X-Ray Photon Correlation Spectroscopy (XPCS) Beamline overview

Eric Dufresne, Suresh Narayanan
APS User meeting Worksop 7
With slides from Alec Sandy, Dana Capatina (CAM/XPCS)
Kevin Wakefield (Project Engineer) and Matt Highland (WA-XPCS lead)

Friday May 7, 2021

Outline

- Beamline overview and science case
- Functional Requirements
- X-ray optics
- Operational modes
- 8-ID-E WA-XPCS station
- 8-ID-I SA-XPCS station
- Summary of main beamline and 8ID instrument status

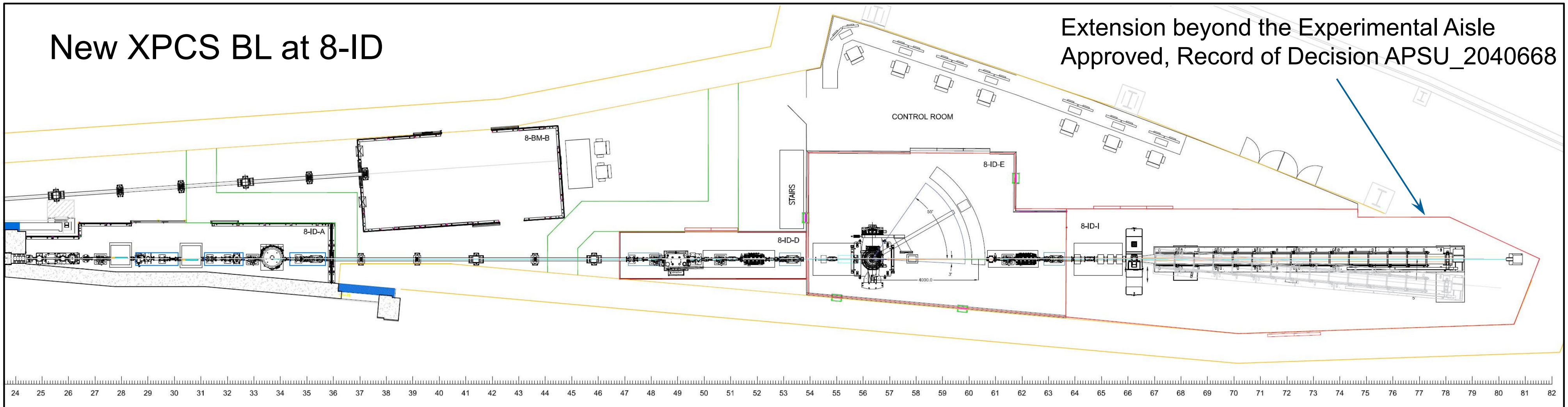
XPCS Science

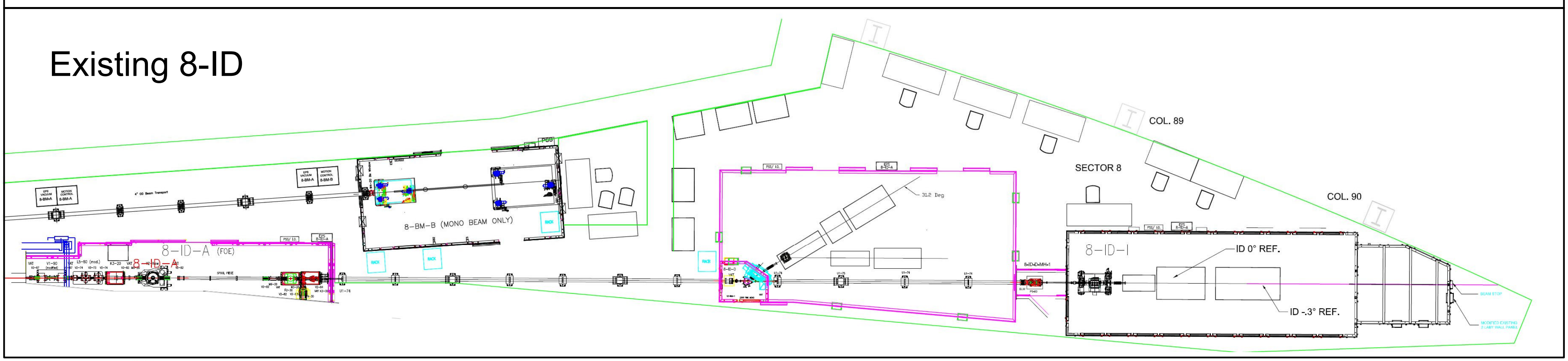
Lead Scientists: Eric Dufresne and Suresh Narayanan

- Goals
  - The XPCS beamline will be optimized for time-resolved coherent x-ray scattering techniques used to examine spatial and dynamic heterogeneity in emergent materials.

- Supported techniques
  - Small-angle XPCS
  - Wide-angle XPCS
  - Ultra-small-angle (pinhole) XPCS
  - Coherent diffraction

