THE AUTOMATED XPDF BEAMLINE AT DIAMOND LIGHT SOURCE

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Senior Beamline Scientist, XPDF (I15-1)

diamond

IUCr2017, MS-005: Total Scattering, 22/08/2017

ACKNOWLEDGEMENTS

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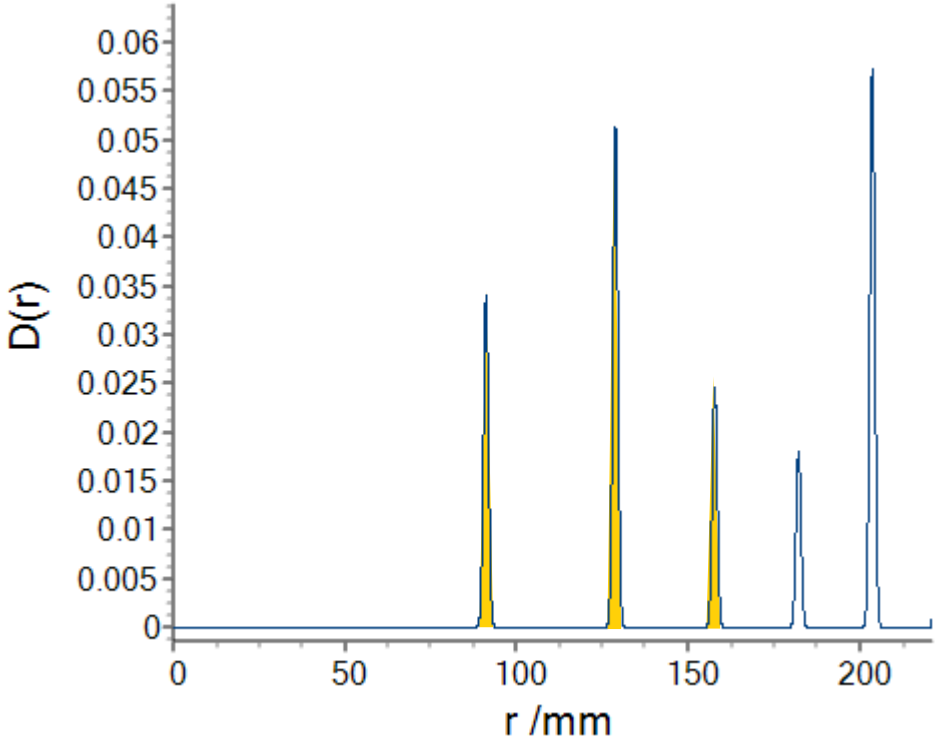
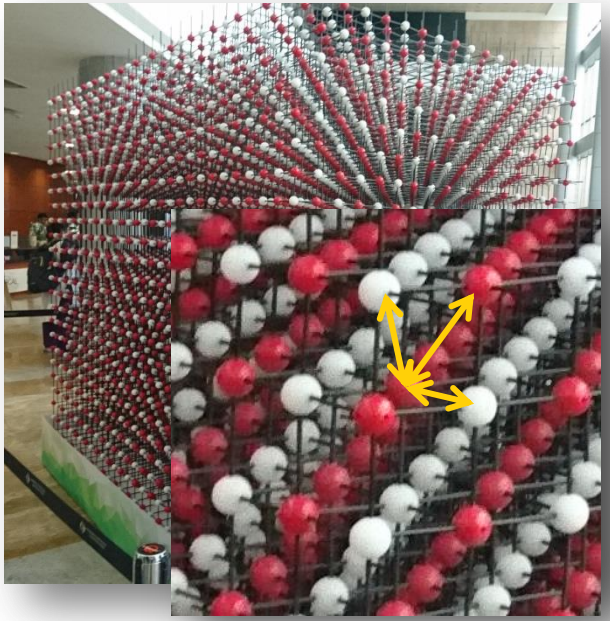
  - Yue Wu (Oxford)
  - Zeyu Deng (Cambridge)
  - Gregor Kieslich (Munich)
  - Fengxia Wei (Cambridge)

WHAT IS A PDF?

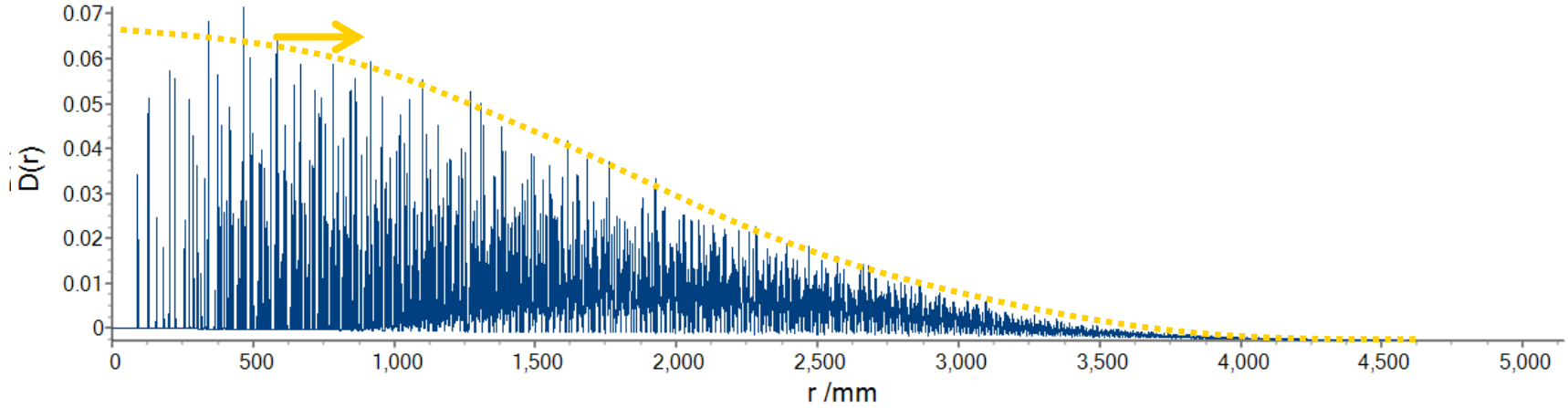
- Probability of finding two atoms at a given separation, *r*





WHAT IS A PDF?

- Peak positions: Bond lengths, geometry, unit cell
- Peak widths: Vibrations, phonons, disorder
- Peak areas: Coordination number, occupancies
- Peak damping: Size, shape

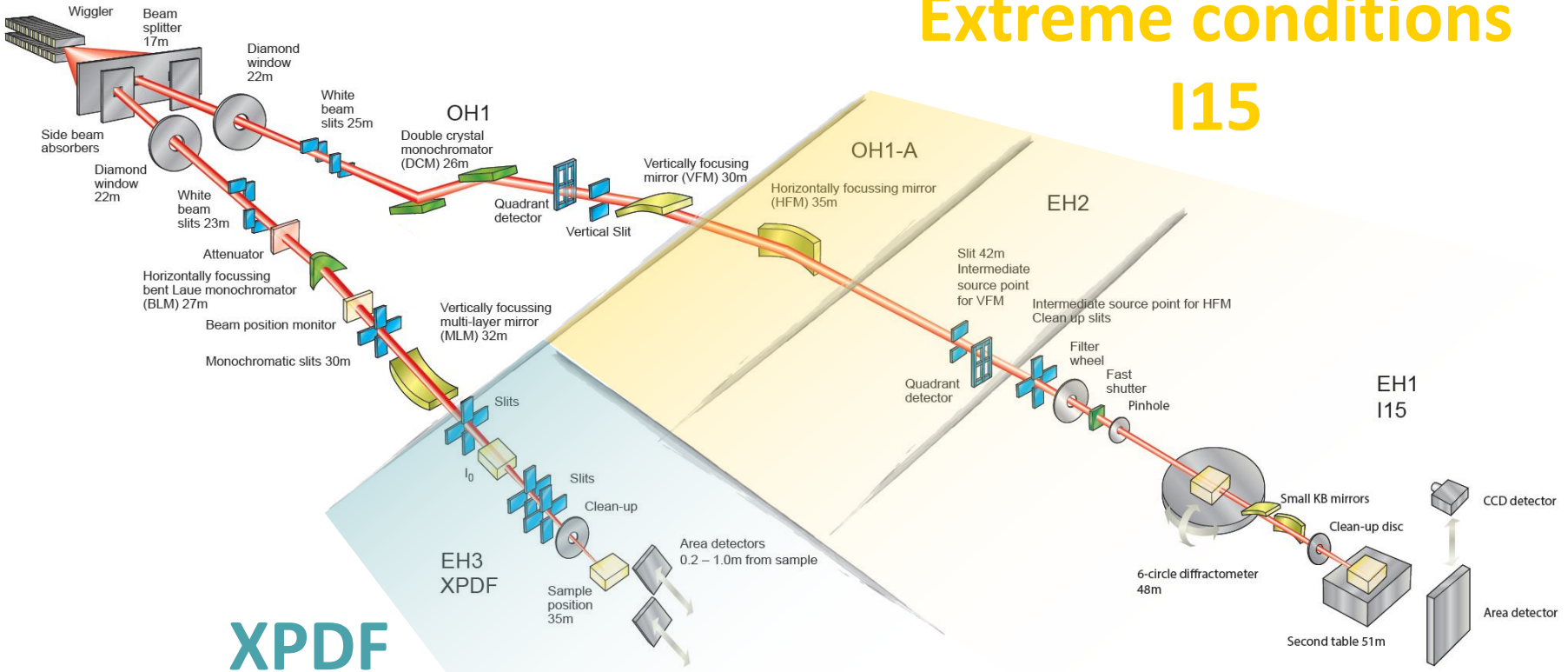


- XPDF (I15-1)

