CCLRC
Daresbury Laboratory

Radiation Damage, Micro-crystals and the Cornell ERL

John Cowan, Colin Nave

SRS, Daresbury

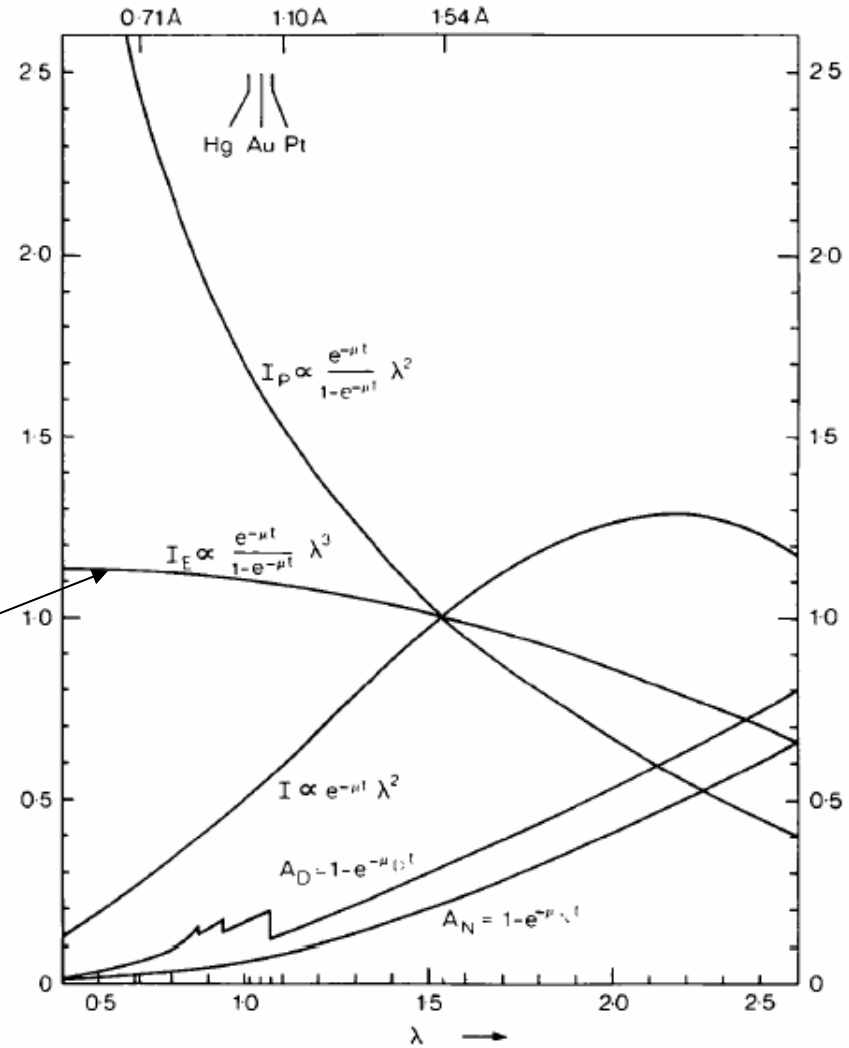
CCLRC Daresbury Laboratory

Overview

- *Calculations of optimum photon energy*
- *Use of pulsed x-ray sources*
- *Beam emittance required*

Assumption – Many protein crystallography projects are compromised because crystals of sufficient size are not available

Arndt J. Appl. Cryst. 1984



Diffracted photons/dose (normalised)

Henderson, 1990

Predicted "X-rays of about 2×10^7 Gy will always destroy the crystalline diffraction from protein crystals"

- *Less than a minute on the most intense sources.*

Assumptions

- *Photo-electron absorption is High*
- *Ignore Compton Scattering*

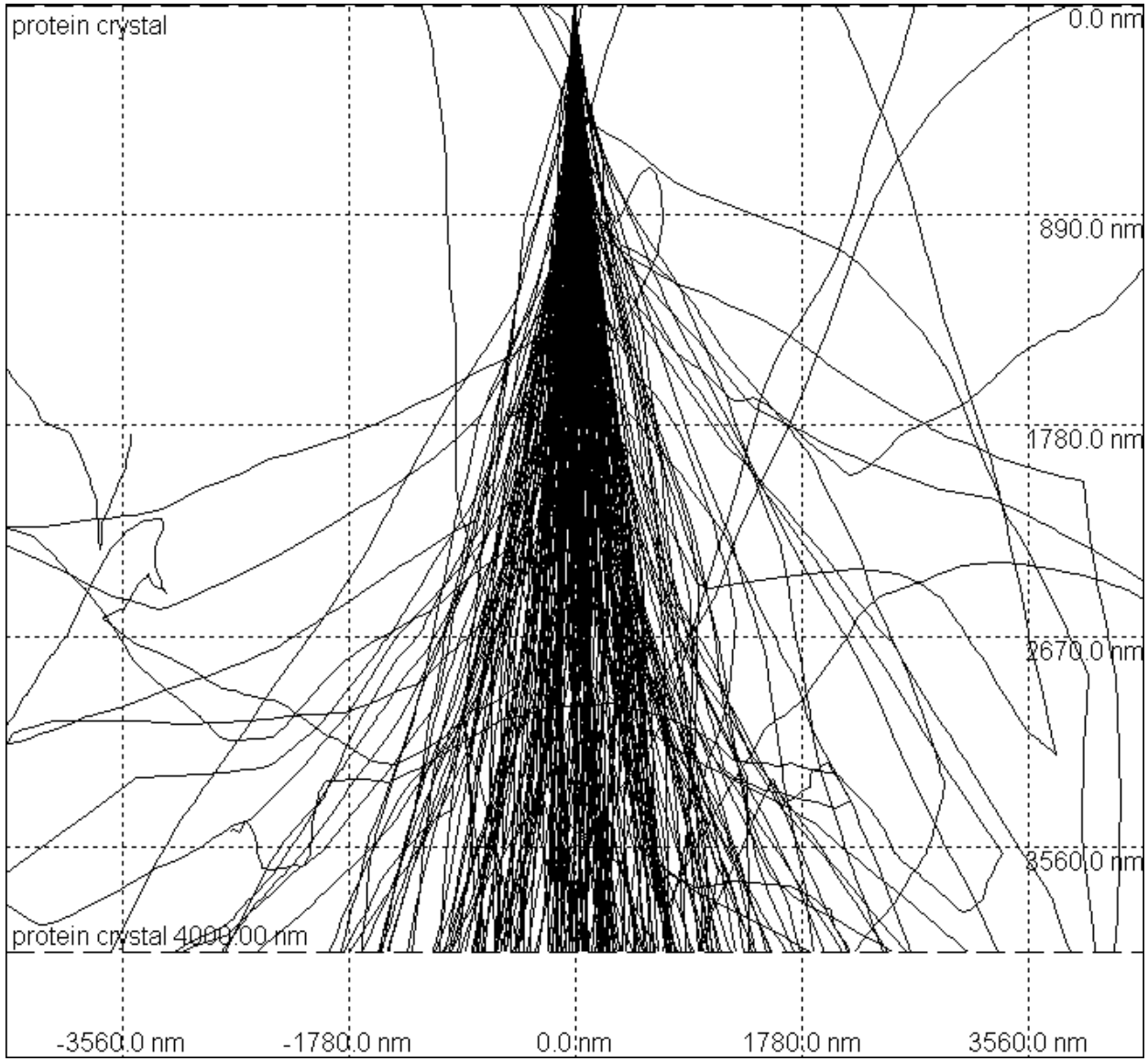
What happens to the photo-electron for very thin specimen?

Colin Nave and Mark Hill
Journal of Synchrotron Radiation, 2005, 12, 299-303.

