CMOS Pixels for Electron Microscopy: Requirements and R&D Results at TEAM

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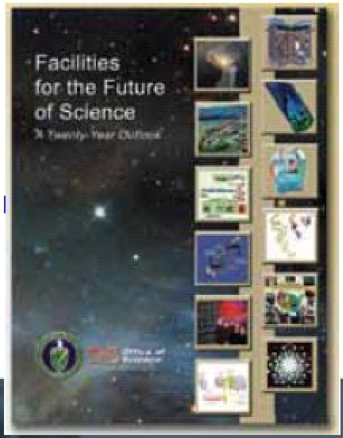
UCSC and LBNL

with contributions by P. Denes

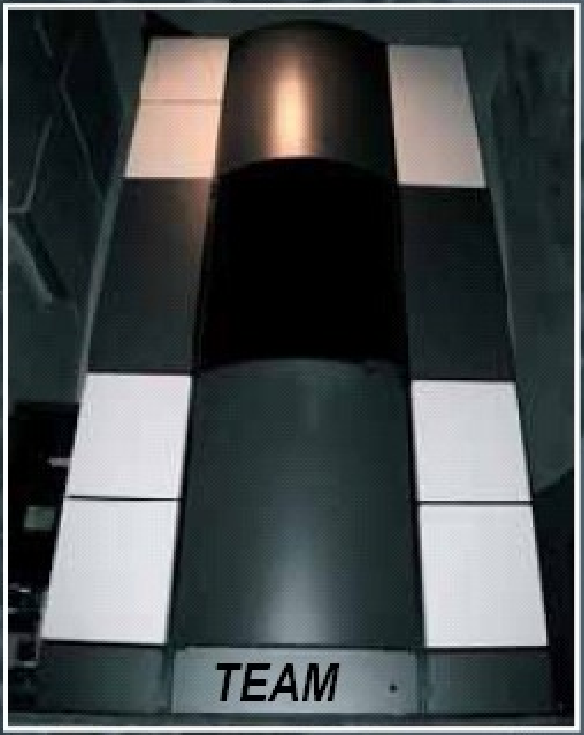
Workshop on CMOS Active Pixel Sensors for Particle Tracking CPIX14
Bonn, September 15th 2014

A New Frontier in Electron Microscopy

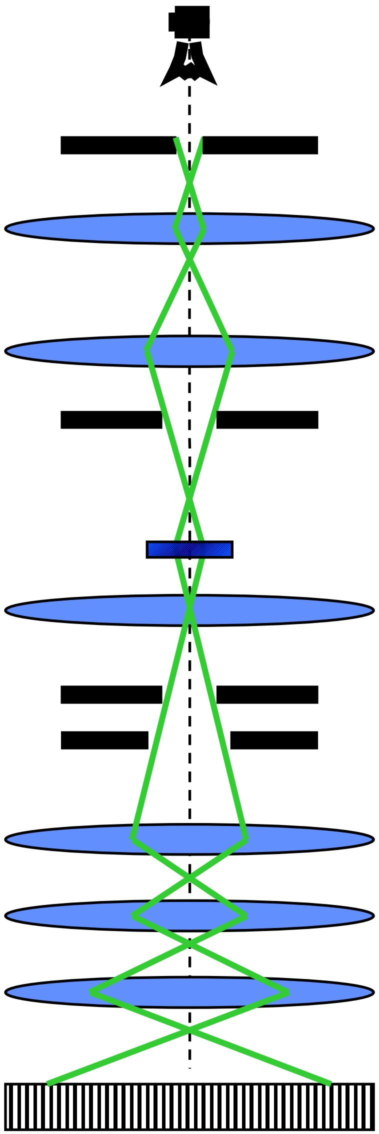


TEAM Project at NCEM

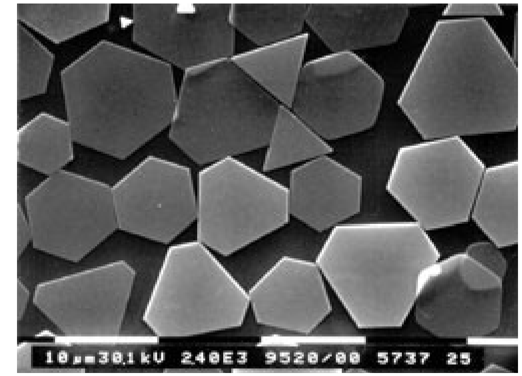
- 80-300 keV e^- beam
- 0.5 A spatial resolution
- Monochromator for $\Delta E = 0.1$ eV
- "quiet" site
- Fast Imaging



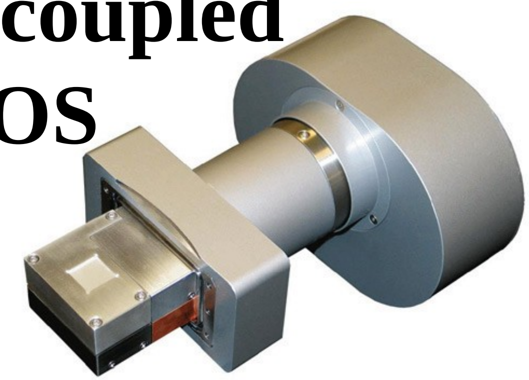
Imaging in Transmission Electron Microscopy



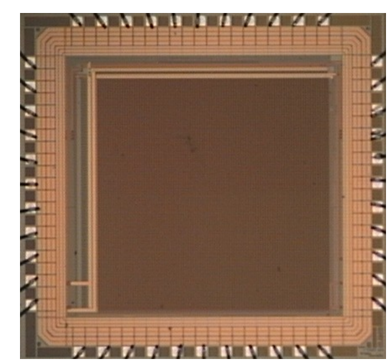
Film



Optically-coupled CCD/CMOS



Direct Detection CMOS



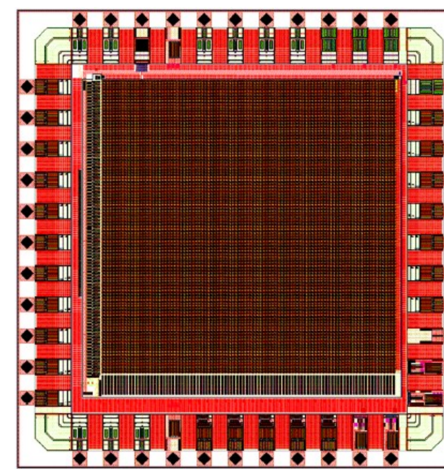
Basic requirements: at least 1k x 1k pixels to get 200 A field of view, pixel size 5-10 μm, PSF < 1 pixel, thickness ~ 50 μm, frame rate > 100 f/s, radiation tolerance > 1 Mrad (~ 1 year of use)

Fast CMOS Pixel Imager for TEAM

Develop new sensor to match TEM capabilities in terms of position and temporal resolution with direct detection, single electron sensitivity, high resolution and fast imaging capabilities based on monolithic CMOS technology.

