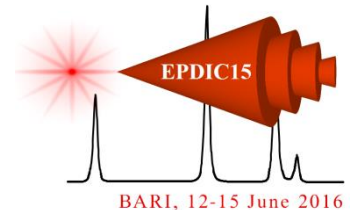# XPDF

The New X-ray Pair Distribution Function Beamline at Diamond Light Source

Phil Chater
Beamline Scientist, XPDF (I15-1)

diamond

EPDIC15, Bari, Italy, 14/06/16

MS06: Progress in instrumentation

EPDIC15

BARI, 12-15 June 2016

XPDF | Acknowledgements

- **diamond**
  - Heribert Wilhelm (PBS)
  - Matt Tucker (now Oak Ridge)
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  - John Sutter (Optics)
  - Dean Keeble (Support sci.)
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  - Steve Usher (Tech.)
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- **THALES SESO**
- **FMB Oxford**
  - Steve Syme
  - Dave Barber
  - Nanna Heiberg
  - Abi Marchant
- **Crystal Scientific**
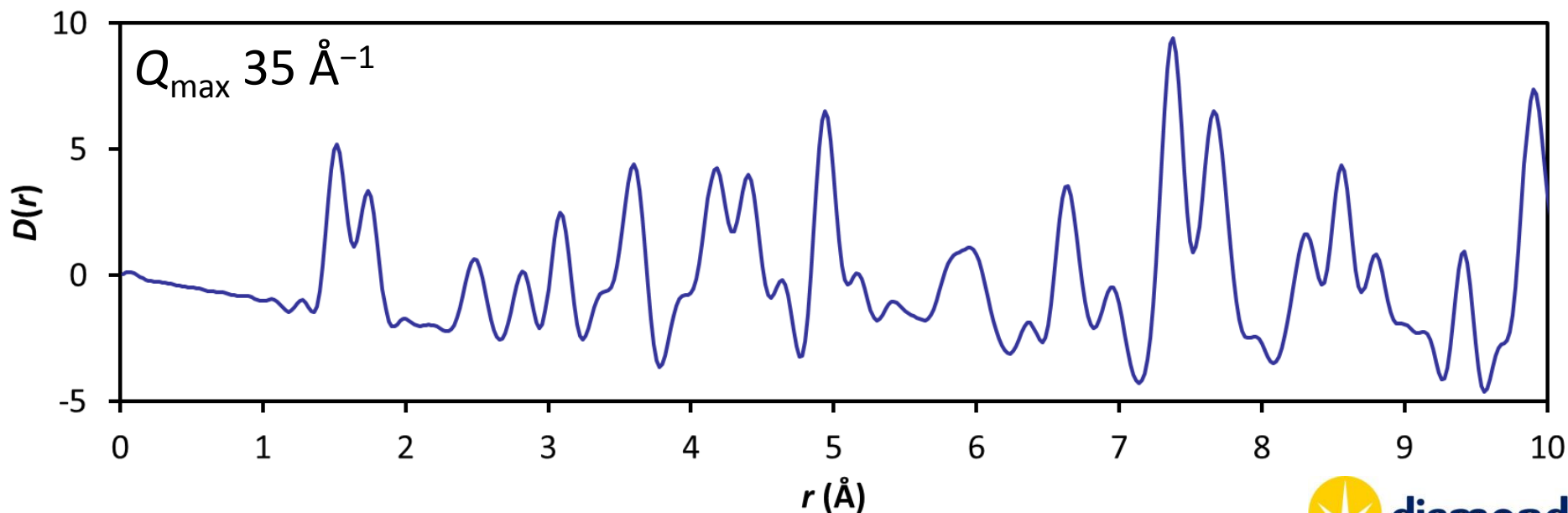 - Simon Cockerton
- **Andrew Goodwin** (Oxford, UWG)

XPDF | New X-ray PDF beamline at DLS

- *"…to produce robust X-ray PDF data in a user friendly, automated way"*
- Study of short- and medium-range order in crystalline, semi-crystalline and amorphous solids and liquids
- Applicable to a wide range of disciplines, e.g.
  - Materials chemistry
  - Solid-state physics
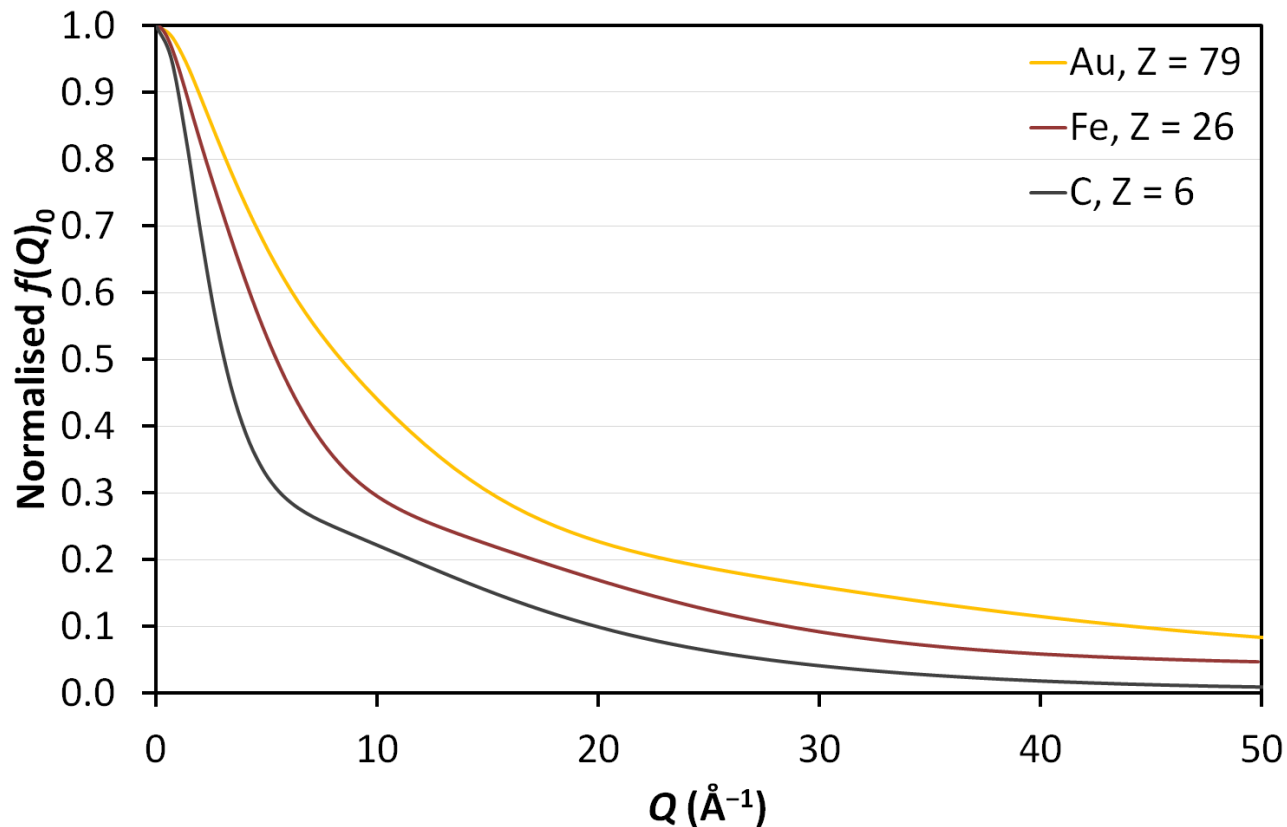 - Earth sciences
  - Pharmaceuticals
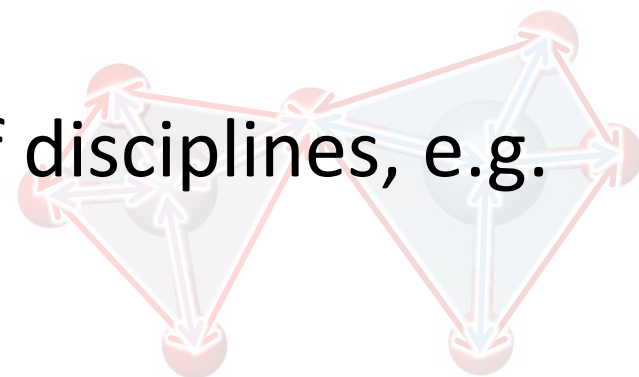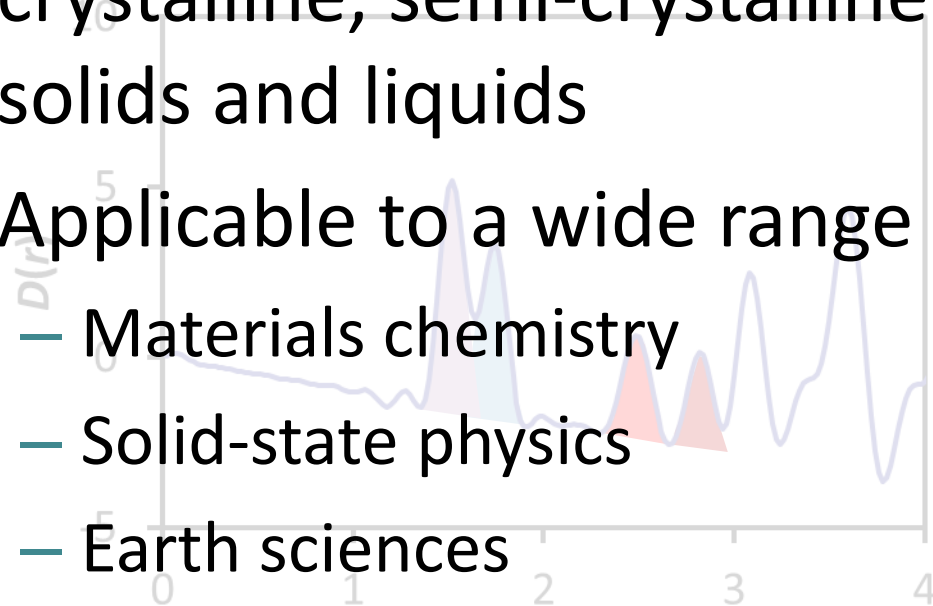
XPDF | Design requirements