CCLRC Daresbury Laboratory

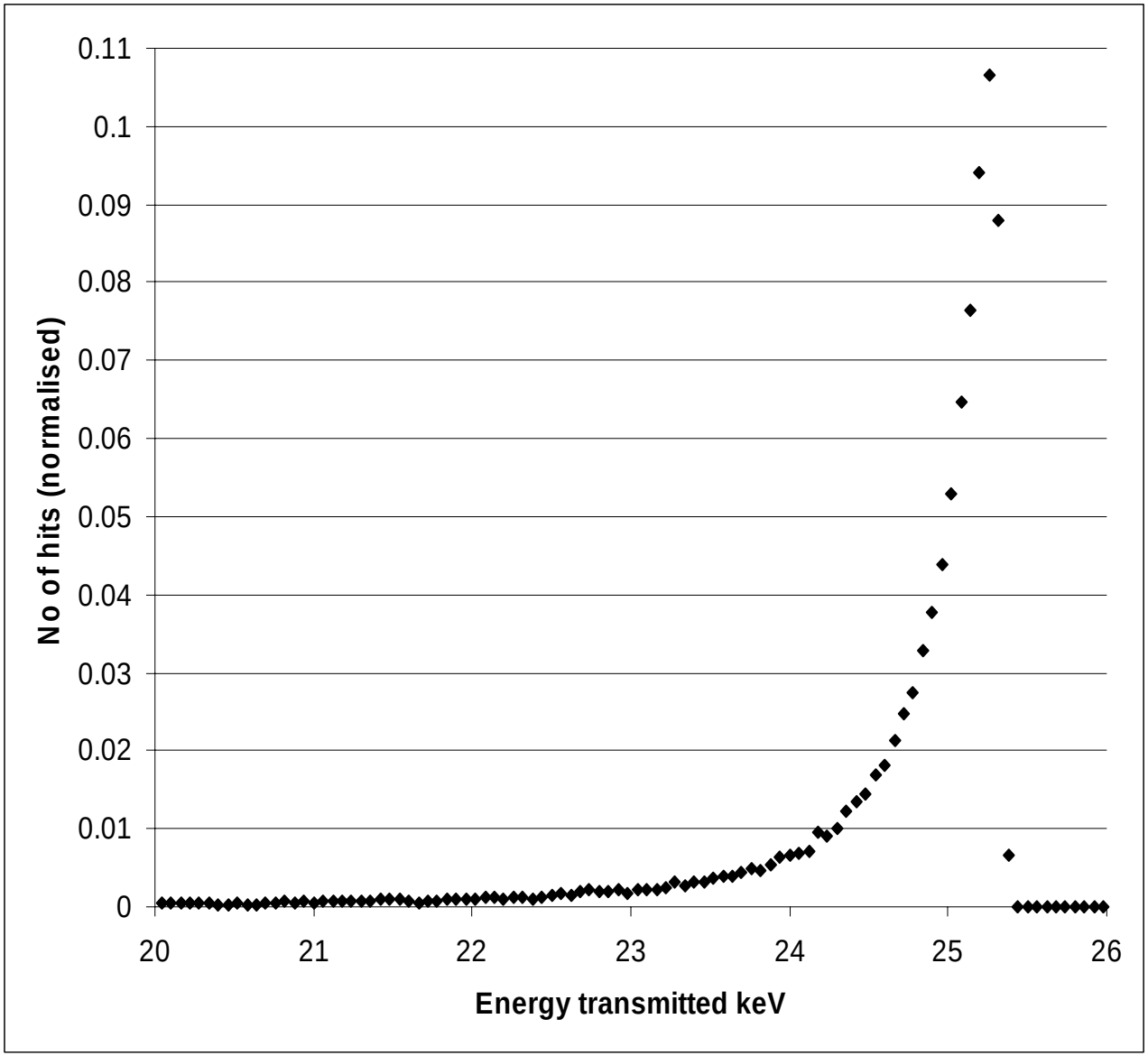
Simulation of Electron Tracks

Casino
monte CArlo SImulation of electroN trajectory in sOlids



4 mm from Surface.
30keV

Spectrum 4μm from source of 30keV electrons



CCLRC Daresbury Laboratory

Escaped energy

Distance from surface (μm)	Transmitted energy (%) at given initial energy			
	10 keV	20 keV	30 keV	40 keV
0.5	79	95		
1	23	89		
2	0	74	90	94
4		34	76	87
8		0	37	57
16			0	23
32				0

Gains of up to 10 theoretically attainable

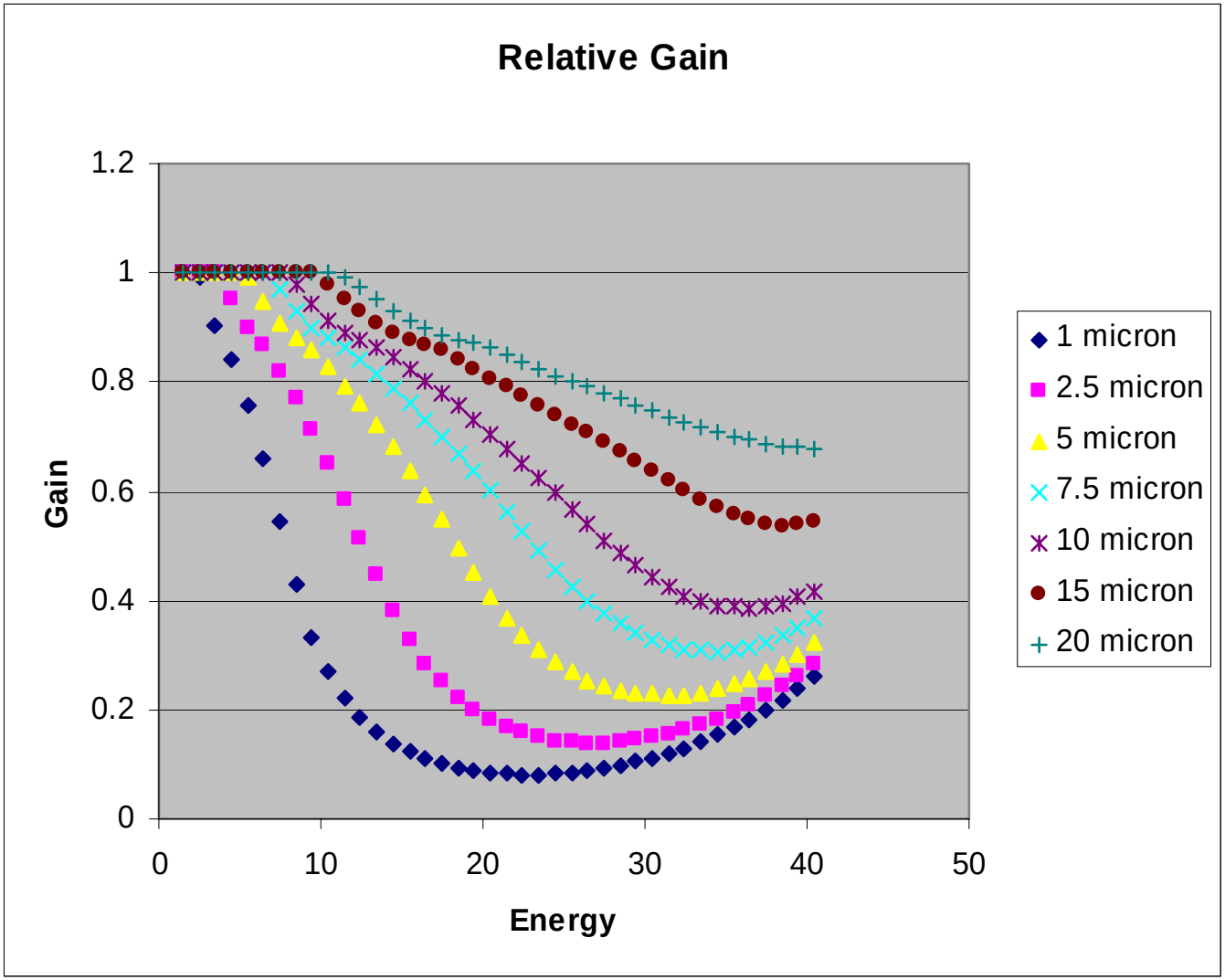
Inclusion of Compton Scattering

Compton and Photoelectric cross sections approximately equal at 25keV.