THE XPDF BEAMLINE

Extreme conditions

I15

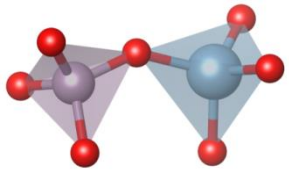


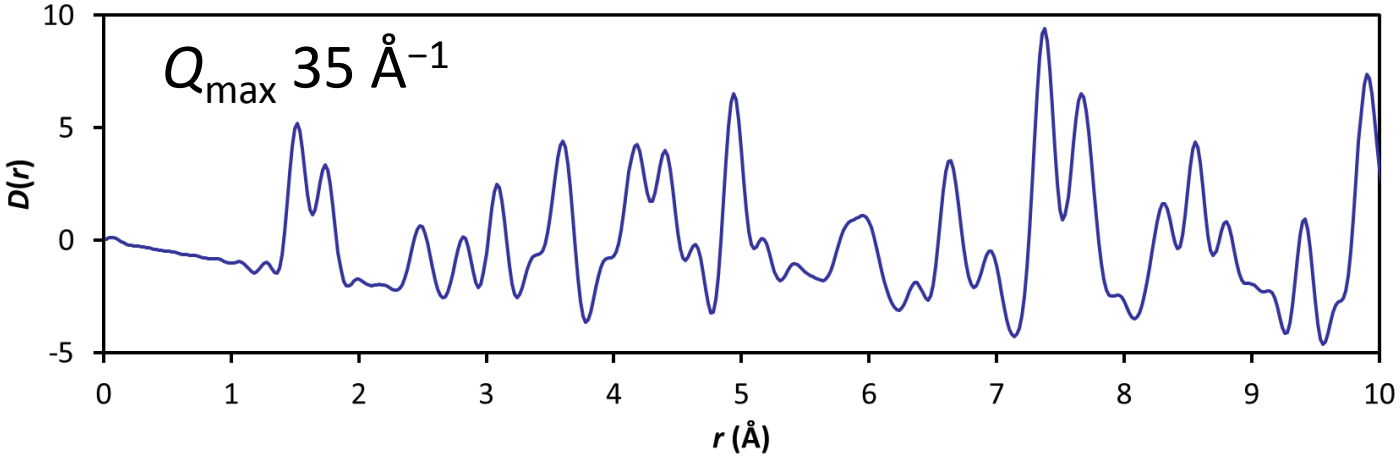
XPDF

(I15-1)

REQUIREMENTS FOR QUALITY PDF DATA

- 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 $Q_{max} > 3/\langle\langle u^2\rangle\rangle^{½}$ †





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

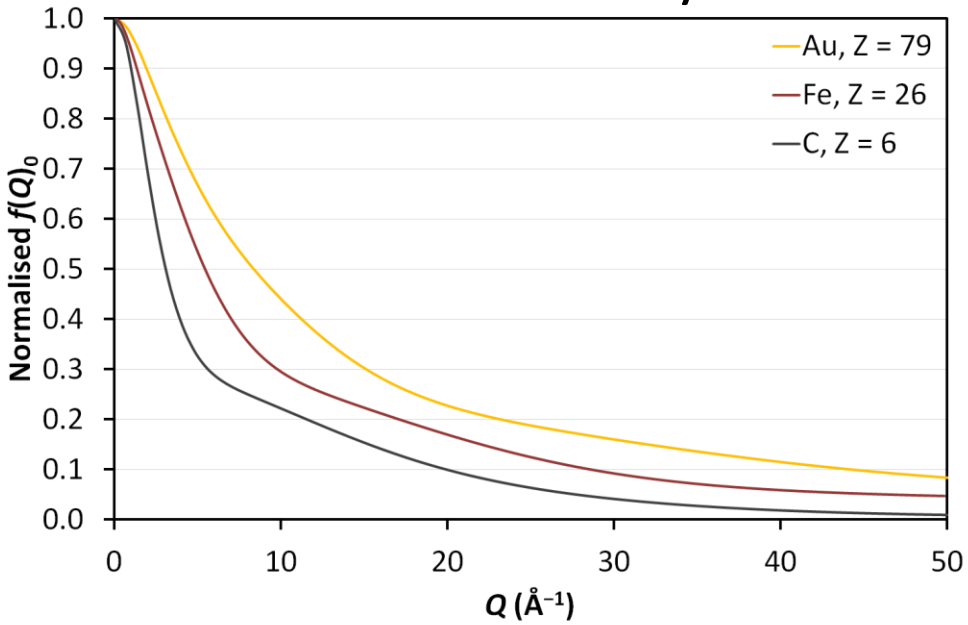
REQUIREMENTS FOR QUALITY PDF DATA

- High Q_{max} ✓
  - Bent-Laue monochromator FMB Oxford
    - 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
  - Large area detector(s)

Q_{max} /Å^{-1}	E /keV	Q_{max} /Å^{-1}
21	40.0	17
35	65.4	28
40	**76.7**	33

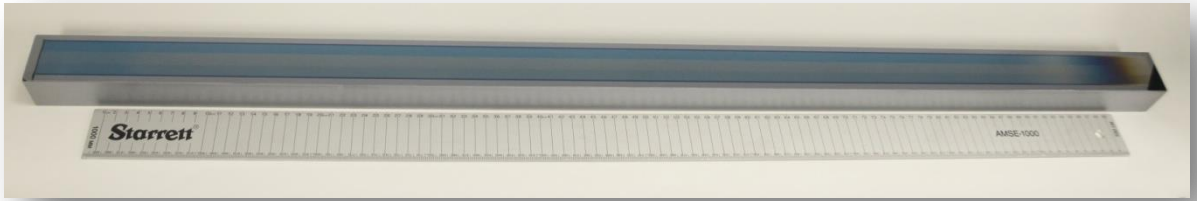
REQUIREMENTS FOR QUALITY PDF DATA

- High Q_{max} ✓
- High flux
  - X-ray form factors fall off dramatically with Q



REQUIREMENTS FOR QUALITY PDF DATA

- High Q_{max} ✓
- High flux ✓
  - 3.5 T superconducting wiggler source
  - Bent Laue crystal to increase bandwidth
  - 1 m long vertical focussing mirror to focus X-rays onto sample