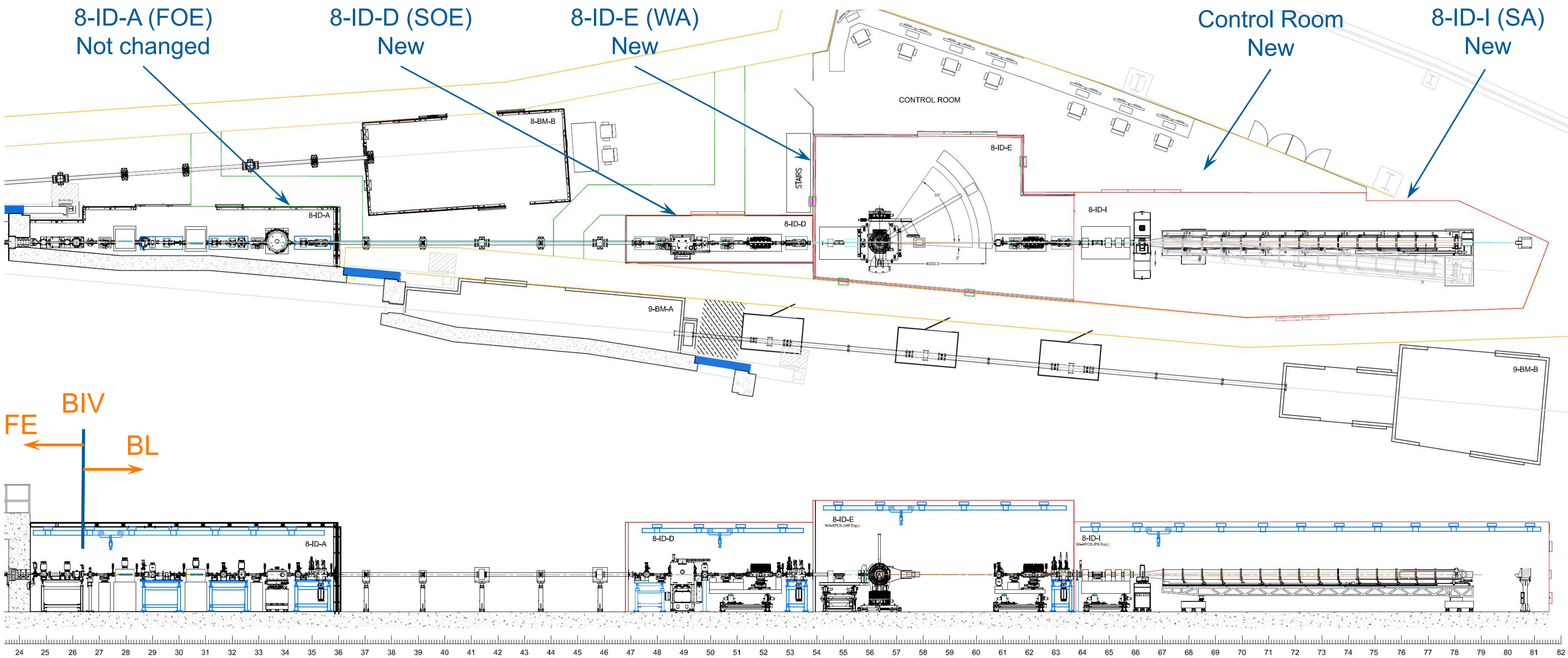
Shielded Enclosures – New & Existing





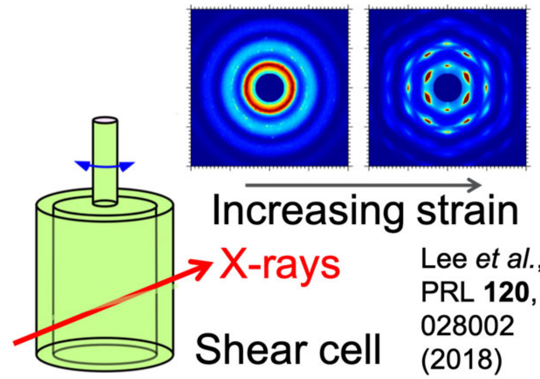
Beamline Scope

- XPCS is a complete rebuild of the 8-ID beamline for time-resolved coherent scattering techniques.
- The infrastructure consists of two optical, two experimental enclosures with these operating one-at-a time, and a control room.



Functional Requirements

- Energy range:
  - 8-25 keV
- Energy bandwidth
  - Monochromatic (Si 311 and 111): 0.005 – 0.014 %
  - Pink: 0.5 %
- Source
  - 2 revolver undulators, 21 and 25 mm period length
- Beam spot sizes
  - 0.3 – 3 μm at the dedicated WA-XPCS experiment
  - 3 – 10 μm at the dedicated SA-XPCS experiment
  - 15-45 μm for USAXS mode akin to beamline 2-ID of ESRF
- Maximize, preserve and efficiently deliver APS-U coherent flux
- Sufficient oversampling in reciprocal space
  - Long sample to detector length (>10 m)
- Maximize beamline and experiment stability



Functional Requirements and Interface Control Document (ICD) (APSU_2011244) approved