Average energy deposition by Compton effect approx 1.5keV at 30 keV photon energy

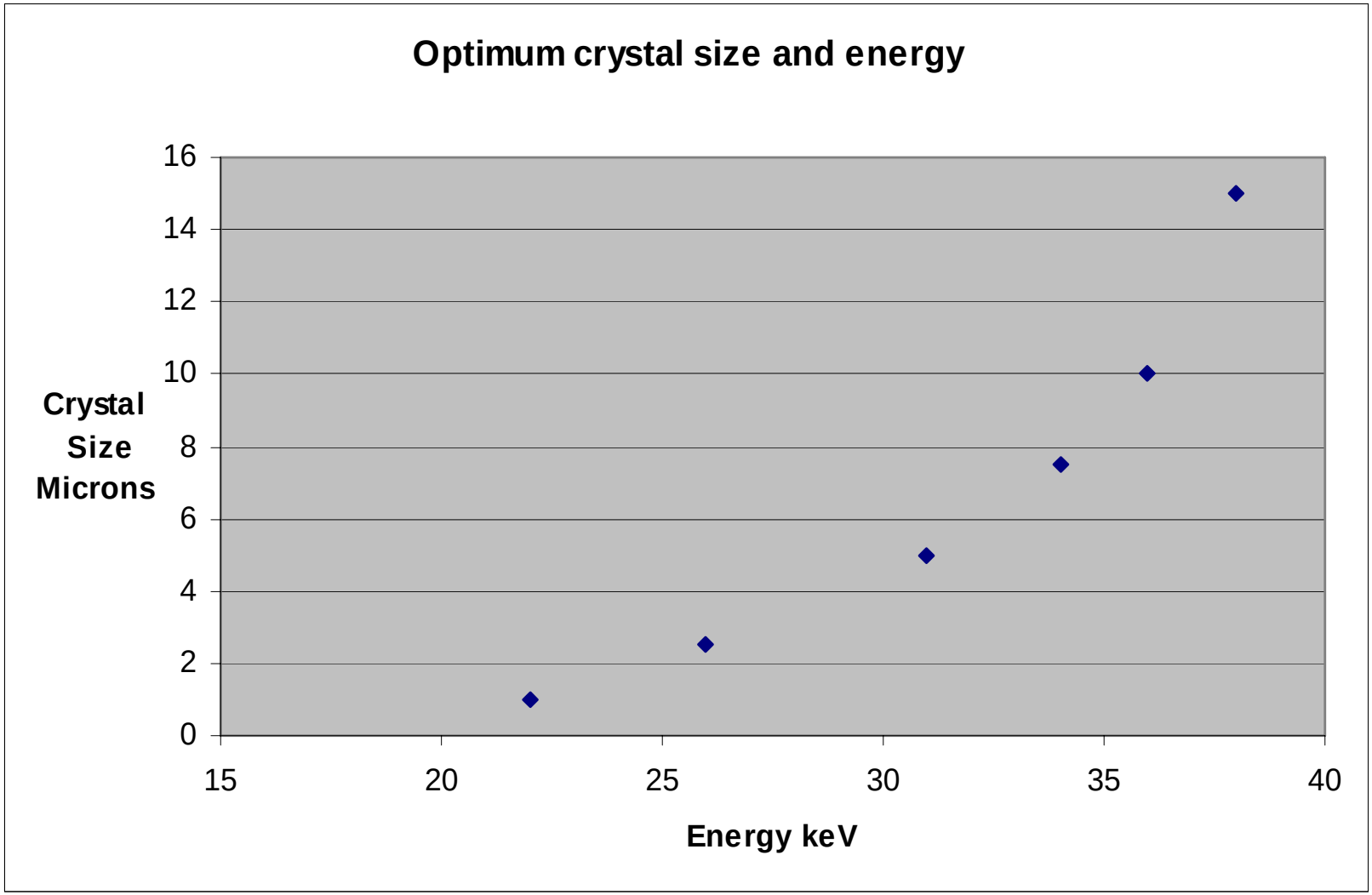
CCLRC Daresbury Laboratory

Possible reduction in radiation damage



CCLRC Daresbury Laboratory

Choice of Energy



Possible modifications to "Rule of Thumb"

One high-resolution diffraction pattern, 1° of rotation), can be obtained from a protein crystal if the crystal size (in micrometers) is 1/10 the unit cell size (in Angstroms).

Robert Glaeser, Marc Facciotti, Peter Walian, Shahab Rouhani, James Holton, Alastair MacDowell, Richard Celestre, Daniela Cambie, and Howard Padmore
Biophys J, June 2000, p. 3178-3185, Vol. 78, No. 6

Could obtain 7 patterns from 2.5 micron crystal, 25Å cell at 25keV energy

4 patterns from 5 micron crystal, 50Å cell at 30keV energy.

1 pattern from 5 micron crystal, 200Å cell at 30keV

Possible Experimental Set Up

em Grid
holey carbon
amorphous ice
thin carbon film?
protein crystal

x,y scanning

Helium Gas
100K

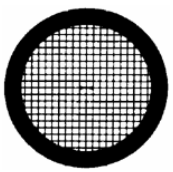
Diffracted x-rays

Detector

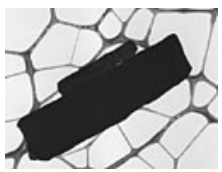
Electron shower

X-ray Beam

Similar set up in paper by Schertler et. al.