Large surface (~1 → 4 cm^2), rad-hard monolithic CMOS pixel imager with fast readout (up to 400 frames s^{-1}), for deployment at TEAM microscopes, inheriting R&D originally intended for ILC applications;

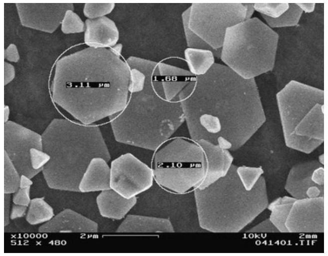
Single e- sensitivity;
< 10 μm Point Spread Function;
Radiation tolerant.

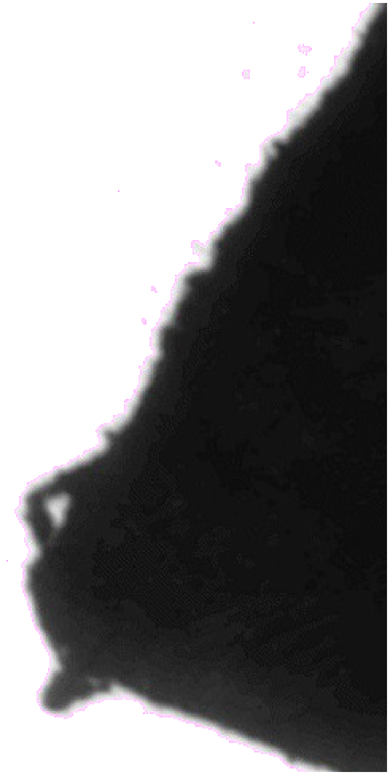


Nano characterisation of dynamics and mechanisms of reactions:
- formation and growth of materials
- visualise catalysis
- reduce beam-induced motion/damage of biological samples.

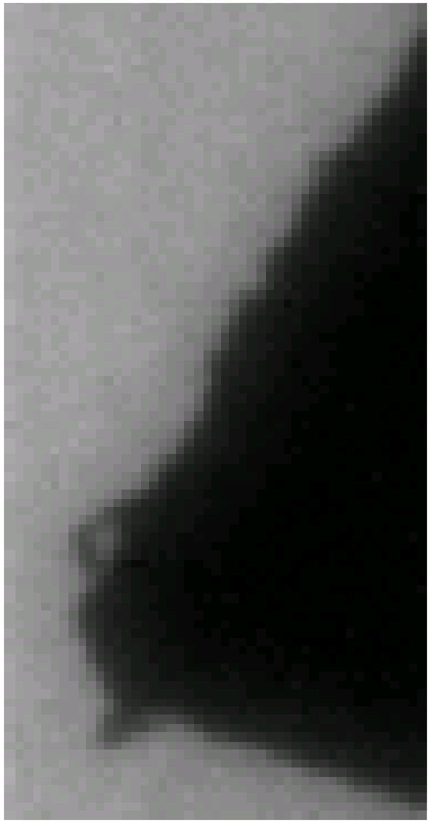
From Still Pictures to Dynamical NanoImaging