- High Q_{max}
  - Resolution of a PDF is dominated by Q_{max}
    - $Q = 2\pi/d = 4\pi\sin\theta/\lambda$
    - $\Delta r \approx 2\pi/Q_{max}$
    - Sample limited resolution if $Q_{max} > 3/\langle\langle u^2 \rangle\rangle^{\frac{1}{2}}$ [†]

Q_{max} 35 Å^{-1}

[†]B. H. Toby and T. Egami, *Acta Cryst. A*, **48** (1992) 336

XPDF | Design requirements

– High Q_{max}

– High flux

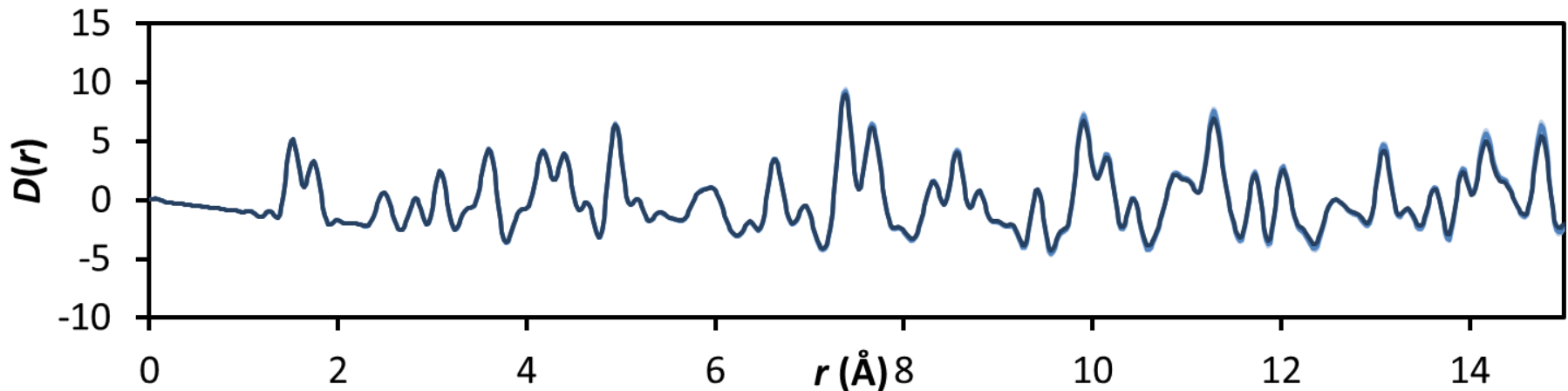 – X-ray form factors fall off dramatically with Q

XPDF | Design requirements

- High Q_{max}

- High flux

- Low (and reproducible) background

  - Need to isolate the weak $S(Q)$ signal from the sample

  - Compton scattering dominates at high Q

XPDF | Design requirements

- High Q_{max}
- High flux
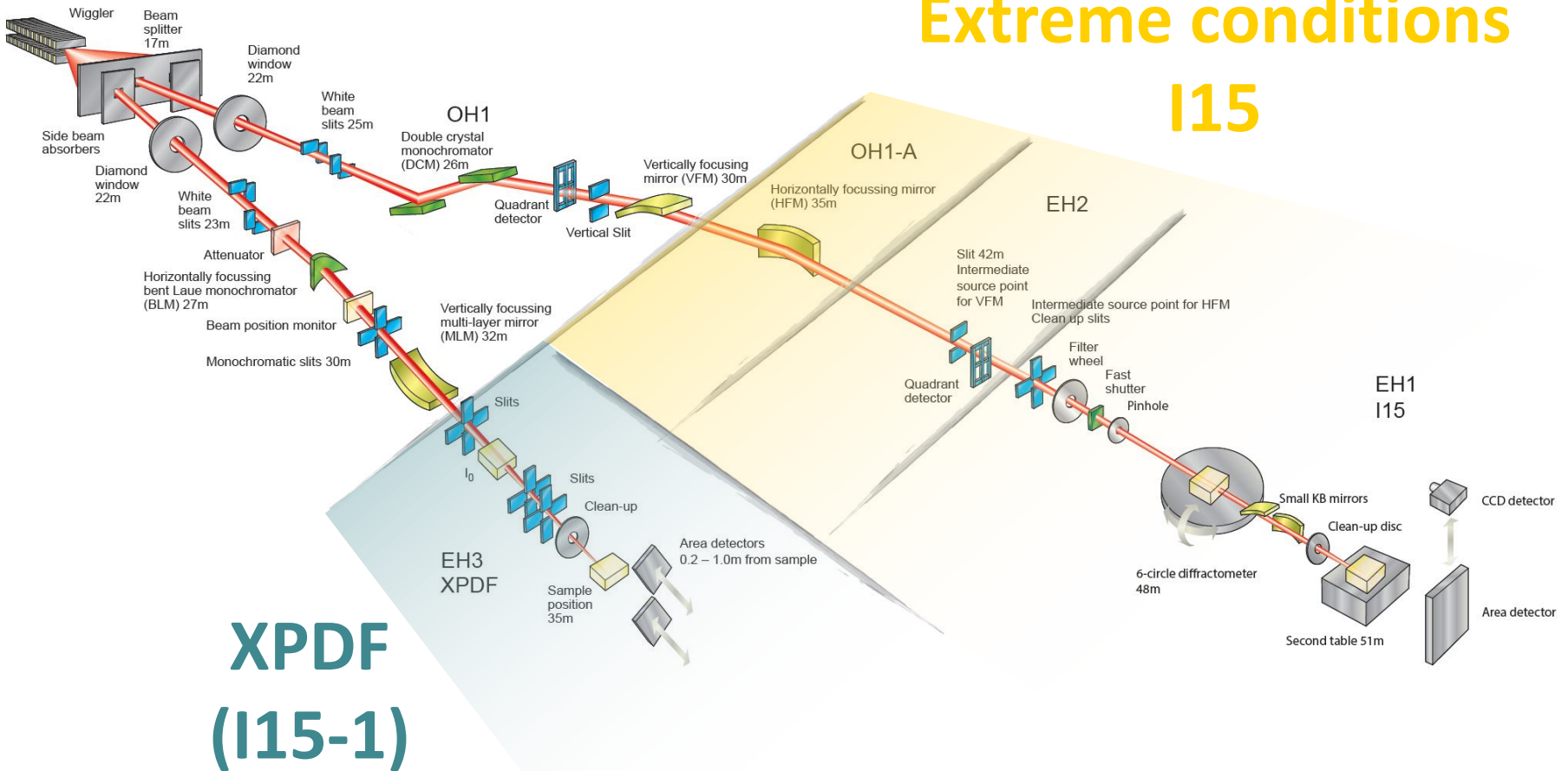- Low (and reproducible) background
- Moderate Q resolution

