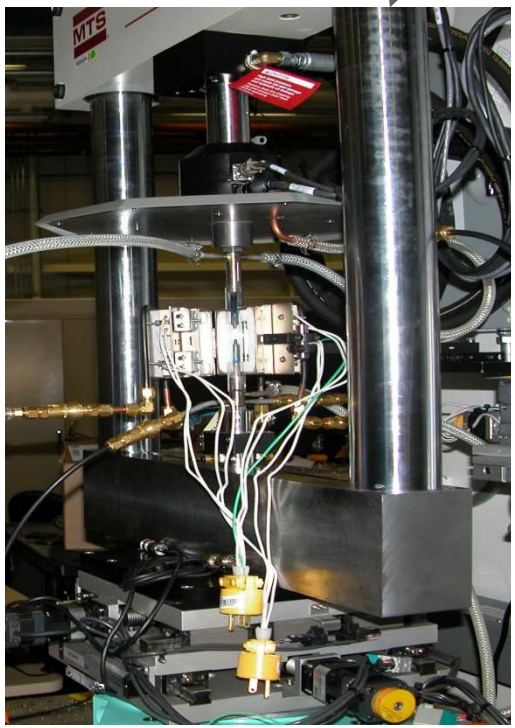


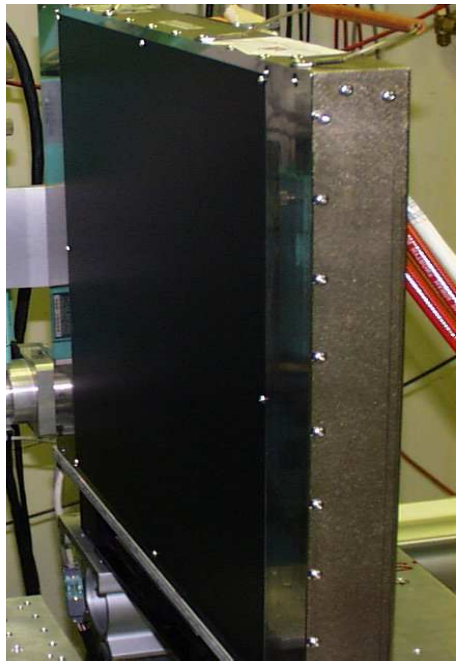
Multiple modes

- WAXS (down to d~1 A)
- SAXS (up to d~5000A)
- Imaging / radiography
- Flourescence

Transverse beam size down to ~1x10 μm^2 using lenses

Diffraction tomography under development





Slide(s) courtesy of Stephen Weigand

5-ID SAXS/WAXS
Materials Science, polymers, solutions, and biology

ultra-low-background pinhole-camera SAXS data down to 0.001 Å-1.

Energy (wavelength)	8 keV to 18 keV (1.5 Å to 0.7 Å)
Beam size V/H	50 µm × 50 µm to 1 mm × 2.5 mm
Camera length	136 mm to 10,000 mm
q range (d spacing)	0.001 $Å^{-1}$ to 5 $Å^{-1}$ (6200 Å to 1.26 Å)
SAXS detector	162 mm marCCD (Rayonix)
SAXS/WAXS detector	Roper Scientific®, up to 2 frames/sec

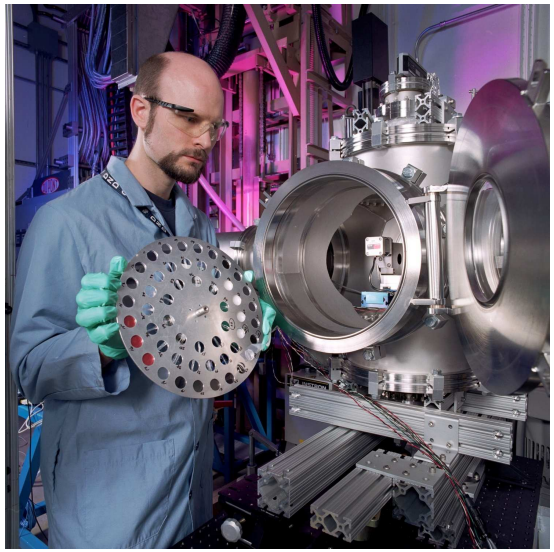
Dynamic studies of polymer crystallization, melting, and deformation

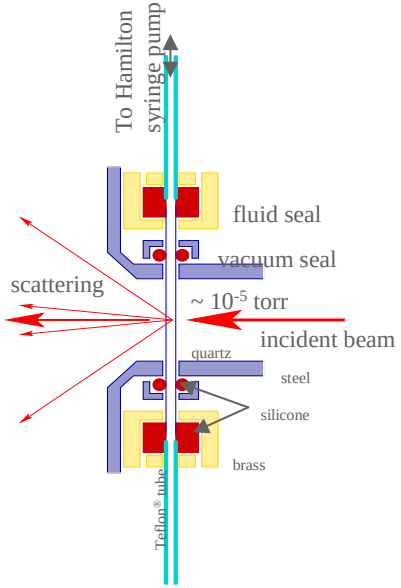
Five sets of slits
camera lengths up to 10 m
remote in-vacuum sample changer

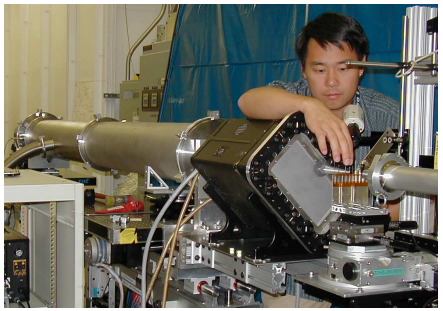
Linkam thermal stage,
Differential Scanning Calorimetry (DSC) cell,
or Instron servo-hydraulic system

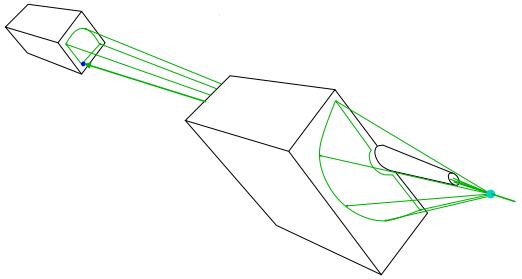
Uses Fit2D and user's preference software; Needs instrument software!