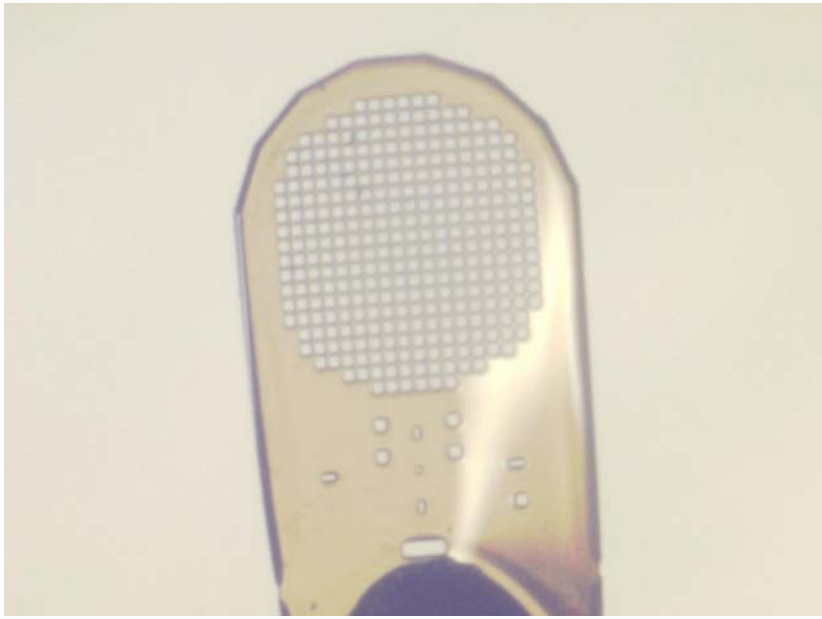
Em grid



Lacey film

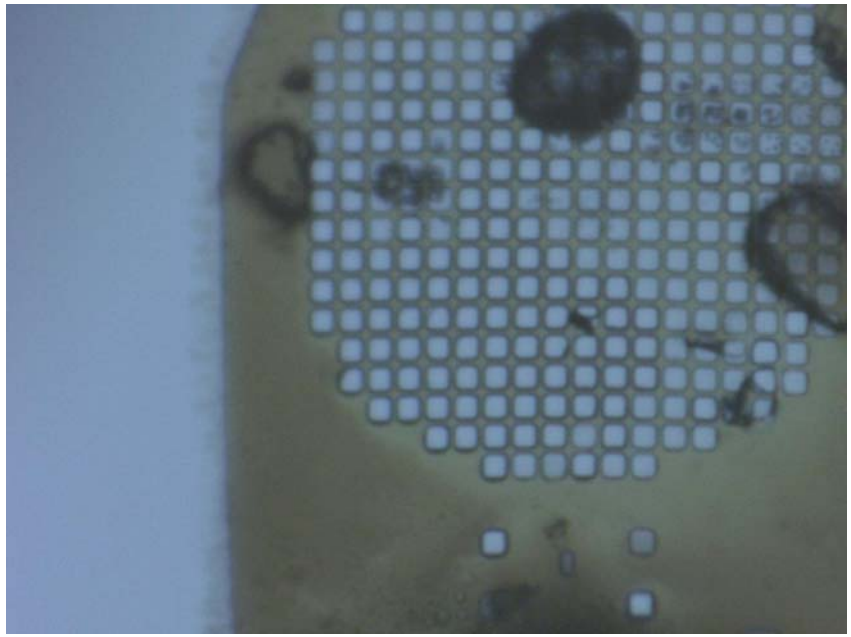
CCLRC
Daresbury Laboratory

Crystals Mounted on Micromesh



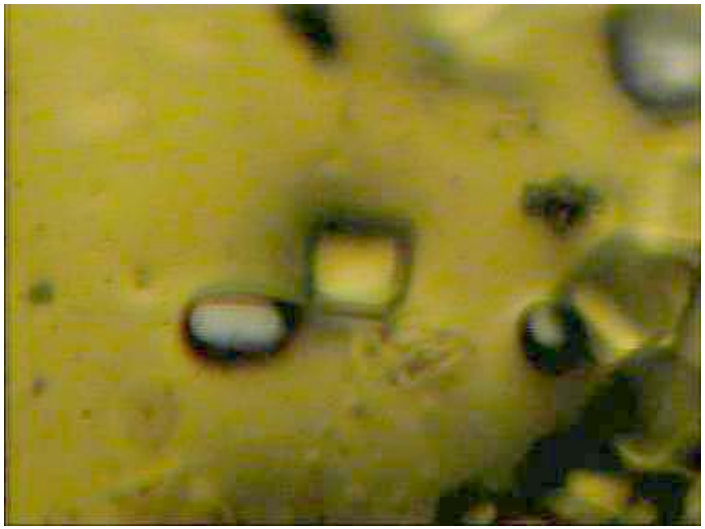
MiTeGen Micromesh Mounts

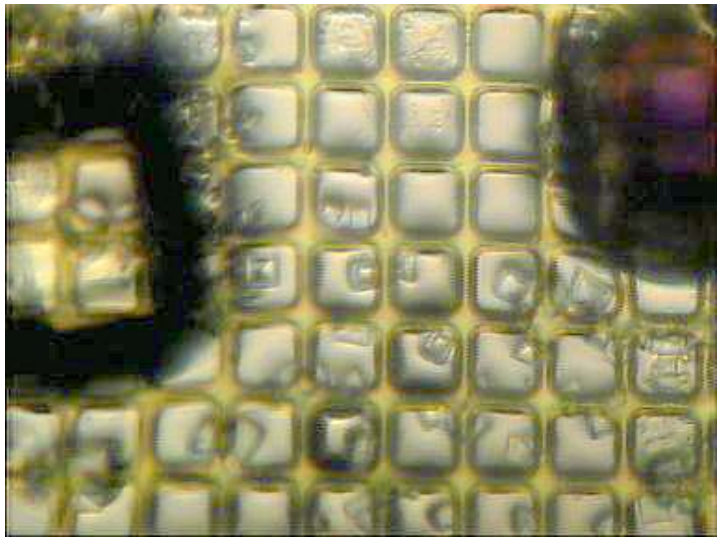
700 microns across,
25 micron holes.