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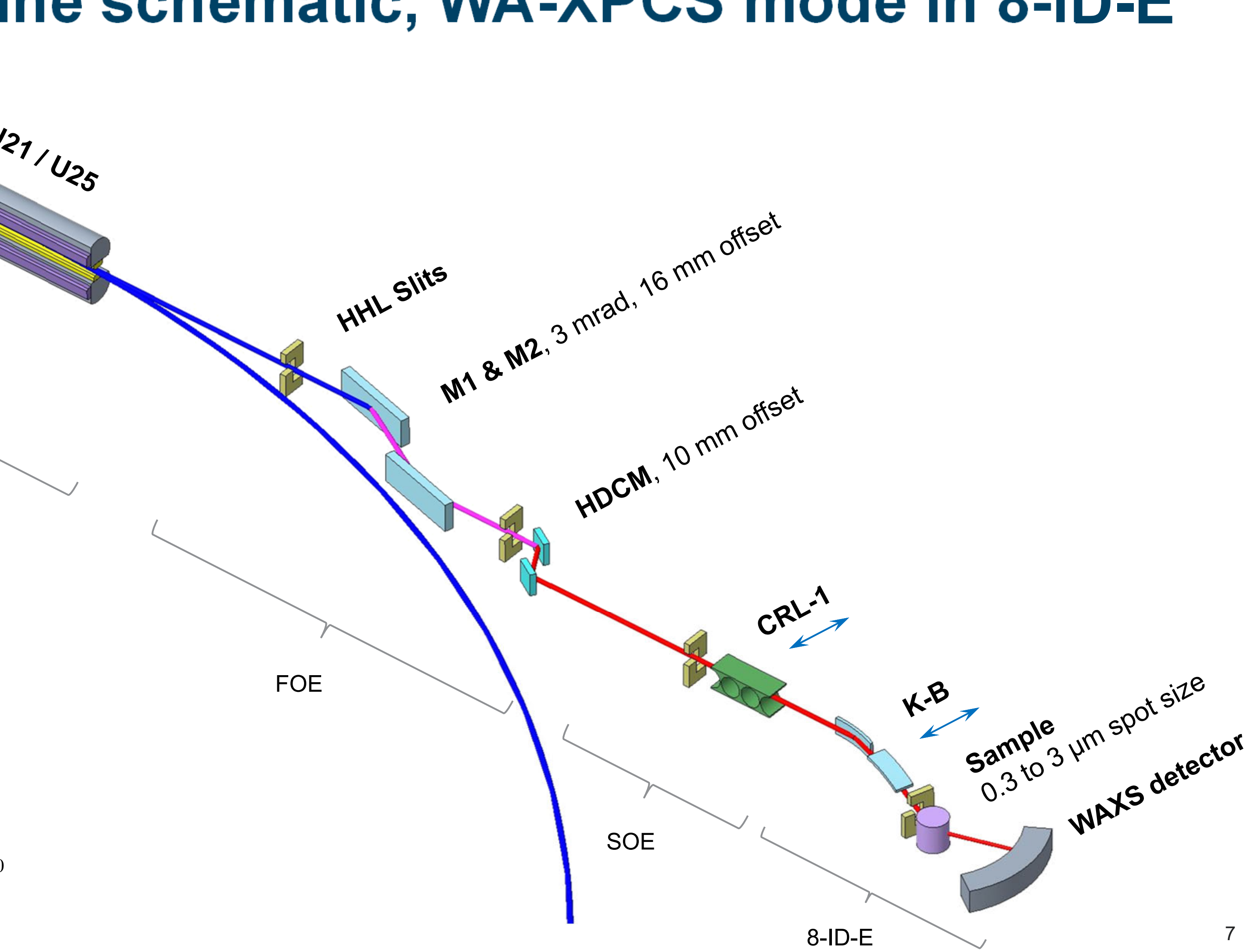
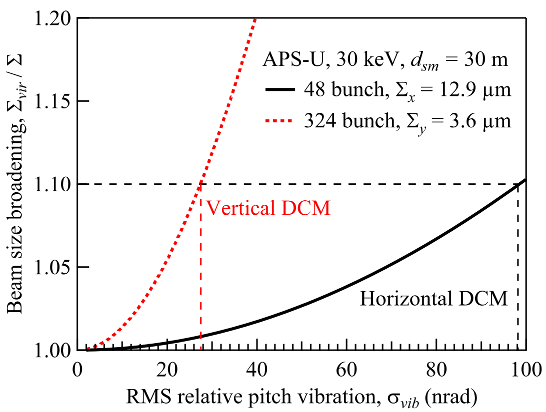
Beamline schematic, WA-XPCS mode in 8-ID-E



H-bounce mirrors/mono



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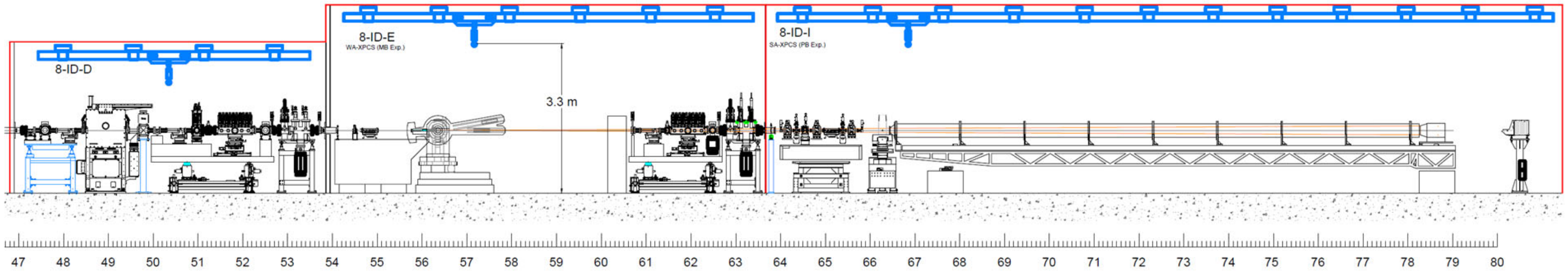
XPCS Beamline Operational Modes

- Wide Angle XPCS (WA-XPCS) Mono
  - Samples in 8-ID-E on diffractometer
  - Detectors in 8-ID-E on diffractometer or LDDP
- Small Angle XPCS (SA-XPCS) Mono/Pink
  - Samples in 8-ID-I on sample support stage
  - Detectors in 8-ID-I at the end of the flight tube
- Pinhole U-SAXS Mono
  - Samples in 8-ID-E on diffractometer
  - Detectors in 8-ID-I at the end of the flight tube (and simultaneously in 8ID-E) (22 m distance)



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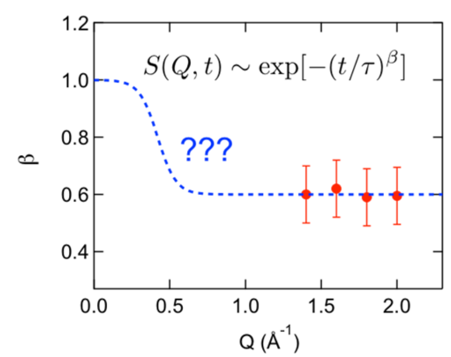
WA-XPCS Instrument

- Science driver
  - Dynamic and structural heterogeneity in complex materials revealed through XPCS
- Wide Angle XPCS (WA-XPCS): XPCS measurements at many locations in reciprocal space
- Experimental Requirements
  - Diffractometer for orientation and positioning in reciprocal space
  - Smaller sample to detector distance for orientation and positioning in reciprocal space
  - Longer sample to detector distance for speckle resolution with horizontal diffraction
  - Vertical arm for mitigating polarization effect at high Q
  - Sample environments covering 4 – 1300 K