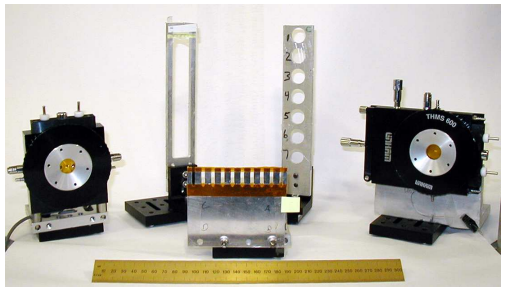
5-ID has many different sample environments

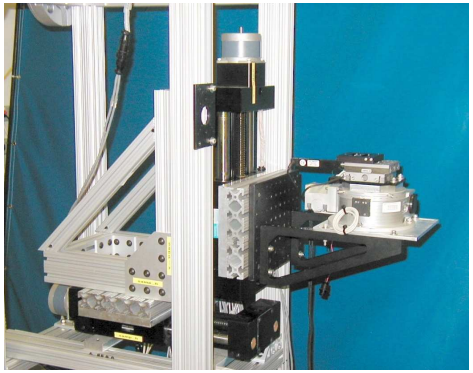


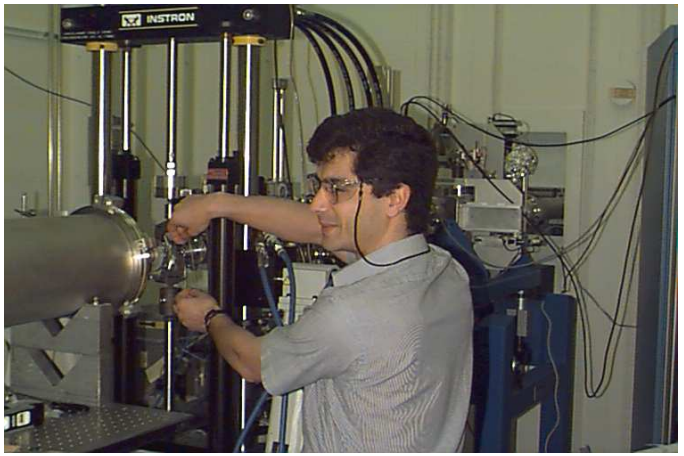


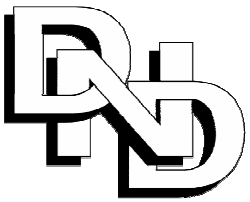








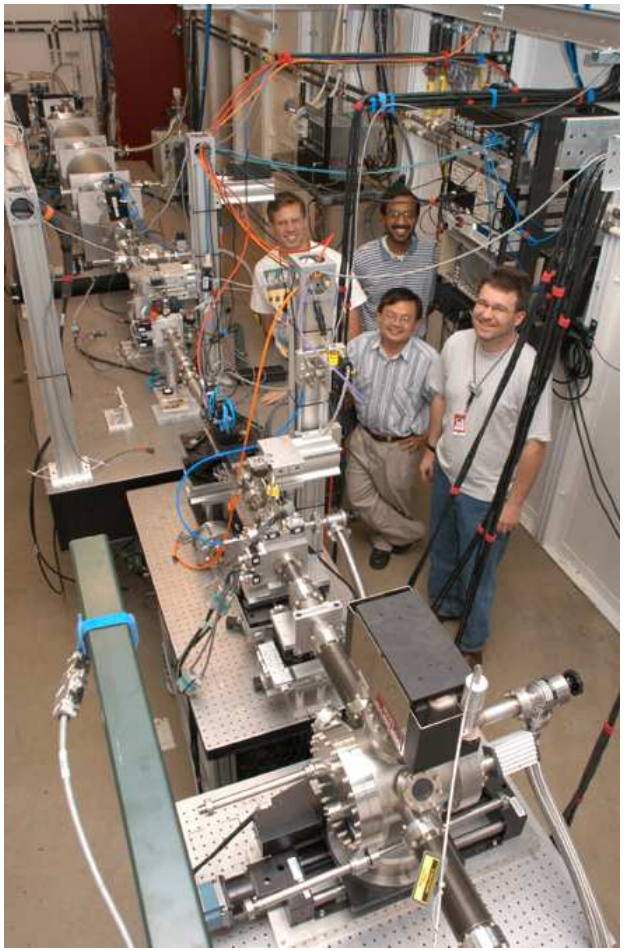




Denis T. Keane
Steven Weigand
Qing Ma
Diane Sandberg
W. Mike Guise

Slide(s) courtesy of Alec Sandy

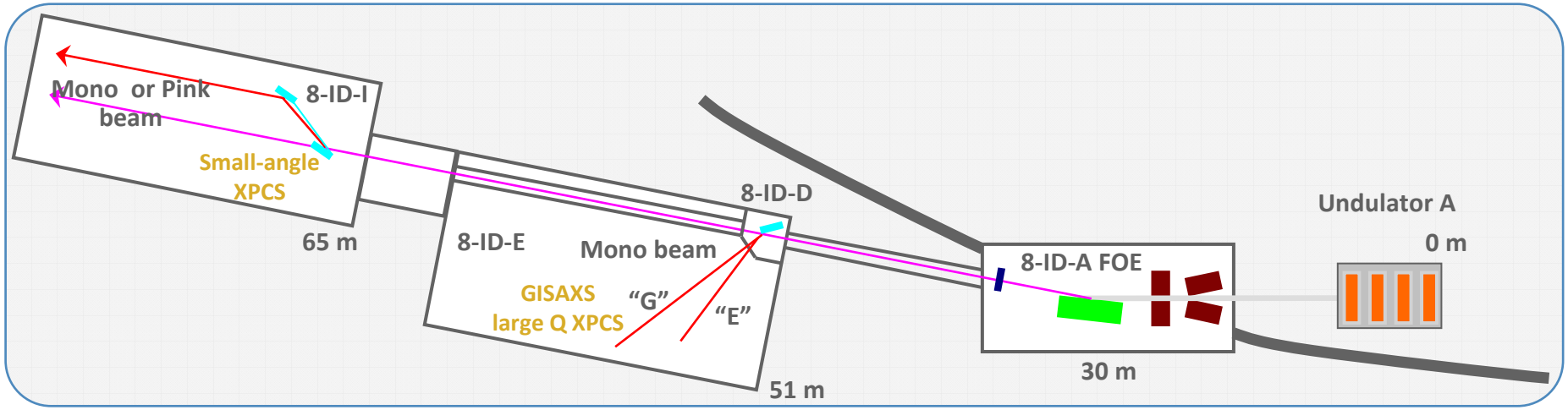
8-ID has XPCS and GISAXS



- APS X-ray Operations and Research (XOR) beamline since 2003
- Part of the APS Time Resolved Research (TRR) Group managed by Jin Wang
- Fully operational with 80% General User (GU) time each cycle
- Staffed with 3 beamline scientists with complementary expertise in the beamline's scientific theme areas
  - Suresh Narayanan
  - Alec Sandy
  - Michael Sprung

