



DECTRIS

Next Generation X-Ray Detectors

DECTRIS Solid State Position Sensitive Detectors in Synchrotron Applications

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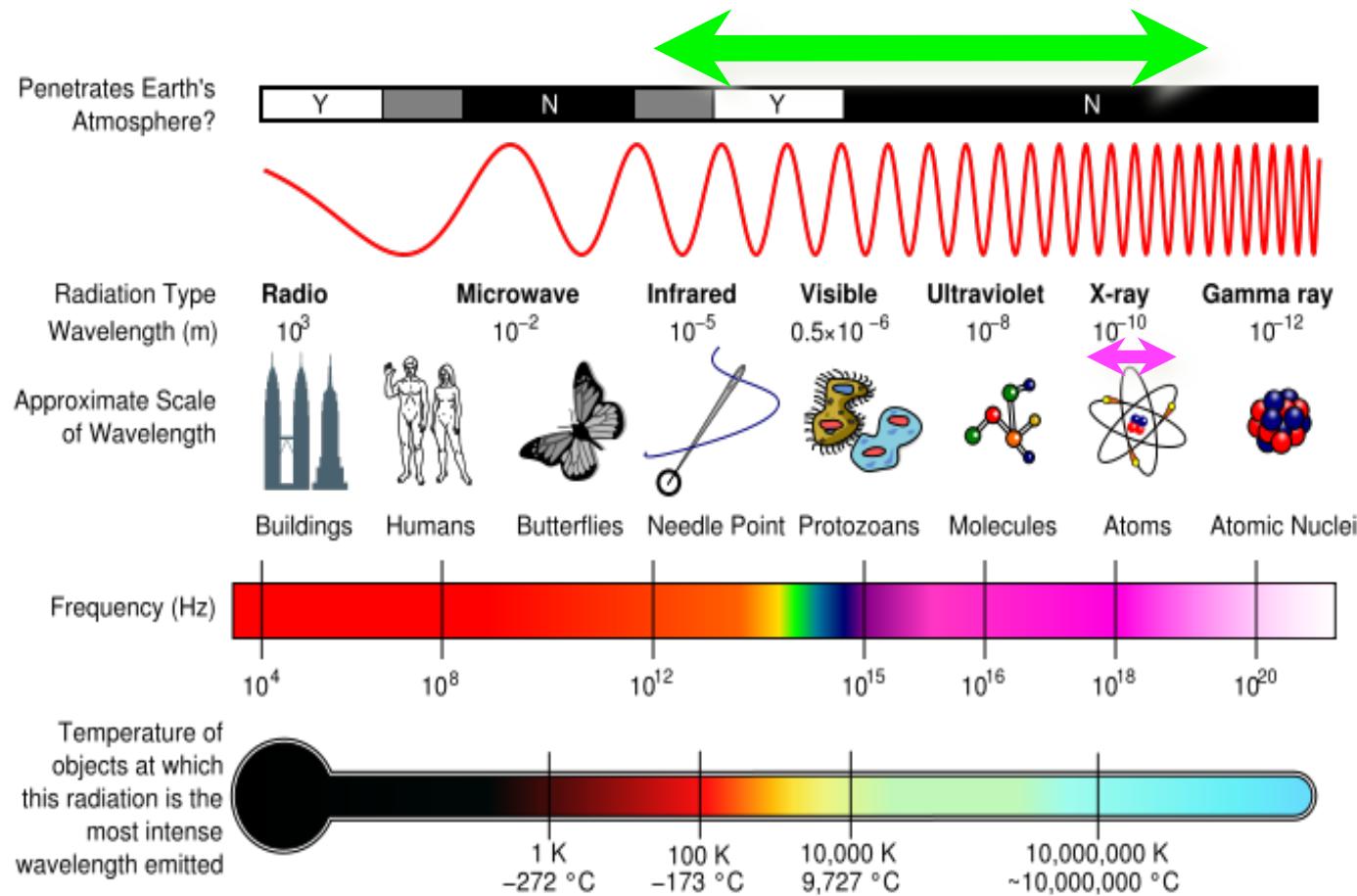
www.dectris.com

CONTENT

SYNCHROTRON RADIATION
EXPERIMENTAL REQUIREMENTS
ADVANTAGE OF COUNTING SSDE
APPLICATIONS
SUMMARY



Energy Range

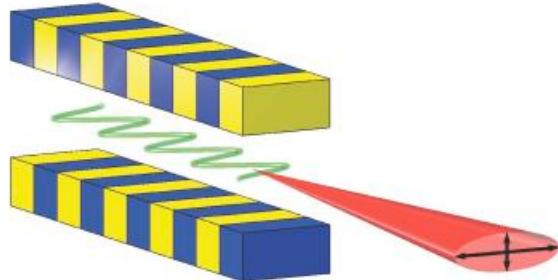


Flux and Brightness

Flux: number of photons / sec

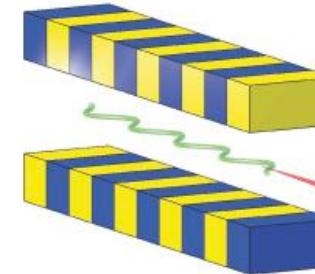
Brightness: $\frac{\text{Number of photons}}{\text{sec} \cdot \text{mrad}^2 \cdot \text{mm}^2 \cdot 0.1\% \text{ bw}}$

(b) Wiggler



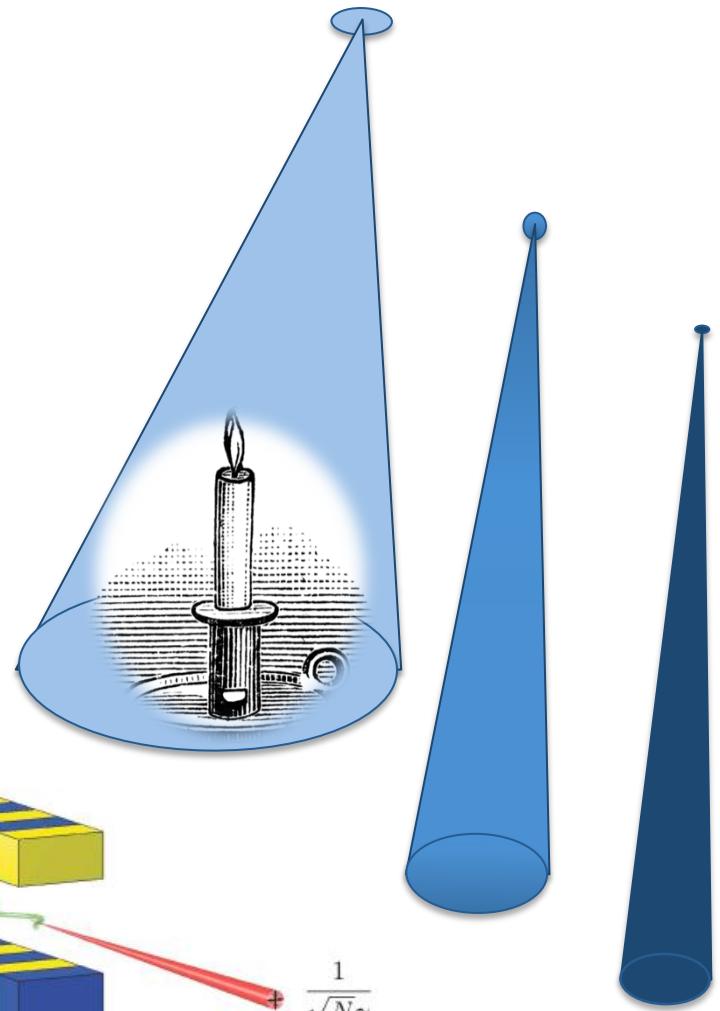
$$\frac{K}{\gamma}$$

(a) Undulator



(b) Wiggler

$$\frac{1}{\sqrt{N}\gamma}$$



Why do users come to synchrotrons ?

	Rotating anode ^a	Bending Magnet ^h	Undulator ^b
• Diode signal ^c	0.060 µA	21.2 µA	84.8 µA
• Flux	$1.4 \cdot 10^9$	$5 \cdot 10^{11}$	$2 \cdot 10^{12}$
• Focal spot size ^{d,e}	$0.3 \times 0.3 \text{ mm}^2$	$0.085 \times 0.045 \text{ mm}^2$	$0.085 \times 0.008 \text{ mm}^2$
• Divergence ^{d,e}	$2.37 \times 2.57 \text{ mrad}^2$	$2.0 \times 0.23 \text{ mrad}^2$	$0.42 \times 0.06 \text{ mrad}^2$
• Bandwidth ^{f,g}	$0.18 \cdot 10^{-3}$	$0.14 \cdot 10^{-3}$	$0.16 \cdot 10^{-3}$
• Brightness	$1.4 \cdot 10^{10}$	$2 \cdot 10^{15}$	$7.2 \cdot 10^{17}$
• Flux ratio	I	350	1420
• Brightness Ratio	I	$1.4 \cdot 10^5$	$5 \cdot 10^7$

a Nonius FR 591 (50 KV, 100 mA, 5 KW, 6000 rpm, slit size: 0.75 mm² and 0.55 mm²)

b SLS UI9 at 8.048 keV and 400 mA

c Sintef 12 µm PIN diode with 20 µm Al

d MSC Blue-3 Optics configuration (Yang et al., Acta Cryst. (1999), D55, 1681 - 1689)

e for focus at the High Resolution Diffractometer

f A.Thompson, CCP4 Study Weekend, 1999

g Si(111) monochromator

h SLS X06DA

Time Structure

Synchrotron sources are stroboscopic sources with a well defined time structure:

- circumference
- RF frequency and bunch filling pattern

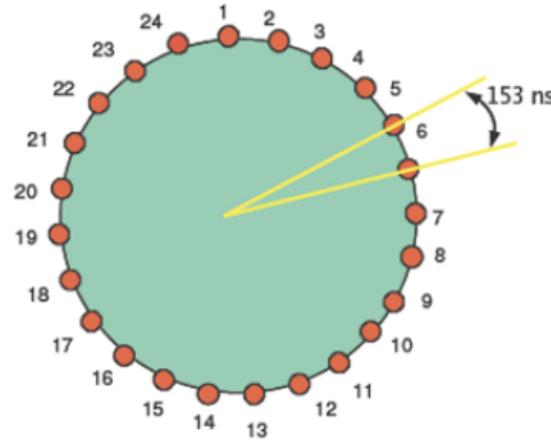


Figure 1. The APS storage ring configured in 24-bunch mode.