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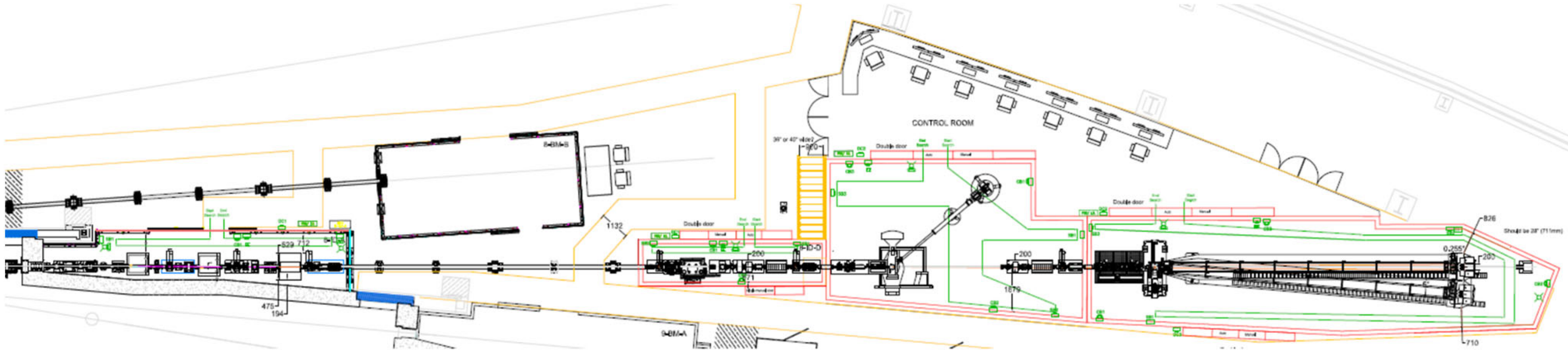
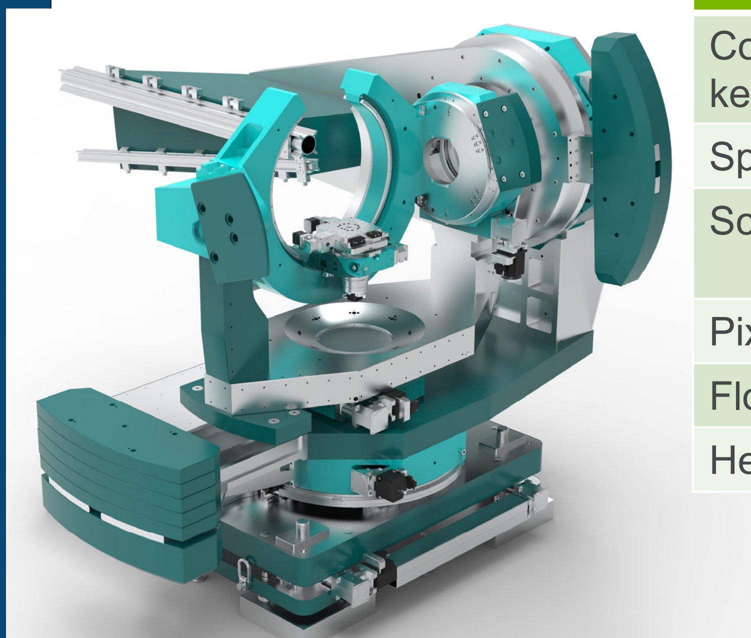
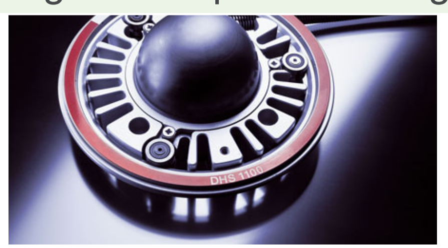
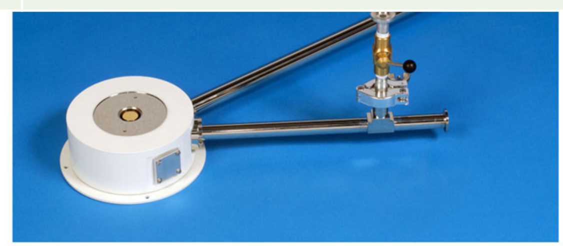


X-ray energy (keV)	4.8-25
Coherent flux (1 mode) (ph/s) (8-25 keV)	$2x10^{12}$ – $1x10^{12}$ with Si (111). Si (311) also available with lower bandwidth.
Spot size (μm)	0.3 to 3 (KB and/or CRL)
Scattering geometry	6 circle with 2 m arm, horizontal 4m arm
Pixel-Array Detector sampling	Up to 50 kHz continuous, 1 MHz burst
Flow cryostat temperature range (K)	4-475
Heating cell temperature range (C)	20-1100