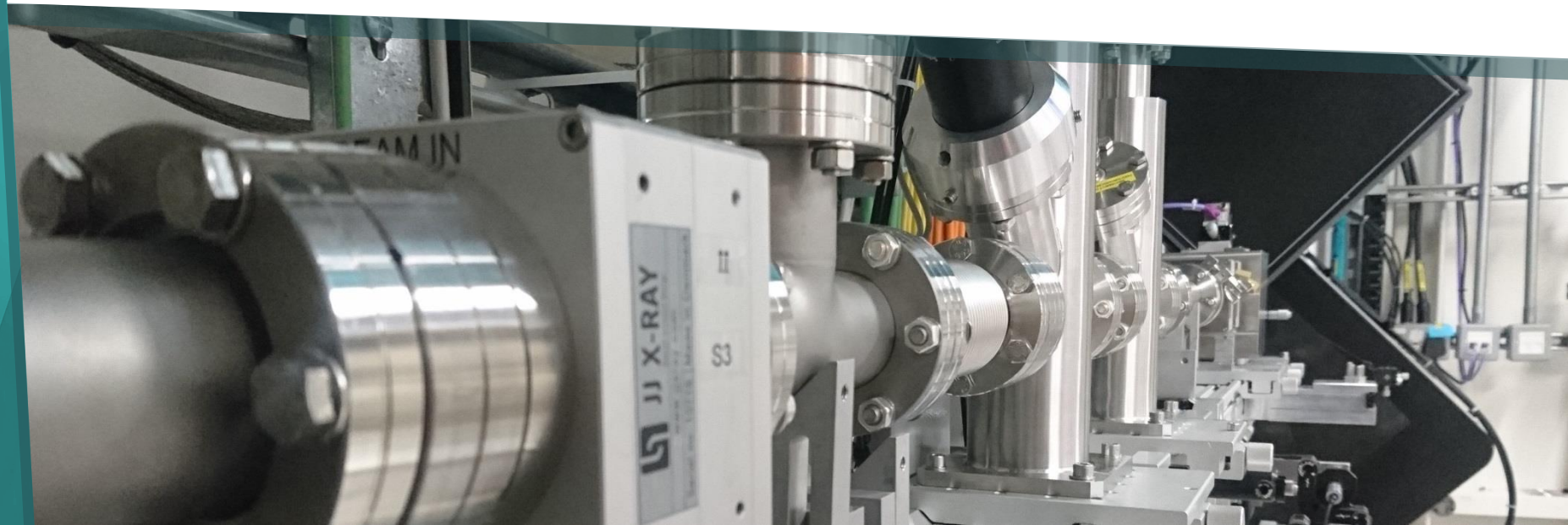


REQUIREMENTS FOR QUALITY PDF DATA

- 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

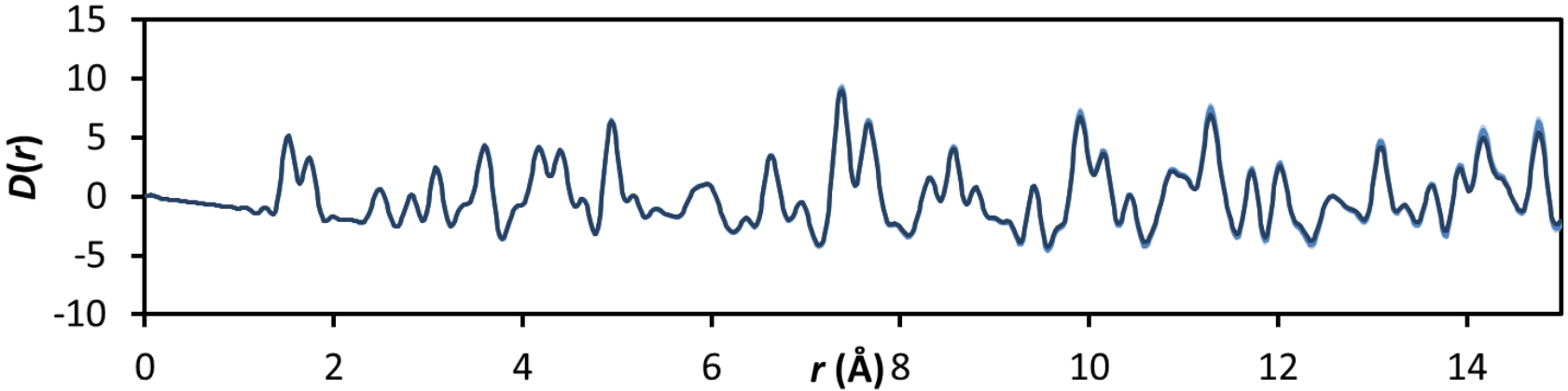
REQUIREMENTS FOR QUALITY PDF DATA

- High Q_{max} ✓
- High flux ✓
- Low (and reproducible) background ✓



REQUIREMENTS FOR QUALITY PDF DATA

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



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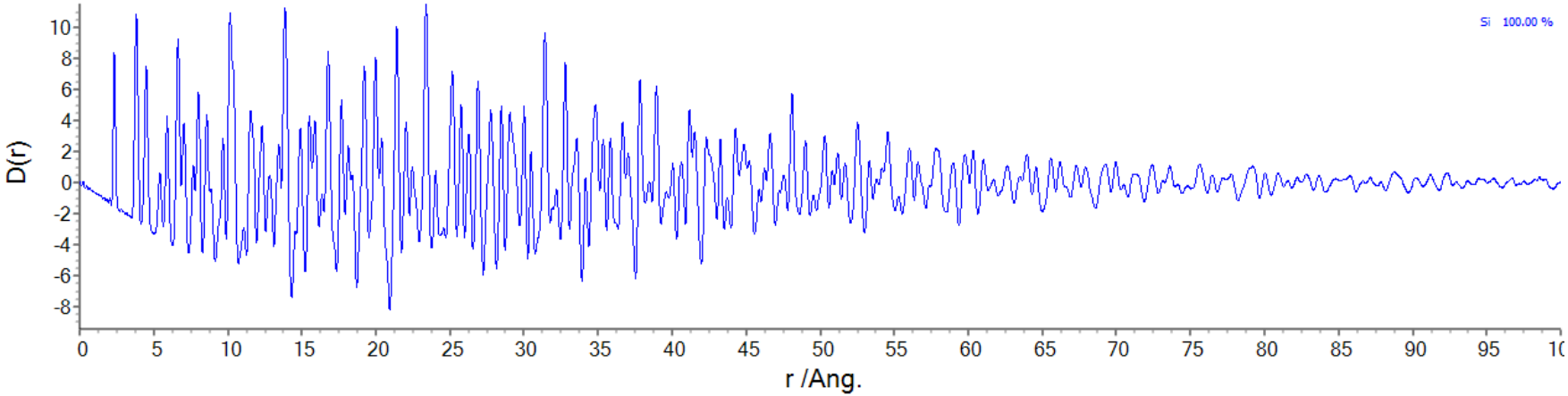
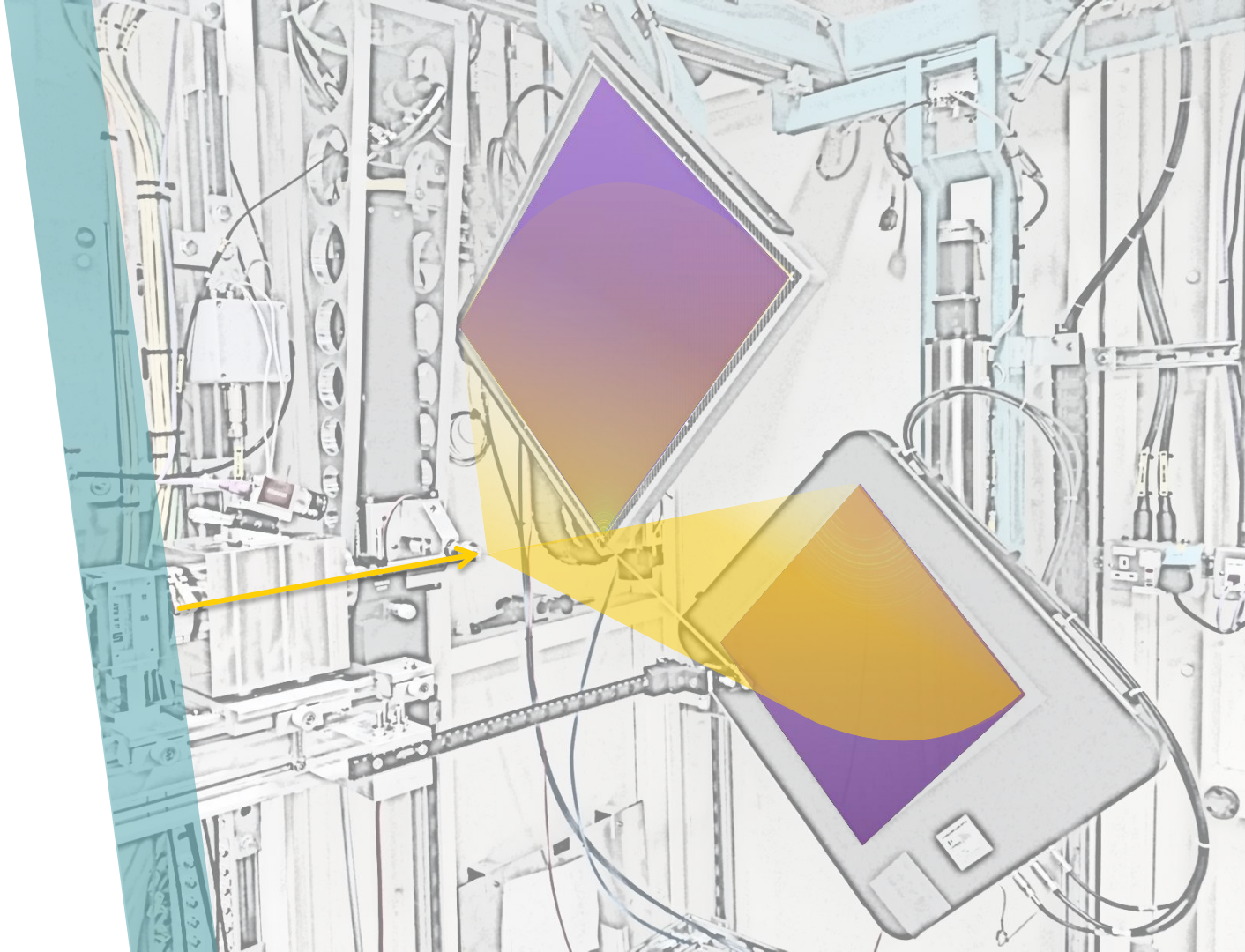
- High Q_{max} ✓
- High flux ✓
- Low (and reproducible) background ✓
- Moderate Q resolution ✓