Source	Bunch Spacing	Bunch Width
ALS ^a	2 ns	65 ps
ALS ^b	328 ns	65 ps
APS ^c	153 ns	20 ps
CHESS ^d	280 ns	5-bunch train
ESRF ^e	2.82 ns	20 ps (RMS)
ESRF ^f	176 ns	48 ps (RMS)
EXFEL ^g	200 ns	100 fs
NSLS ^h	18.9 ns	290 ps (2σ)
NSLS ⁱ	94.5 ns	290 ps (2σ)
NSLS ^j	567.2 ns	290 ps (2σ)
SLS ^k	1.88 ns	43 ps (RMS)
SPring-8 ^l	23.6 ns	70 ps (FWHM)
SPring-8 ^m	145.5 ns	11 bunch train
SPring-8 ⁿ	342.1 ns	70 ps (FWHM)
SLS low α	1.88 ns	8 ps (RMS)
SLS Femto	1 ms	150 fs

- Ideally suited for time resolved experiments
- More challenging for counting detectors

Current detectors

Imaging plates

- + large area, cheap
- + good resolution ($\sim 50 \mu\text{m}$)
- calibration 1 % (ghosting, drifts, deexcitation)
- very slow readout (minutes)

• CCDs

- + large areas
- lower read-noise and dark current
- fast readout (0.1 - 10 s)
- PSF and DQE limited by light scattering and fibre optic taper
- limited dynamic range (10^4)
- low high energy efficiency

• Energy dispersive detectors

- low count rate capability
- small effective area and bulky
- expensive

Detector	Energy range (keV)	$\Delta E/E$ at 5.9 keV (%)	Dead time/event (μs)	Maximum count rate (s^{-1})
Gas ionization (current mode)	0.2–50	n/a	n/a	10^{11}a
Gas proportional	0.2–50	15	0.2	10^6
Multiwire and microstrip proportional	3–50	20	0.2	$10^6/\text{mm}^2$
Scintillation [NaI(Tl)]	3–10,000	40	0.25	2×10^6
Energy-resolving semiconductor	1–10,000	2.1	0.5–30	5×10^5
Surface-barrier (current mode)	0.1–20	n/a	n/a	10^8
Avalanche photodiode	0.1–50	20	0.001	10^8
CCD	0.1–70	n/a	n/a	n/a
Superconducting	0.1–4	< 0.5	100	5×10^3
Image plate	4–80	n/a	n/a	n/a

^a Maximum count rate is limited by space-charge effects to around 10^{11} photons/s per cm^3 .

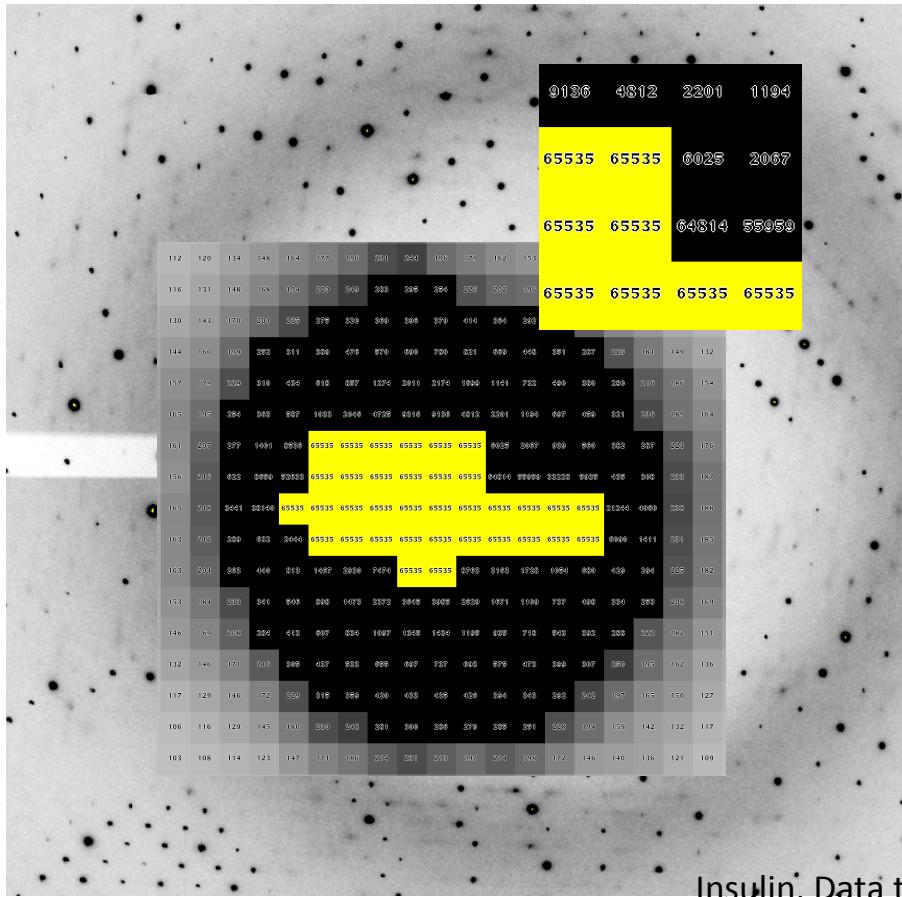
- Synchrotron experiments are almost always detector limited and future XFEL and ERC experiments will be even more, unless new dedicated detectors will be developed
- Innovative experiments cannot be optimally implemented with current detector technologies

Dynamic Range	$> 10^6$	SAXS, PX, CDI, Diffuse Scattering, Surface Diffraction
Count rate	$> 1 \text{ MHz}$ $> 10 \text{ MHz}$	PX, CDI Material science
Single Photon Counting		SAXS, PX, CDI, Diffuse Scattering, Surface Diffraction
Pixel Size	$< 50 \mu\text{m}$ $< 10 \mu\text{m}$	PX micro diffraction, CDI, tomography
Point Spread Function	$< 1\% @ 1.1 \text{ pix}$ $< 0.01\% @ 2\text{pix}$	PX, diffuse scattering, CDI
Frame rates	100 Hz full frame 25 kHz subframe	PX, diffuse scattering, surface diffraction XPCS, sSAXS, CDI, tomography
Triggering/Gating	$\sim 1 \text{ ns}$	Time resolved diffraction, XAS, XFS
Energy Resolution	window $\sim 300 \text{ eV}$ analog spectrum	Diffraction Experiments, XAS X-ray absorption spectroscopies
Energy range	5 - 20 keV 5 - 100 keV	PX, CDI, SAXS Material Science, powder diffraction
Engineering	seamless vacuum compatible	PX, CDI, tomography SAXS, nano-micro-diffraction, surface diff
Radiation Hardness	$> 100\text{MRad}$	Diffraction (HP), CDI, SAXS

Dynamic Range and Point Spread Function

CCD

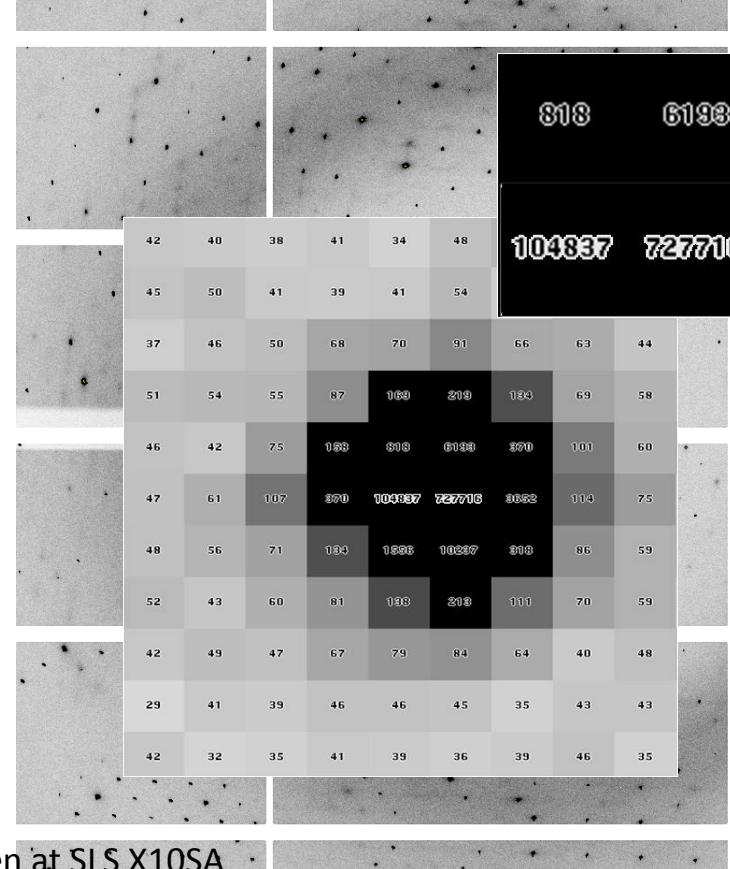
16 bit (65 535 ADU)



Insulin, Data taken at SLS X10SA

PILATUS

20 bit (1 048 575 counts)

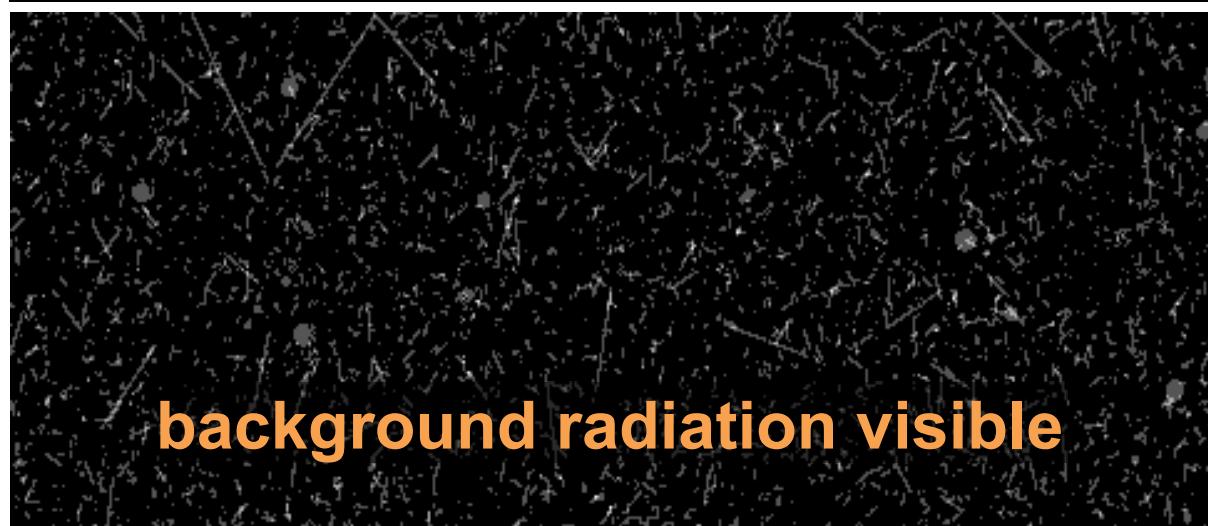


Hybrid pixel: noise free detection

100 ms

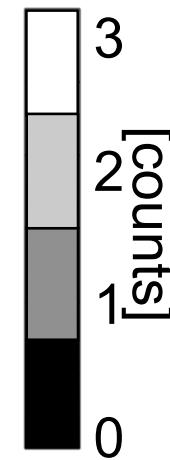


1 hour



100K detector

No readout-noise
No dark-current.