    – Low-r region unaffected by Q resolution

XPDF | Beamline layout

– Built within I15, but operated independently

Extreme conditions I15

XPDF (I15-1)

XPDF | Beam characteristics

- Three fixed energies
  - Bent-Laue monochromator
    - Si (111), 40.0 keV, 0.310 Å
    - Si (220), 65.4 keV, 0.190 Å
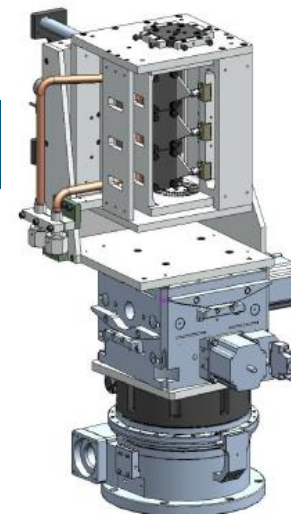   - Si (311), 76.7 keV, 0.162 Å
    - Horizontal focussing to 700 μm

XPDF | Beam characteristics

- Three fixed energies

  - Bent-Laue monochromator

    - Si (111), 40.0 keV, 0.310 Å
   - Si (220), 65.4 keV, 0.190 Å
    - Si (311), 76.7 keV, 0.162 Å

  - Multi-layer mirror

    - Three multi-layer stripes
    - Bimorph substrate
   - Vertical focussing to 20 µm, due Sep. 2016

XPDF | Beam characteristics

- Three fixed energies

- Focal spot size 700 μm (h) × 20 μm (v) calculated
  - Smaller beams achieved with collimation

- Variable bandwidth
  - High flux mode
    - BW 1% (76 keV) to 2% (40 keV)
  - High resolution mode
    - Decrease BW at expense of flux

- Flux expected to be 10^{12} ph/s
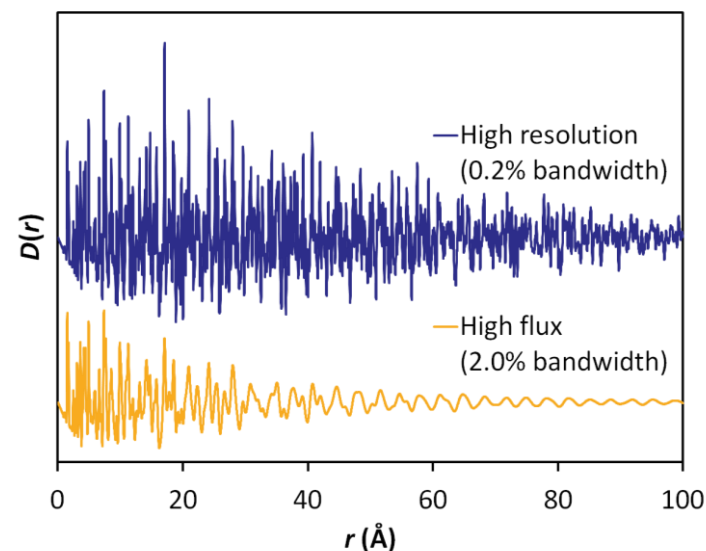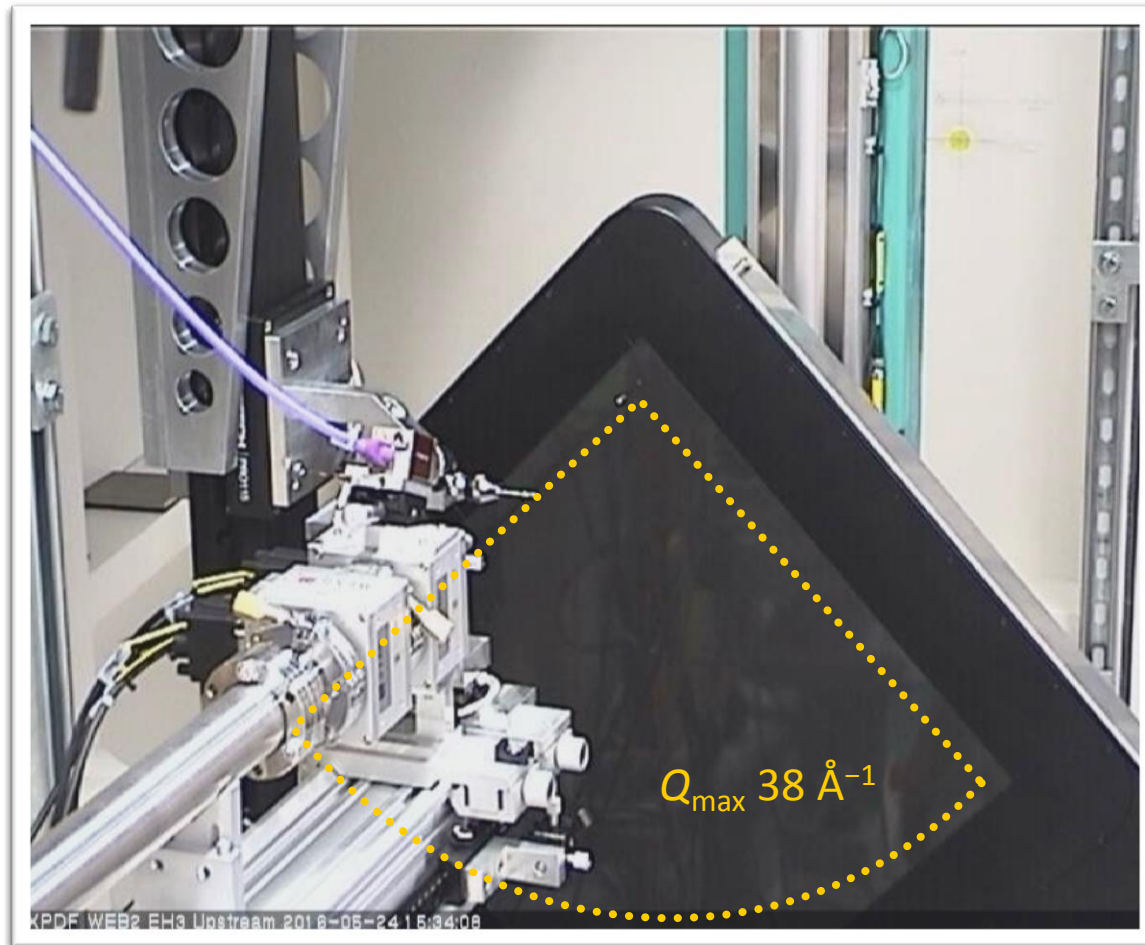
XPDF | Transmission geometry