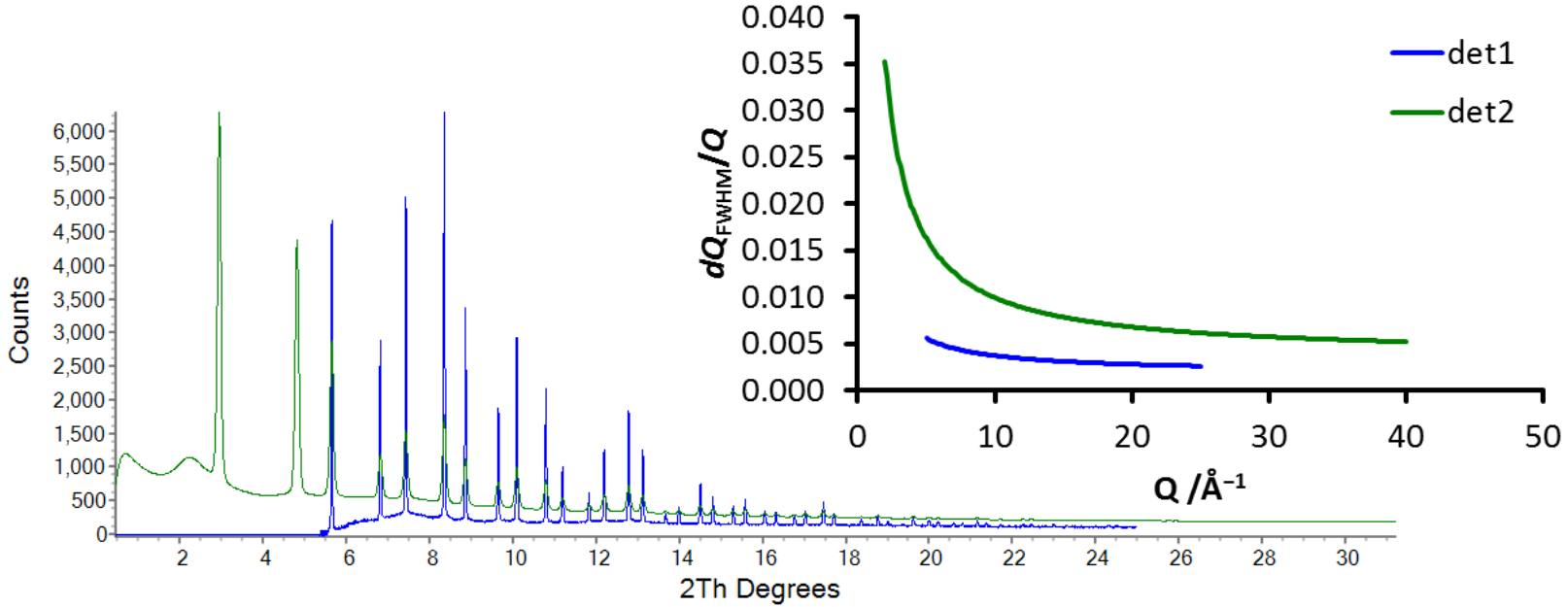
PDF +
Bragg

PDF + Bragg

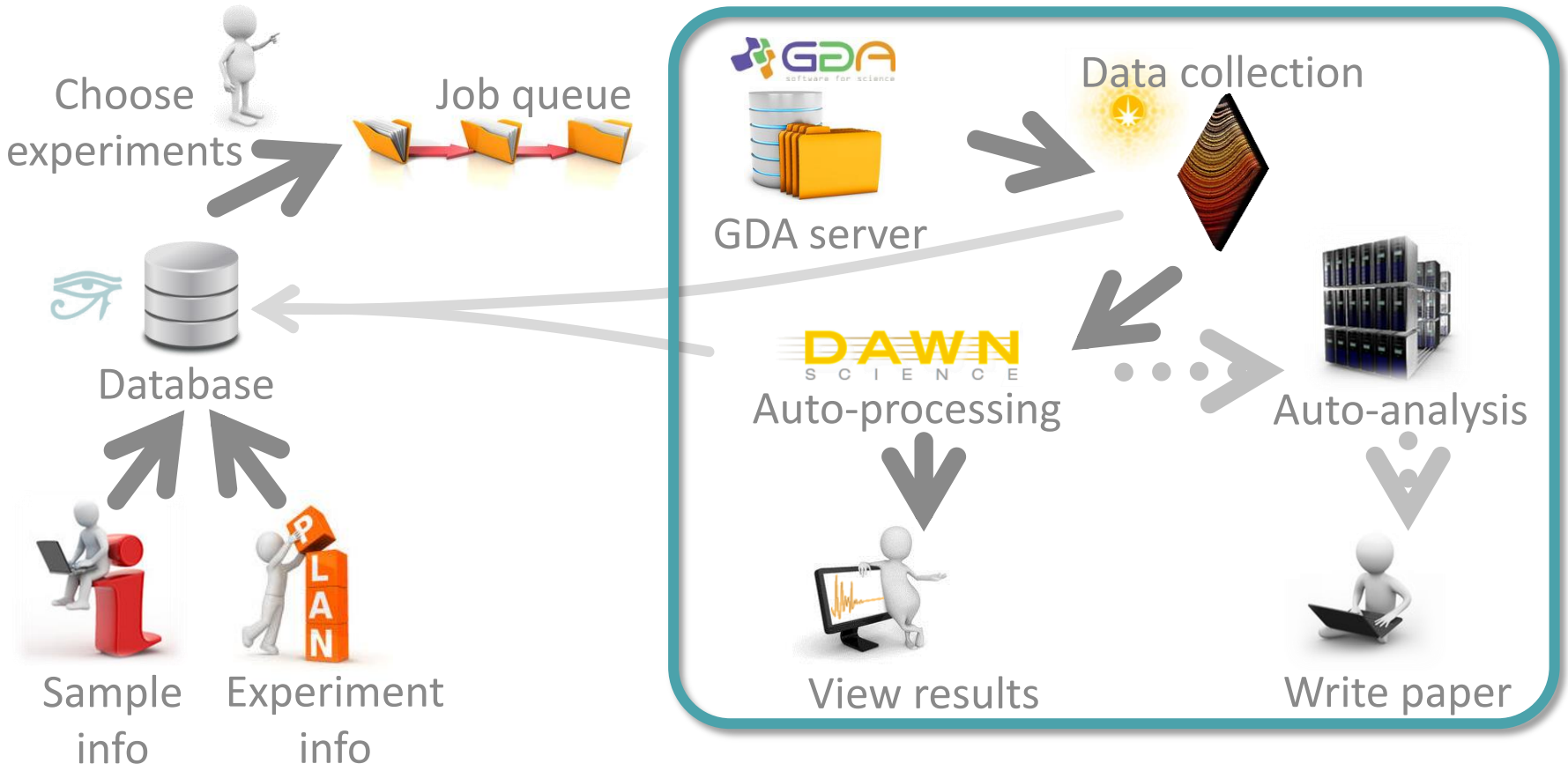


PDF + Bragg

- Allows quality PDF collection alongside higher resolution Bragg data for joint refinements

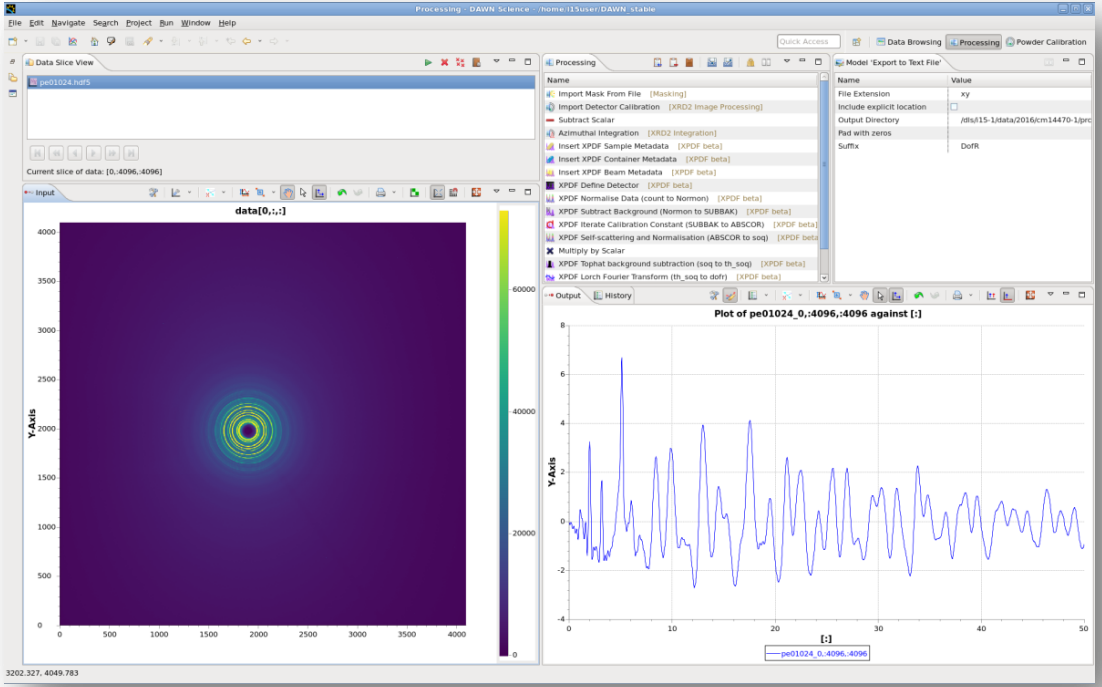


XPDF-SOFTWARE



AUTO-PROCESSING

- Flexible processing of 1- and 2-D data[†]

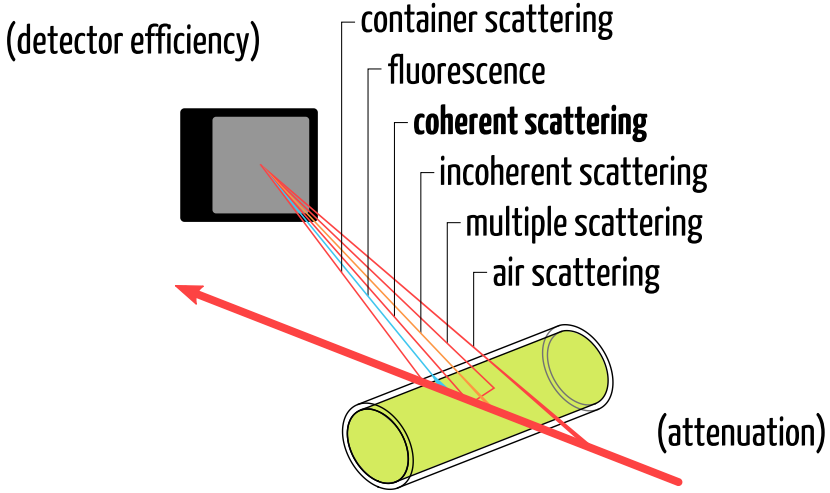


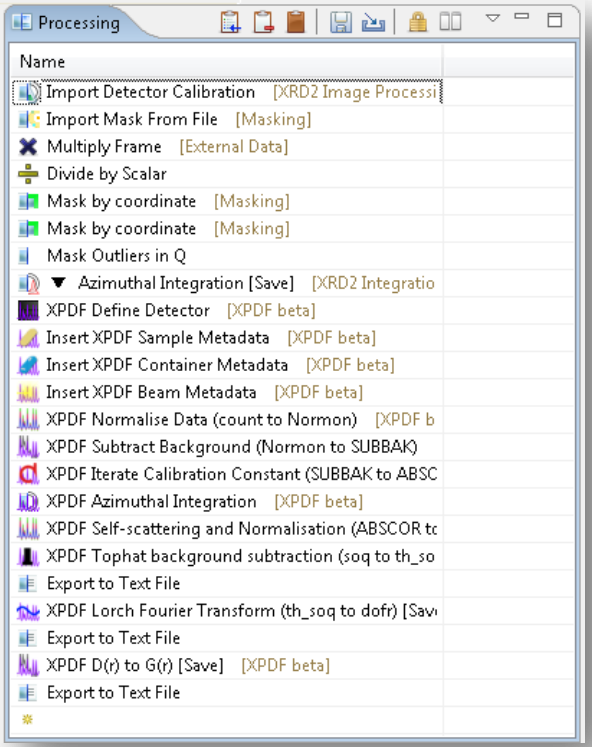
[†] Filik J. *et. al. J. Synchrotron Rad.*, 2017, **50**, 959.

AUTO-PROCESSING

DAWN SCIENCE

- Flexible processing of 1- and 2-D data†
- PDF processing steps based on GudrunX‡ methodology





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‡ A. K. Soper and E. R. Barney, *J. Appl. Cryst.*, 2012, **45**, 1314–1317.

AUTO-PROCESSING

- Flexible processing of 1- and 2-D data[†]
- PDF processing steps based on GudrunX[‡] methodology
- DAWN is free to download from www.dawnsci.org
- Frequent updates, so check back every few months or follow @DAWNScience

[†] Filik J. *et. al. J. Synchrotron Rad.*, 2017, **50**, 959.

[‡] A. K. Soper and E. R. Barney, *J. Appl. Cryst.*, 2012, **45**, 1314–1317.