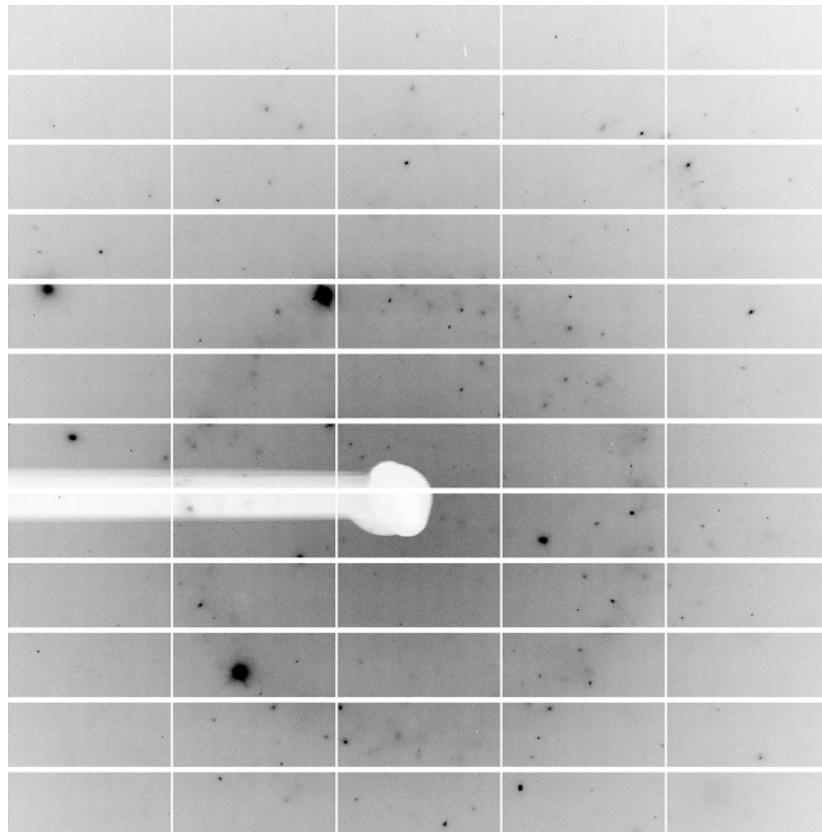


Energy Discrimination

Sample: Icosahedral Al-Cu-Fe quasicrystal
Weber *et al.*, *J. Appl. Cryst.*, **41** (2008)

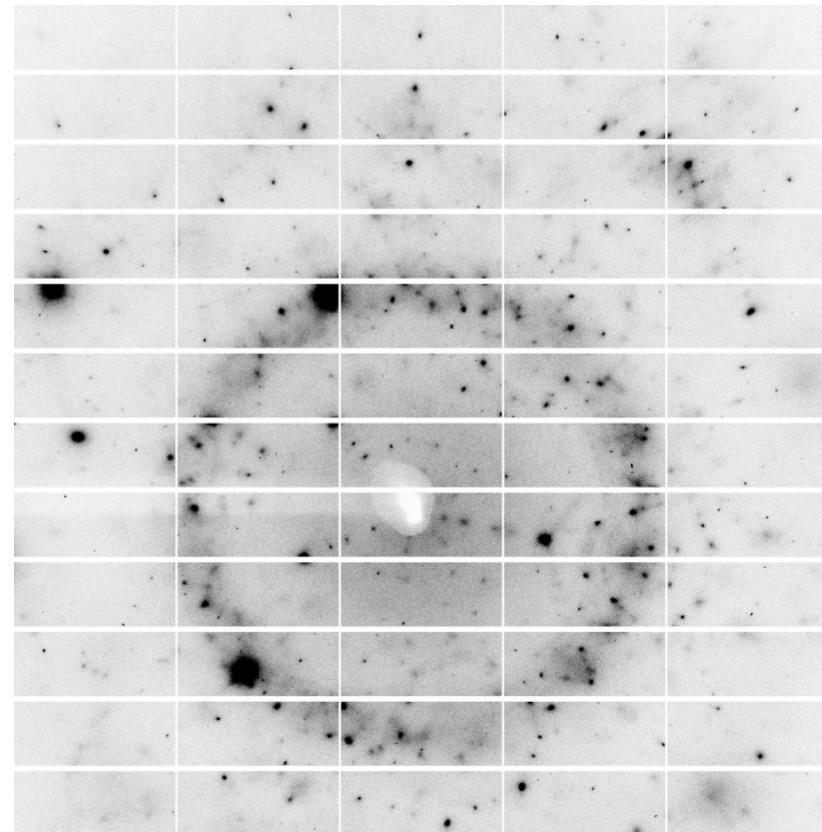
Energy threshold of 8 keV

Fluorescence suppression of:
Al (1.5 keV), Fe (6.4 keV)

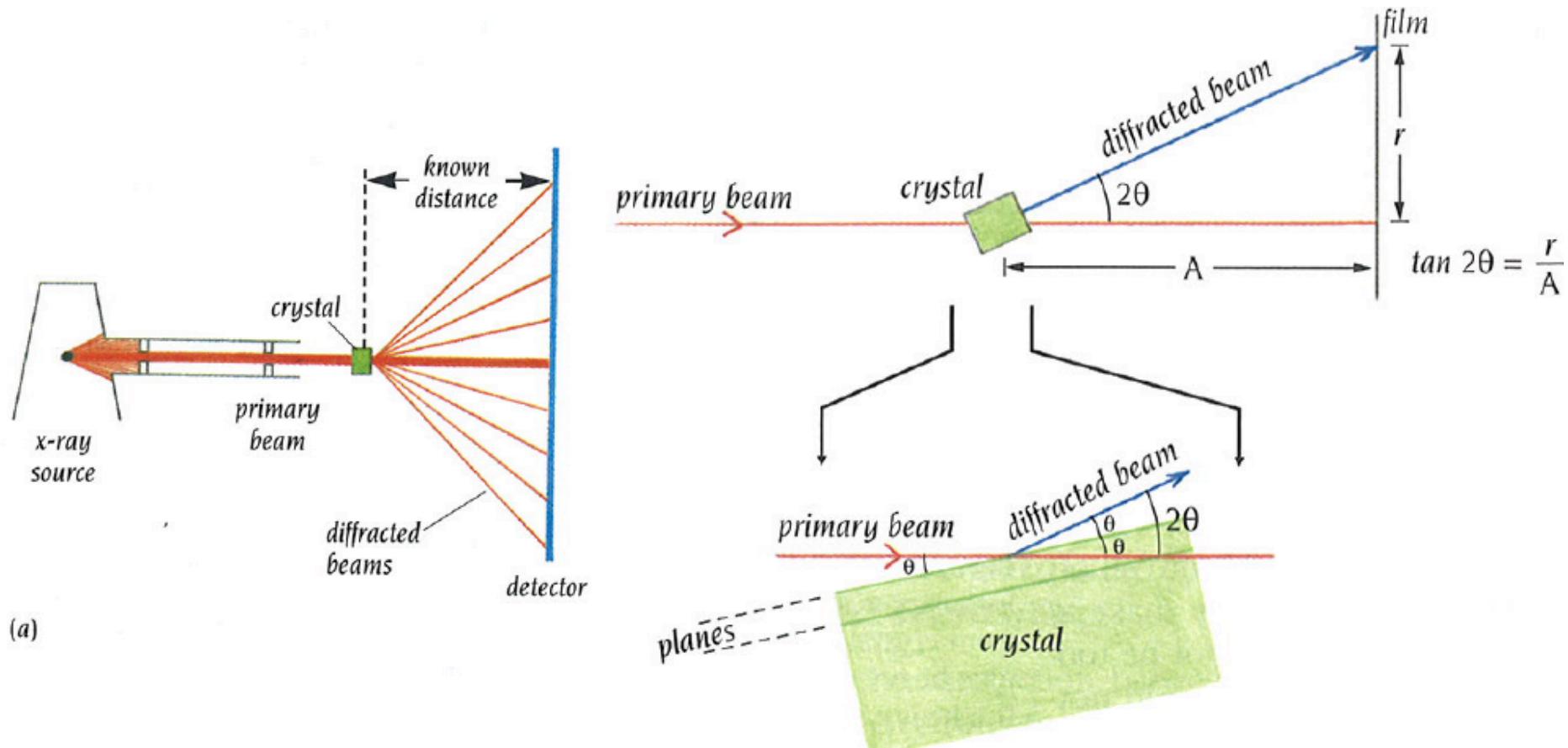


Energy threshold of 10 keV

Fluorescence suppression of:
Al (1.5 keV), Fe (6.4 keV), Cu (8 keV)