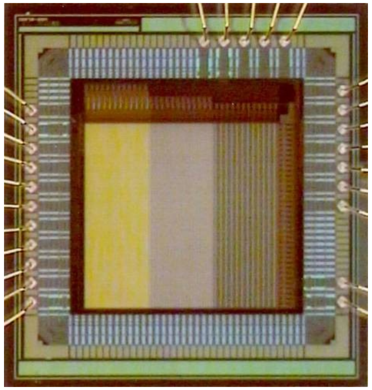
Film

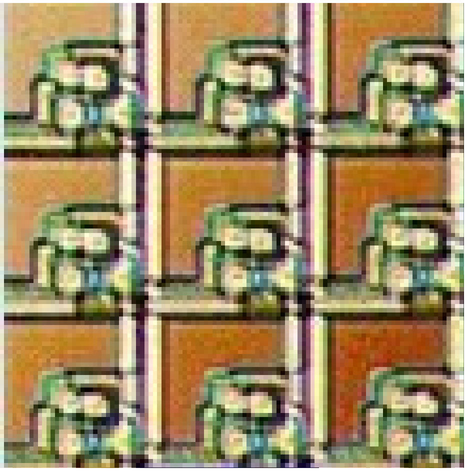




CMOS Pixels







10 μm
CMOS Pixels

Fast CMOS Pixel Imager for TEAM

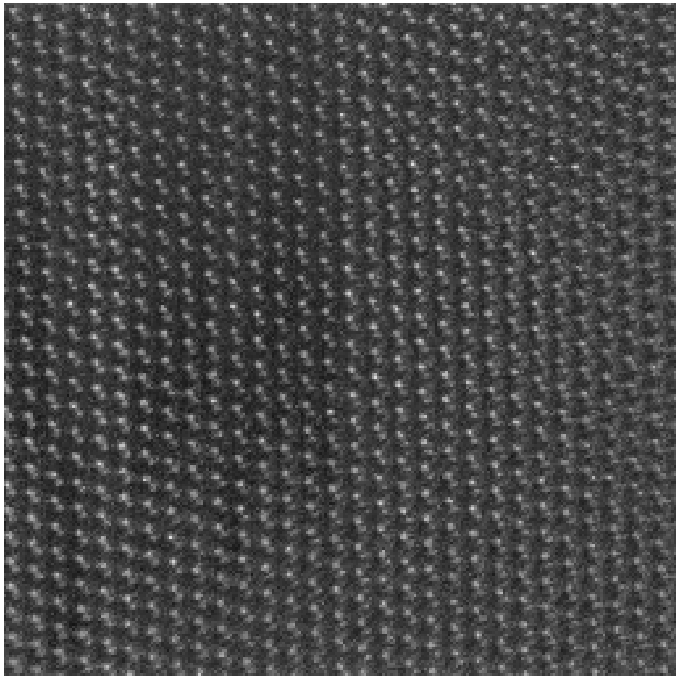
<u>TEAM1k</u>: CMOS Imager demonstrator

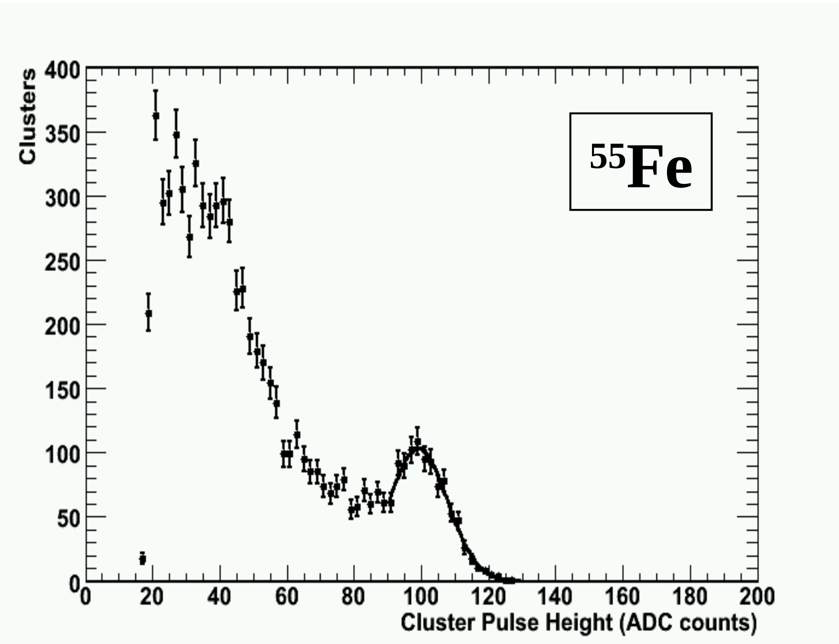
9.5 μm pixels ;

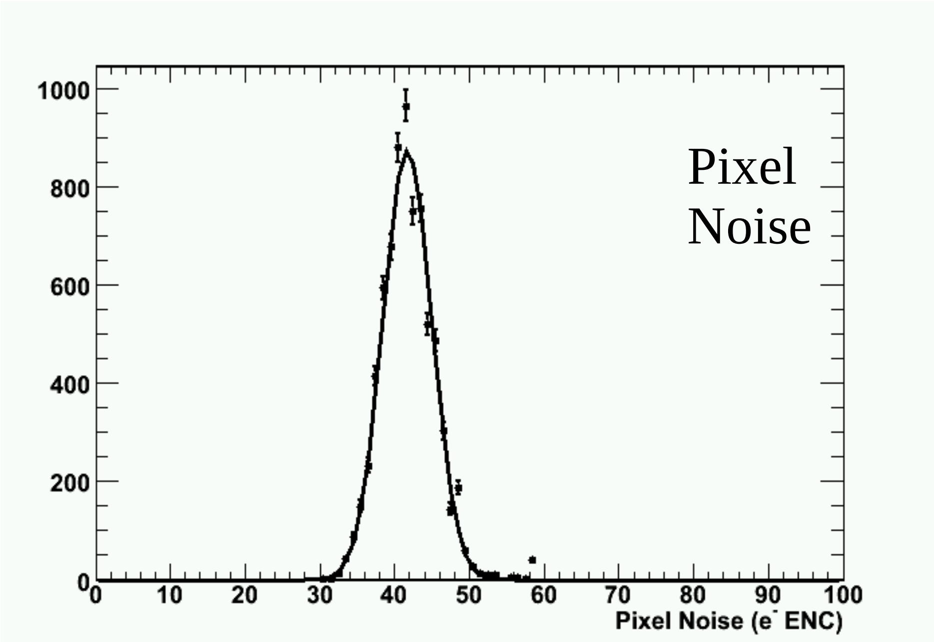
1k x1k matrix ;

fast readout (~500 f s^{-1}) ;