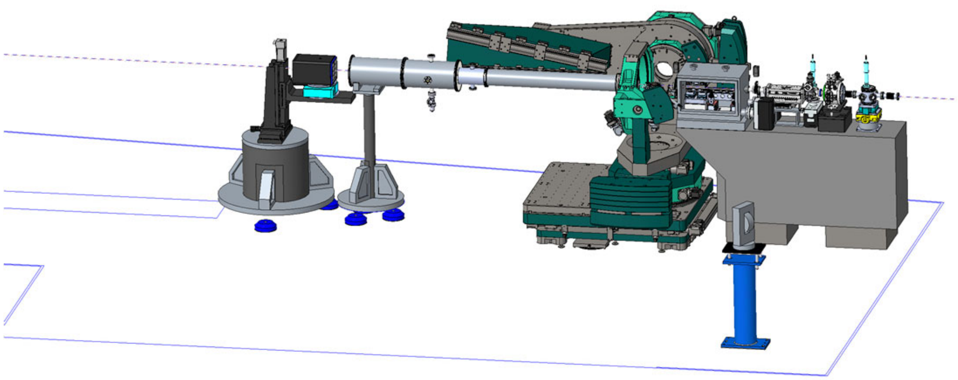


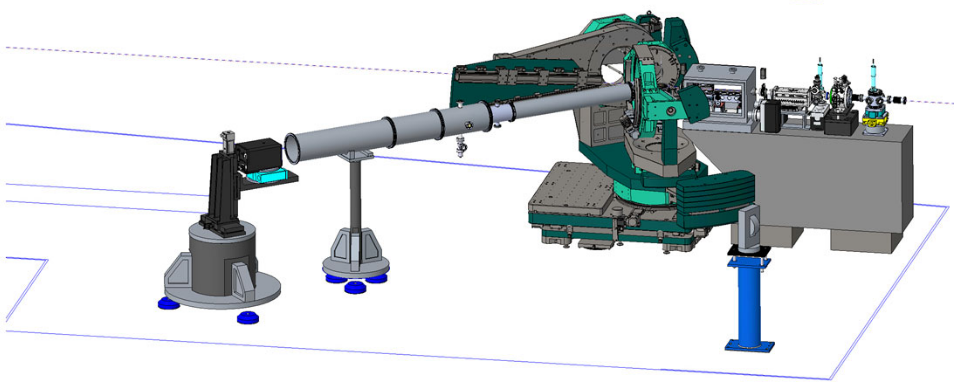
Hot cell procured, arriving soon!





Example of LDDP and 6-circle use





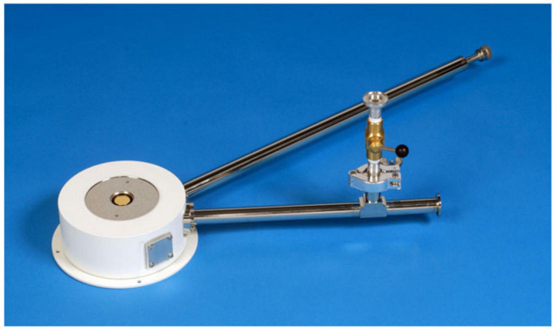
- Could combine simultaneously the vertical WAXS detector and provide low contrast but high-Q SAXS above the USAXS range. (e.g. PNAS Petra III on ice)
- Stand alone, 25 keV, highest wavevector, horizontal scattering.
- You can imagine also vertical WAXS and USAXS mode together, with optimized focusing for one of the mode for XPCS.

Sample Environments

- High temperature cell: Anton Paar DHS 1100
  - Up to 1373K
  - Graphite Dome
  - Controlled atmosphere
  - Standard design does not reach sufficient vacuum (Standard: 10^{-2}, Required: 10^{-5})
- Cryostat: Janis ST-500
  - 4K to 450K
  - Low vibration ($\pm$15 nm)
- Cooling Stage Anton Paar DCS-500
  - 93K to 773K

High Temperature Cell

Cryostat

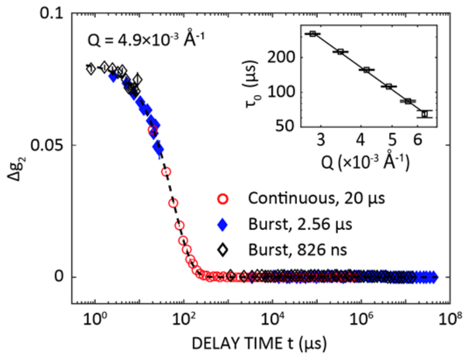


Cooling Cell

Pixel Array Detectors

- Pixel Array Detectors (PADs) are required for each instrument
- Key Specifications
  - Pixel Size (55 um commercially available)
  - Dynamic range
  - Software compatibility
- Currently available options
  - Lambda
  - Eiger
  - Rigaku XSPA
  - Xspectrum Sparta

Lamdba 750K



Eiger 1M

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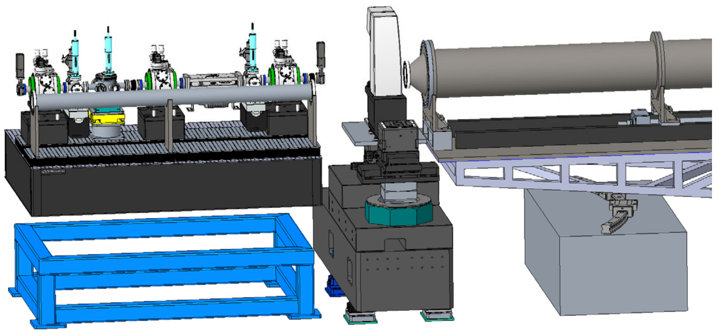
SA-XPCS 8-ID-I Instrument



KEVIN WAKEFIELD

Mechanical Engineer - XSD

XPCS TEAM

Science Lead – Suresh Narayanan and Eric Dufresne

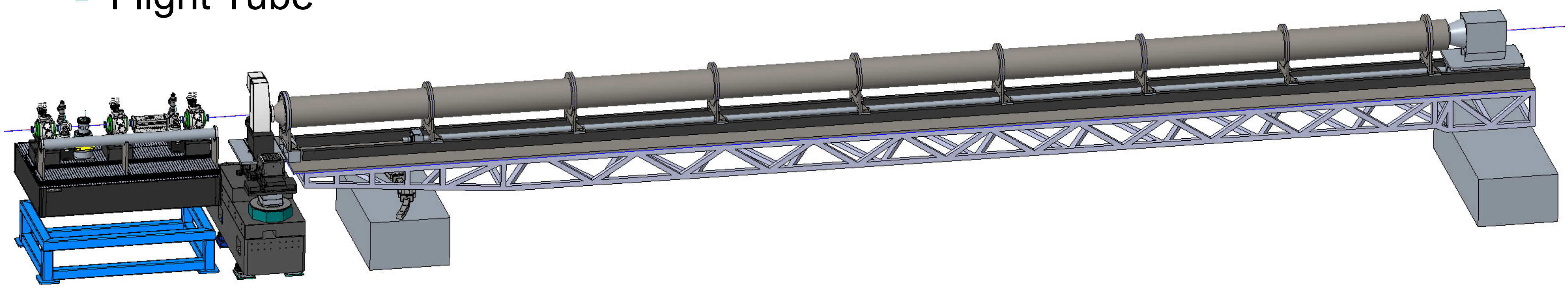
Instrument Engineering – Matt Highland, Kevin Wakefield, and Dana Capatina