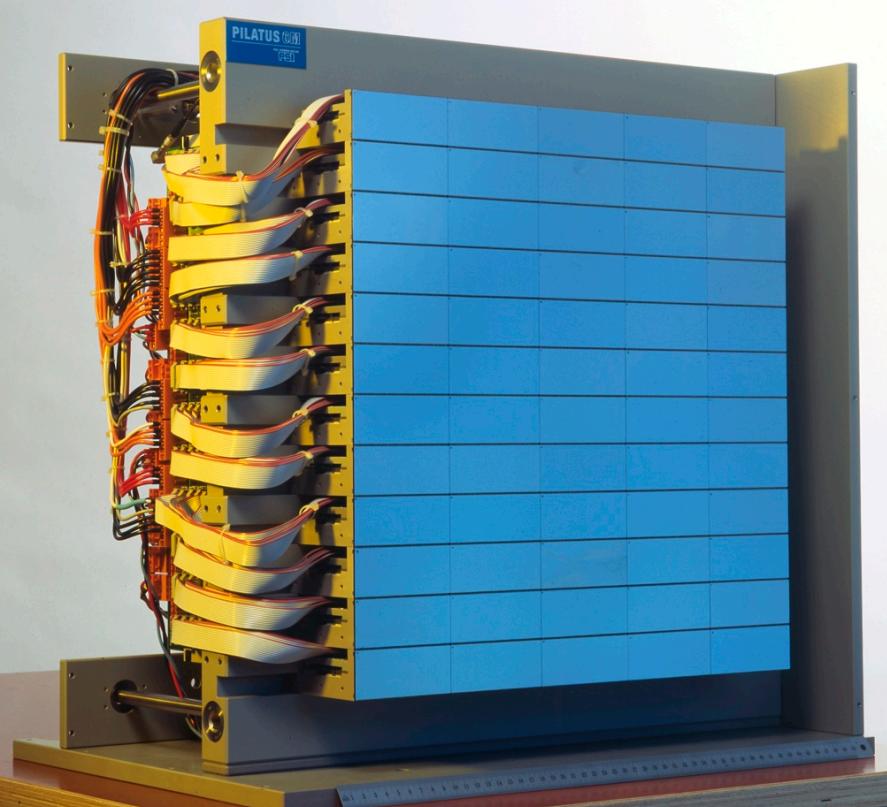


Diffraction Experiment



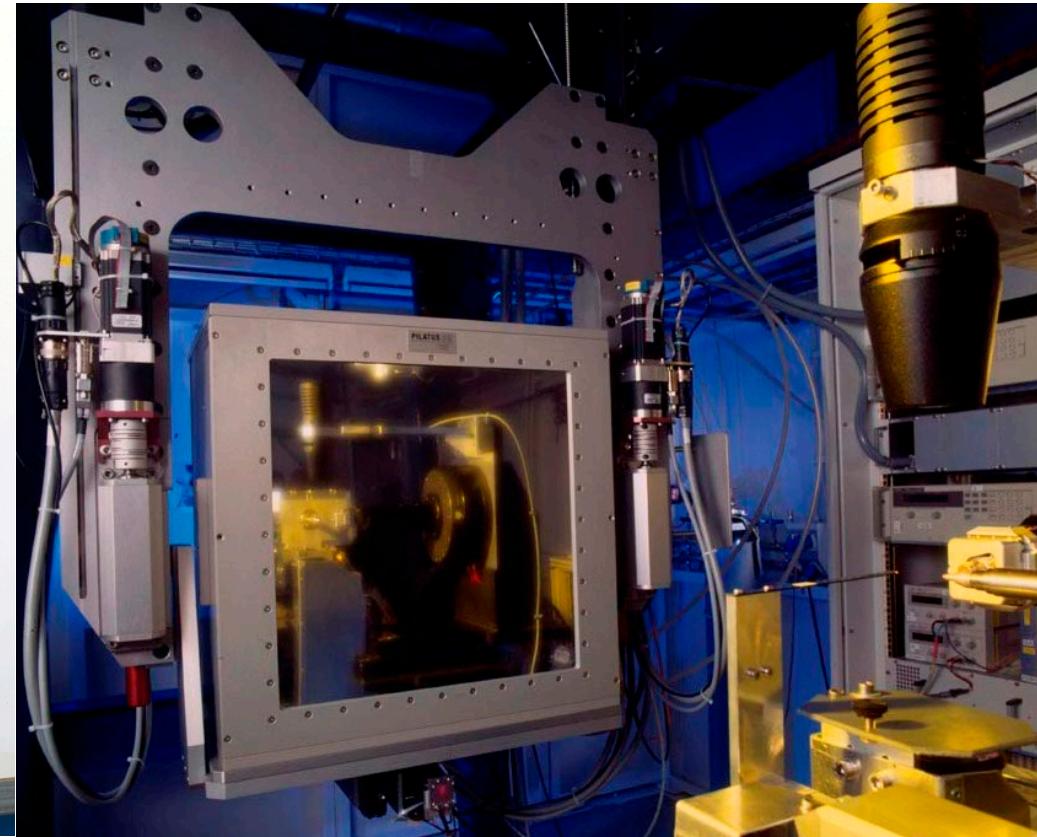
PILATUS 6M Pixel Detector

6M at Detector Lab



Pixels: 2' 463 (x) • 2' 527 (y) = 6' 224' 001
Active area: 424 • 435 mm²; dead area: 8.4 %
Frame rate: 12.5 Hz; with 2.3 ms dead time
Noise free counter

6M at SLS beamline X06SA



In operation since June 2007

PILATUS : New Opportunities in MX

- ⇒ Continuous shutter-free data acquisition
- ⇒ elimination of systematic errors
- ⇒ fastest way to collect data (1-2 min/180° to 3 sec/60°)
- ⇒ Fine ϕ -slicing in continuous mode
- ⇒ Possibility to take high redundancy, low exposure data
- ⇒ Simultaneous collection of low and high resolution data
- ⇒ Diffuse scattering experiments
 - T. Weber et al., J. Appl. Cryst. (2008), 41, 1
 - A. Bosak et al., PRL 103, 076403, (2009)
- ⇒ Room temperature data collection with less radiation damage

PILATUS 2M-F 450

The fastest detector for MX

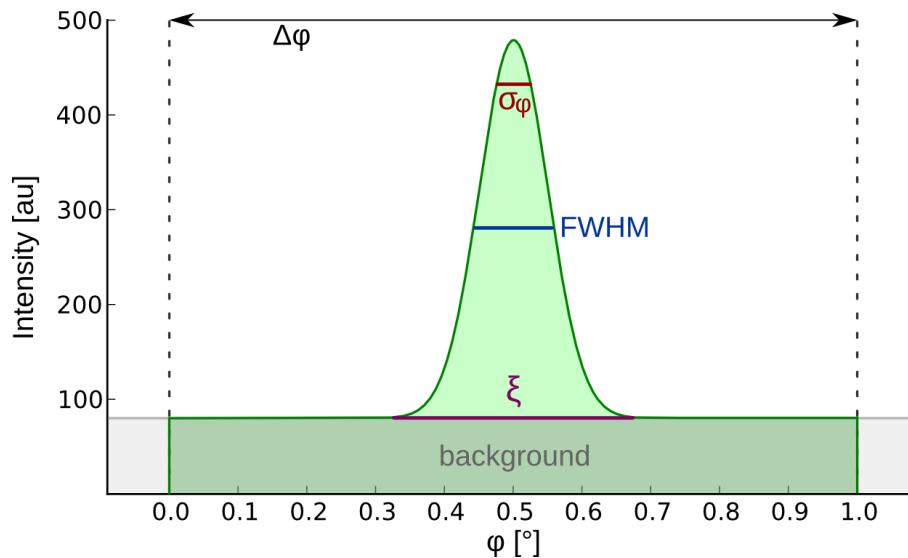
Up to 60 Hz data collection

Example with Insulin: 20 deg/sec, i.e. 0.0167 sec / 0.33° image, 180 images in 3 sec !!!

SUBSET OF INTENSITY DATA WITH SIGNAL/NOISE >= -3.0 AS FUNCTION OF RESOLUTION												
RESOLUTION LIMIT	NUMBER OF REFLECTIONS OBSERVED	NUMBER OF UNIQUE REFLECTIONS	NUMBER OF POSSIBLE REFLECTIONS	COMPLETENESS OF DATA	R-FACTOR observed	R-FACTOR COMPARED expected	I/SIGMA	R-meas	R _{rmeas} -F	Anomal Corr	SigAno	Nano
5.94	1476	395	401	98.5%	1.9%	1.9%	1459	59.18	2.3%	1.4%	43%	1.294
4.23	2635	680	684	99.4%	2.0%	1.9%	2620	60.67	2.3%	1.4%	24%	1.090
3.46	3166	875	897	97.5%	2.2%	2.0%	3126	52.31	2.6%	1.7%	18%	0.938
3.00	3156	1038	1056	98.3%	2.4%	2.4%	3059	38.74	2.9%	2.2%	11%	0.895
2.68	3904	1169	1176	99.4%	3.7%	3.7%	3877	28.08	4.4%	3.9%	4%	0.860
2.45	4696	1339	1341	99.9%	4.7%	4.7%	4675	22.58	5.6%	5.2%	5%	0.849
2.27	5031	1436	1438	99.9%	6.8%	6.8%	5008	16.45	8.1%	7.7%	4%	0.830
2.12	5434	1531	1532	99.9%	8.8%	8.9%	5416	12.81	10.4%	10.5%	-1%	0.774
2.00	5913	1651	1653	99.9%	12.9%	13.4%	5888	8.84	15.2%	17.2%	0%	0.786
total	35411	10114	10178	99.4%	3.2%	3.1%	35128	26.85	3.7%	4.8%	5%	0.867
												4522

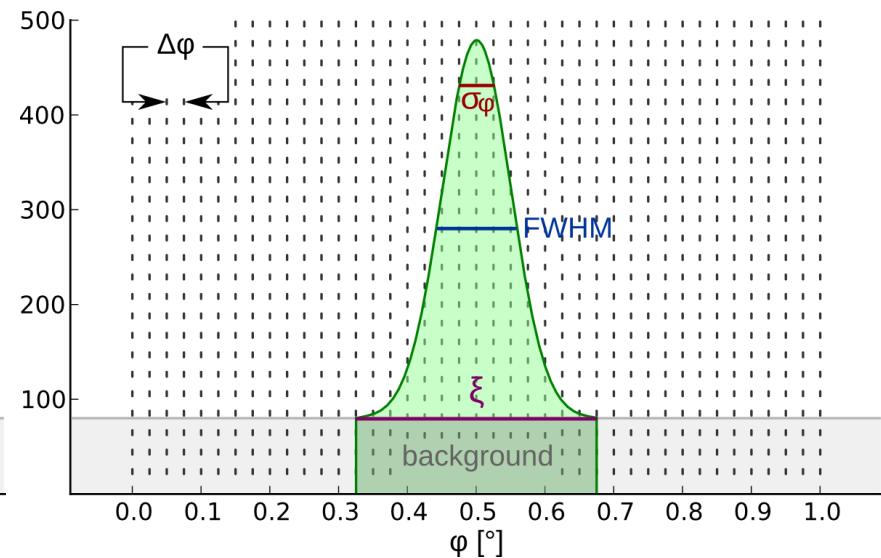
With the same dose, one can collect a complete dataset instead of few test images

Fine φ -slicing and background



Wide φ -slicing

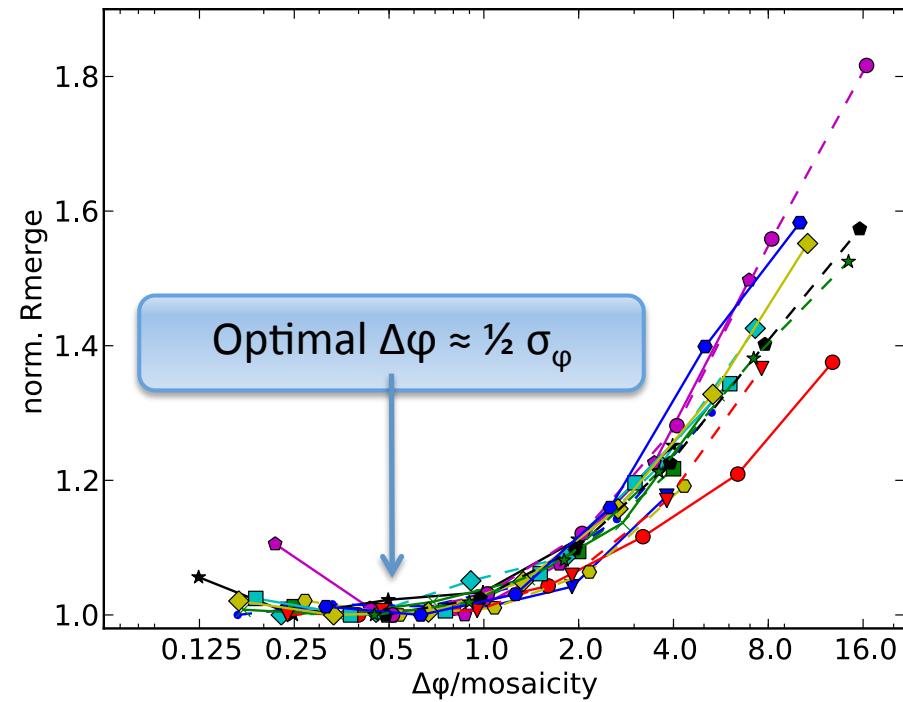
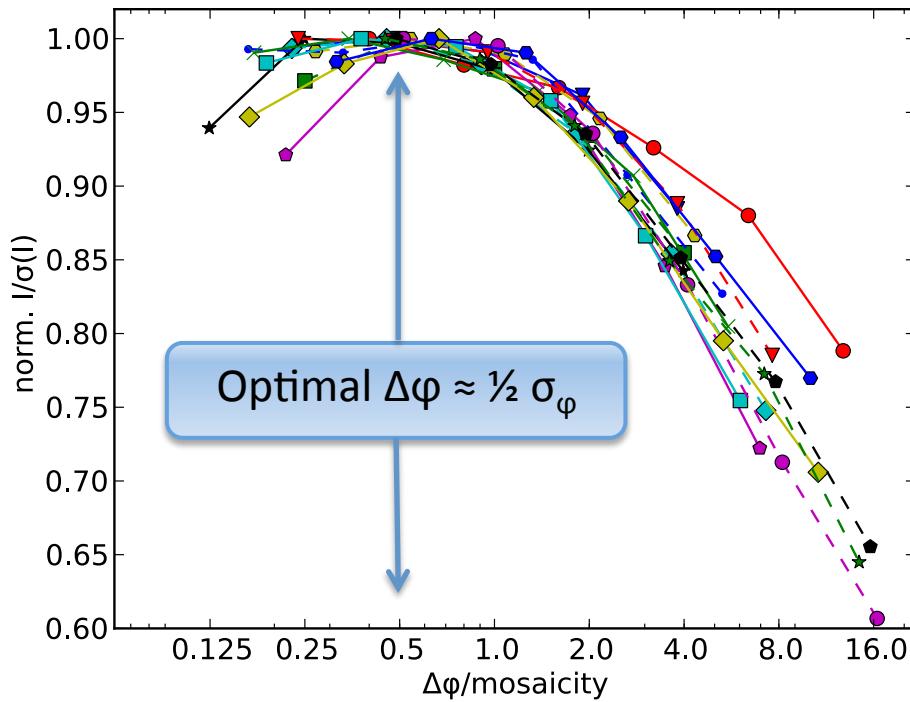
- Large $\Delta\varphi$ ($\Delta\varphi > \xi$)
- Large overlap of reflections and background along φ
- Few images