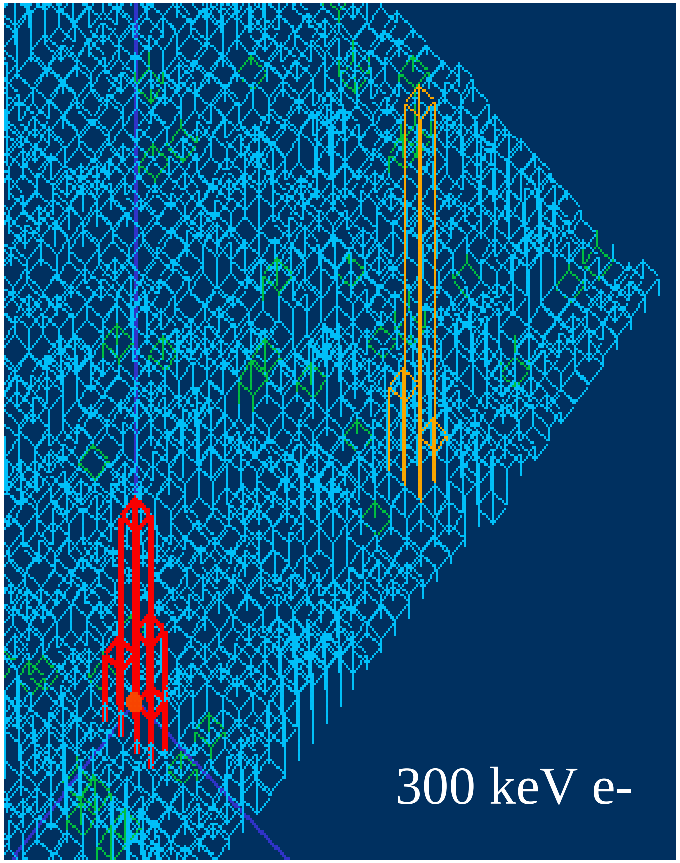
thinned to 50μm

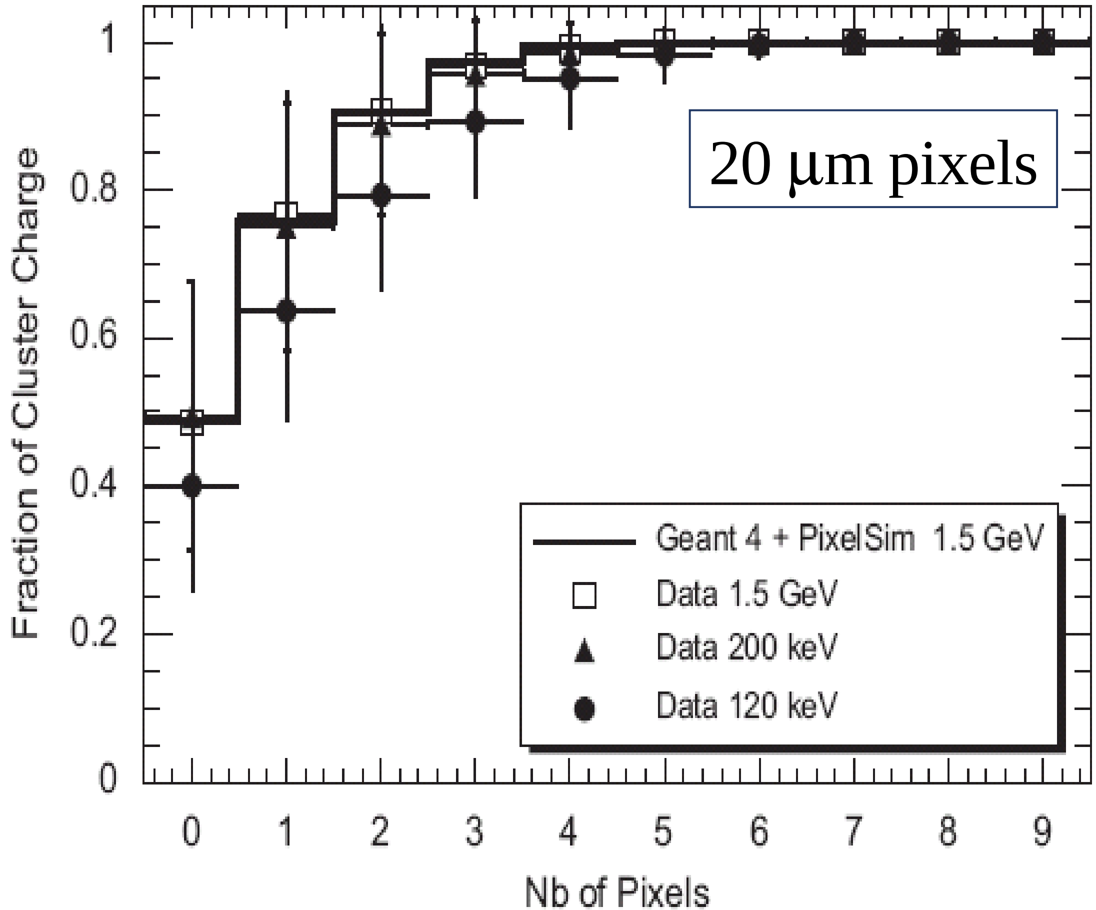






Monolithic CMOS Pixels and TEM Imaging





In HEP charge spread beneficial to improve single point resolution with charge centre interpolation but reduces two-track separation;
in TEM charge spread contributes to point spread function.

Charge Diffusion in epi-layer

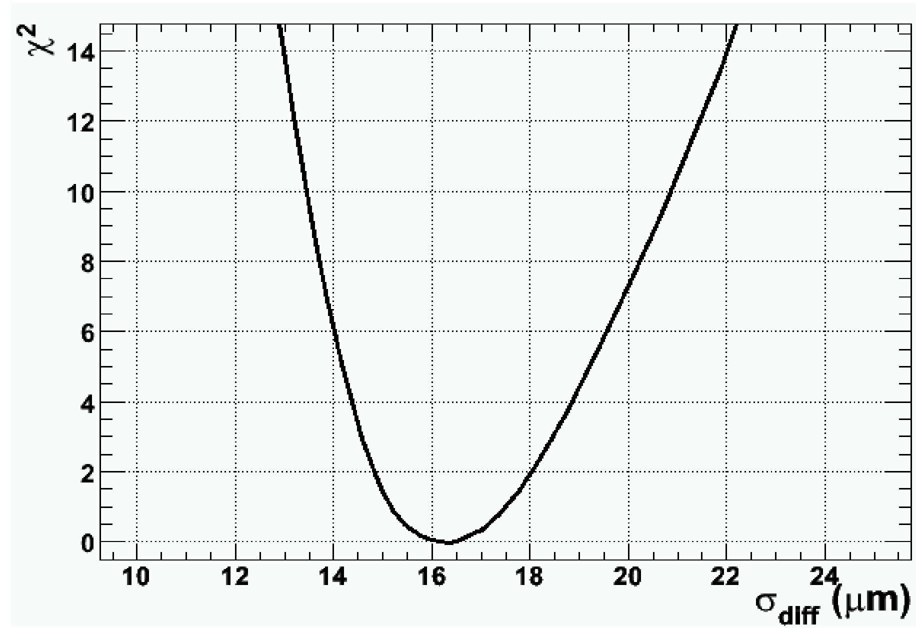
Multiple Scattering in Si

Charge Diffusion in CMOS Pixel sensor

Pixel Multiplicity in Clusters

Extract diffusion coefficient from data by 1-D fit of pixel multiplicity in clusters to G4+pixel simulation varying σ_{diff}