Beam Delivery – Kevin Wakefield and Dana Capatina

CAM – Dana Capatina

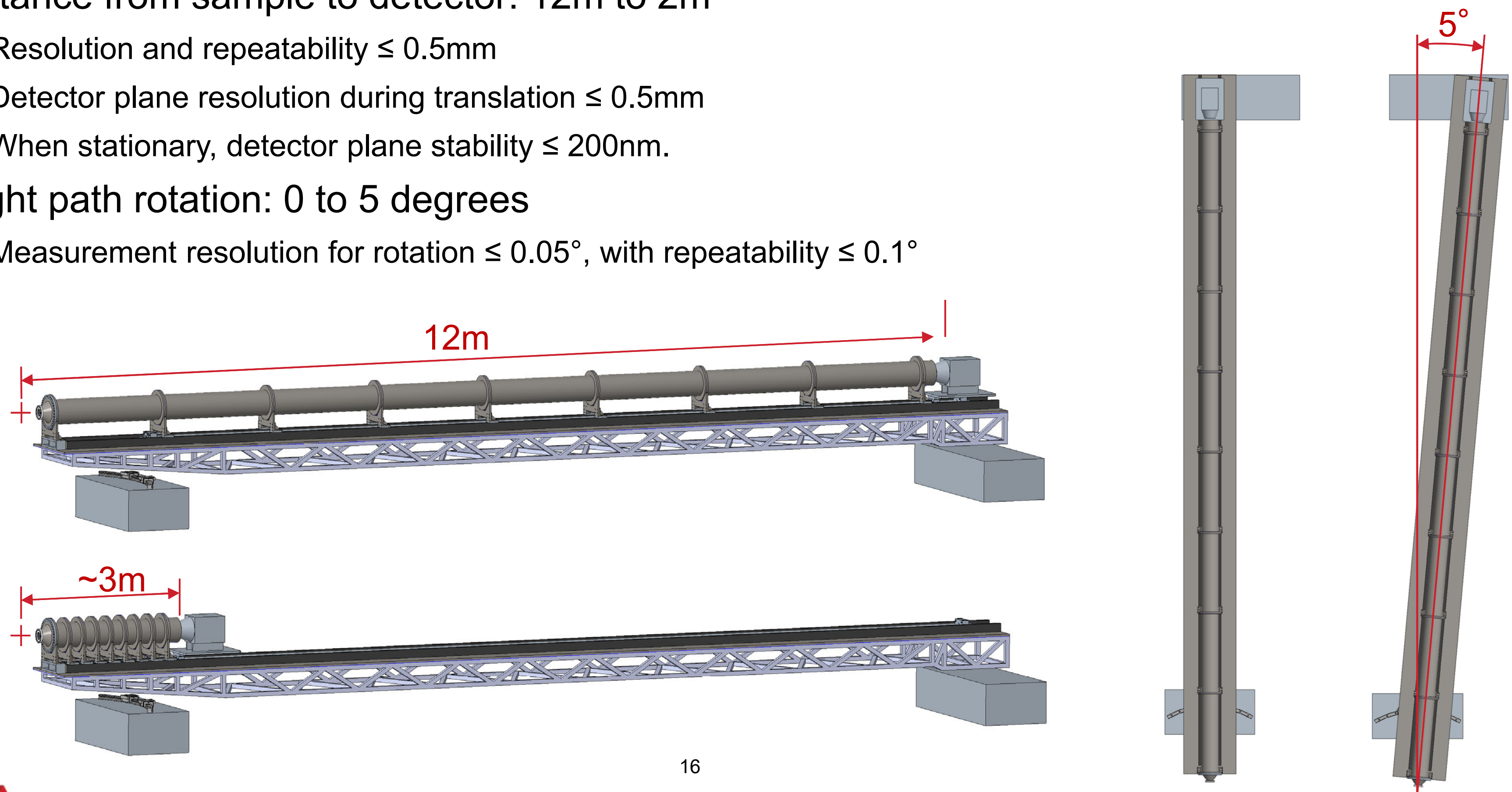
8-ID-I Instrument

- Upstream optical table
- Beam conditioning optics
  - Slits, Shutter, Attenuators, Be windows and valves
- Sample Environment Table
- Sample Environments
- Flight Tube



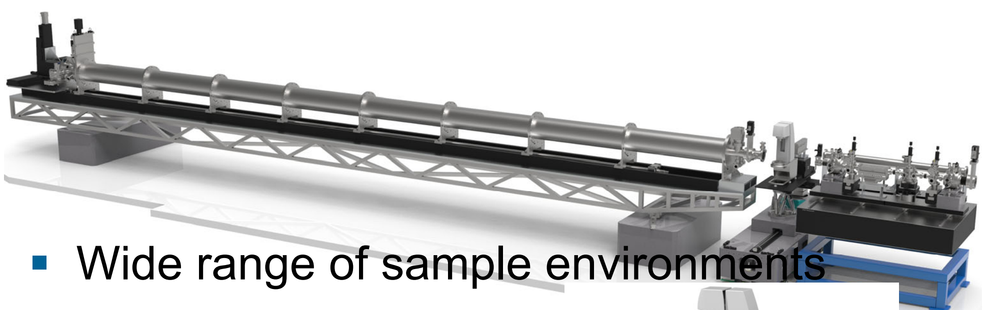
Flight Tube – 8ID-BI-5 – Motion Requirements

- Distance from sample to detector: 12m to 2m
  - Resolution and repeatability ≤ 0.5mm
  - Detector plane resolution during translation ≤ 0.5mm
  - When stationary, detector plane stability ≤ 200nm.
- Flight path rotation: 0 to 5 degrees
  - Measurement resolution for rotation ≤ 0.05°, with repeatability ≤ 0.1°