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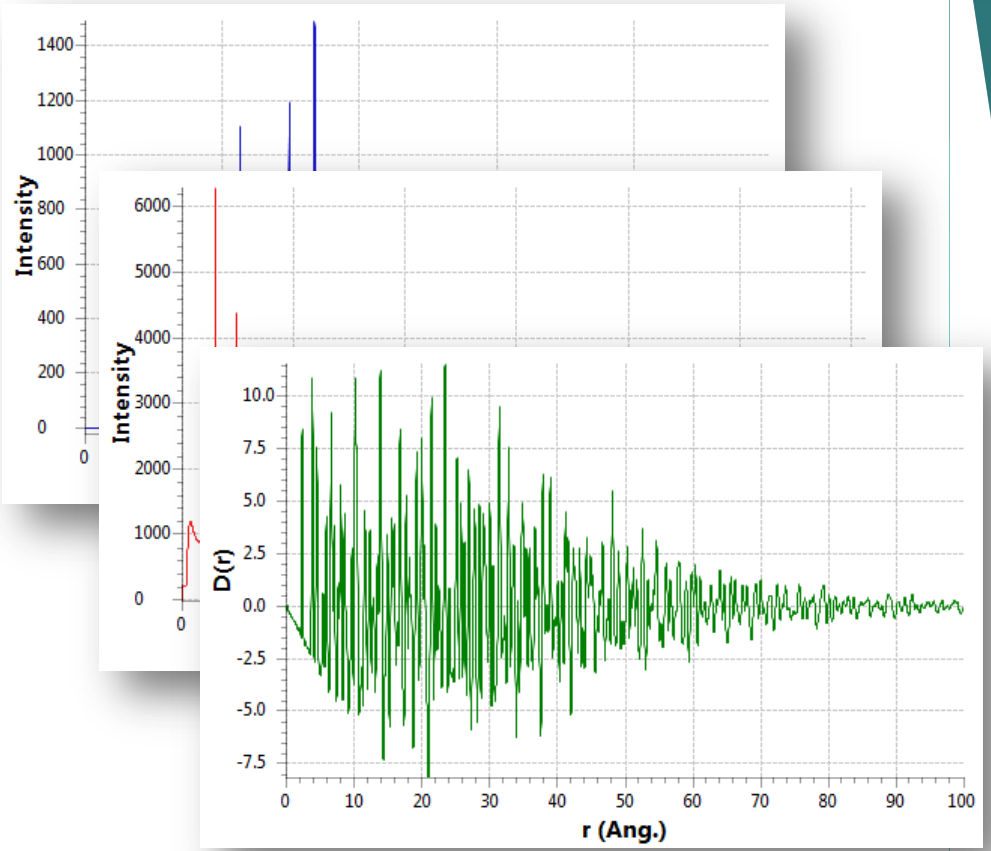

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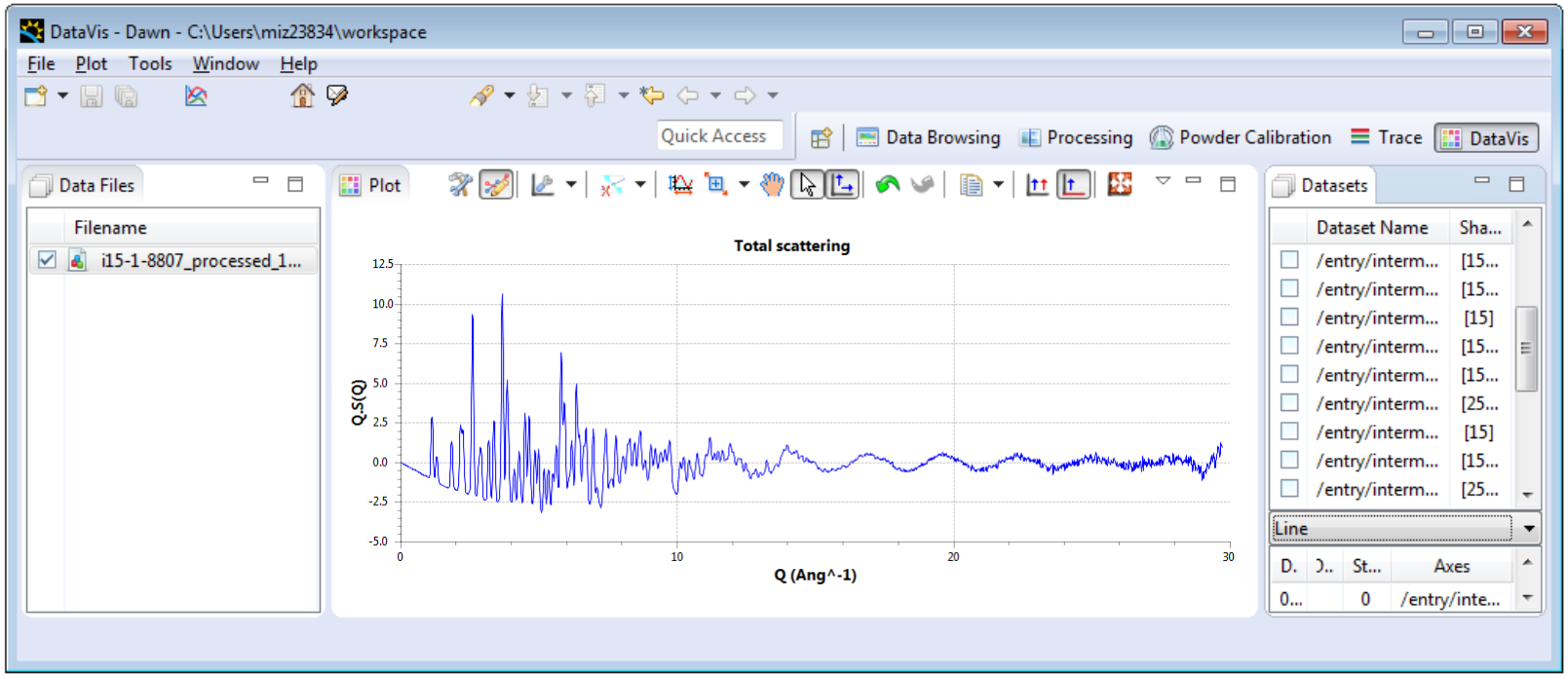