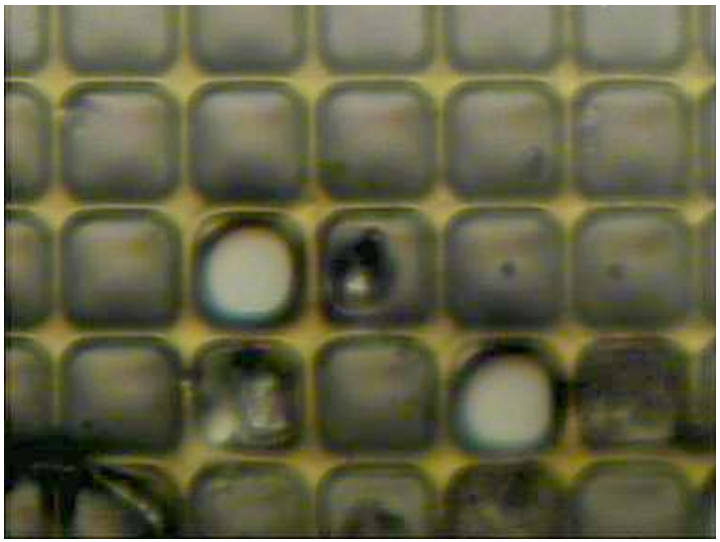


CCLRC
Daresbury Laboratory

Crystals on ID23-2 ESRF

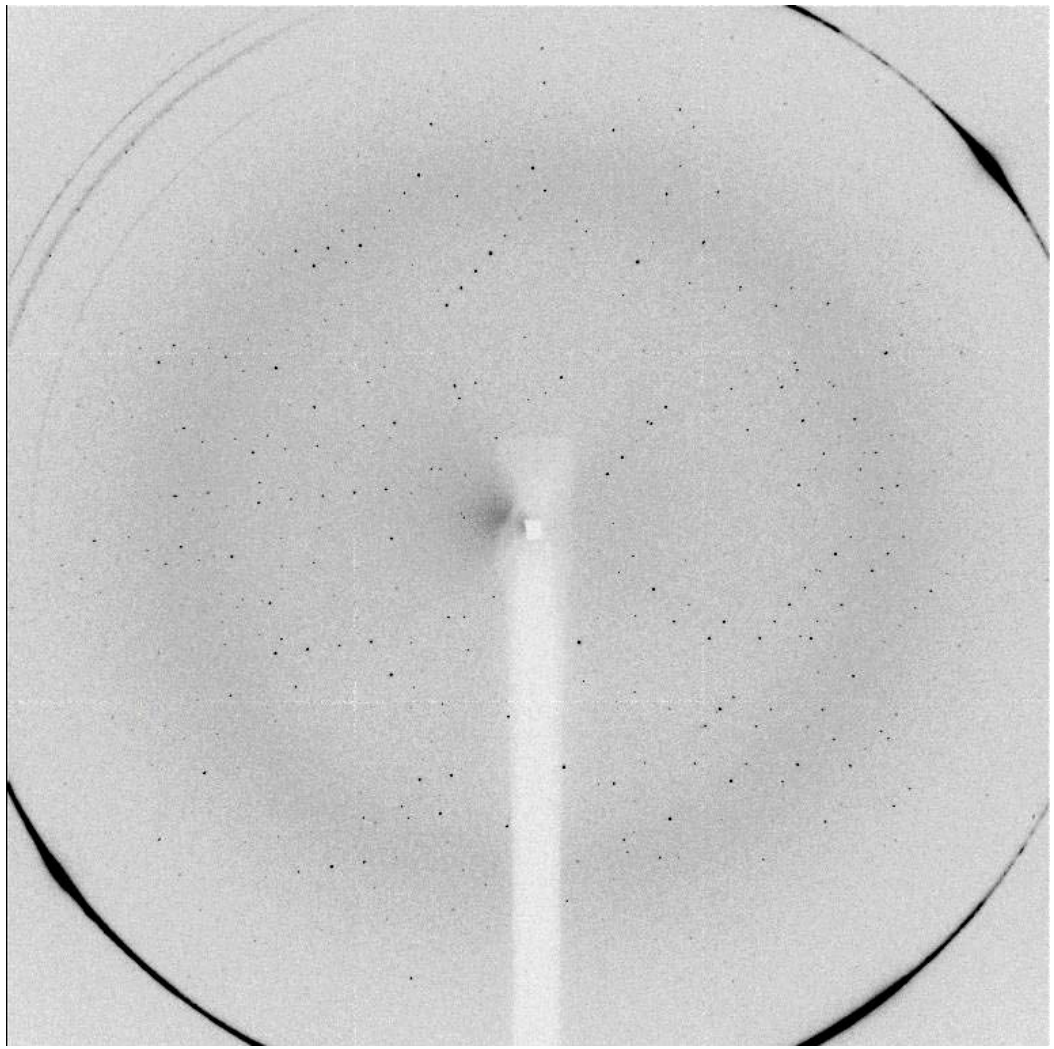






CCLRC
Daresbury Laboratory

Diffraction Patterns from ID23



Em Grid

Nitrogen v Helium from CHESS newsletter

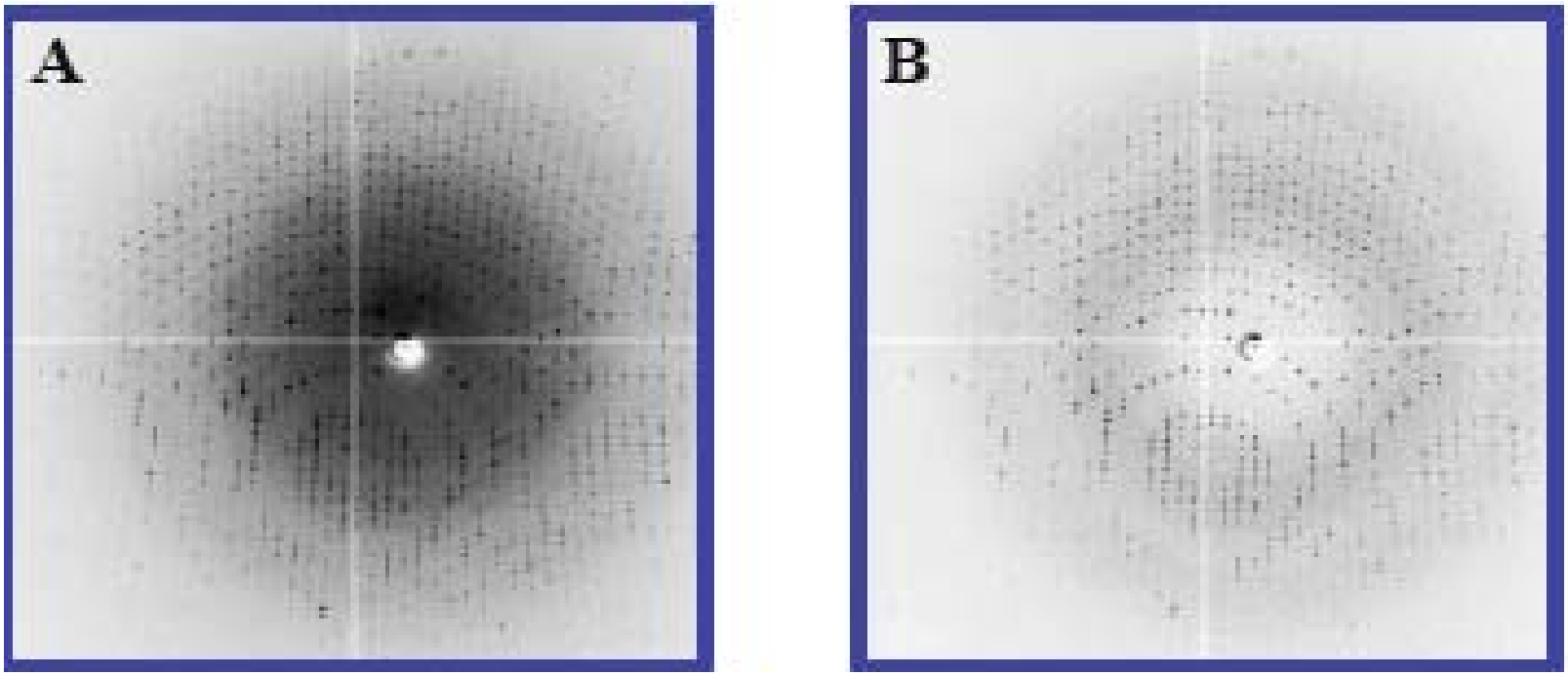


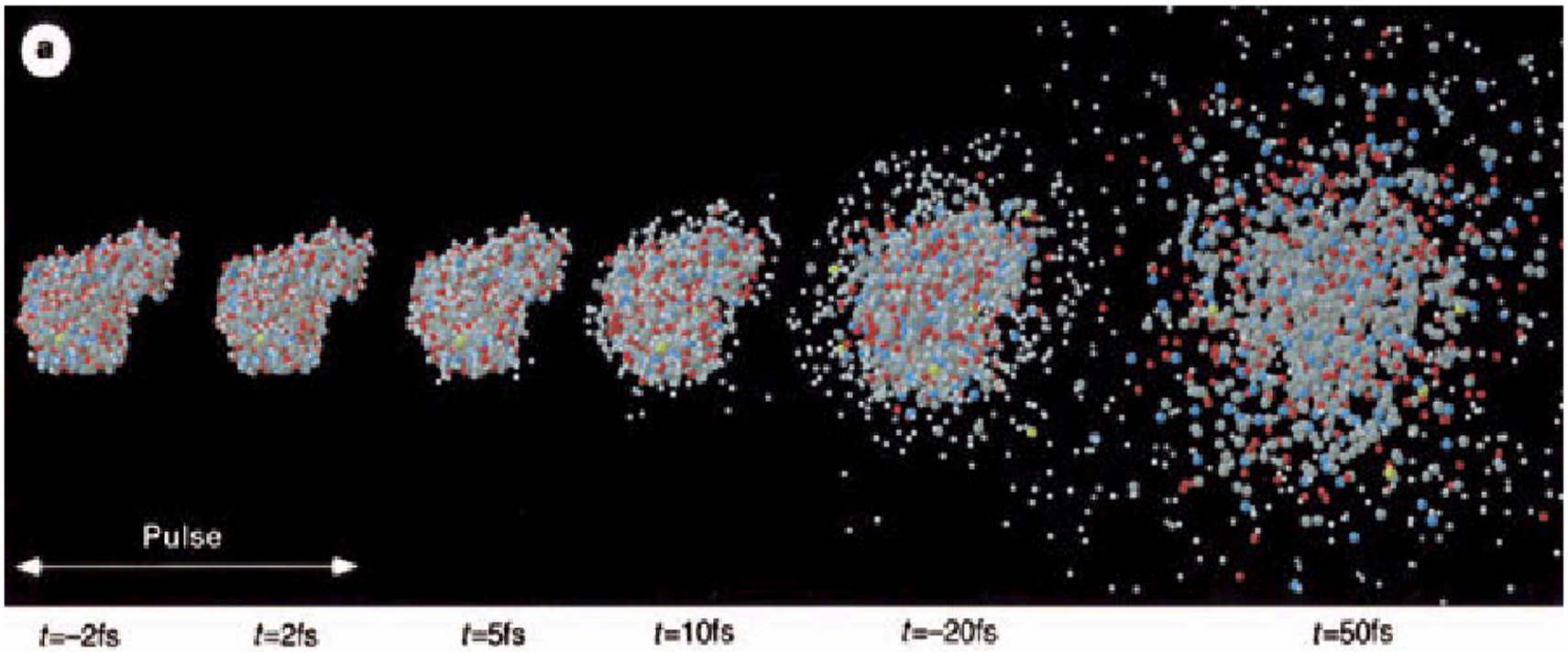
Fig 4: Comparison of diffraction images produced from a rhodamine-dyed lysozyme crystal in an enclosure filled with different gases.
A. Nitrogen-filled enclosure.
B. Helium-filled enclosure.