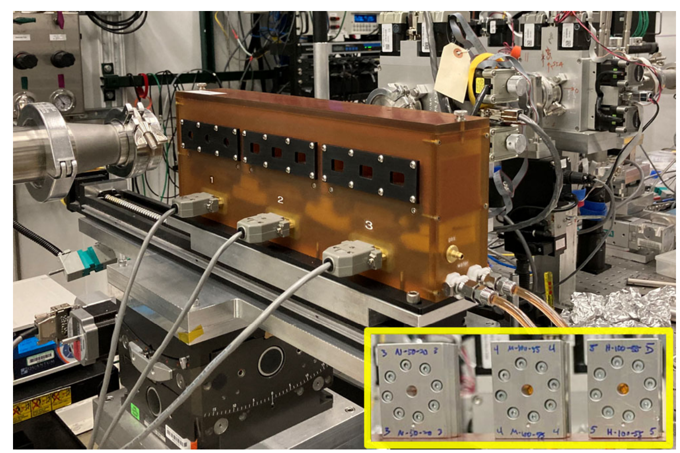
SA-XPCS Sample Environments

- Dynamical time scales: <1 microsec – 1000 sec with state-of-the-art pixel array detectors
- Coherence at higher energies for penetration into *in situ/operando* environments: 8-25 keV
- State-of-the-art Pixel Array Detectors



- Wide range of sample environments

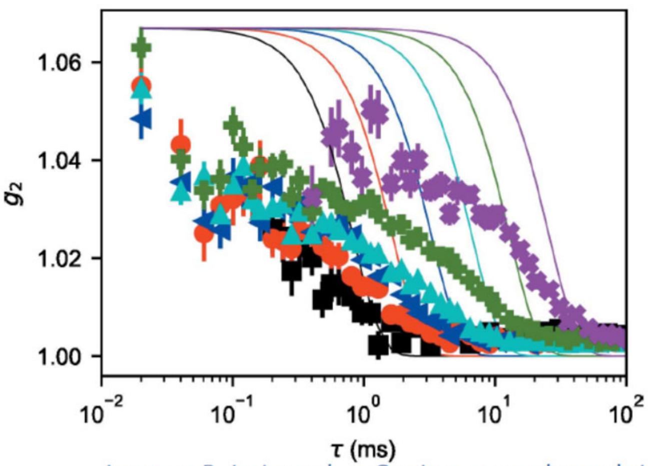
  – Rheometer and robotic sample changer (small-angle)
- Precision temperature-controlled sample environment – 9 samples with 3 temperature zones (-20ºC – 150ºC)
  - Gels, liquids
- Polymer melts in vacuum (-20ºC – 225ºC)



Sample Environments for Setup-2

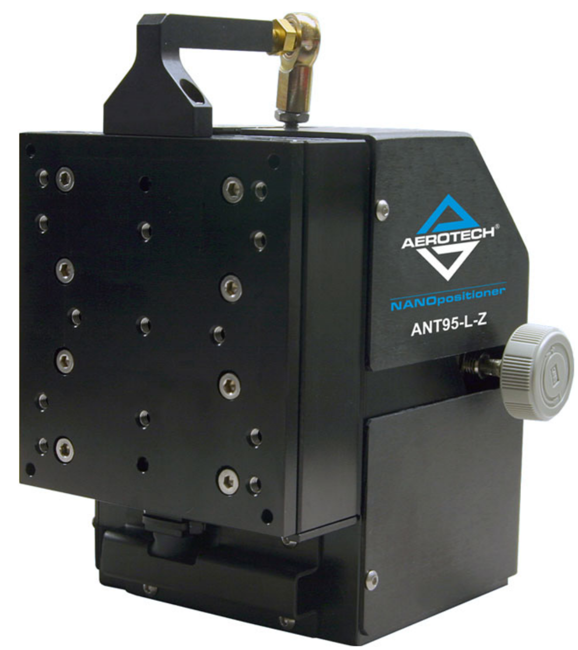
2. In vacuum motion stages with fly scanning capability

1. Horizontal – vertical translation stage – HV compatible
2. Build a chamber around it
3. Details to be dealt with
    1. Upstream of sample will be in UHV or very high HV
    2. How to avoid the window between sample and upstream
    3. Duty cycle of operation due to heating issues



Laurence B. Lurio *et al.* • Continuous sample translation to reduce radiation damage

J. Synchrotron Rad. (2021). **28**, 490–498



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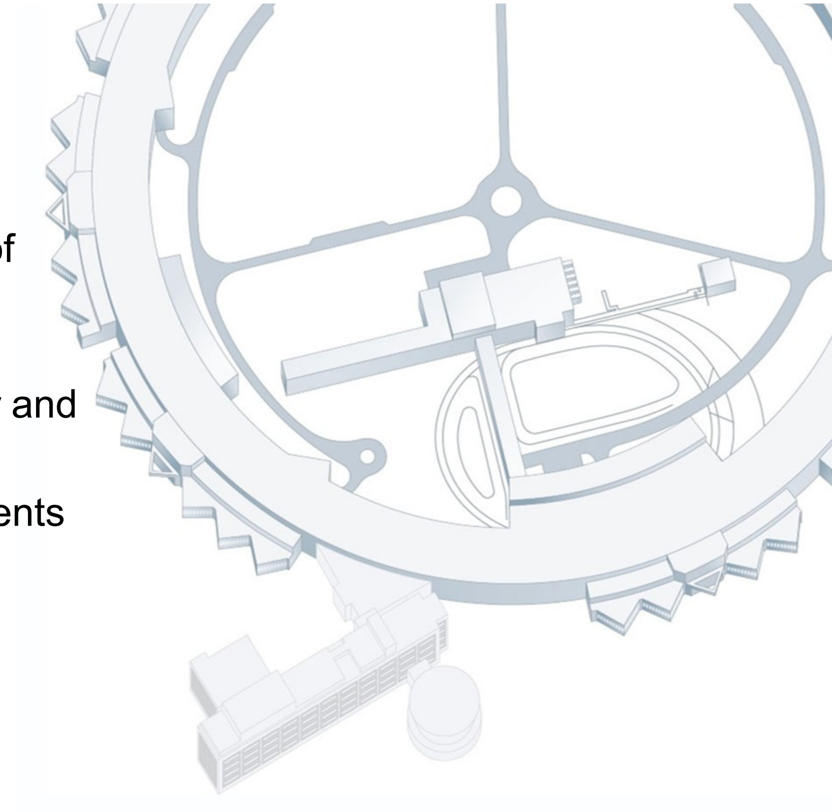
Status and summary