Fine φ -slicing

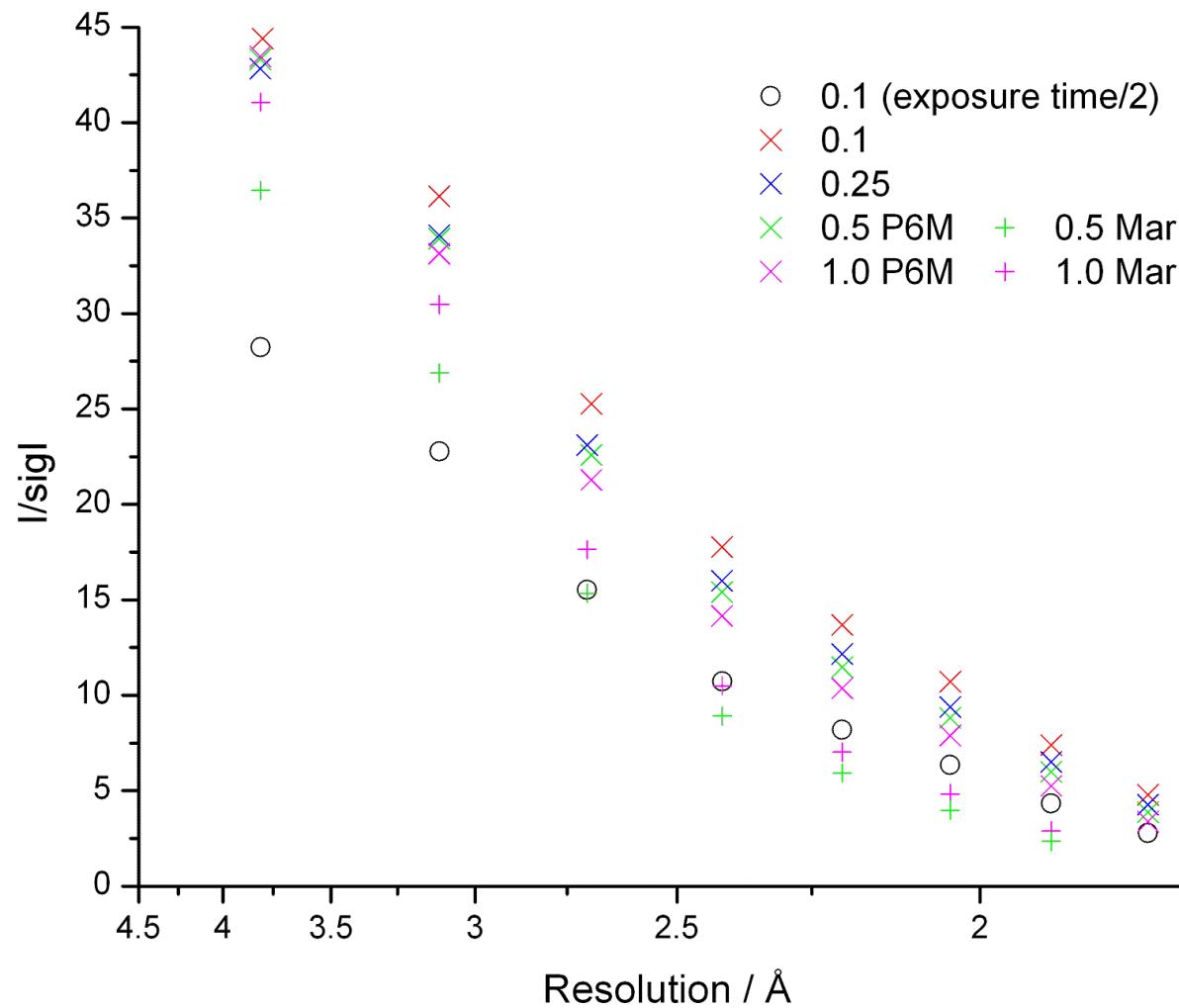
- Small $\Delta\varphi$ ($\Delta\varphi \ll \xi$)
- Minimal overlap of reflections and background along φ
- Many images

Highest shell statistics



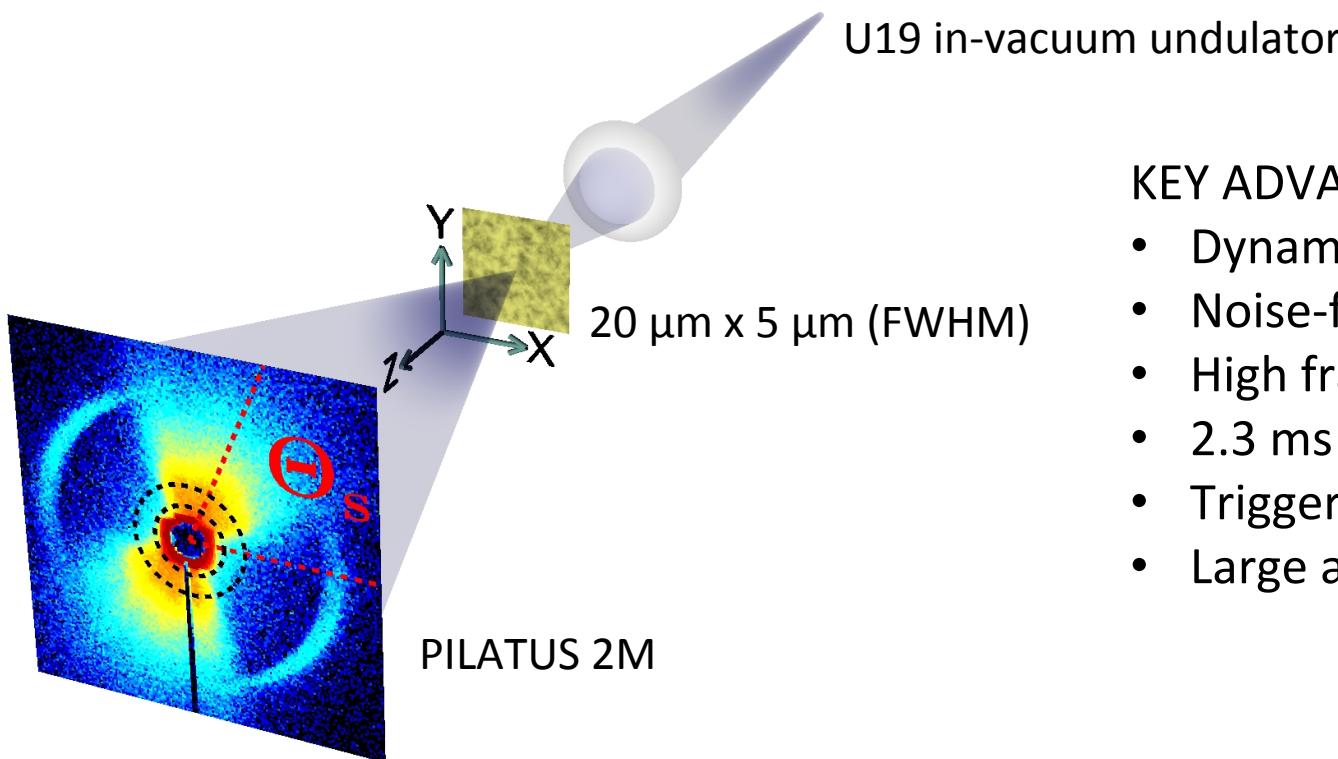
- Best data at $\Delta\varphi = \frac{1}{2}$ mosaicity (σ_φ)
- Highest shell statistics improve substantially
=> fine-slicing extends maximum resolution of data set

Comparison with CCD



Scanning SAXS at SLS X12SA

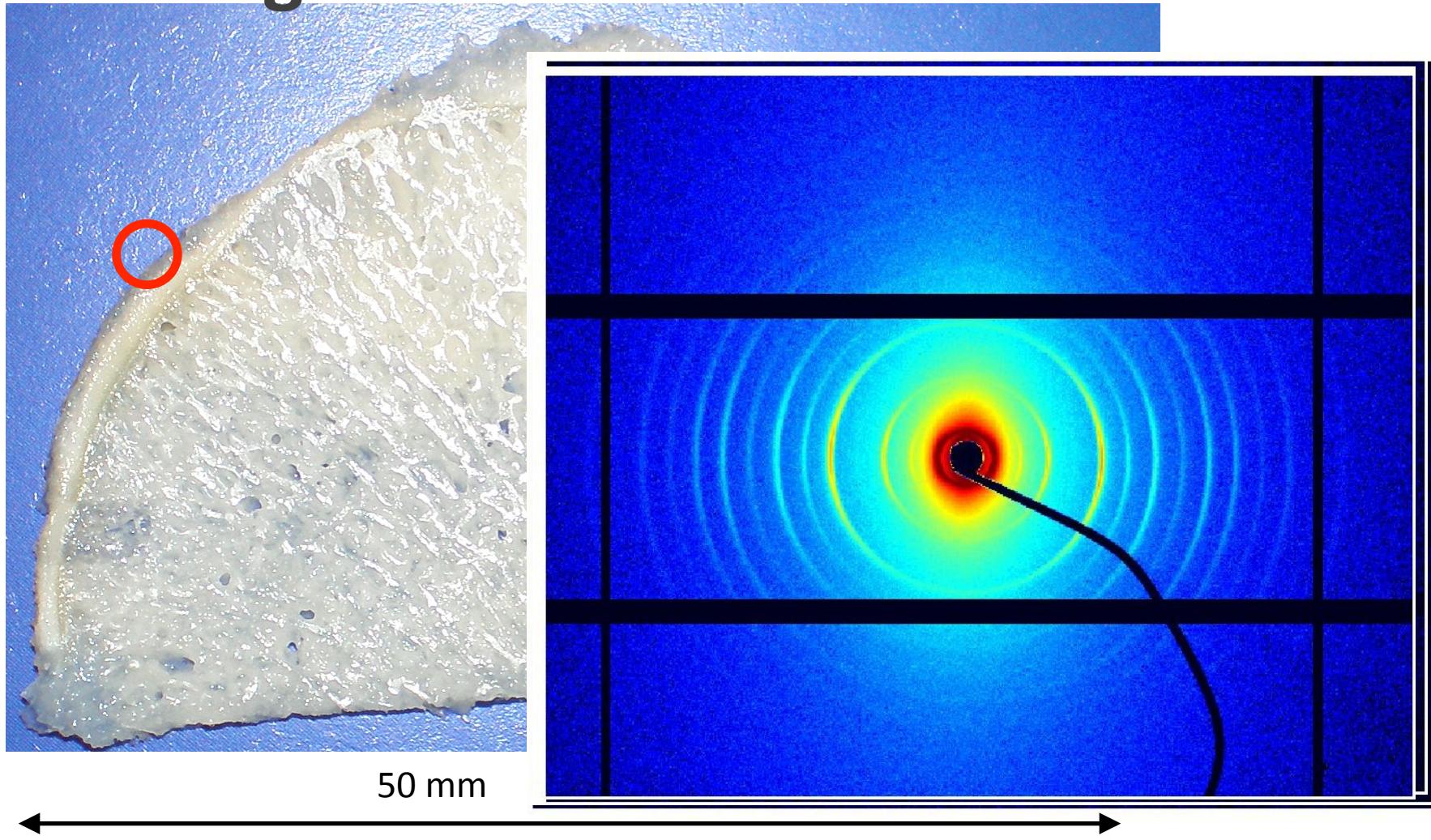
- high brightness
- moderate focus
- fast, high dynamic range, low noise detector