– RA-PDF[†] geometry data collection
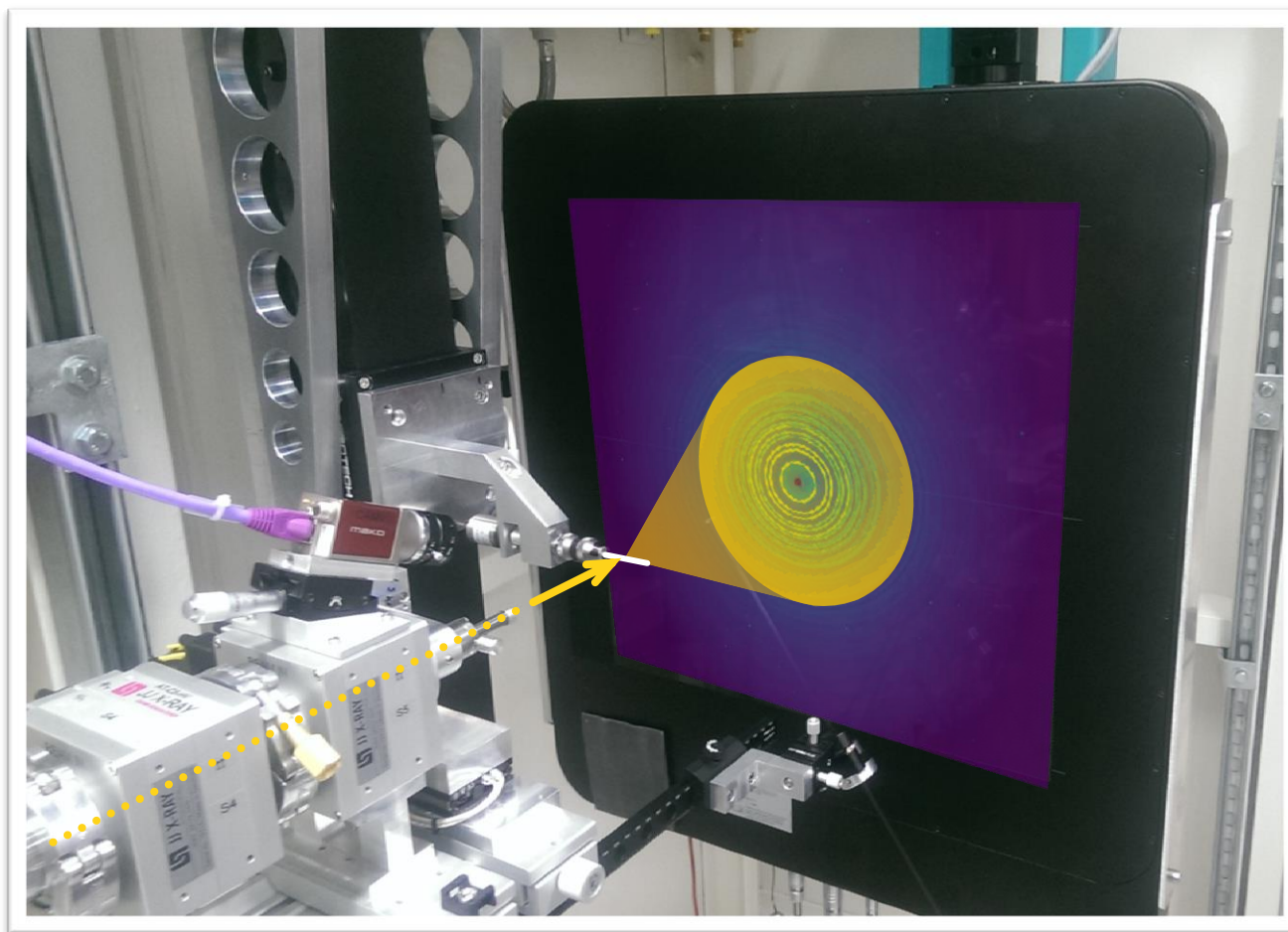
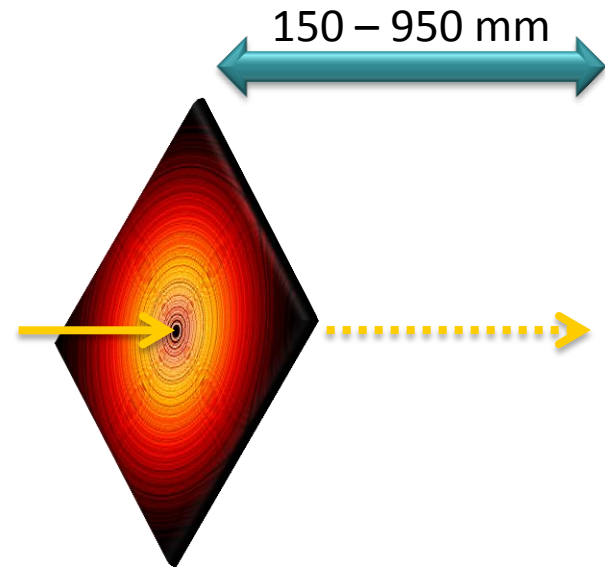†P. Chupas *et al.*, *J. Appl. Cryst.*, **36**(6) (2003) 1342

XPDF | Endstation

– Detectors: Perkin Elmer a-Si TFT/CsI detector
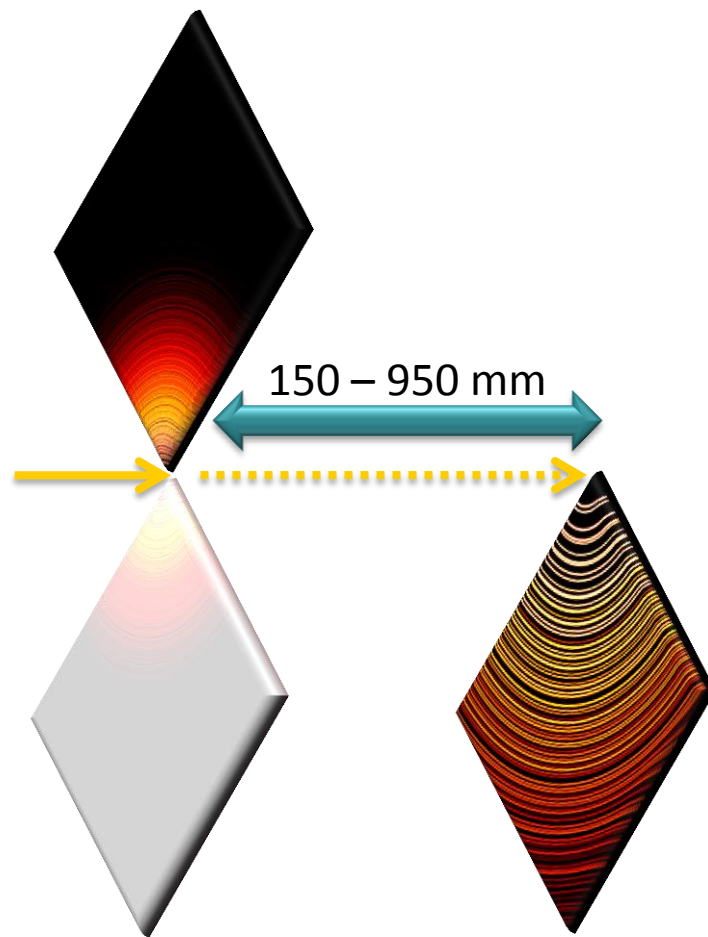
   – Flexible detector positioning