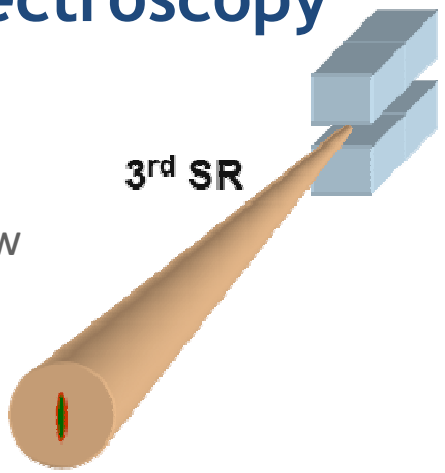
8-ID layout

- Undulator beamline supporting 2 scientific theme areas:
  - Grazing-incidence small-angle x-ray scattering (GISAXS)
    - ≈ 30% General User (GU) time
    - Dedicated in-vacuum set-up
  - X-ray photon correlation spectroscopy (XPCS)
    - ≈ 70% General User (GU) time
    - Only other similar facility worldwide is at the ESRF
- Most 8-ID user groups from physics, chemistry, chemical engineering, polymer and materials science and engineering disciplines

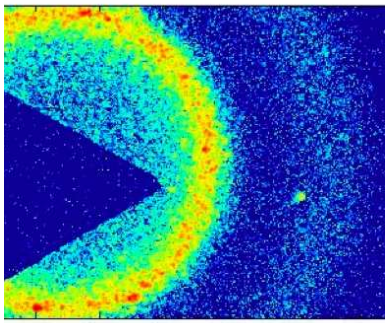


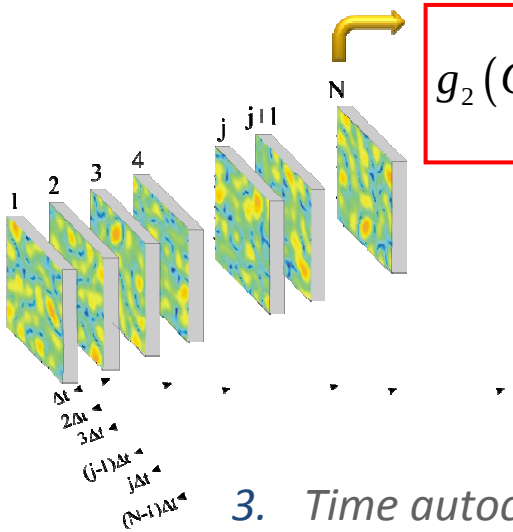
8-ID XPCS: X-ray Photon Correlation Spectroscopy dynamic studies

- X-ray photon correlation spectroscopy (XPCS) is the x-ray analog of dynamic light scattering permitting characterization of the slow dynamics of condensed matter at the nanoscale

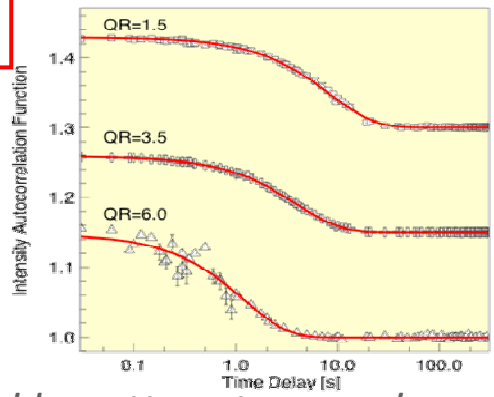


1. *Illuminate disordered sample with a (partially) coherent x-ray beam*
2. *Collect speckle pattern versus time with a high resolution and high gain area detector*





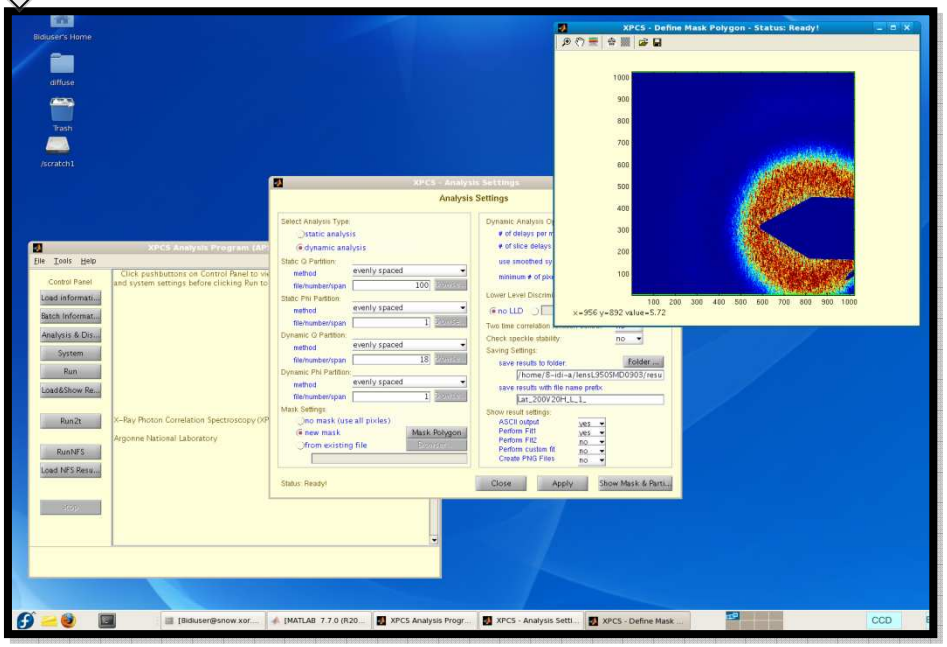
$$g_2(Q,\tau) \equiv \frac{\langle I(Q,t)\, I(Q,t+\tau)\rangle}{\langle I\rangle^2}$$