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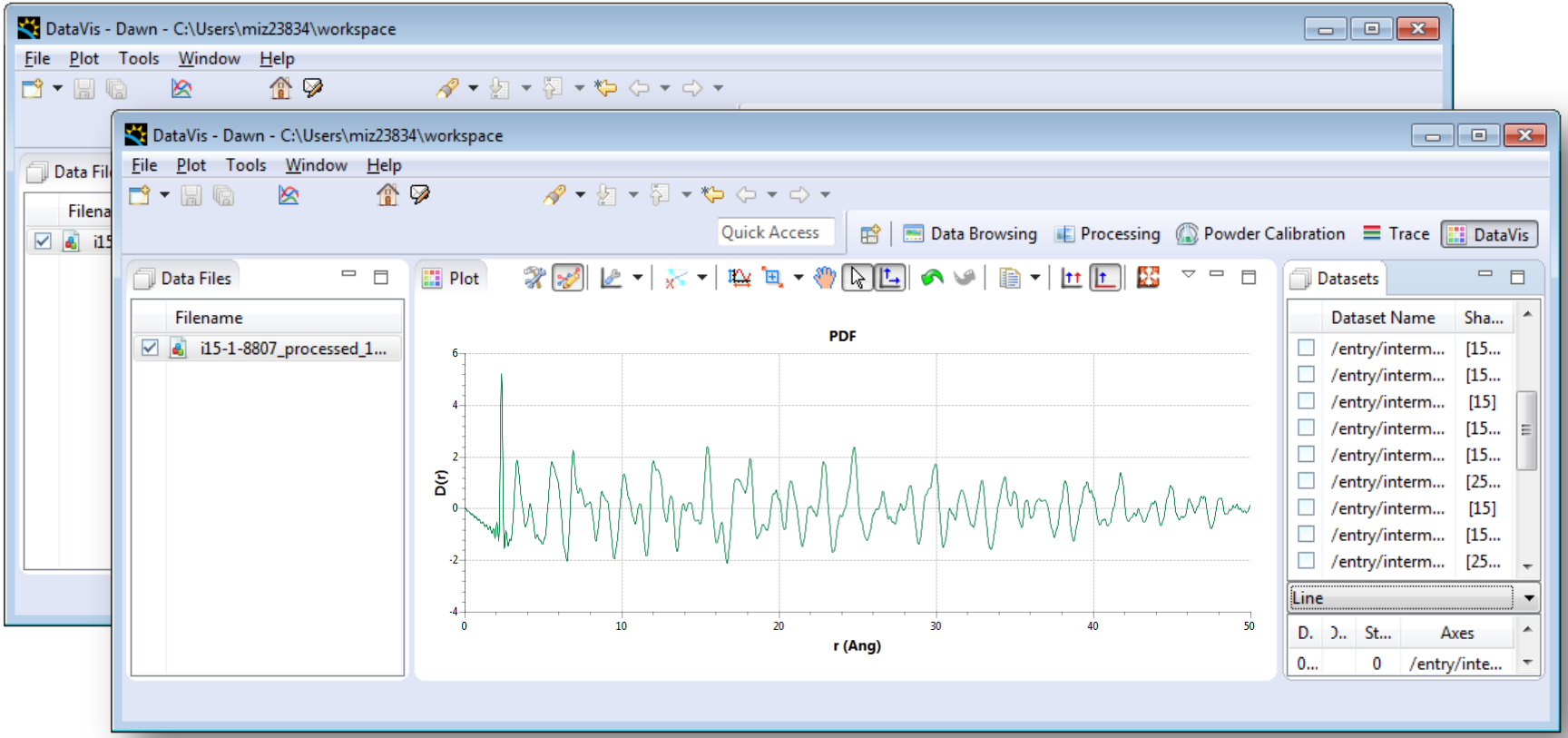
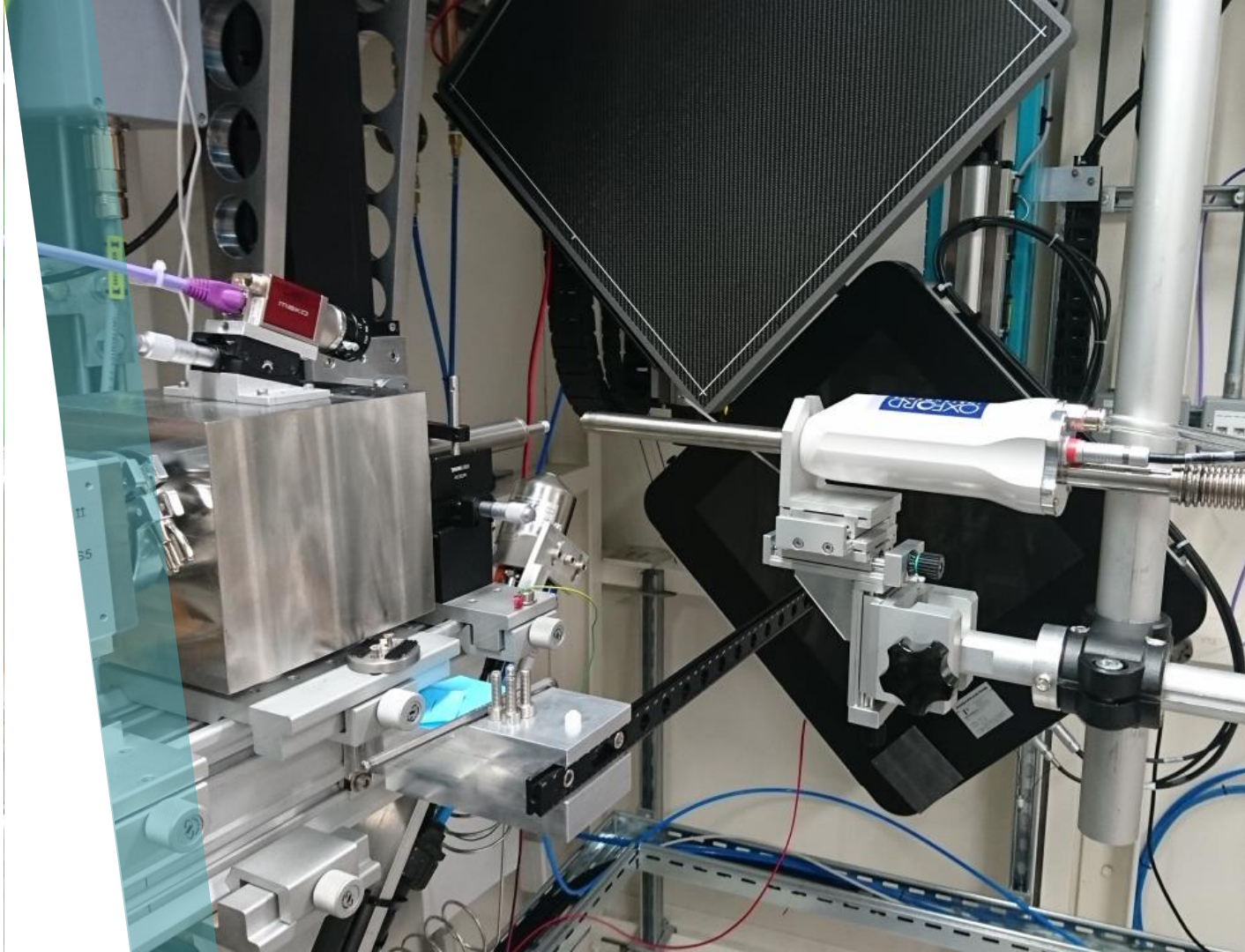
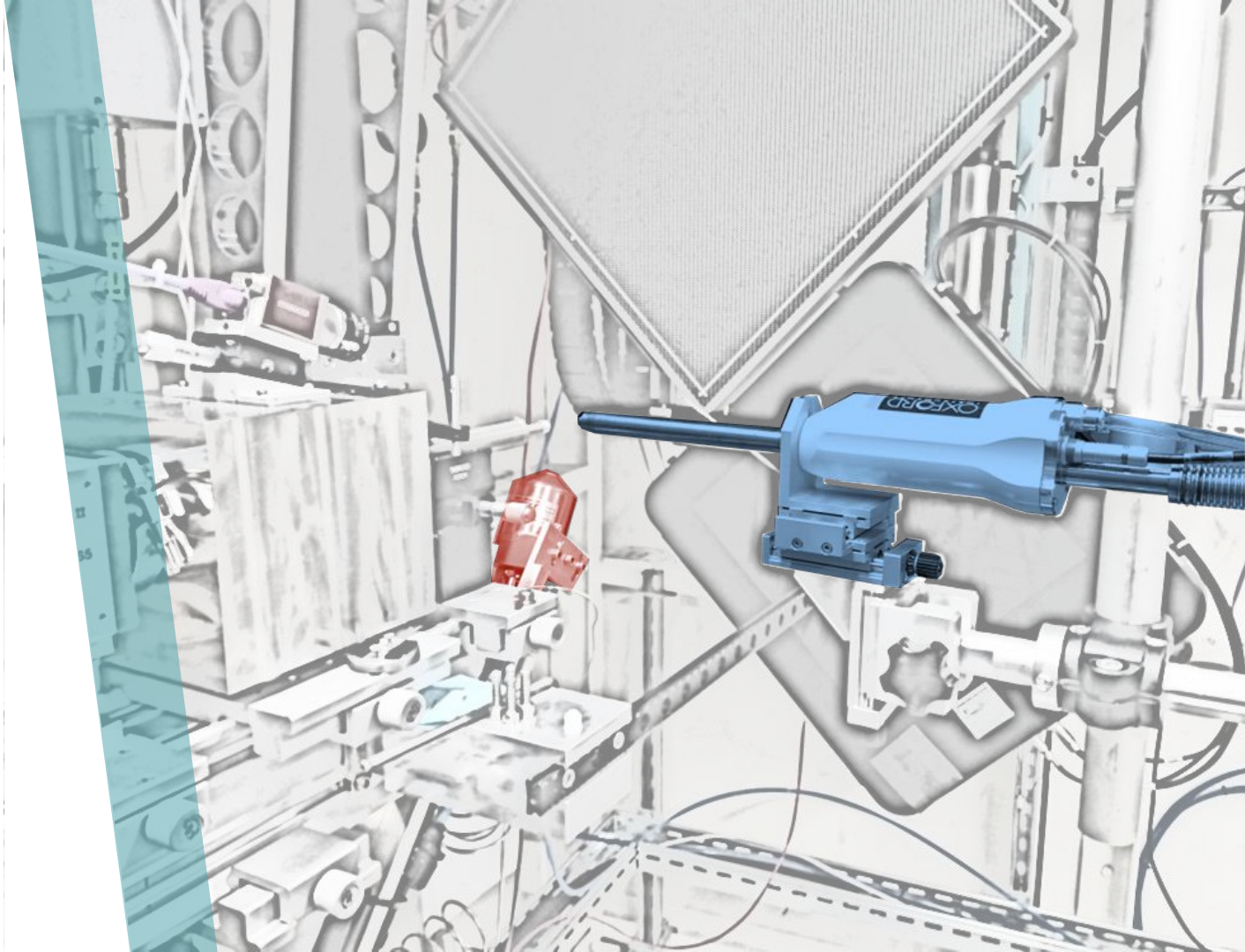