Q_{max} 38 Å^{-1}

XPDF | Endstation

- Detectors
  - Flexible detector positioning
  - "Symmetrical" PDF / Bragg

150 – 950 mm
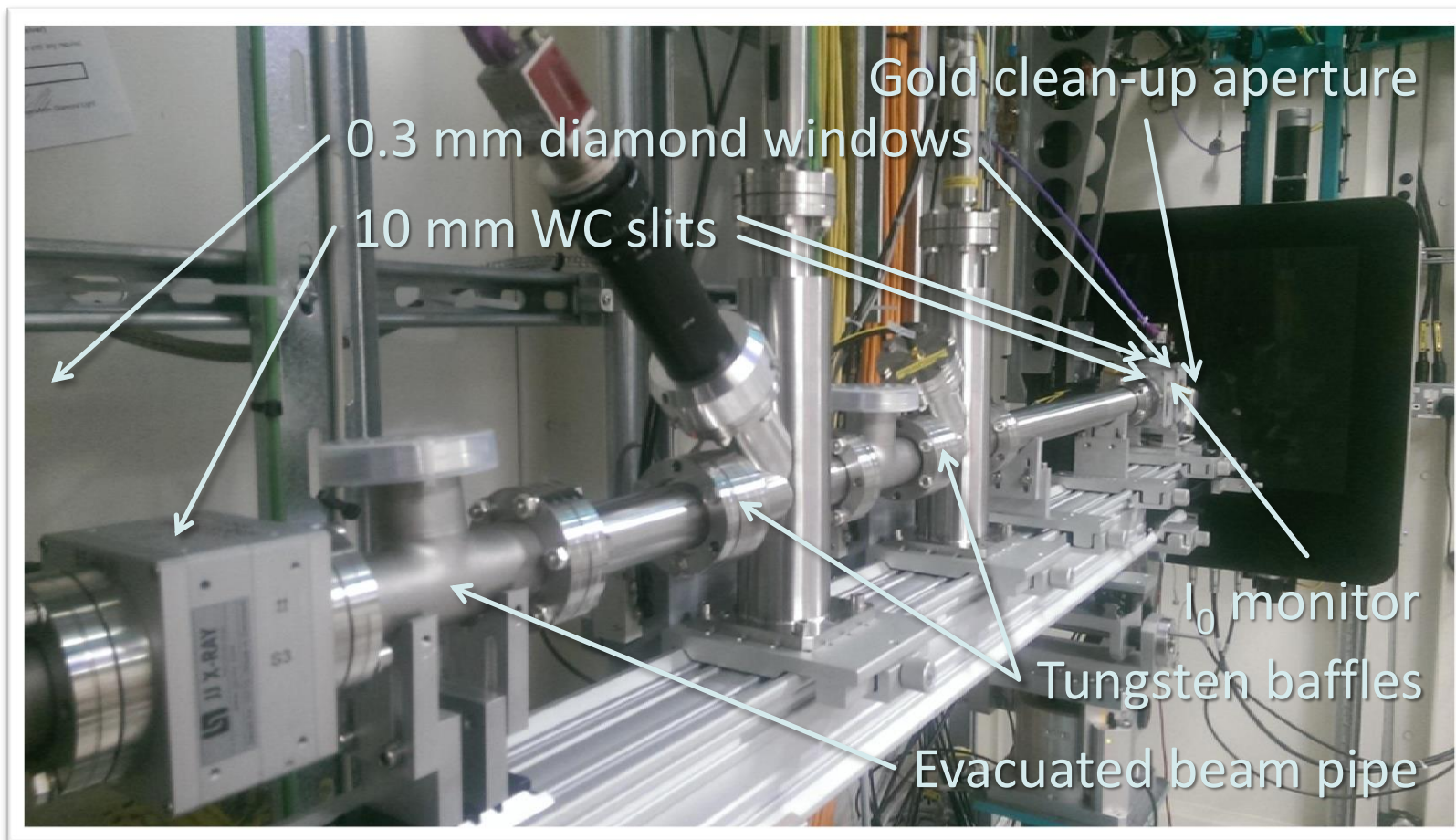
XPDF | Endstation

– Detectors

  – Flexible detector positioning

  – "Symmetrical" PDF / Bragg

  – 2nd detector due December

    • PDF + Bragg

150 – 950 mm

XPDF | Endstation

– ## Low background

Gold clean-up aperture

0.3 mm diamond windows

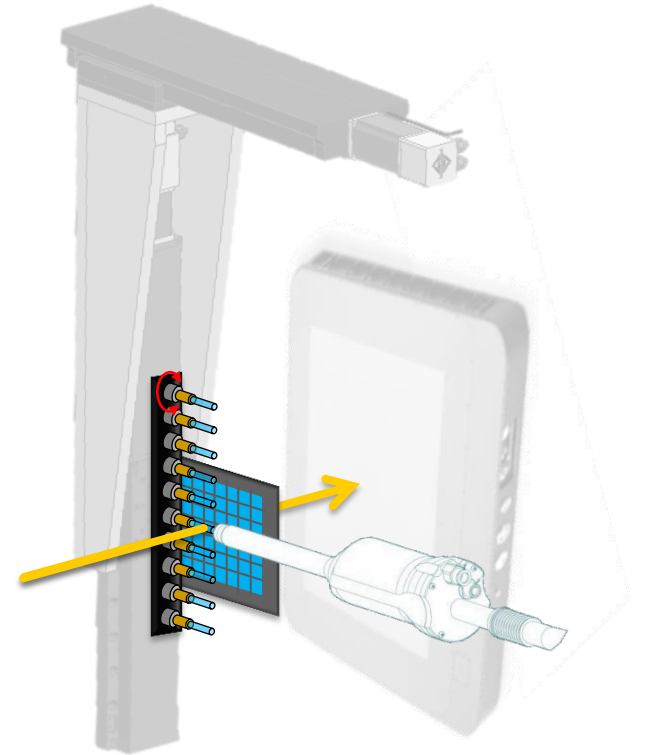10 mm WC slits

I_0 monitor

Tungsten baffles

Evacuated beam pipe

XPDF | Endstation

- Sample environments
  - 250 mm × 450 mm XY travel
  - Flat plate samples
  - Capillary spinners
  - Hot air blower (1200 K)
  - Cryojet (85 – 500 K)
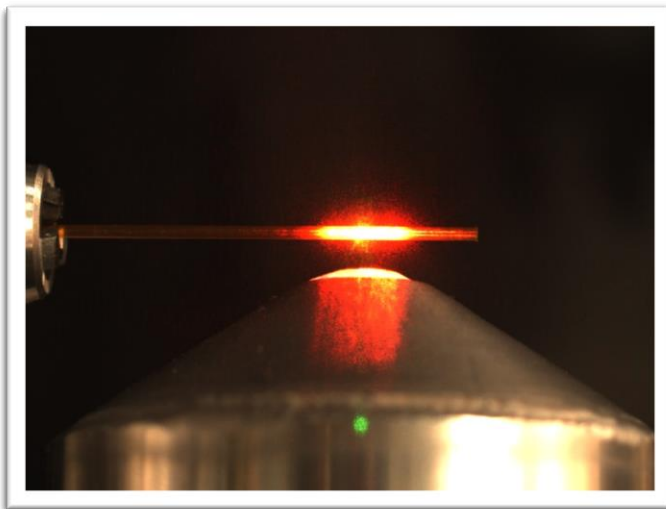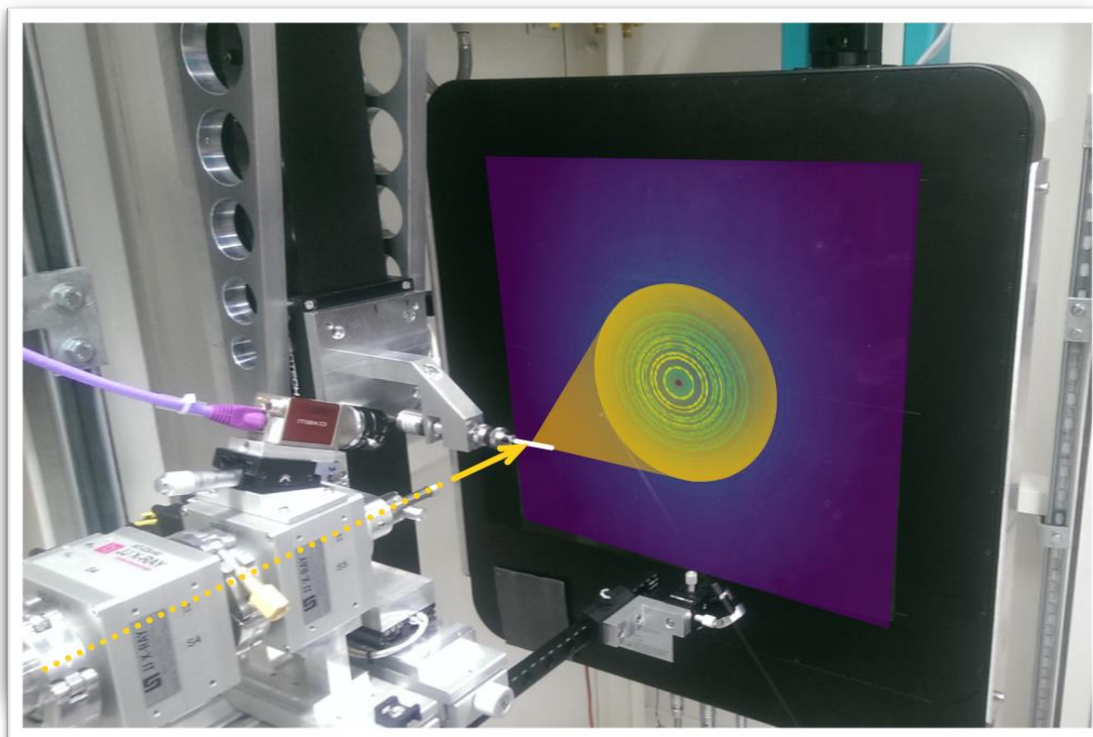
XPDF | Endstation