KEY ADVANTAGE

- Dynamic range of 20 bit
- Noise-free counter
- High frame rate
- 2.3 ms dead time
- Triggerable
- Large area

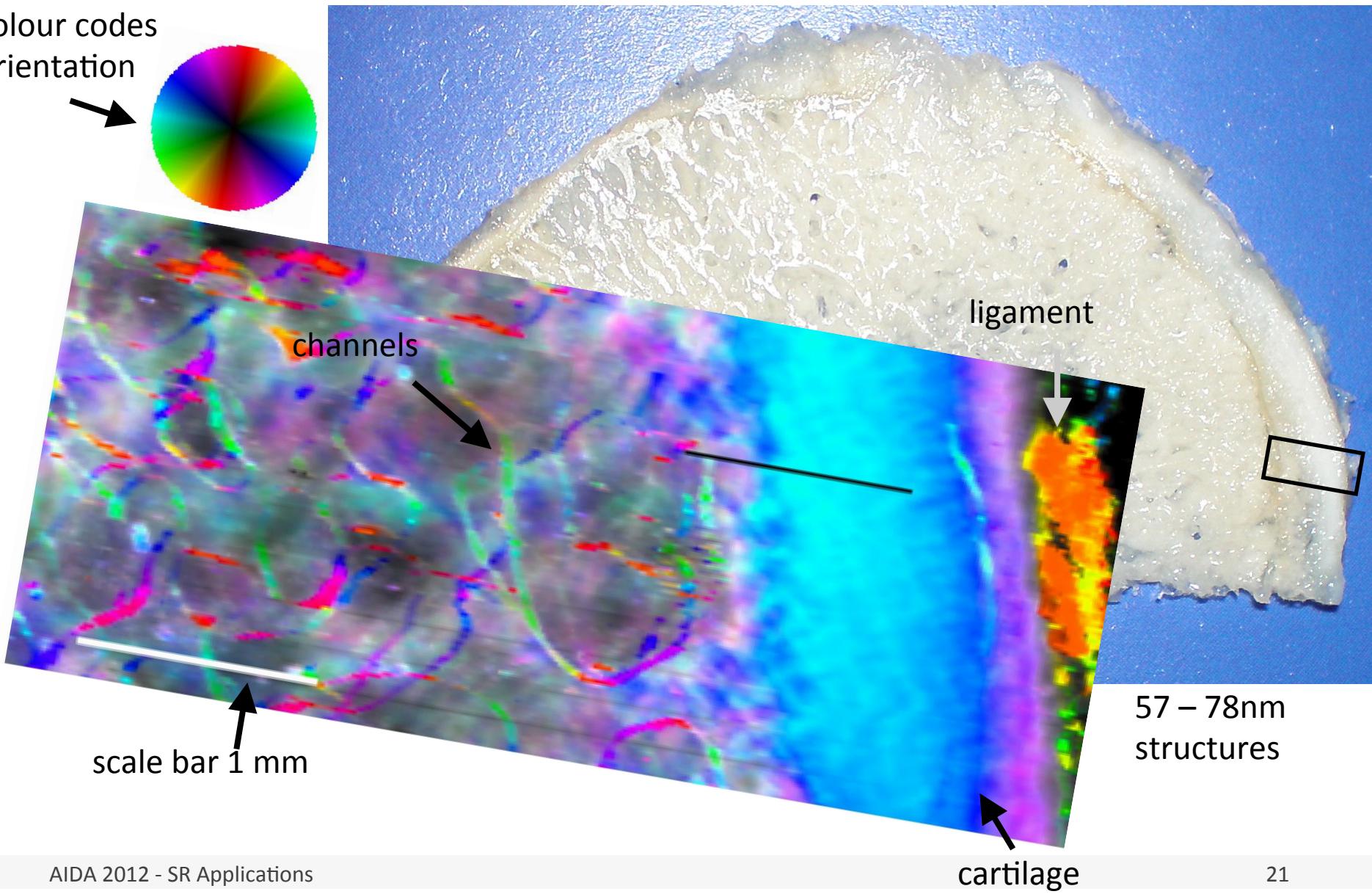
Scanning SAXS of human femoral head



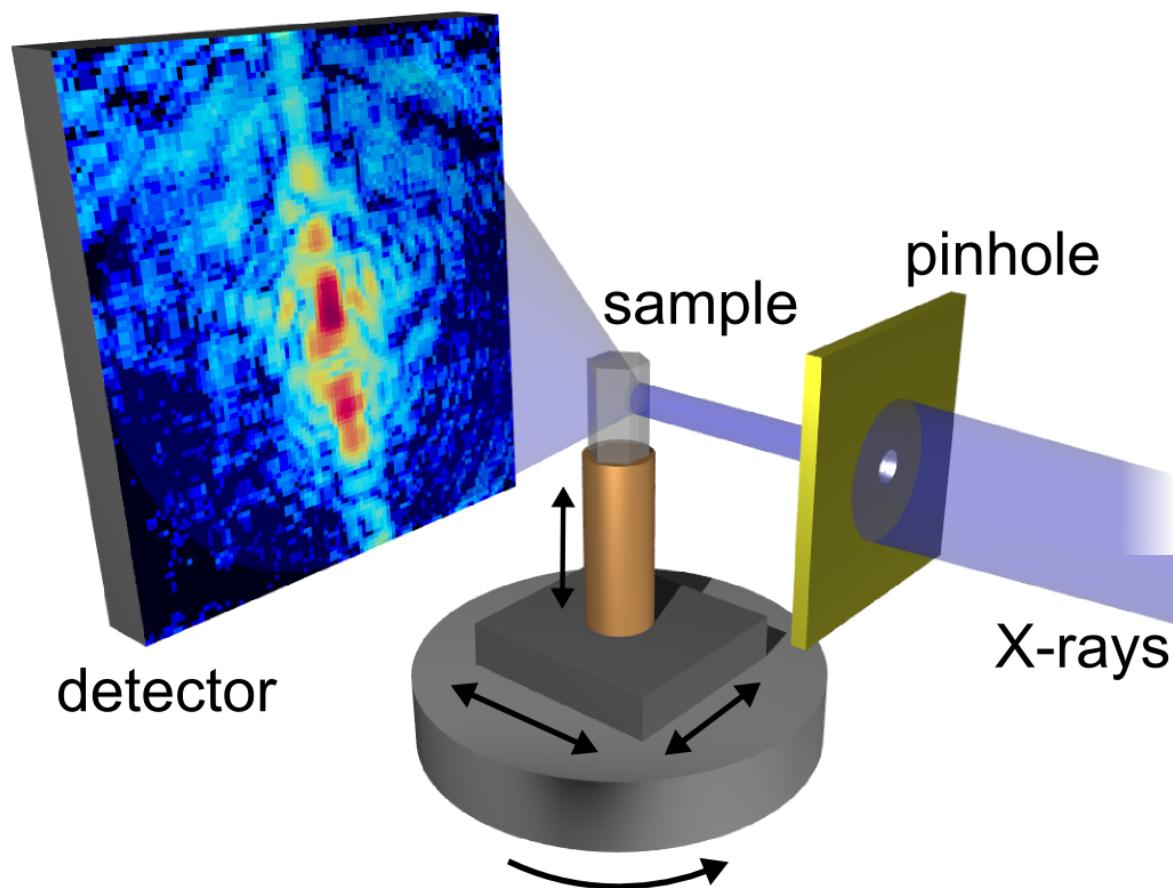
Beam size : 20 micrometer

Human femoral head: area with thick cartilage

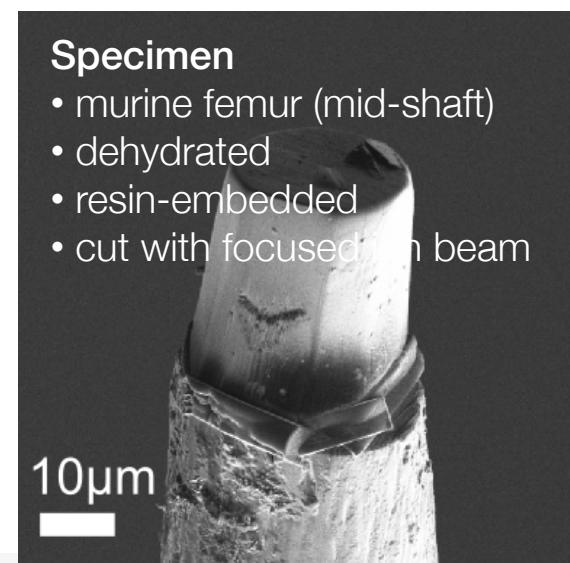
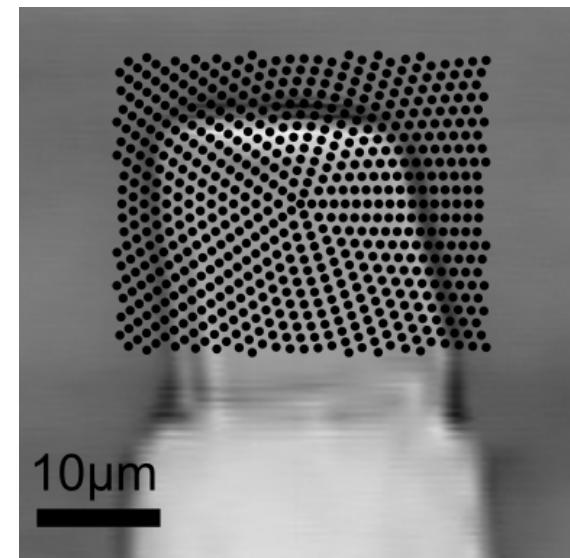
colour codes
orientation



Ptychography and Tomography



704 scan points \times 181 angles ($\pm 90^\circ$)
 $\approx 125'000$ coherent diffraction patterns