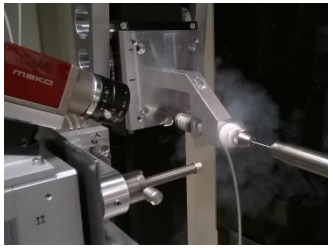
HIGH T AND LOW T



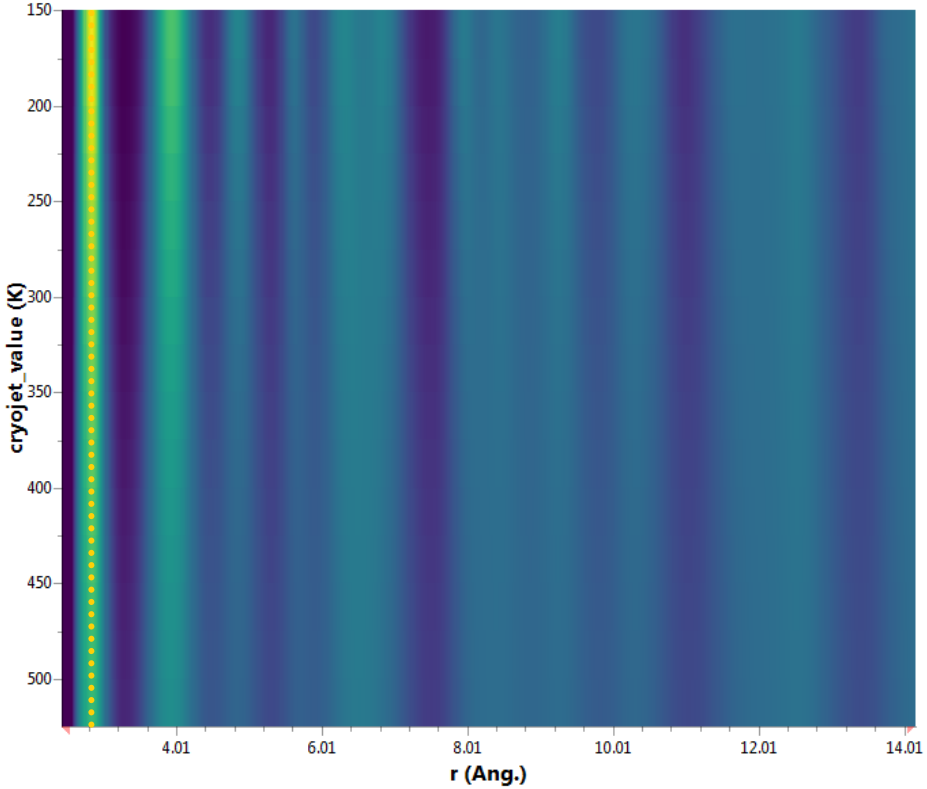
HIGH T AND LOW T

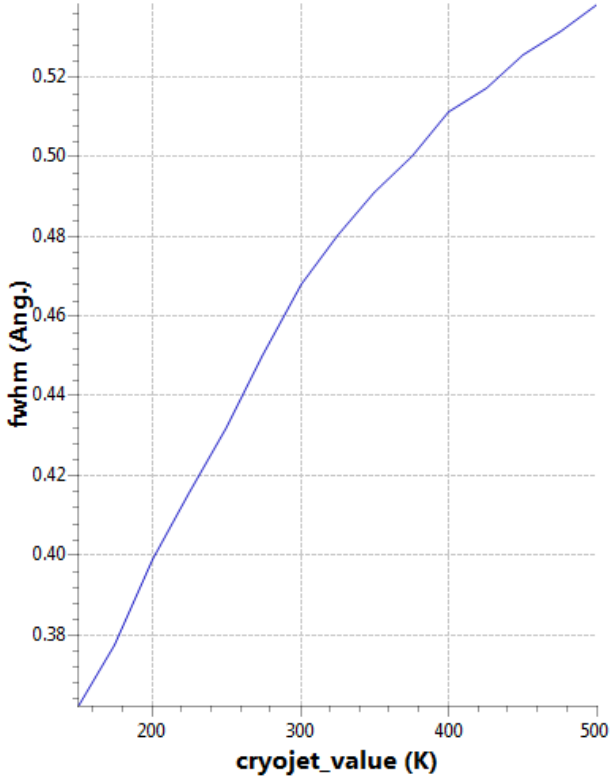


LOW T PEROVSKITE DYNAMICS



- Variable temperature data collection





IN-SITU BATTERY CYCLING

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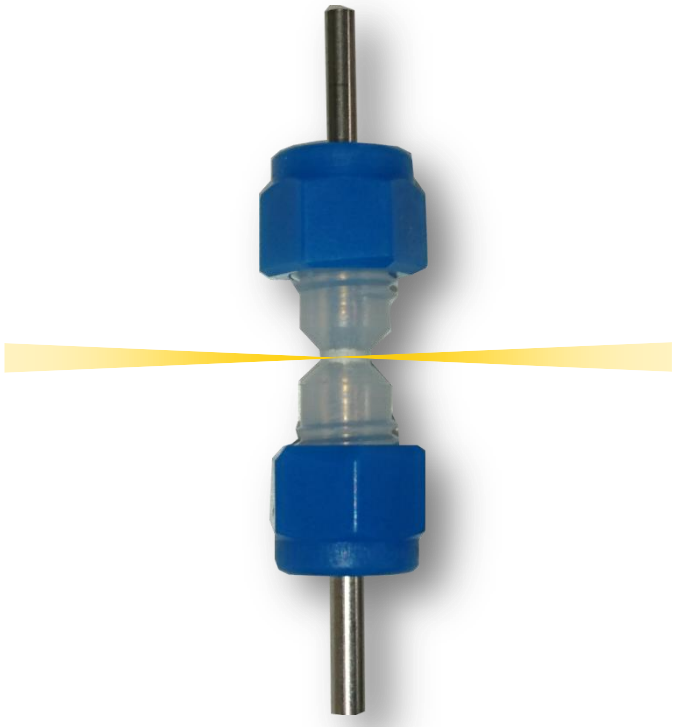

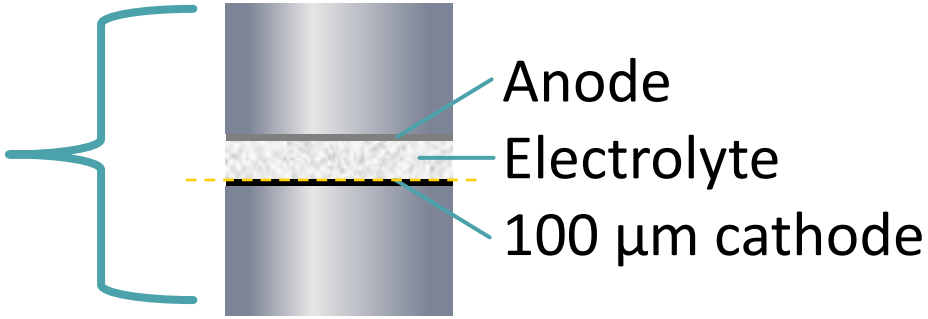

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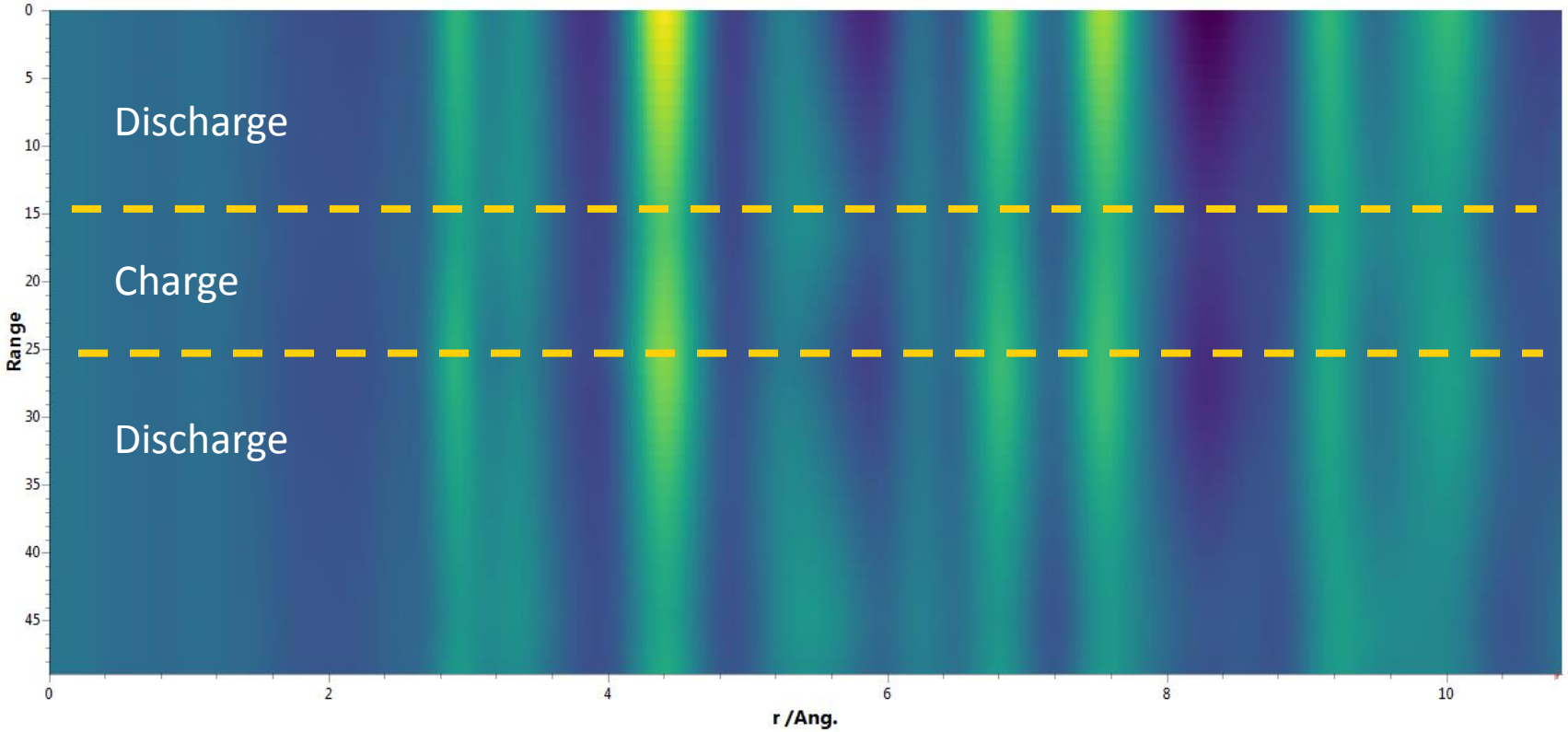
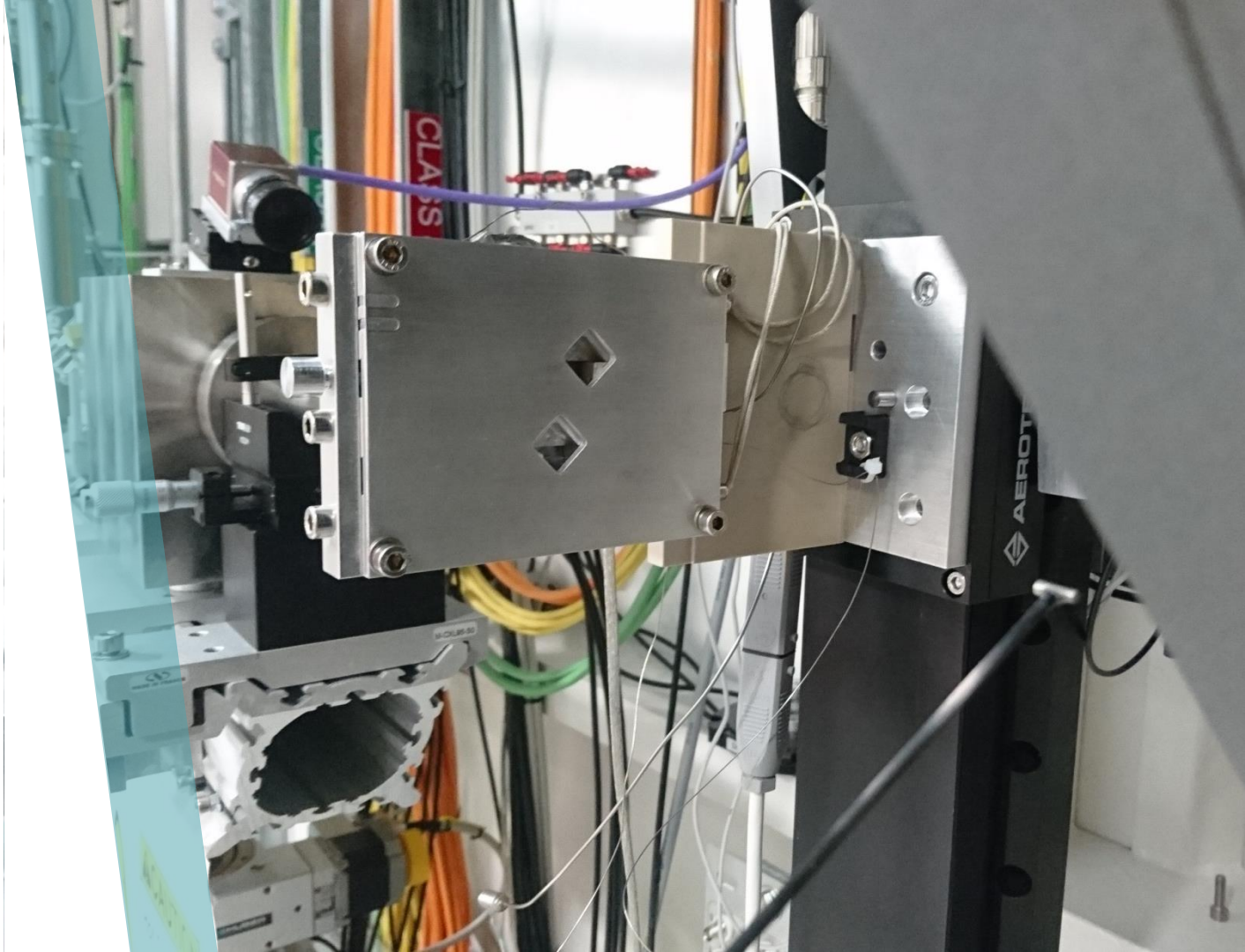
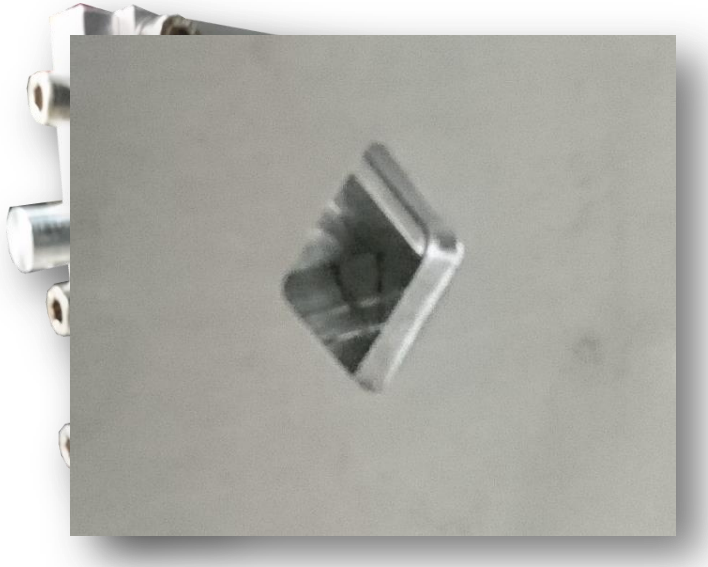