- Sample environments
  - 250 mm × 450 mm XY travel
  - Flat plate samples
  - Capillary spinners
  - Hot air blower (1200 K)
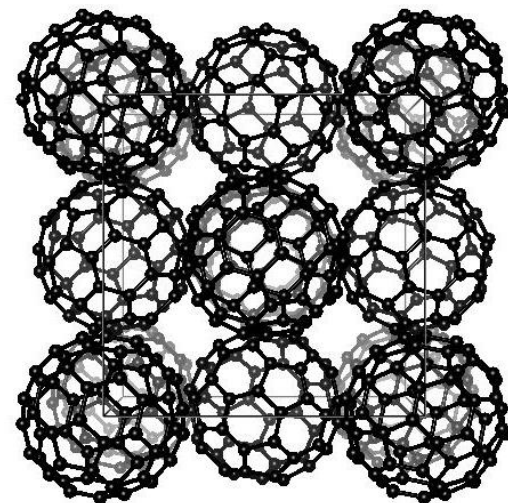 - Cryojet (85 – 500 K)
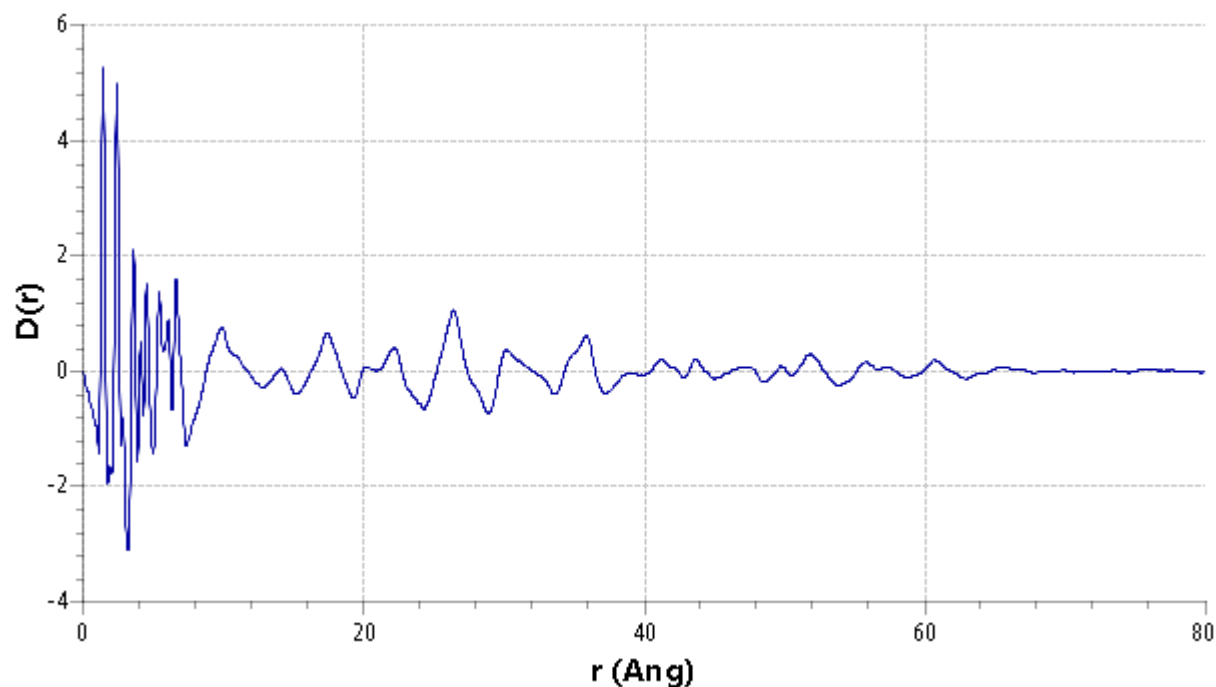 - Electrochemical cells

XPDF | Beamline status

- First light in XPDF endstation on 8th April 2016
- First users on 14th April 2016
  - PDF data processed using Diamond's DAWN software
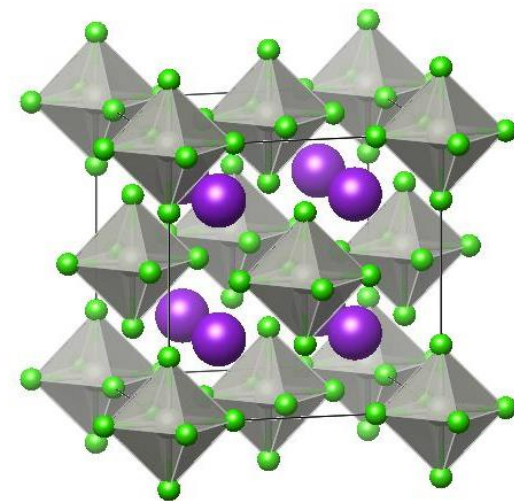- Commissioning call should by end of June

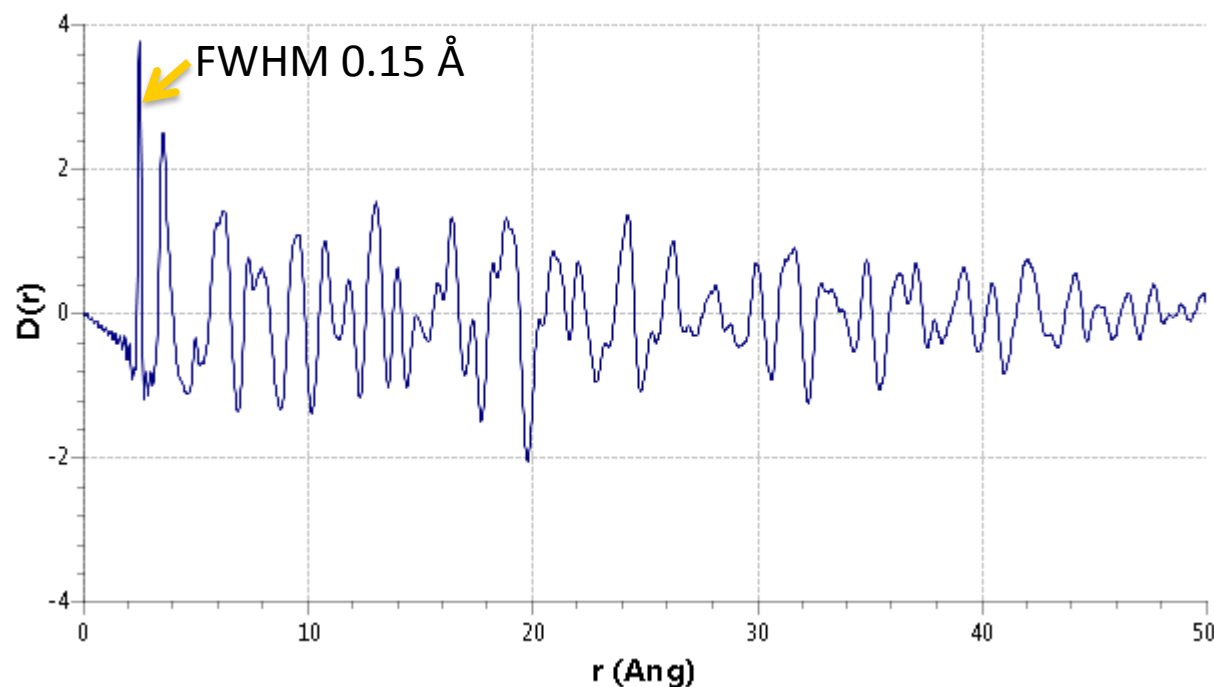# XPDF | First data

- Un-focussed data, 76 keV
  - C_{60}
  - 15 mins, 4 mm cap., symmetric collection, Q_{max} 22 Å^{-1}