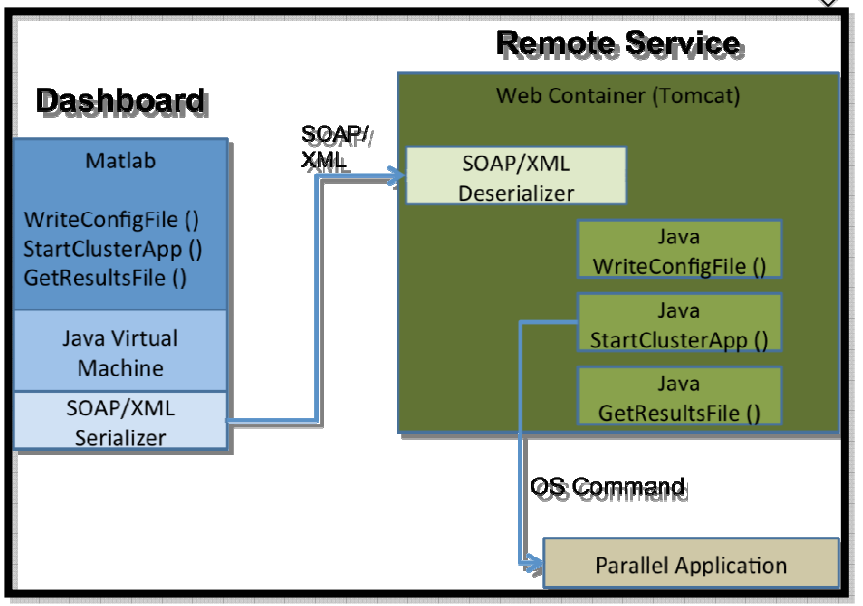


3. *Time autocorrelate fluctuating speckle pattern to reveal sample dynamics*

8-ID XPCS software overview

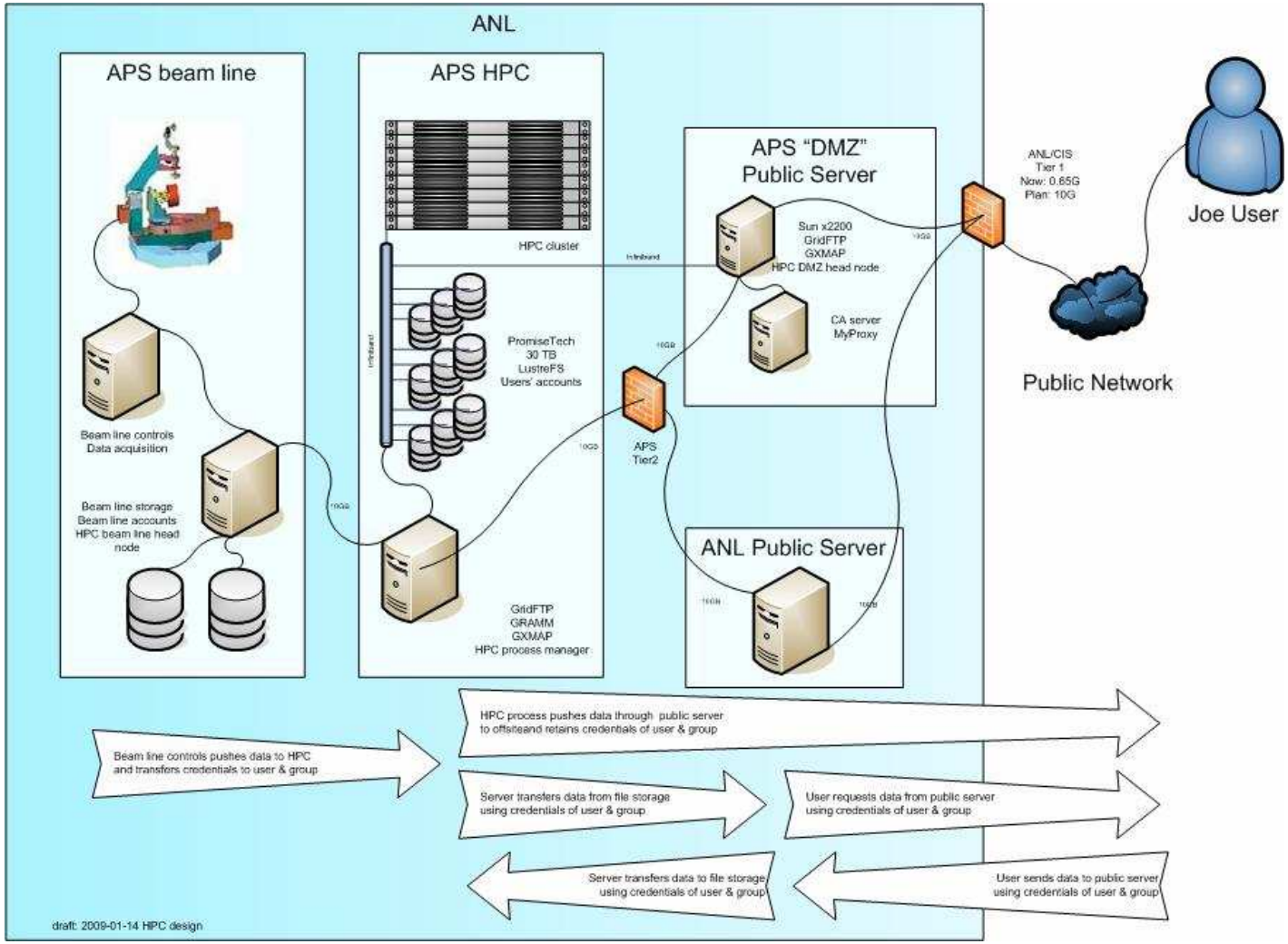
- Matlab used to create an overarching user-friendly interface to underlying hardware, firmware and software (Zhang Jiang)
  - EPICS channel access for camera/FPGA control
  - Java ➔ SOAP/XML calls for directing and monitoring cluster calculations
  - Time autocorrelations continue to be available locally in Matlab





APS XOR High-Performance Computing Environment

Supports: Tomography, XPCS, 3-D HE-XRD, 3-D microdiffraction



12-ID Upgrade

Two Beamlines with Canted Undulators

materials science, chemistry

- **C/D Line (4.5 – 36 Kev, pink beam)**

  SAXS/WAXS/GISAXS/GIXAS - in situ, time resolved [12-ID-C]

  Surface Scattering – MOCVD, surface diffraction [12-ID-D]