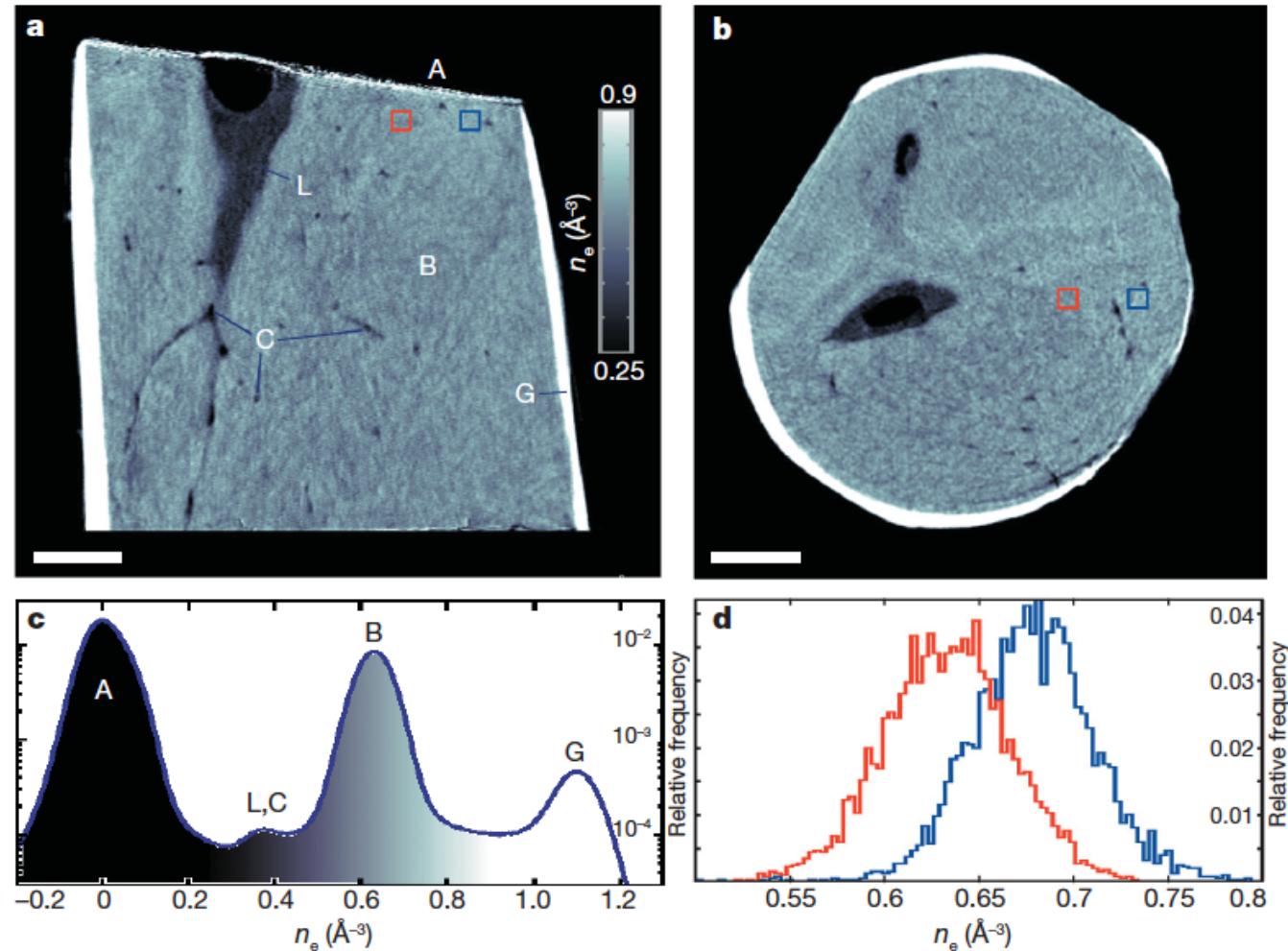
Specimen

- murine femur (mid-shaft)
- dehydrated
- resin-embedded
- cut with focused ion beam

Quantitative Results

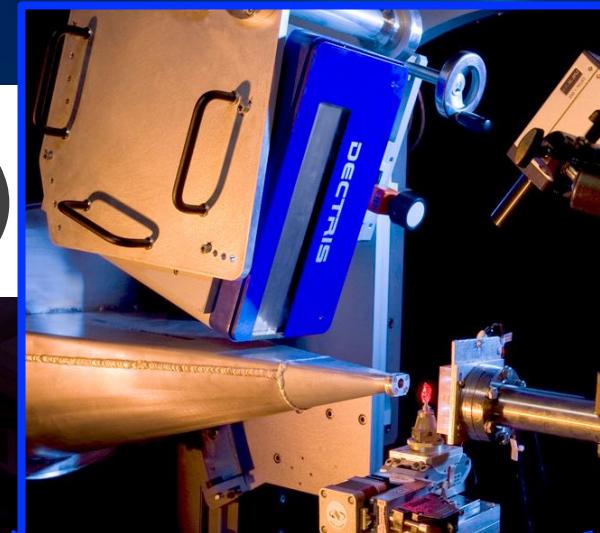
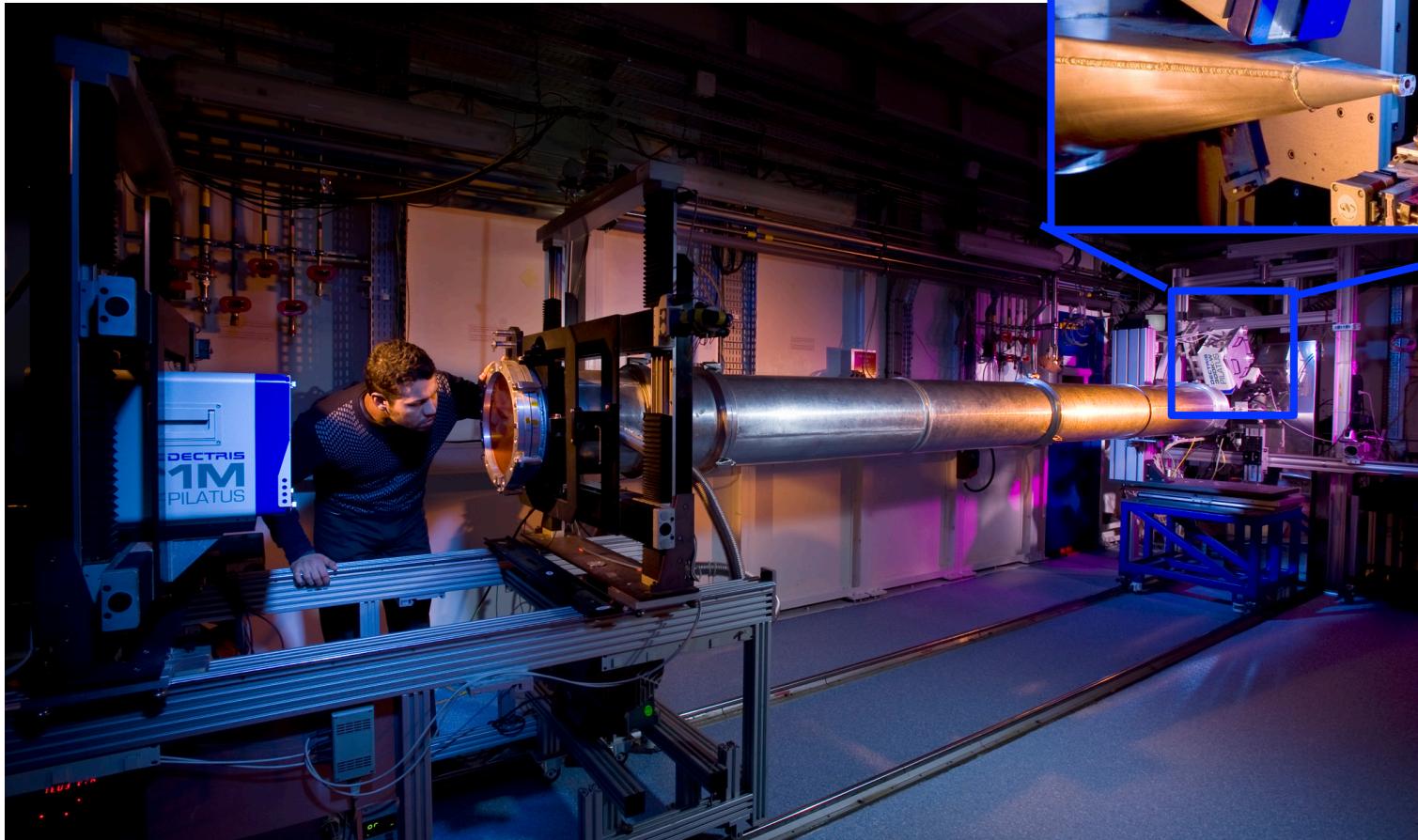
- voxel size (65nm)³
- uncertainty within voxel is 0.04 e⁻/Å⁻³
- significantly higher sensitivity for larger volumes, e.g., 0.2% equiv. to <0.001 e⁻/Å⁻³ (1μm³)

Sample: mouse femur



M. Dierolf *et al.*, Nature, 467, (2010), 436

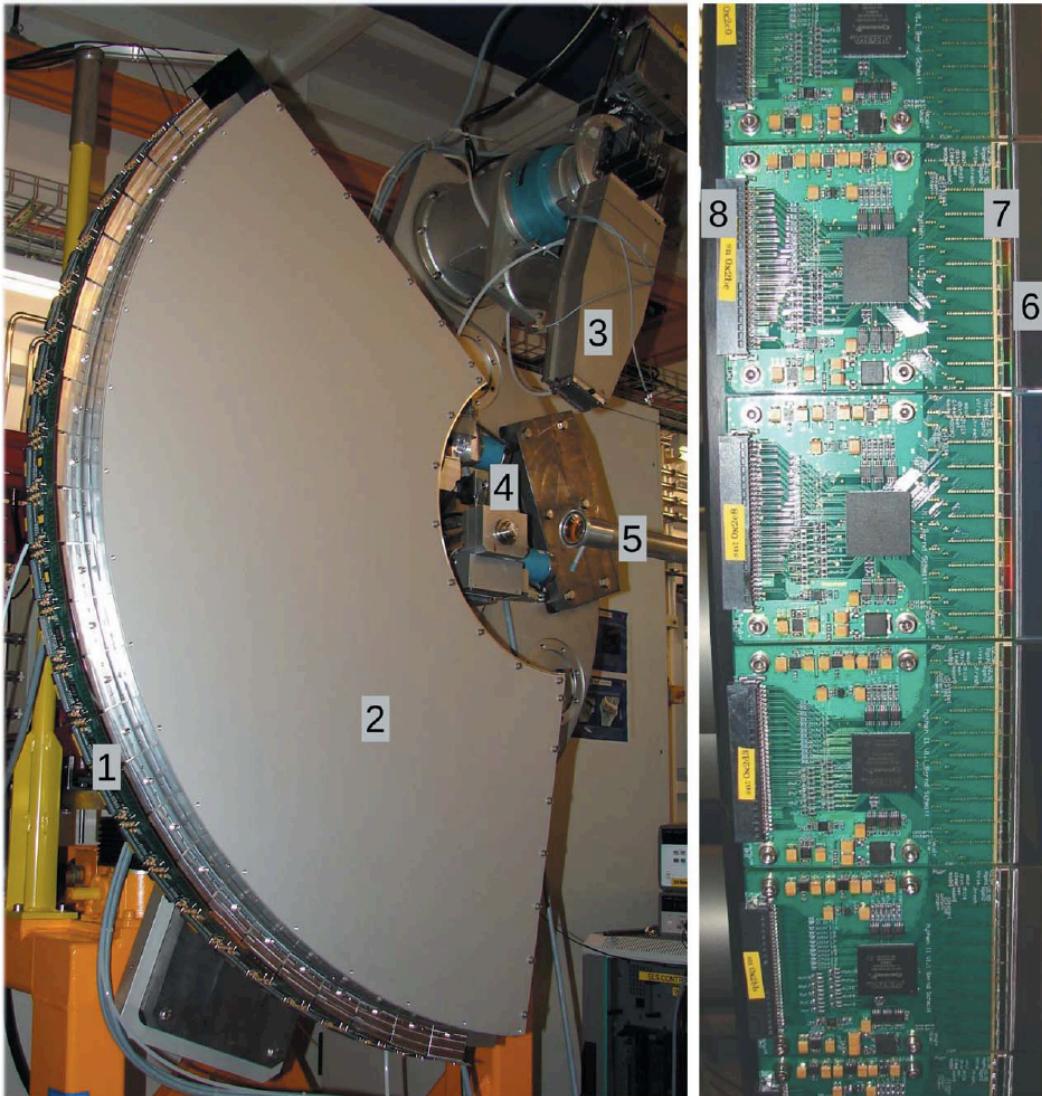
DUBBLE CRG at ESRF (1M & 300K)