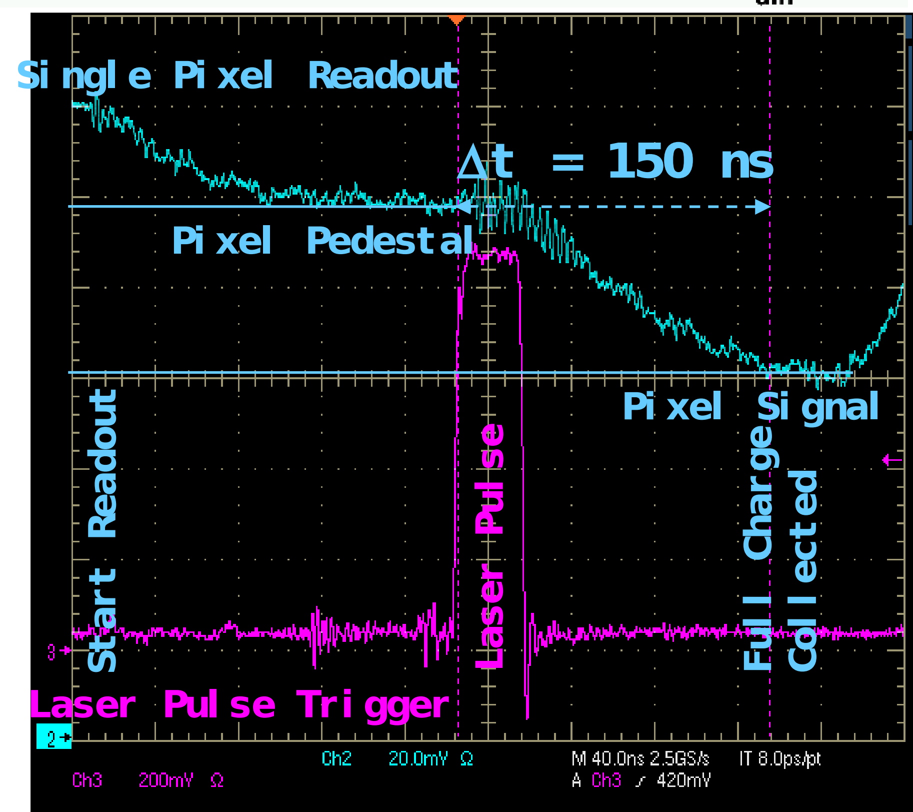
$$\sigma_{diff} = (16.4 \pm 1.5)\,\mu m$$