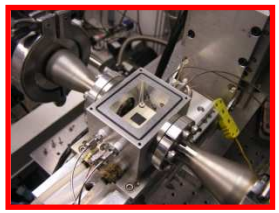
- **B Line (7.4 – 13.9 Kev)**

  SAXS/WAXS/GISAXS - rapid adjustable Q [12-ID-B]

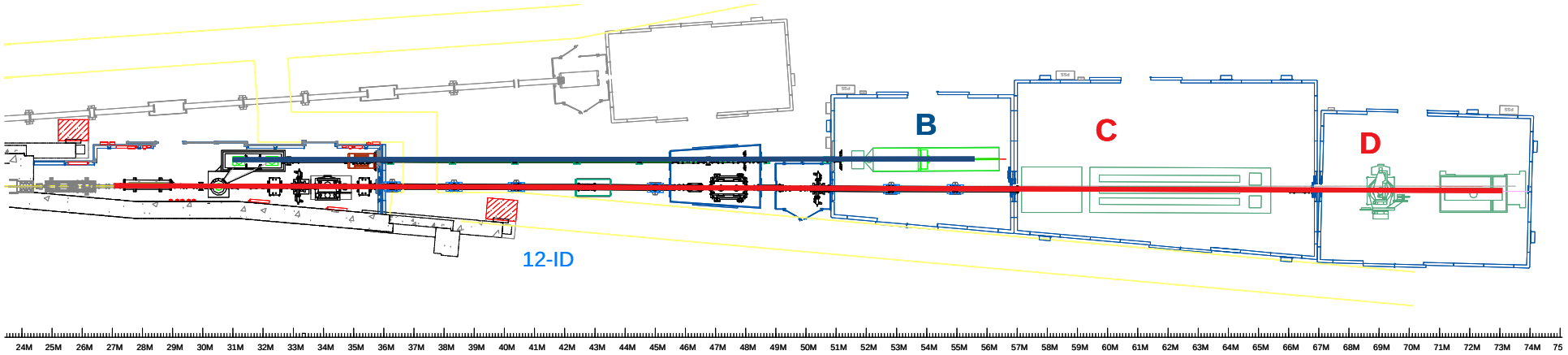
- **Detectors**

  APS/XOR Platinum mosaic CCD

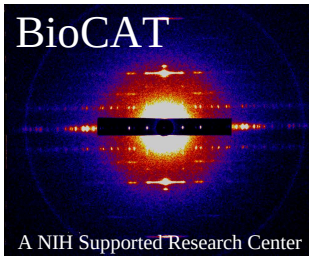
  Pilatus 2M and wide angle (300K)



GISAXS Cell

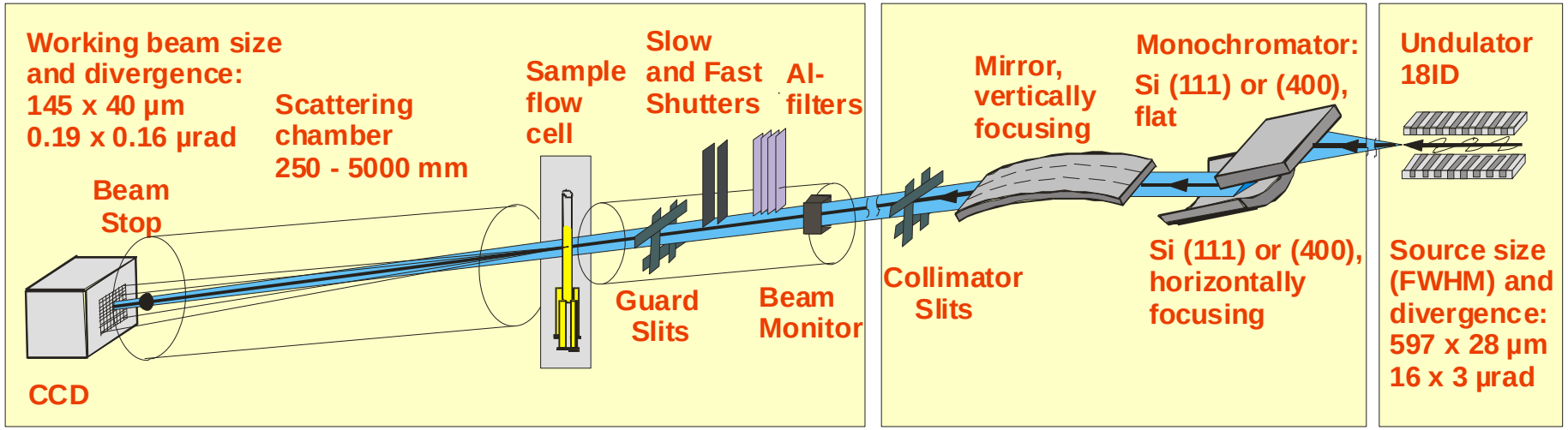


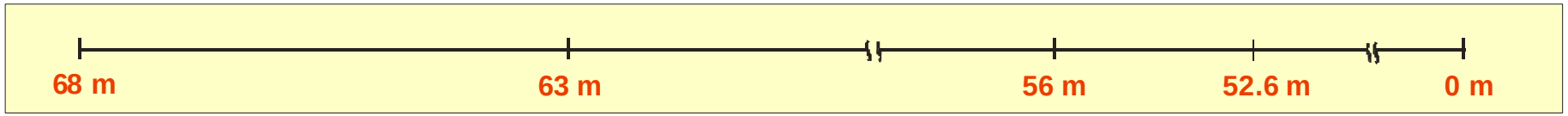
18-ID SAXS biology, solutions



Slide(s) courtesy of Tom Irving

- A NIH-supported research center for the study of partially ordered and disordered biological materials. Operated by the Illinois Institute of Technology
- Comprises an undulator based beamline, (18-ID) associated laboratory and computational facilities.
- Available to all scientists on basis of peer-reviewed beamtime proposals