XPDF | First data

– Un-focussed data, 76 keV

  – K_2PdBr_6

  – 30 mins, 1 mm cap., offset diamond, Q_{max} 35 Å^{-1}

FWHM 0.15 Å

XPDF | First data

– First horizontally focussed data, 65 keV
  – $Zn(CN)_2$
  – 64 seconds, symmetric collection, Q_{max} 25 Å^{-1}
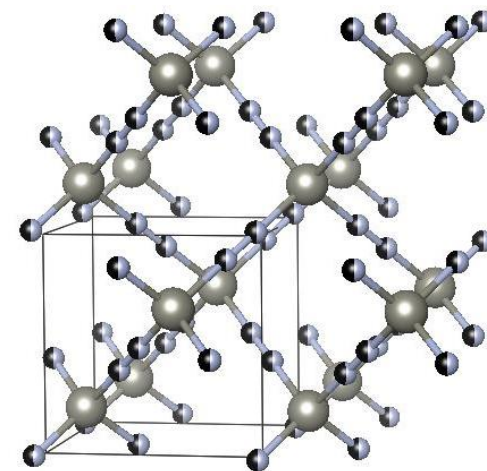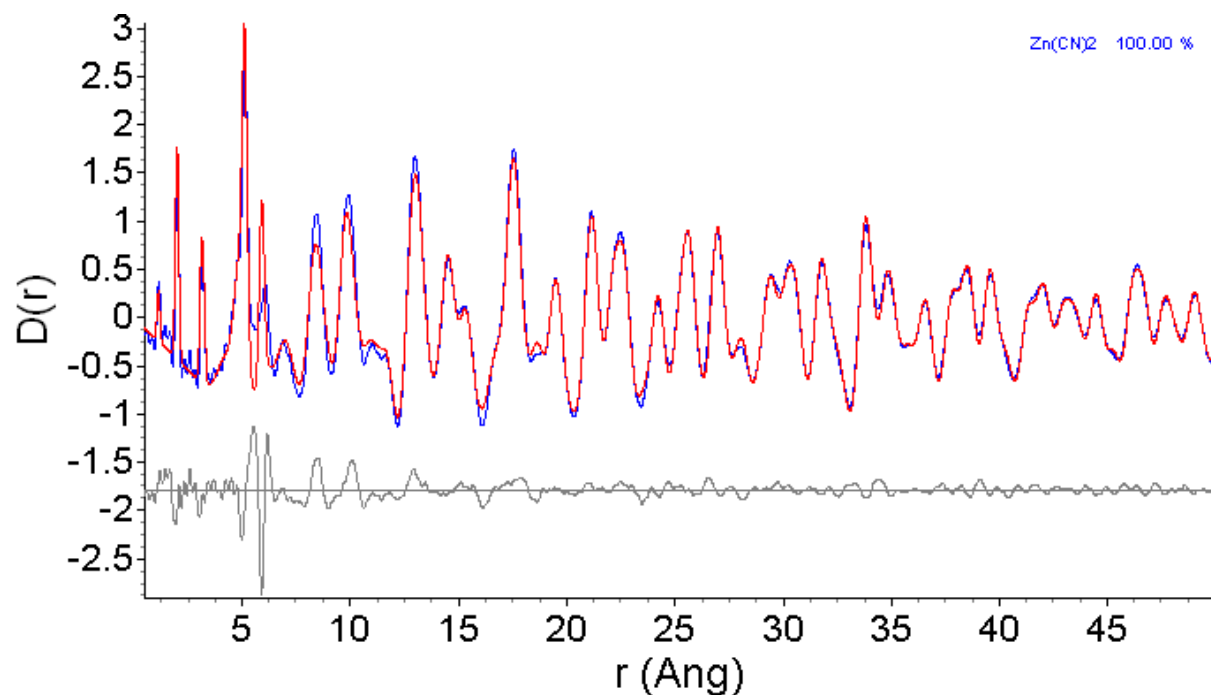
XPDF | Next commissioning steps

– June-Aug. 2016

   – Full characterisation of horizontal focussing

   – Q_{max} optimisation

Detector	40.0 keV	65.3 keV	76.6 keV
⊗	17.4 Å^{-1} [18.8 Å^{-1}]	28.4 Å^{-1} [30.8 Å^{-1}]	33.4 Å^{-1} [36.1 Å^{-1}]
⊗	21.5 Å^{-1} [23.5 Å^{-1}]	35.1 Å^{-1} [38.4 Å^{-1}]	41.2 Å^{-1} [45.1 Å^{-1}]

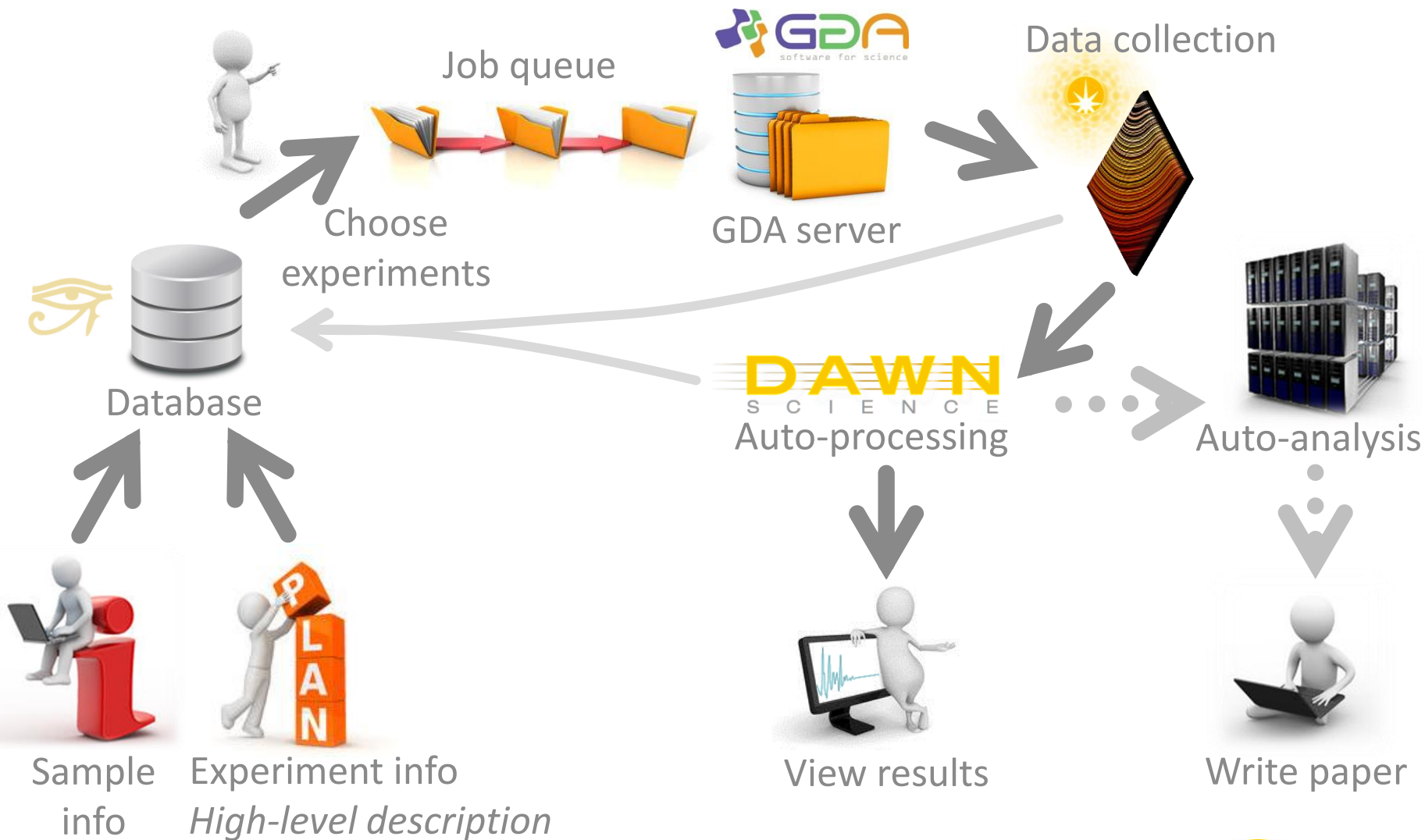[†]Sample-to-detector: 200 mm
Detector size : 409.6 mm × 409.6 mm

XPDF | Next commissioning steps

- June-Aug. 2016
  - Full characterisation of horizontal focussing
  - Q_{max} optimisation
  - Background optimisation
  - Integration of I$_0$
- Sept.-Dec. 2016
  - Vertical focussing
  - Bandwidth selection
 - Integration of software