- The XPCS beamline will be a world-leading facility for the study of dynamic heterogeneity in novel materials.
- The hutches and 8ID-A and D optics (mirror, HDCM, CRL) procurement are proceeding as well as the 6-circle diffractometer and KB mirror.
- We're completing statement of work for the 8-ID-E and I components

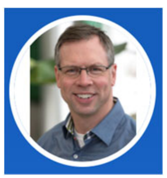


XPCS Beamline Core Team

APS-U

Science Drivers Specifications Development

Suresh Narayanan
(science co-lead)



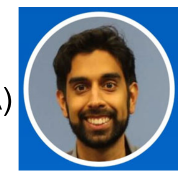
Alec Sandy
(science co-lead)

Eric Dufresne
(WA-XPCS)

Engineering & Project Management

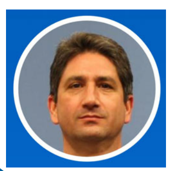
Dana Capatina
(Lead Engineer, CAM)

Altaf Khan (Design and FEA)



Matt Highland (WA-XPCS)

Kevin Wakefield(SA-XPCS)

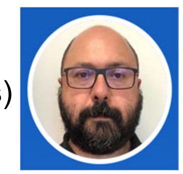


Luis Diaz (Design and Drafting)

Optics Design

Xianbo Shi (Lead)

Luca Rebuffi (Simulations)

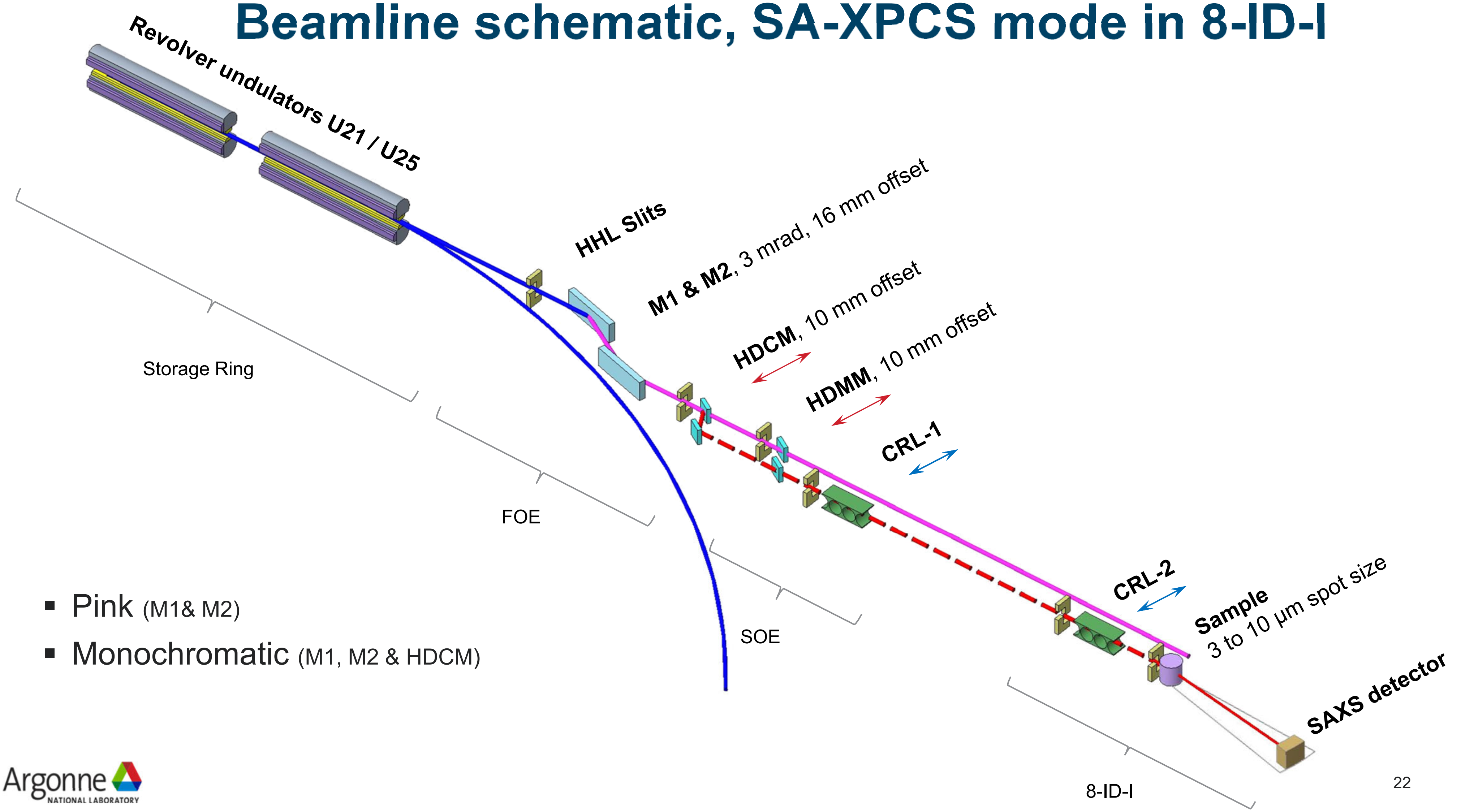


Ruben Reininger (Simulations)

Jonathan Knopp (FEA)

Thank you!

Beamline schematic, SA-XPCS mode in 8-ID-I



- Pink (M1& M2)
- Monochromatic (M1, M2 & HDCM)