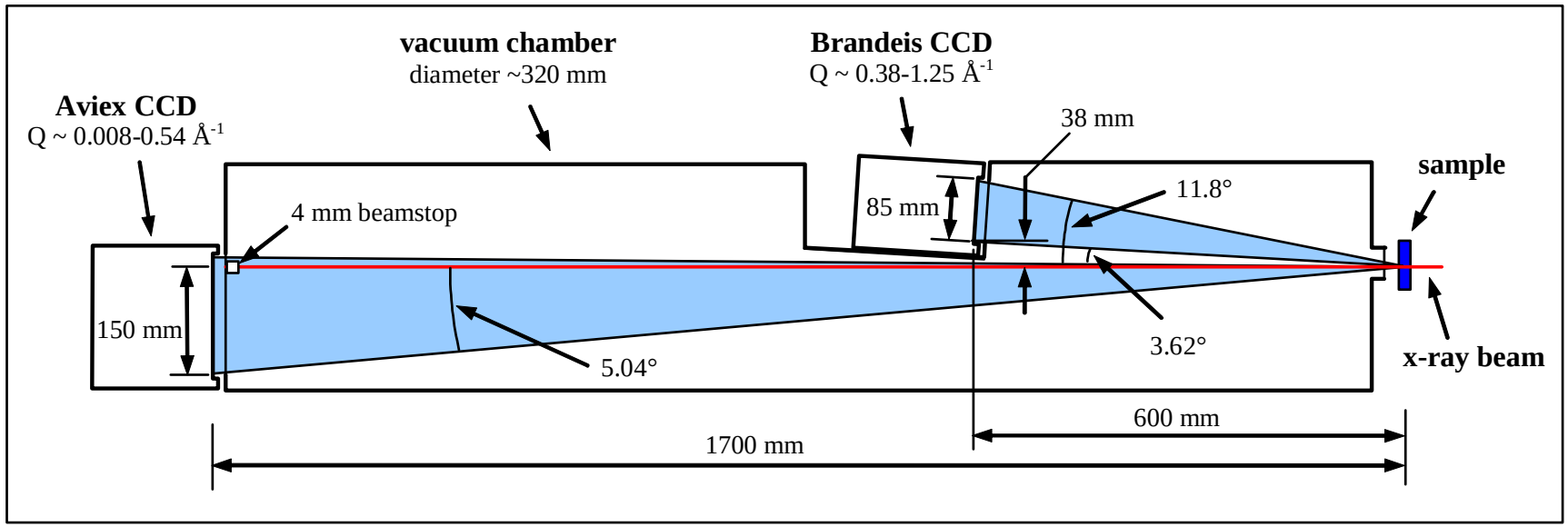




18-ID BioCAT parameters

- Total X-ray flux 1-2.5 x10^{13} photons/s
- Focal spot size ranges from < 50 µm vertical and < 160 µm horizontal to ~3 x 1.5 mm
- Wide energy range (4-39 keV)
- Rapid 1 keV energy scans in < 15 seconds
- First order resolution > 1500 Å (1/d)
- Order to order resolution > 10000 Å (1/d)
- High sensitivity (~photon counting), high spatial resolution (~60 micron psf, 39 micron pixels) CCD detectors
- Pilatus 100k detector for time resolved studies

Future 18-ID BioCAT SAXS/WAXS Instrument



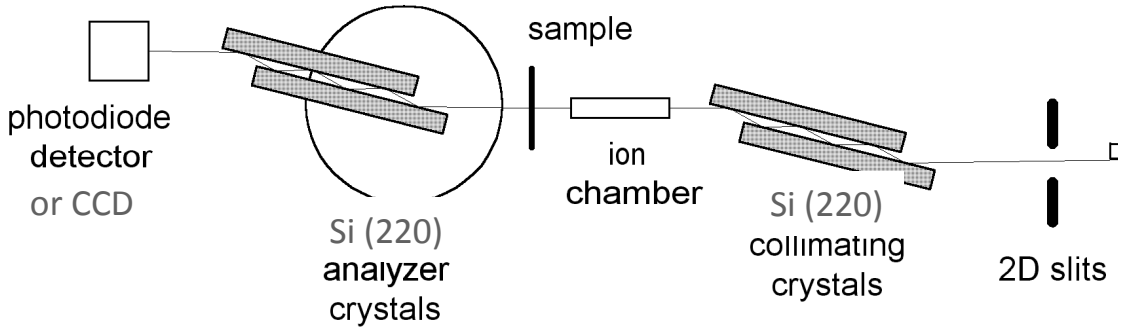
Use two detectors to cover entire q-range from 0.008 $Å^{-1}$ to 1.25 $Å^{-1}$

To be implemented fall 2009/spring 2010

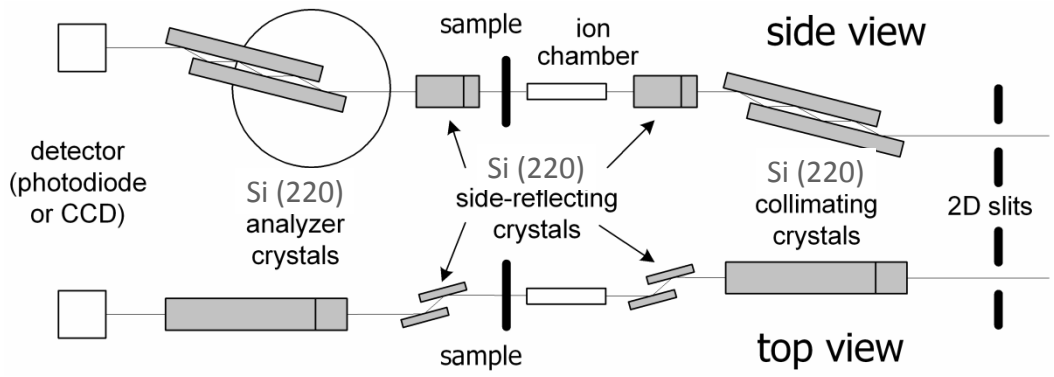
Will be MUCH more efficient than separate SAXS/WAXS runs

Slide(s) courtesy of Jan Ilavsky

32-ID USAXS
materials science, larger structures, USAXS imaging



1-D collimated Bonse-Hart Camera (slit smeared)



2-D collimated Bonse-Hart Camera

32-ID USAXS parameters

- General purpose USAXS instrument using Bonse-Hart design
- Used to examine structures in the few micrometer scale
- Intensity and Q range:
  - ca. 10^{13} ph/s (monochromatic) incident on sample
  - Up to 9 decades of intensity range
  - 0.00015 A^{-1} to 1 A^{-1} Q range (0.5 nm ----> >1 micron)
  - Both 1-D (slit smeared) and 2-D collimated ("2D-USAXS") geometries available
  - 10 min/scan (shortest scans down to 3 minutes)
  - Flexible beam size (1 x 2 mm ---> 0.02 x 0.2 mm)

Mid-2010 USAXS moves to 15ID beamline (ChemMat CARS):
Available pinhole camera SAXS at the same time
Planned upgrade to high-*Q* small pinhole SAXS camera integrated into USAXS (using Pilatus detector)
NO GUP time in 2010-2