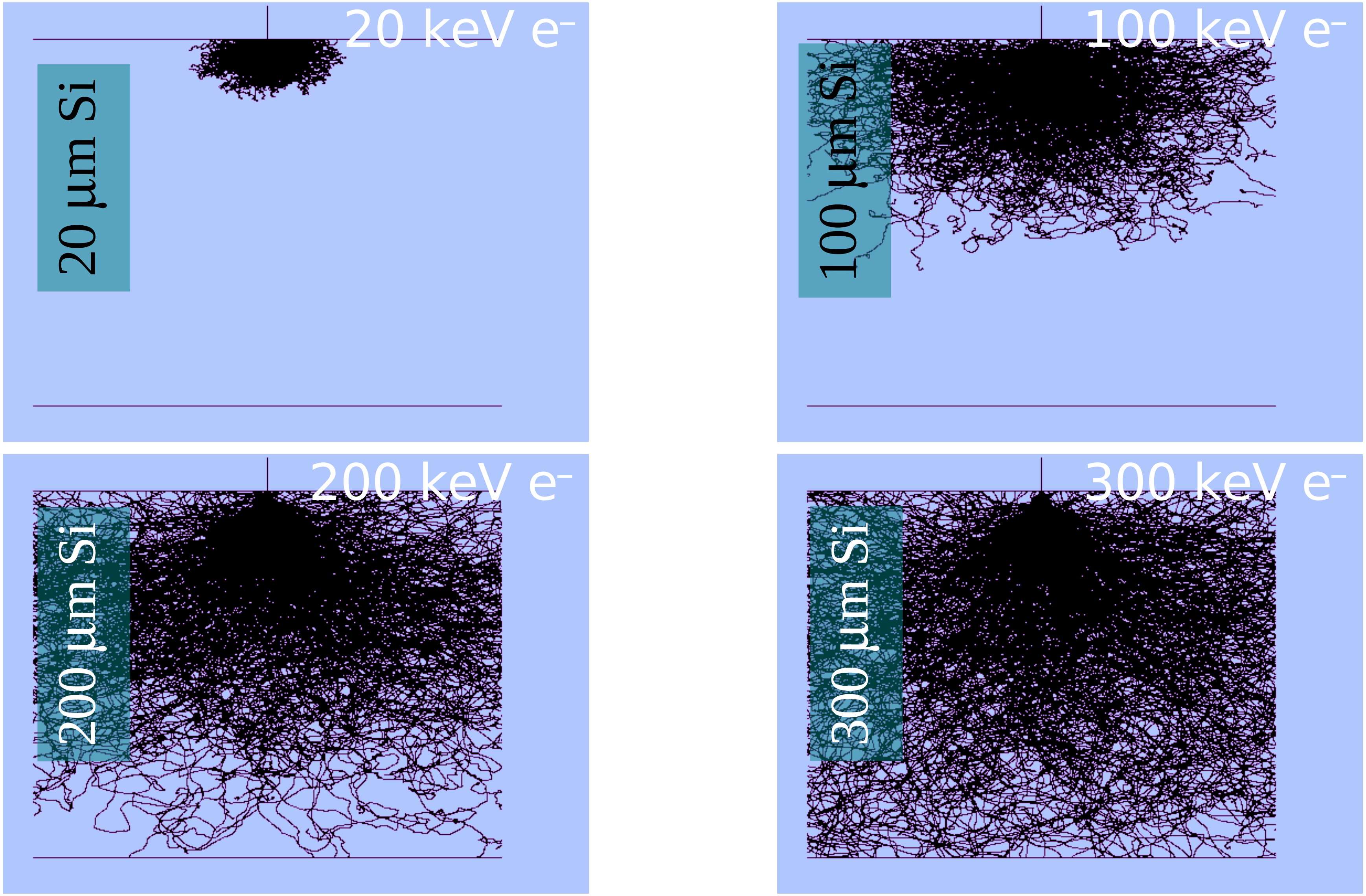
Charge Collection Time

Charge collection time
$\Delta t \sim 150$ ns

Charge diffusion length $L_n = \sqrt{D_n \tau_n}$
$L_n \sim 14\text{-}19\ \mu m$

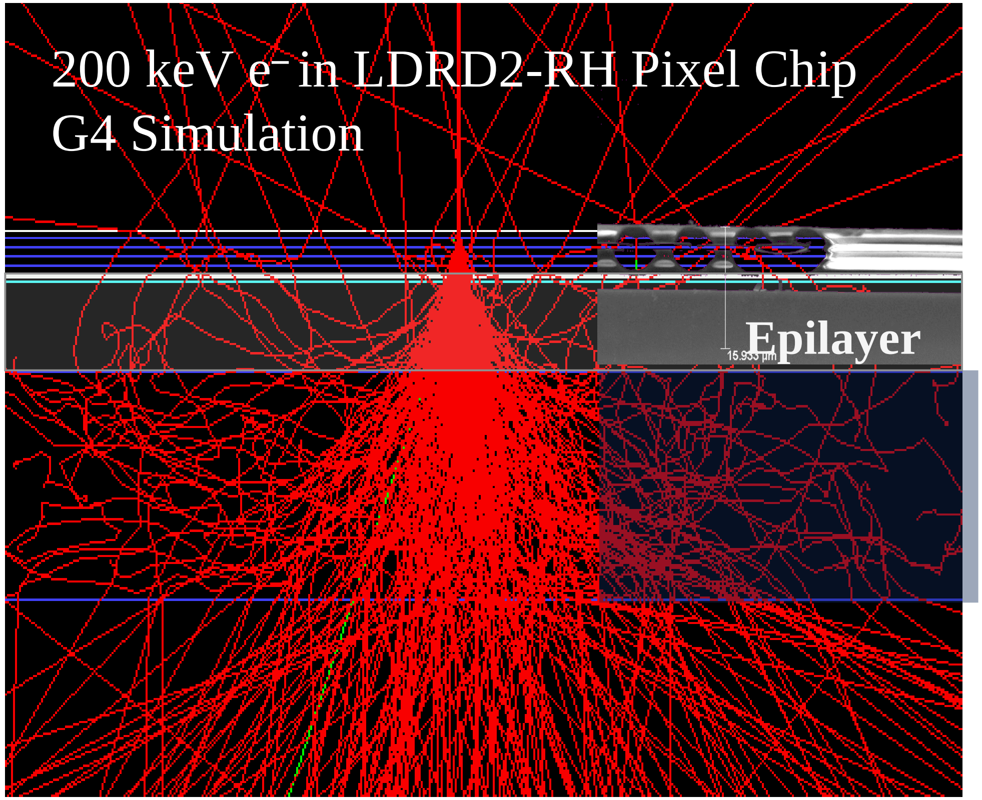


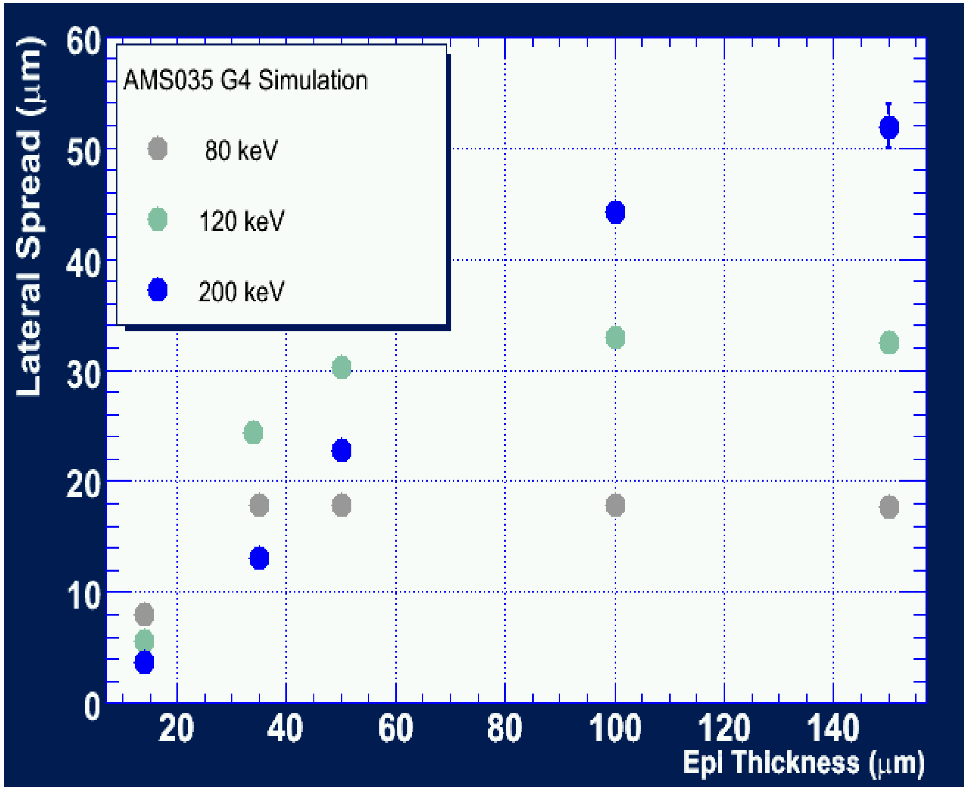
Multiple Scattering