XPDF Software | Overview

Job queue

Data collection

GDA server

Choose experiments

Database

DAWN SCIENCE
Auto-processing

Auto-analysis

Sample info

Experiment info
High-level description

View results

Write paper

XPDF Software | DAWN Processing

– PDF processing from 2D data has been included in the DAWN[†] Processing Pipeline
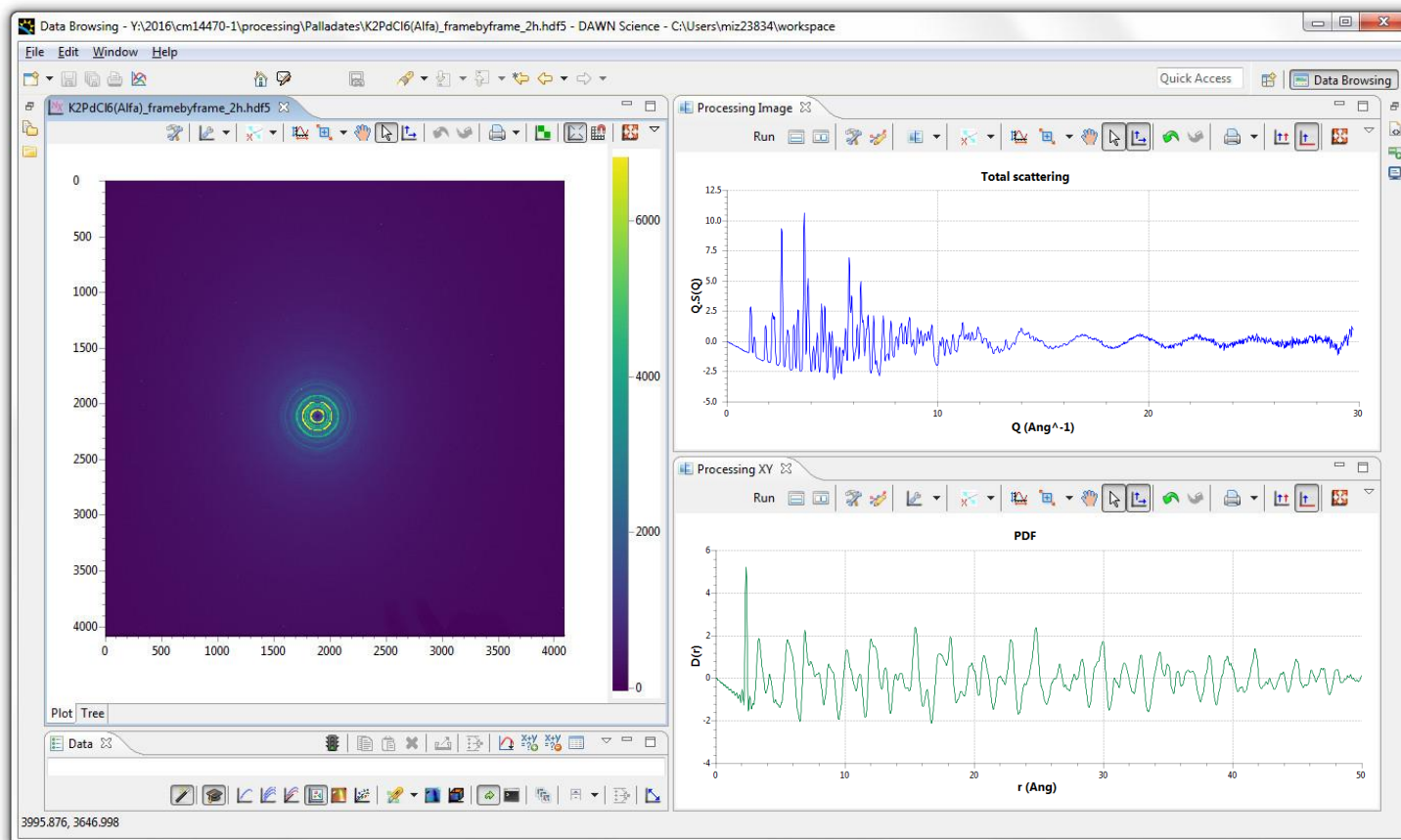
– Follows GudrunX[‡] methodology
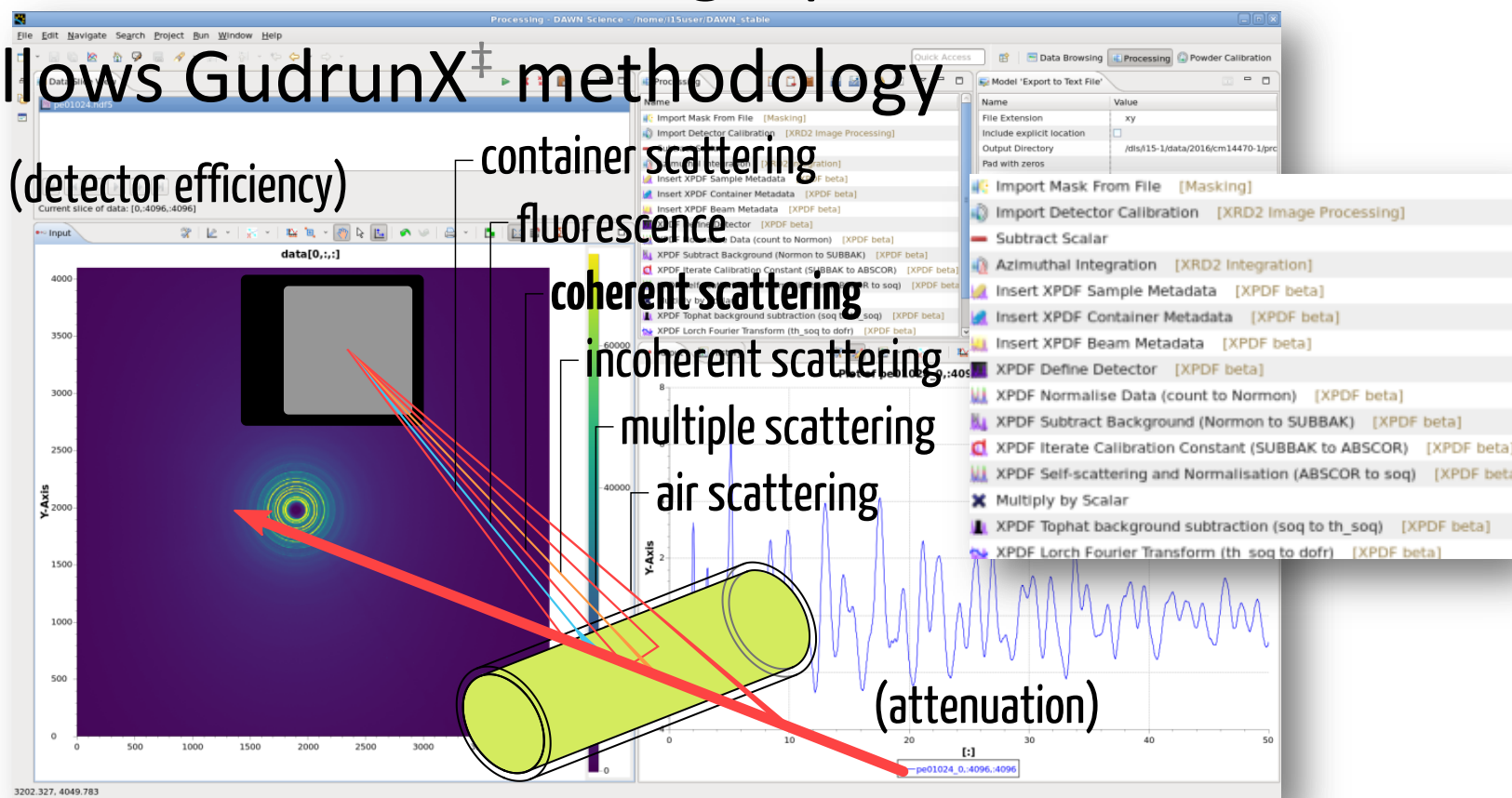
[†] Basham M., Filik J., Wharmby, M. T. *et. al. J. Synchrotron Rad.,* 2015, **22**, 853.
[‡] A. K. Soper and E. R. Barney, *J. Appl. Cryst.*, 2012, **45**, 1314–1317.

XPDF Software | Live PDF data

– A 1D version of the pipeline will show PDF data in real-time (as its collecting)

Data shown at 40× the data collection speed.

XPDF Software | Robust PDF data

– Full 2D data processing is performed on the Diamond Cluster

- Configurable container/sample environment corrections

- Masking handled in 2D

- Errors propagated from the 2D sample, empty and container data

– Beta version of XPDF Processing is already available in DAWN (www.dawnsci.org)

XPDF | Commissioning call

- Call should be open by the end of June

  - Spun capillary samples from 85 K to 1200 K

  - Sample changer for static capillaries / flat plates

  - Likely to be two time slots

    - July-Aug. 2016: Horizontal focussing only

    - Jan.-Mar. 2017: Fully focussed beam

- Full XPDF Software suite will still be undergoing commissioning, so users with some PDF experience recommended

XPDF

The New X-ray Pair Distribution
Function Beamline
at Diamond Light Source

Thank you for your attention

Questions?

XPDF commissioning call open SOON
E-mail: xpdf@diamond.ac.uk
: @xpdfdls