32-ID USAXS software and support

- "One stop shop" –
  user friendly source for absolute calibrated SAS results
- Combine:
  - *USAXS instrument* - robust & reliable hardware
  - *Software support* - quick, user friendly, and scientifically correct method to extract from *measurements* useful & publishable *results*.
    - Data reduction software ("Indra") in IgorPro
    - Data evaluation software ("Irena") in IgorPro
  - *User support* "from cradle to grave" - from exp. preparation to publication.
- Significant fraction of facility users are not SAS experts, but materials scientists, chemists, physicist, geologists… Students…
- **15 – 20 publications/year (with 6 – 9 weeks of beamtime/year)**

Web-based view of live USAXS data acquisition with automated, preliminary data reduction

32ID-B USAXS status

HTML page refresh interval 0:05:00 (h:mm:ss)

Webcam: http://usaxsqvs1.xor.aps.anl.gov

raw / descriptive content updated: Thu Jul 19 07:00:53 PM CDT 2007

shutters:	USAXS CCD: closed	USAXS Ti filter: open	mono: Open	white: Open
APS current = 101.26 mA	ID E = 23.203 keV			
|Q| = 6.53324126293e-05 1/A	I = 4.857143e+02 pA/uA	SAD = 114.00		
mirror position: white beam pass-by	PF4 filter transmission: 2.2e-92 (Al=2.3877 mm, Ti=			

SOFC61 near catho

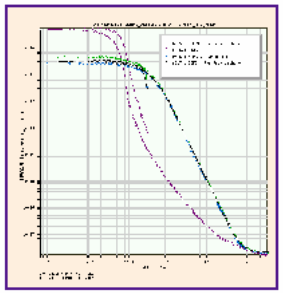
USAXS reset complete

spec macro: usaxs.mac	time stamp: Sun Jul 15 07:44:04 PM CDT 2007	
/share1/USAXS_data/2007-07/07_15.dat	scan #175	Mon J

slits

slits	mm	mm	mm	mm
USAXS (h,v)(gap,center)	0.599976	0.250182	-0.000032	0.000052
white (r,l,t,b)	0.500012	-0.699801	0.600000	-0.400000

USAXS plot



detectors

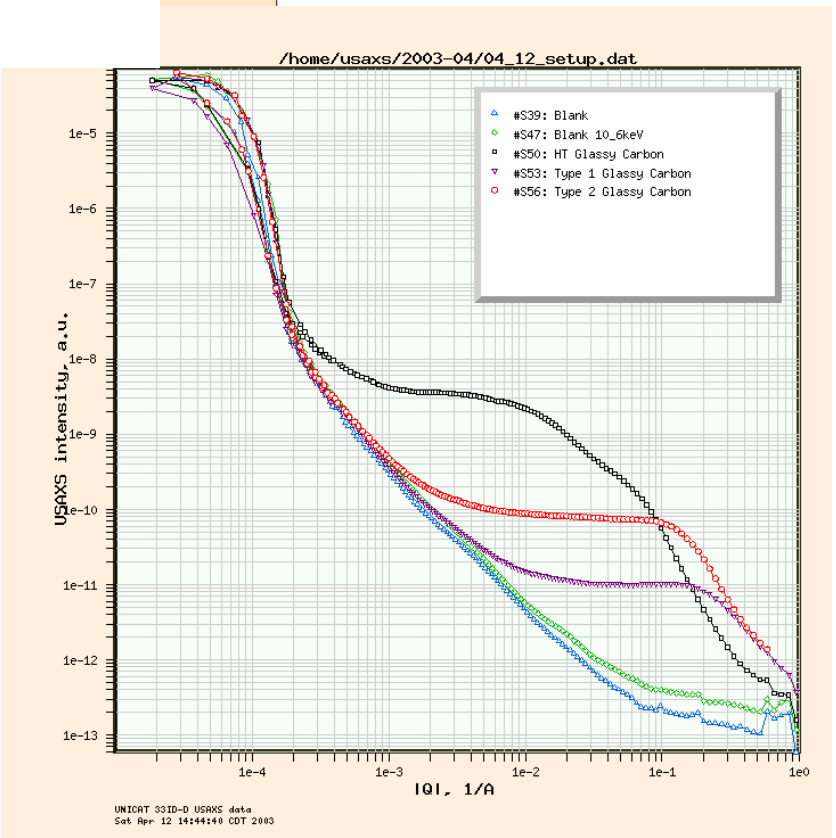
detector	counts	VDC	gain,V/A	current,A
I0	79	-0.0140	1.0e+07	1.400000e-09
I00	78	-0.0160	1.0e+07	1.600000e-09
I000	419286	4.1350	1.0e+08	-4.135000e-08
photodiode	7817	0.0680	1.0e+11	6.800000e-13

motors

stage	rot,deg	encoder,deg	X,mm	Y,mm	Z,mm	tilt,deg
m	11.054822	0.000000	-107.298975	0.000400		
ms	-0.000001		0.000001	3.000000		0.000000
s			-52.349994	1.000001		
as	-0.000001		0.100000	-0.000000		0.000000

Done

Web based live data



Additional software to talk about …

- **canSAS** (Collective Action for Nomadic Small-Angle Scatterers) is a forum of users, software developers, and facility staff who have gathered together to discuss better sharing of SAS data analysis software.
- One recent effort was the standard for saving 1-D data in XML

- Members of the 1-D Format Working Group "Tribe":
- Pete Jemian (APS), Steve King (ISIS), Andrew Jackson (NIST), Ken Littrell (ORNL), Andy Nelson (ANSTO), Ron Ghosh (ILL, retired), Jan Ilavsky (APS)