10μm water ~10mm air

CCLRC
Daresbury Laboratory

Application of X-ray FEL?

R. Neutze, et al., *Nature*, **406**, 752-757, January 17, 2000



Timescales longer if spread incident beam larger area (e.g. 5 μm crystal)

CCLRC Daresbury Laboratory

Necessary flux for a pulsed photon source

Neutze et. al., $3x10^{11}$ photons in 100nm diameter spot gives 3.9Å resolution from lysozyme crystal 5X5X5 unit cells

10^{10} photons incident on 15nm X 15nm area – path length 15nm.

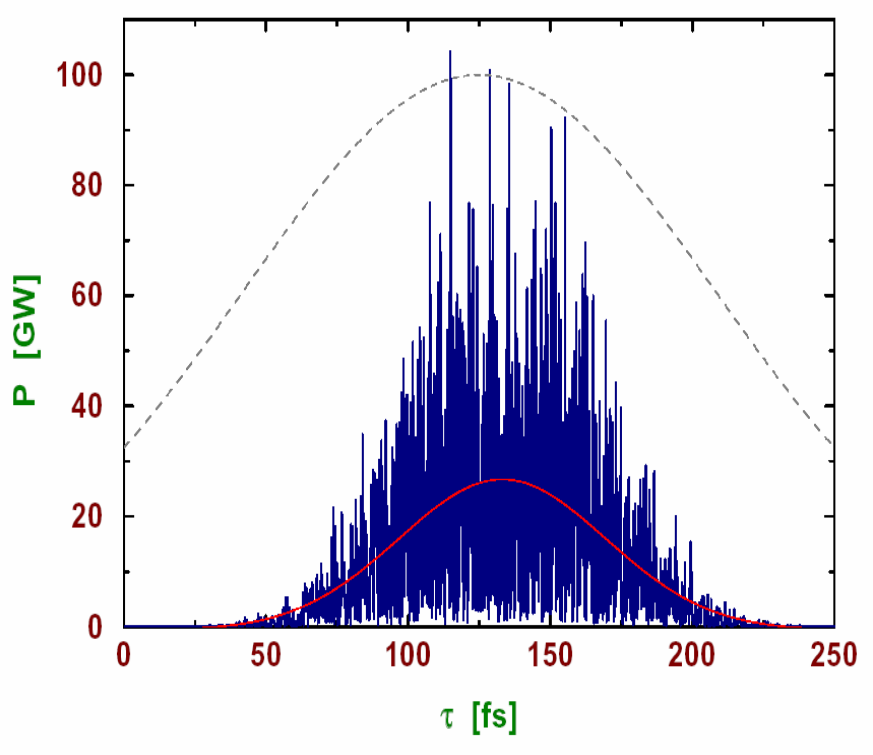
For 1 μm sample would need 1.5×10^{8} photons
For 10 μm sample would need 1.5×10^{7} photons

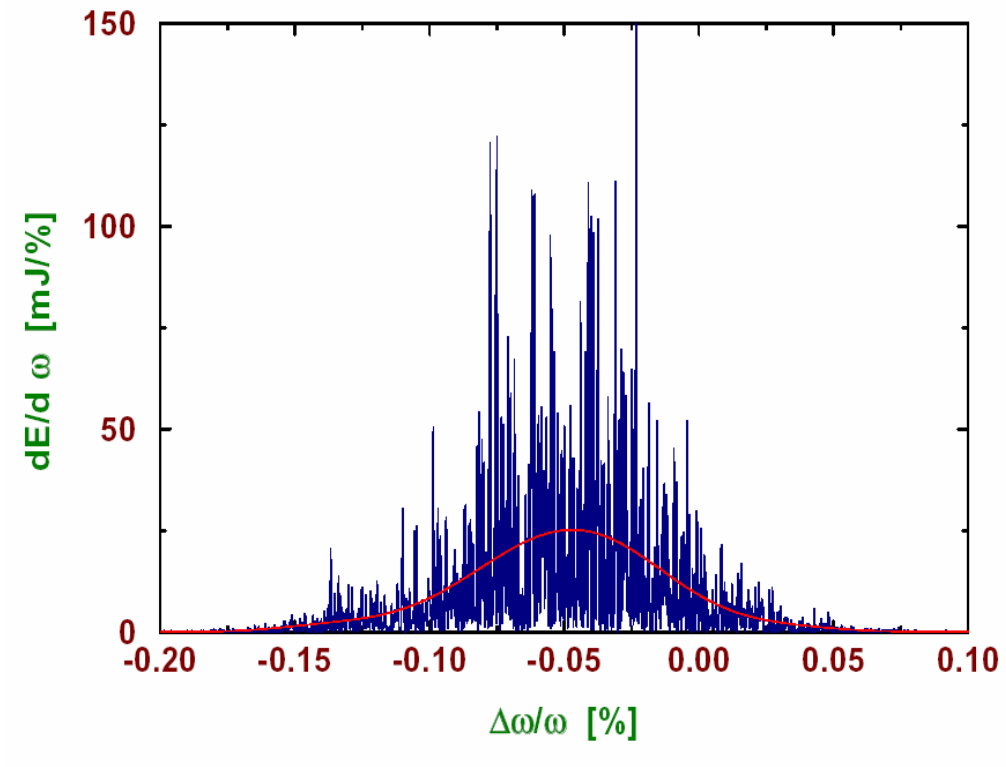
Cornell ERL-IIa, high flux gives $1.36\ 10^{7}$ photons/0.1% per pulse.

XFEL, LCLS, SCSS give over 10^{12} photons/pulse

Note that can get single image from 3 μm sample anyway.