HYDRO-THERMAL CELL



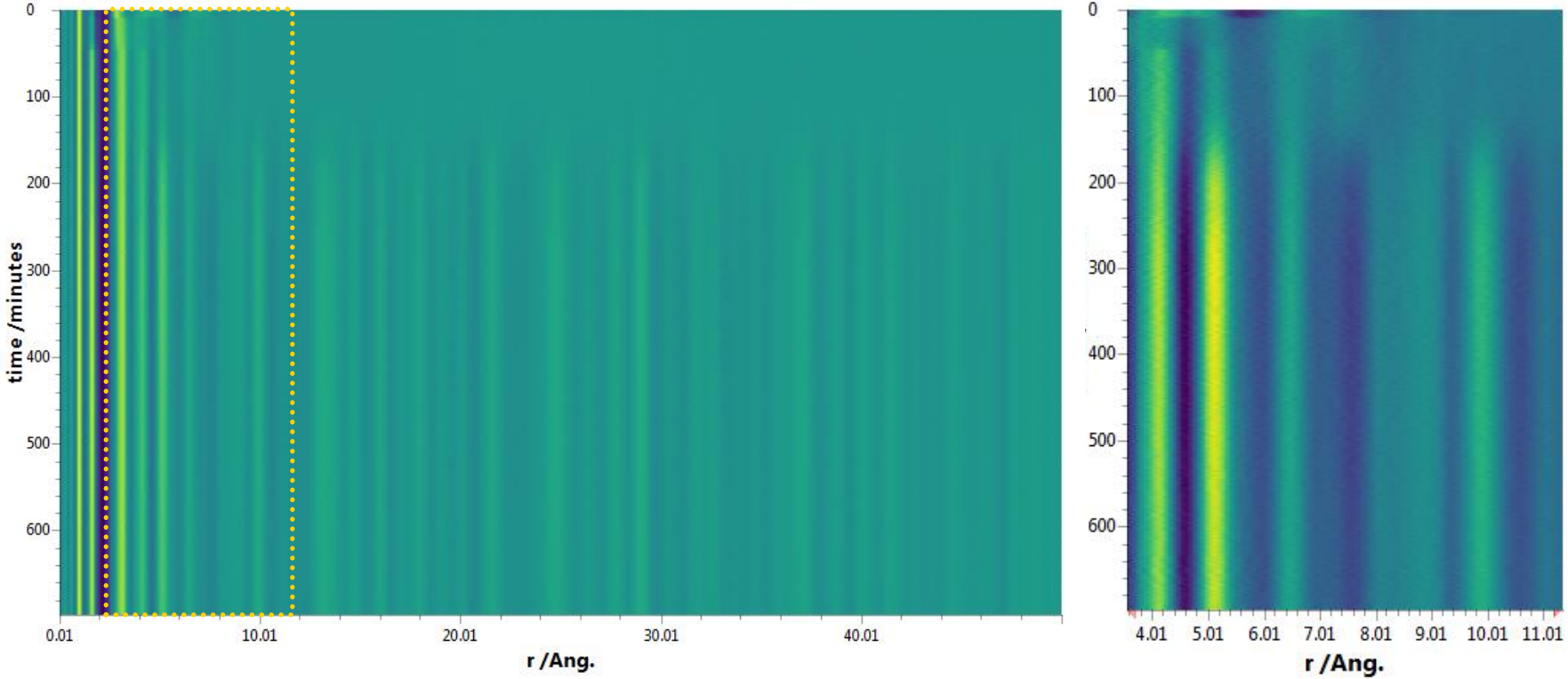
IN-SITU HYDROTHERMAL SYNTHESIS

- Room temperature to 200°C
- Zero background from the heated cell
  - 2.5 mm OD fused quartz capillary



IN-SITU HYDROTHERMAL SYNTHESIS

- Formation of mesoporous aluminosilicate



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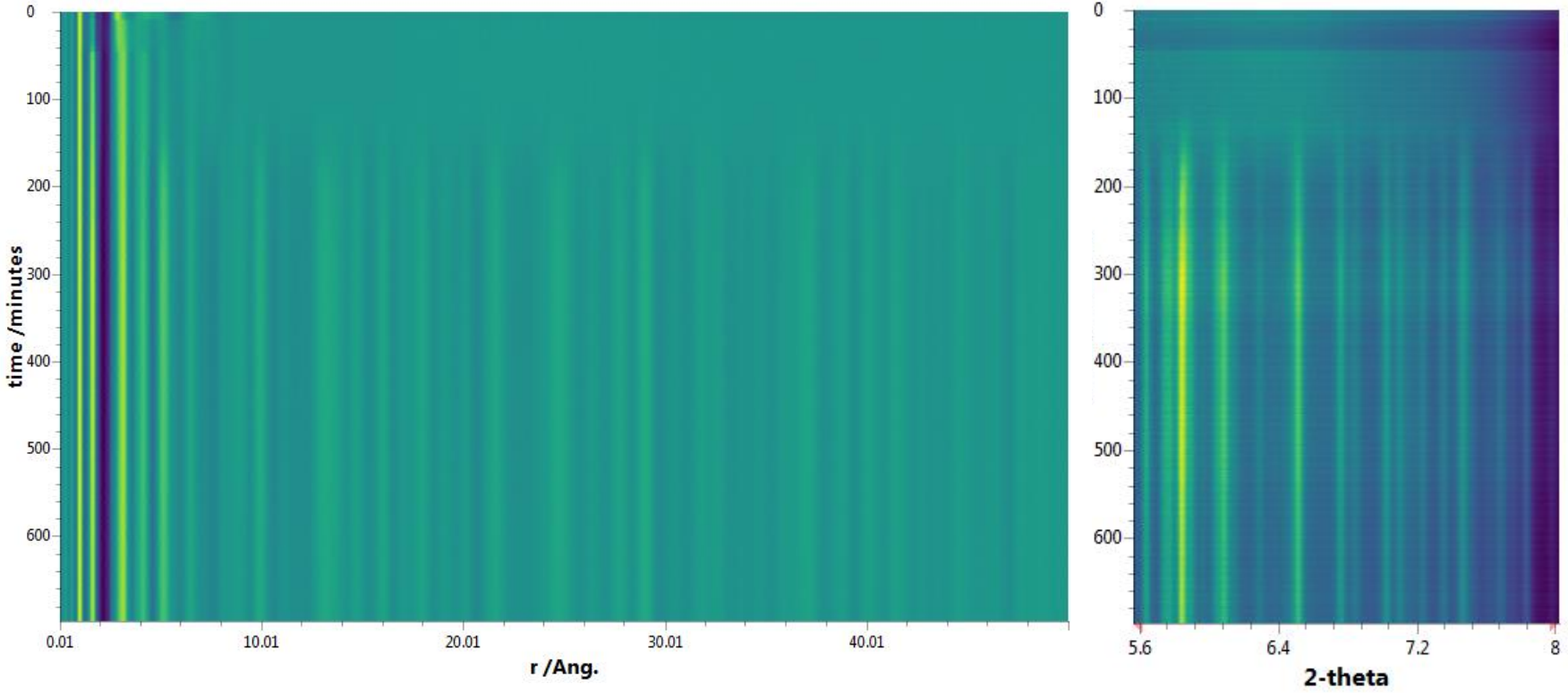

AUTOMATION: NEXT STEPS

- Integrate database
  - Move towards remote beamline operation
- Improve robustness of PDF processing
  - Monte-Carlo calculations of corrections
- More auto-processing
  - Data quality checks
  - Linking to PDFgui, RMCProfile, TOPAS etc…

THANK YOU FOR YOUR ATTENTION
QUESTIONS?

Next XPDF proposal deadline: 1st October 2017

E-mail: xpdf@diamond.ac.uk

: @xpdfdls