...the first publication...concerns an fast crystallization experiment with a data quality that is as nice as the first cool beer after a 15 km hike on a very warm day. (It even makes me poetic...) WIM BRAS, Feb 2011 ESRF

Real-Time WAXD Detection of Mesophase Development during Quenching of Propene/Ethylene Copolymers
Macromolecules 2010, **43**, 10208–10212 DOI: 10.1021/ma1022499

PD with MYTHEN at Synchrotrons



X04SA Powder Diffractometer

- 1 MYTHEN detector layer
- 2 He box
- 3 analyser crystal detector
- 4 diffractometer axis
- 5 beam pipe
- 6 Si microstrip detector
- 7 front-end electronics
- 8 connector to DAQ system

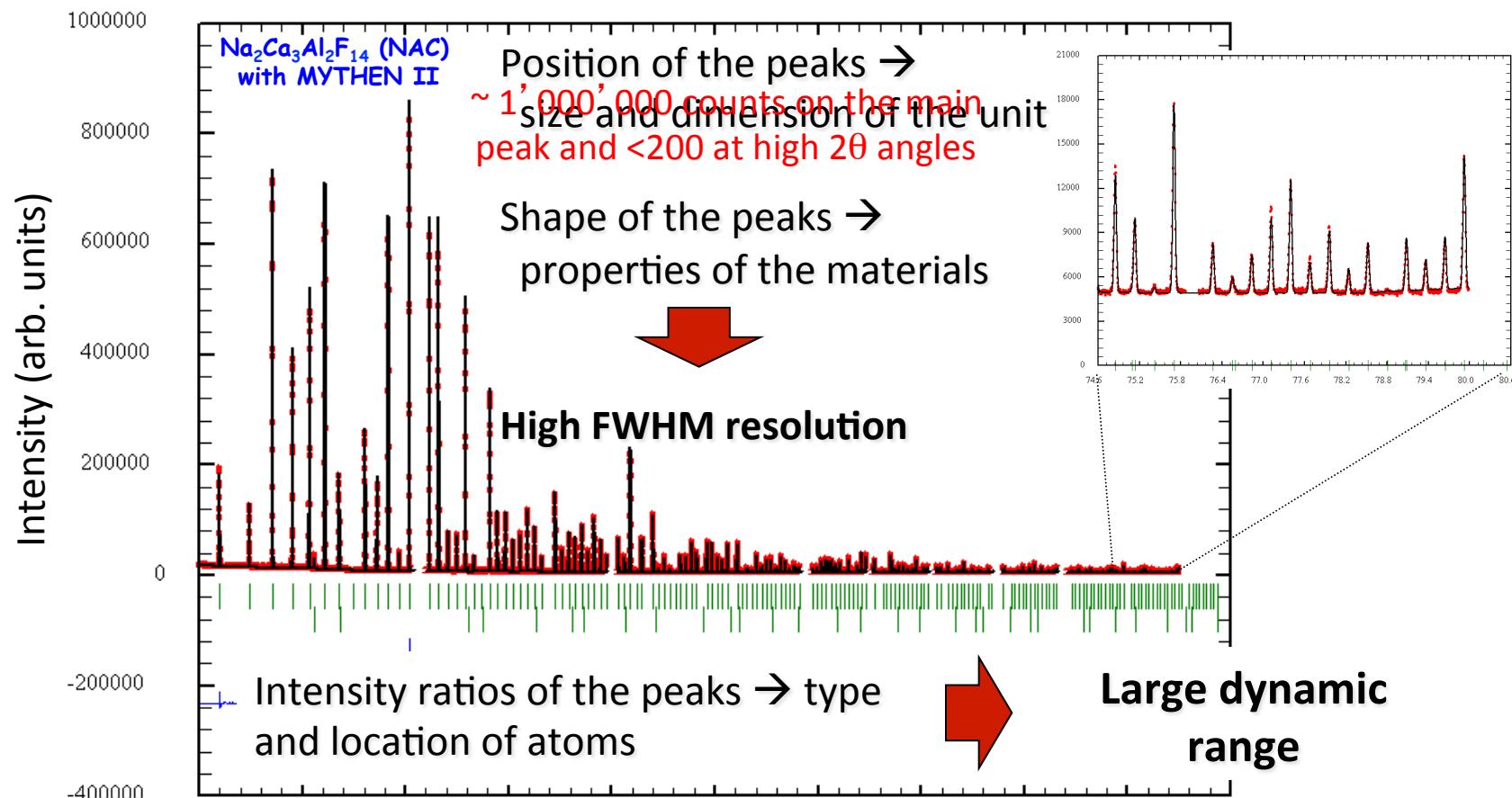
MYTHEN detector

- 120 ° equiv. to 30720 channel
- 50 µm strip pitch (4 mdeg)
- 10 frames per second at 24 bit

A. Bergamaschi *et al.*, JSR 17, (2010), 653

F. Gozzo, Z. Kristallogr. 225 (2010) 616–624

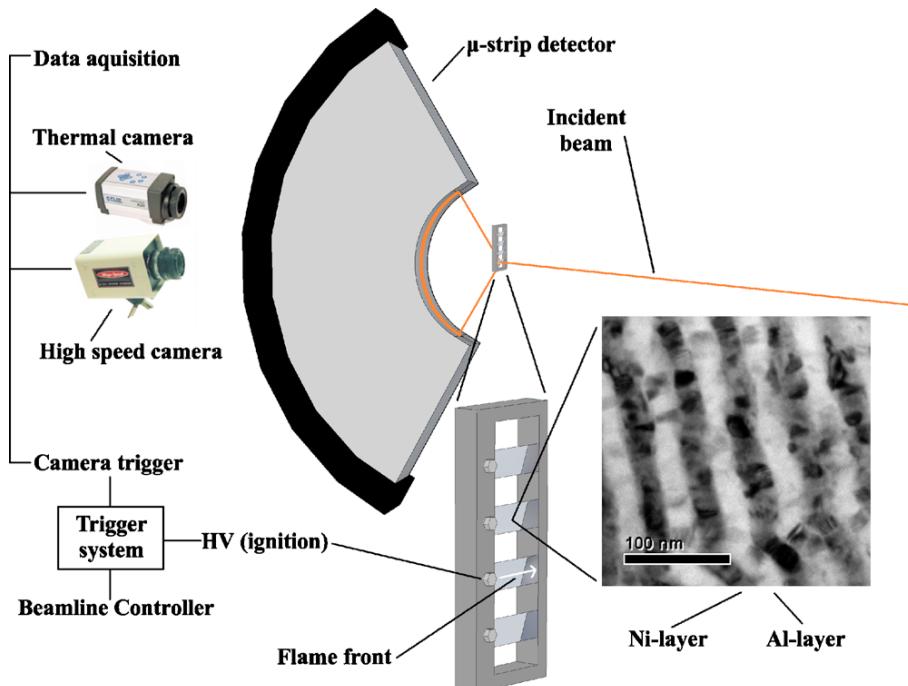
MYTHEN applications at synchrotrons



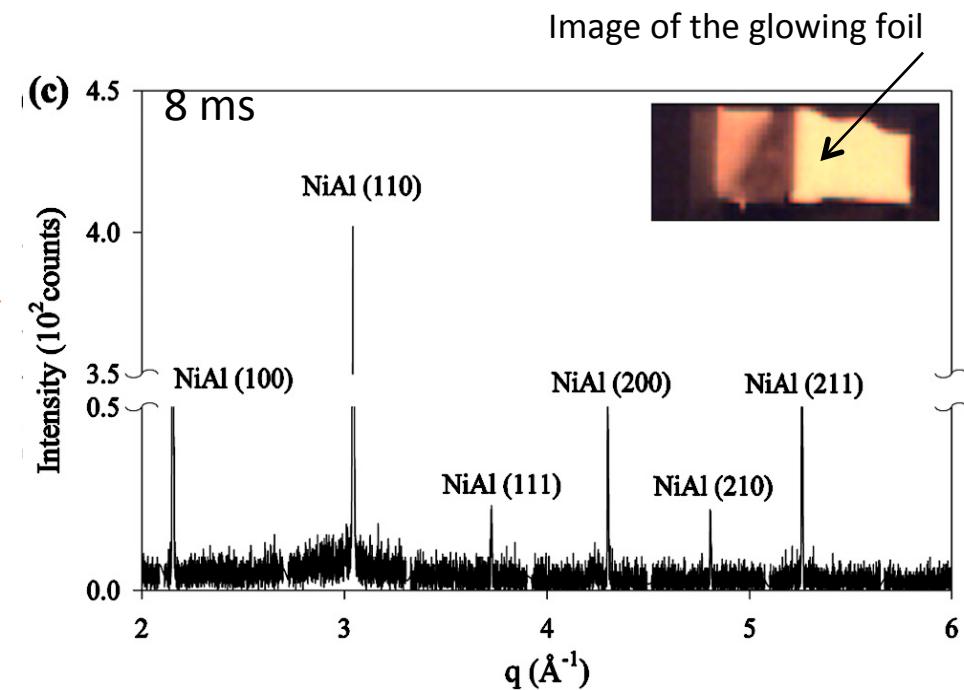
- Parallel detection => Fingerprinting, phase analysis
- High FWHM- and d-spacing resolution => Minimisation of radiation damage
- Temporal resolution => Structural solution and refinement
- Kinetic studies

Self-Propagating Exothermic Reactions

In-situ measurements: Speed and Angular Coverage



60 μm $\text{Ni}_{0.9}\text{V}_{0.1}$ -Al foils



Summary

DECTRIS photon counting detectors overcome most limitations of previous technologies

Advanced data acquisition protocols improve data quality

Novel experiments enabled by detector properties





thank you for your attention