X-ray FEL Temporal and Spectral Pulse Structure

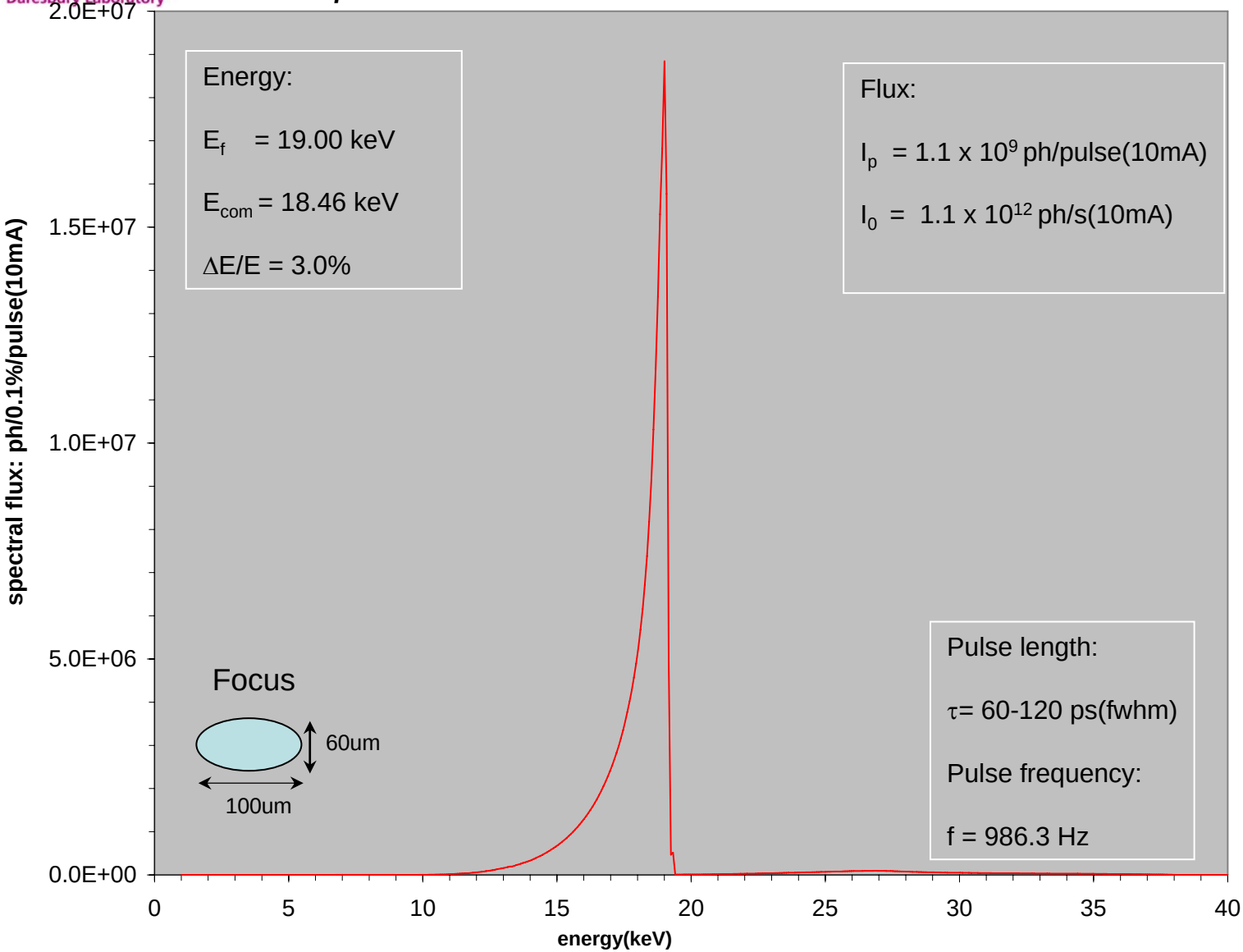




Ideally would like a pulsed x-ray source with 10^{11} photons/ pulse and 1-5% bandwidth ?

CCLRC Daresbury Laboratory

Spectrum of the mono-harmonic undulator U17



From Michael Wulff

CCLRC Daresbury Laboratory

Size and divergence of beam

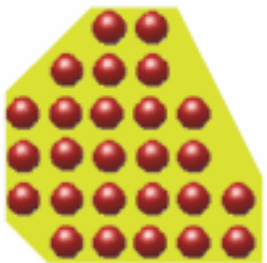
Beam size matched to size of crystal

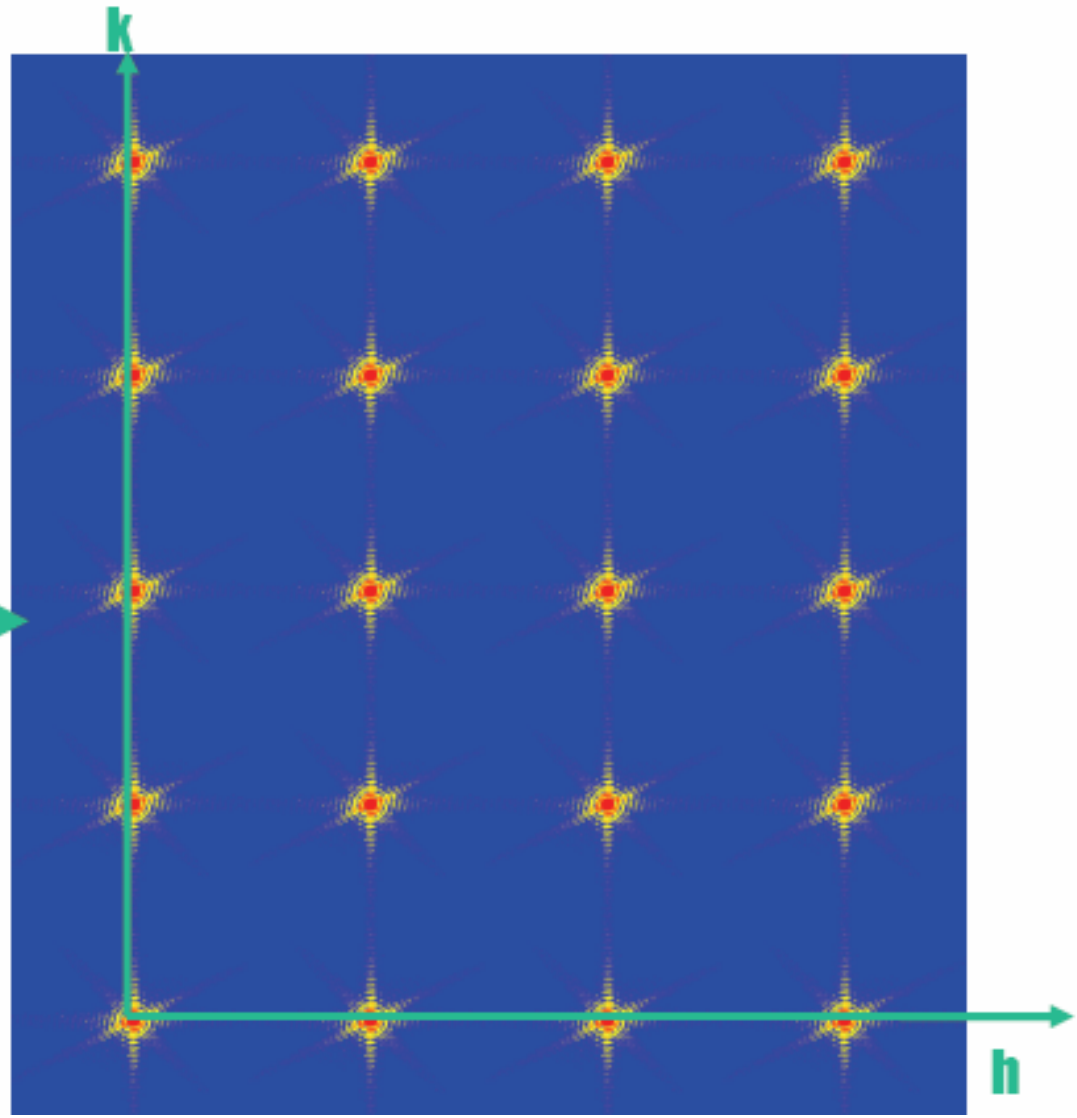
Low divergence for diffracted beam to give sharp spots on detector

Divergence for a perfect crystal given by crystal size - implies highly coherent incident beam.