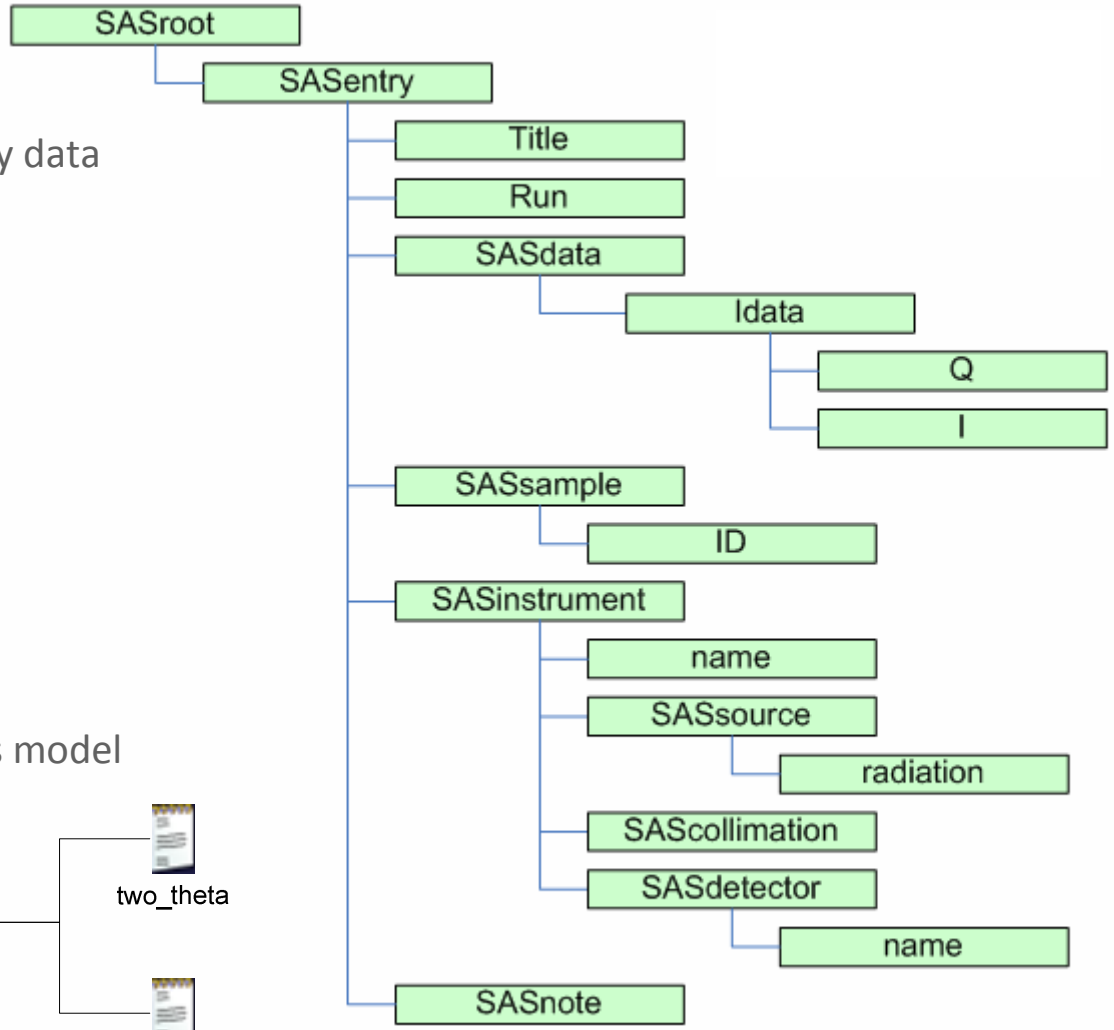
Motivation for canSAS 1-D XML Data Standard

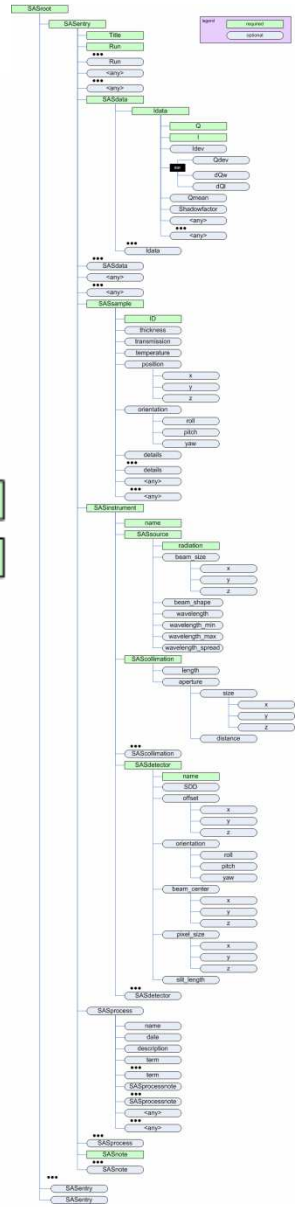
- Better sharing of SAS data analysis software
  - primary data, *I(Q)* : NOTE: small data sets
  - metadata --- any other descriptive information about the sample, measurement, instrument, source, processing, or analysis steps.
- Significant SAS community need satisfied by standardizing a format
  - robust
  - self-describing
  - text-based (human readable, editable, import using simple commands)
  - reduced one-dimensional small-angle scattering data, *I(Q)*
  - Communicate data between users of our facilities
- minimal verbosity
- programs need not:
  - recognise advanced structure in the file
  - require advanced programming interfaces.
- Provenance (record of processing steps and analysis results)

- Compatible, where possible, with NeXus

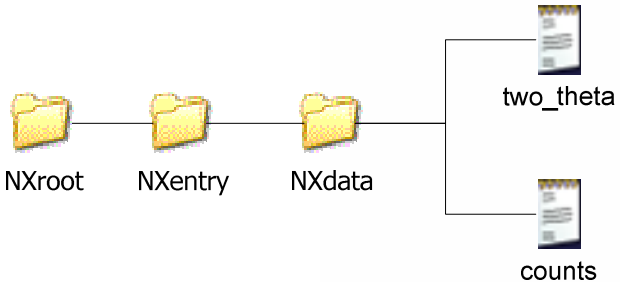
cansas1d/1.0 The Minimum Set of information

- Easy to support legacy data





- Similar to basic NeXus model



canSAS 1-D Standard: Comments & Conclusions

- Good idea long time in the making
  - Agreement on standard between many user facilities
  - Flexible design tailored for specific community (small-angle)
  - Format allows access to data by variety of methods
  - Multiple measurements may be included within a single XML file
- Perceived competition with other standards bodies (NeXus, CIF)
- XML is a good method to store scientific data
  - Rich tools & support exist
  - XSLT to transform between different standards
- **The cansas1d/1.0 standard meets the objectives for a 1D standard, incorporating experiment metadata, and parameters and results of processing or analysis steps**.
- Higher dimensionality
  - 2-D area detectors, Time series, Other parametric studies
  - Rely on NeXus NXsas

http://www.smallangles.net/wgwiki/index.php/cansas1d_documentation

Thank you for your attention!