Multiple Scattering and Thin Sensors for TEM

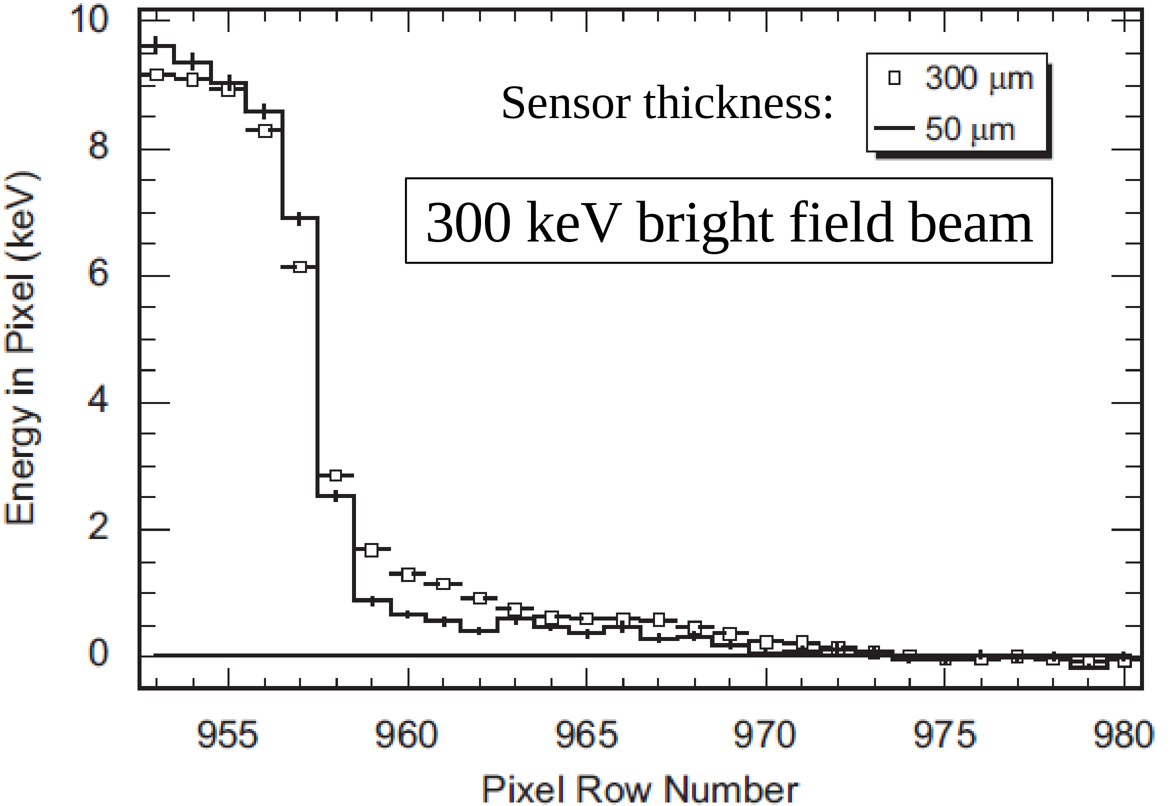
Thin sensitive layer to minimise scattering contribution to PSF;
Thin chip to minimise backscattering:





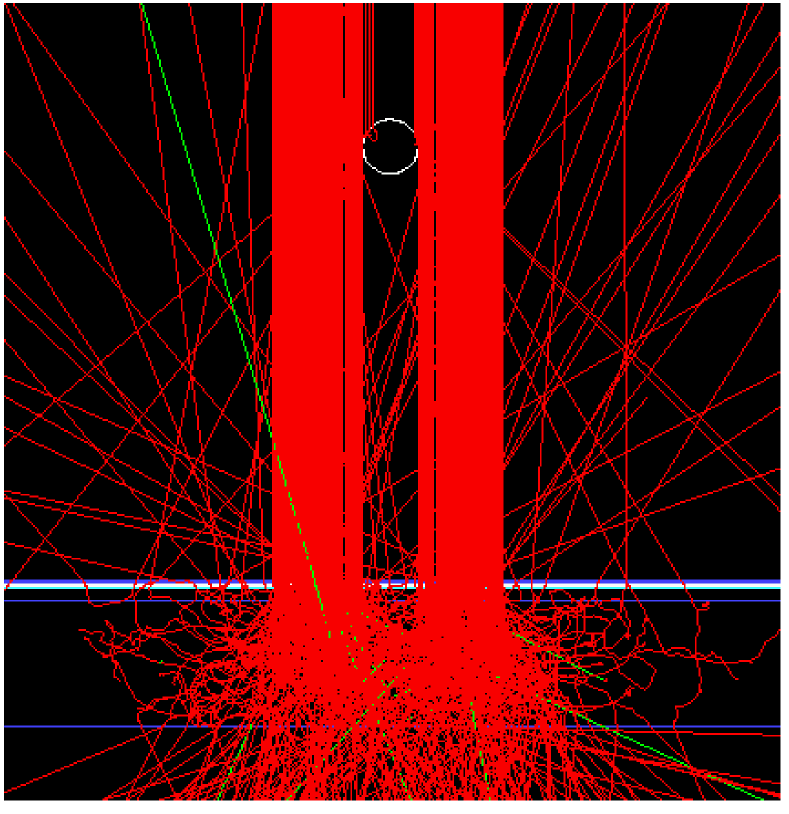
Multiple Scattering and Thin Sensors for TEM