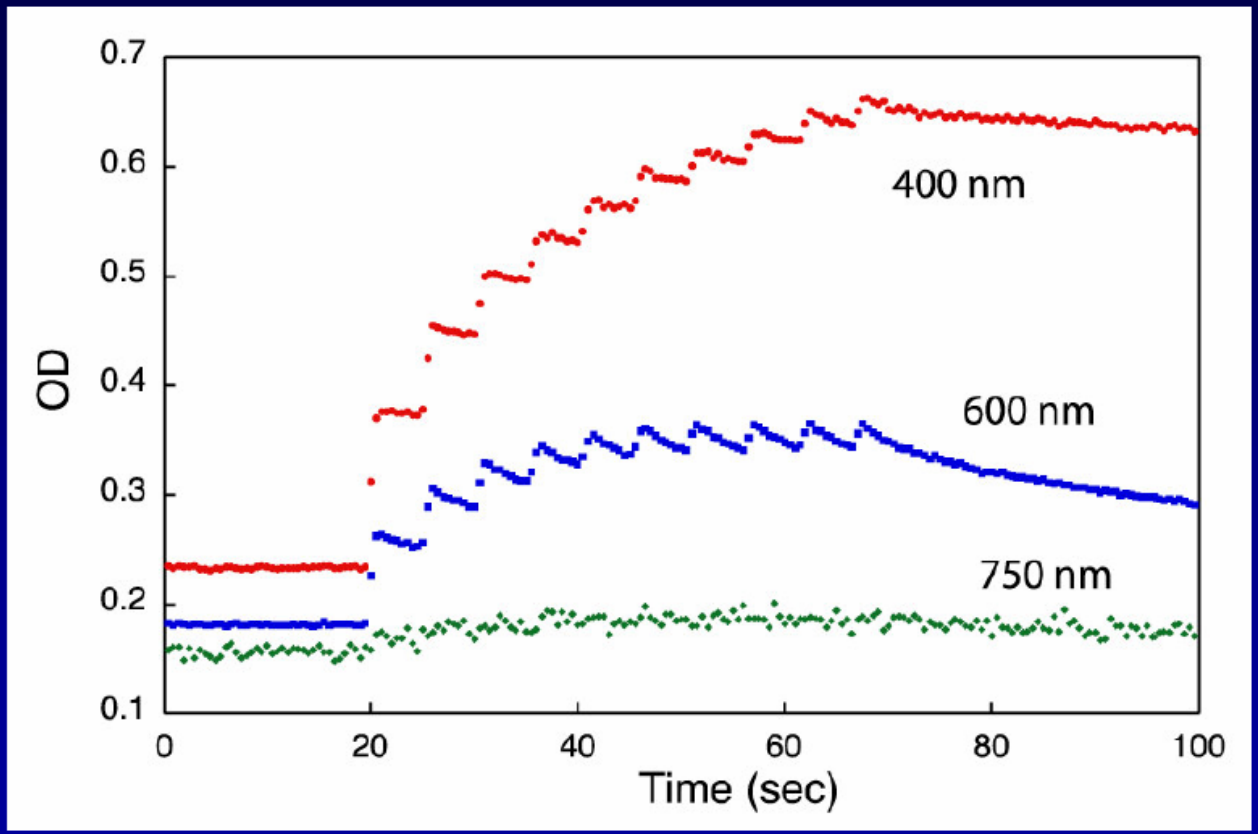
Coherent Diffraction from Crystals

From Ian Robinson, University College London





Formation of Radicals during data collection



John McGeehan, Raimond Ravelli, Florent Cipriani, Franck Felisaz, Raphael Moya
EMBL and ESRF Grenoble

Evidence of dose rate effect?

CCLRC Daresbury Laboratory

Detectors

- *Need detectors optimised for photon energies around 30keV.*
- *Should have large number of pixels to match small, parallel beams from micro-crystals.*
- *Modifications to present CCD or pixel (hybrid or monolithic) based systems required.*

Summary

Could get much more data if chose appropriate wavelength (prediction).

Must lower x-ray background from system.

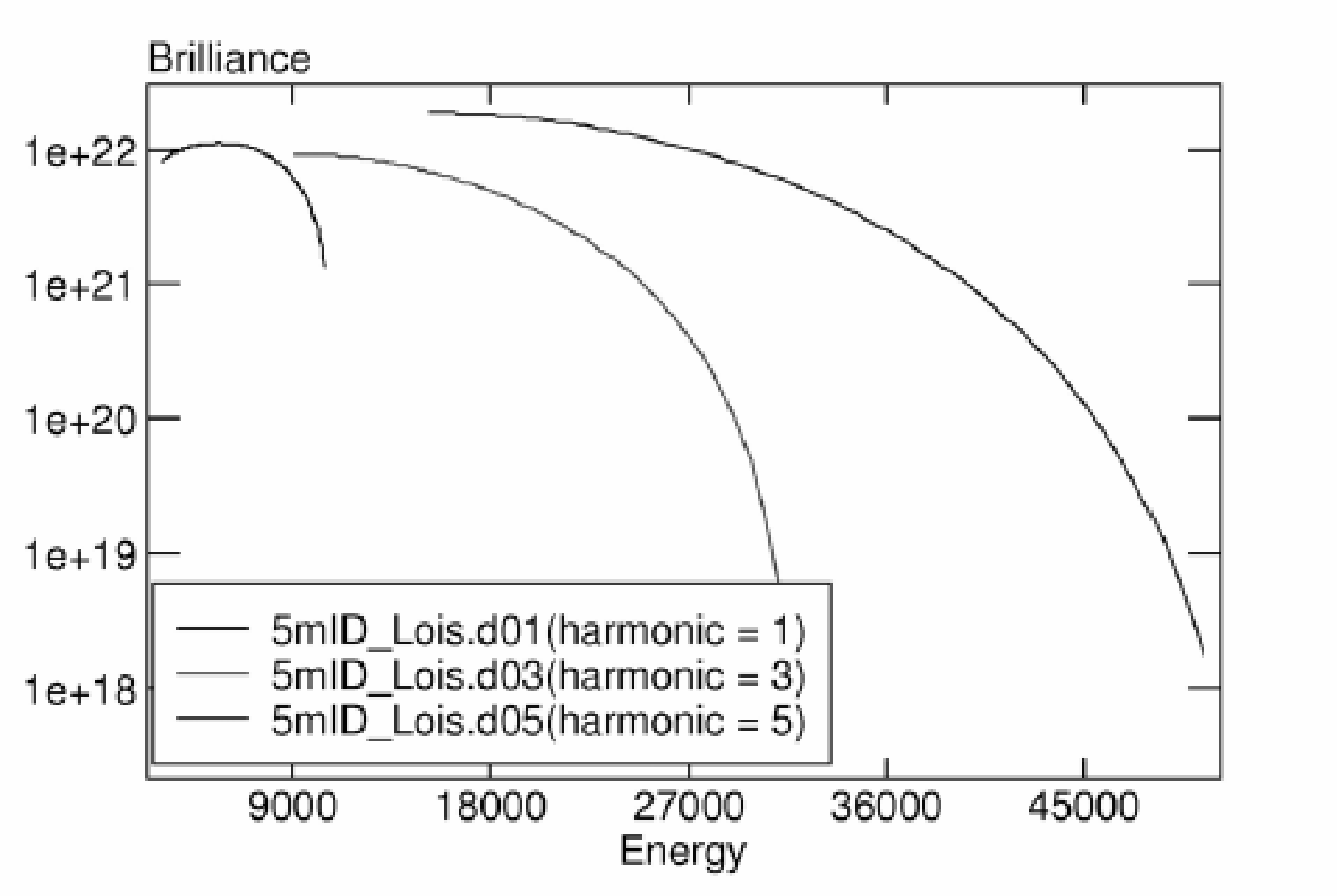
Mounting methods have to be optimised.

An appropriate x-ray source for microcrystal work

- *30keV continuous*
- *Or 8 keV pulsed with 3% bandwidth,*
- *High degree of coherence*

As always detectors also have to be addressed

Cornell ERL


Brilliance
1e+22
1e+21
1e+20
1e+19
1e+18
5mID_Lois.d01(harmonic = 1)
5mID_Lois.d03(harmonic = 3)
5mID_Lois.d05(harmonic = 5)
9000
18000
27000
36000
45000
Energy