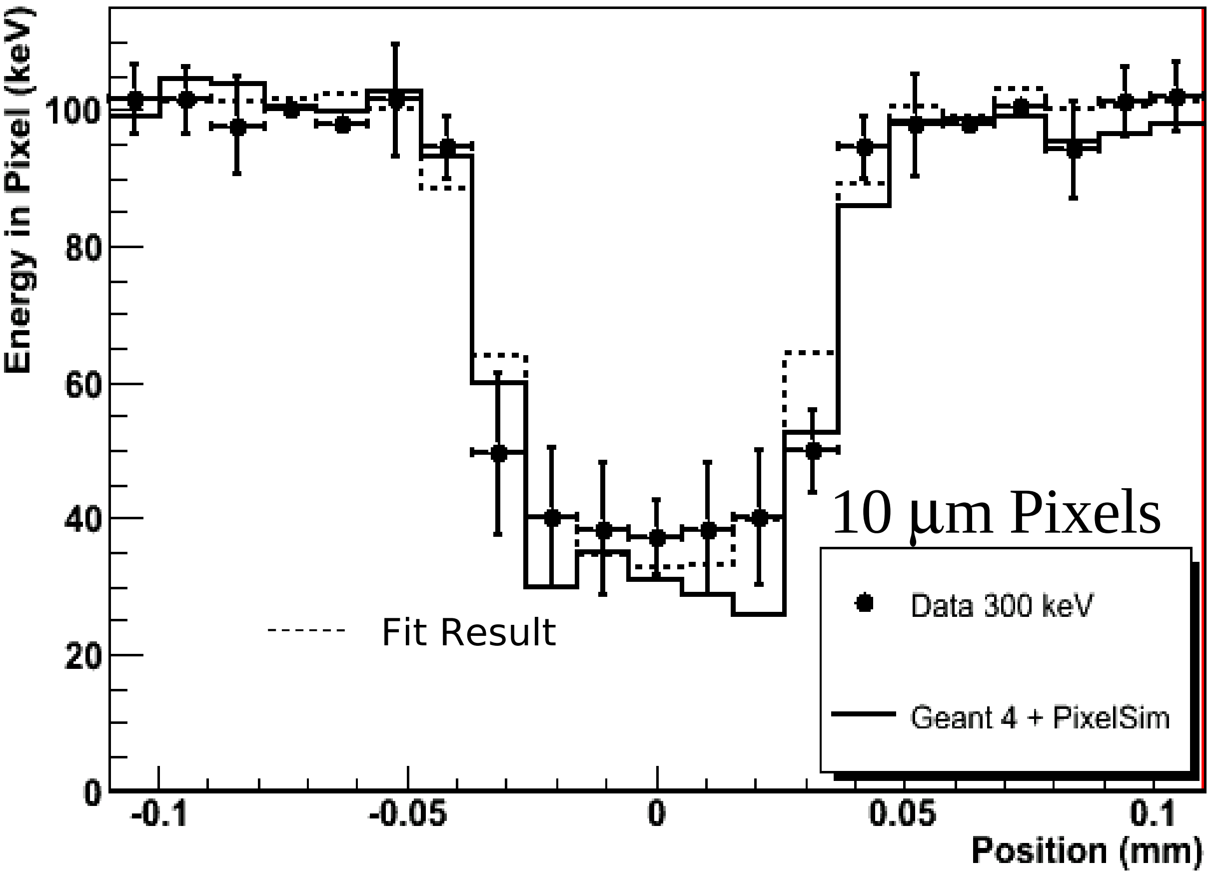
Pulse height across shadow of beam stop edge
on 300 and 50 μm thick CMOS sensor



	300 μm thick	50 μm thick
Contrast Ratio	2.95	4.49
LSF	10.4 ± 0.5	7.7 ± 0.4

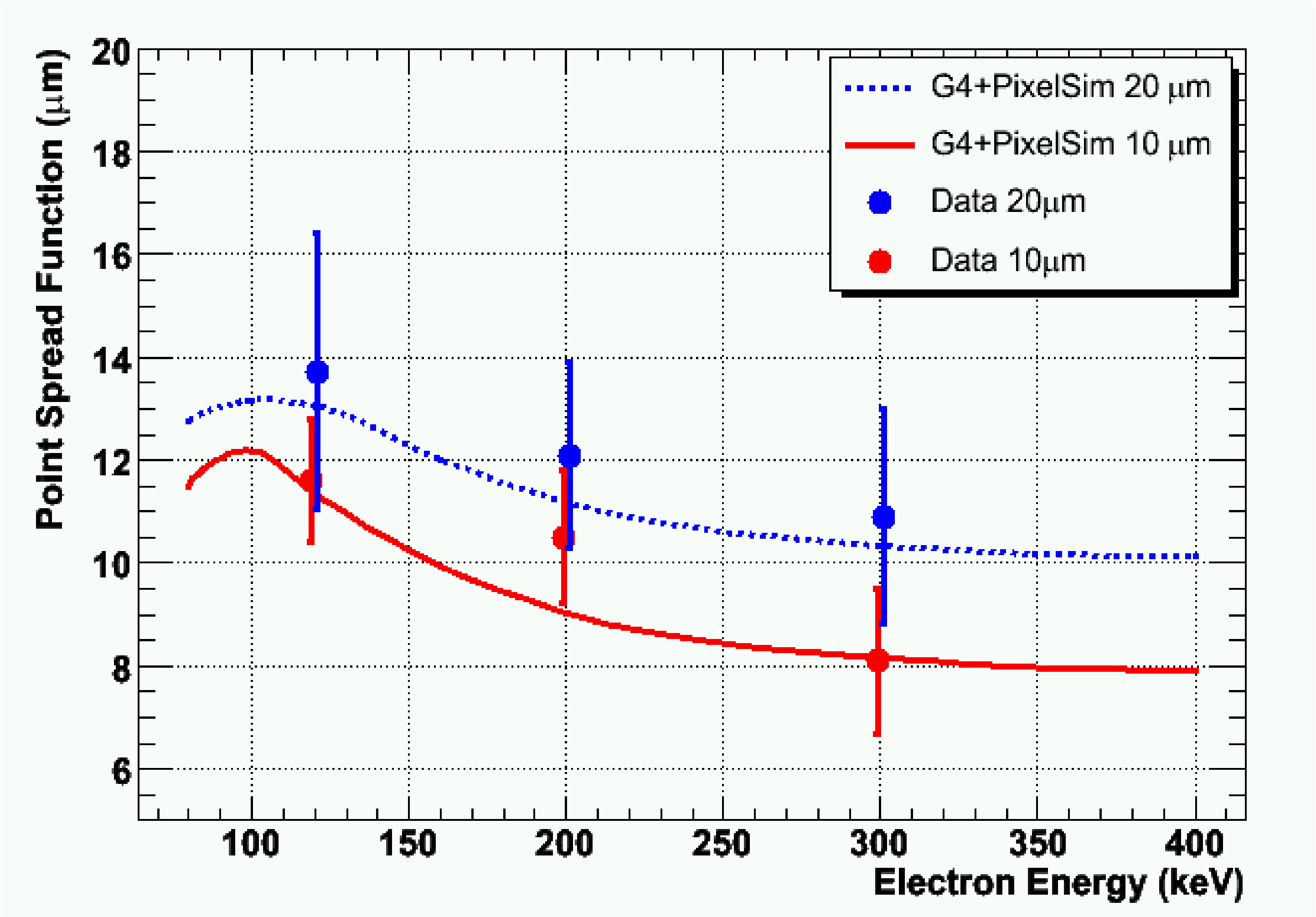
Point Spread Function Determination





1-D Fit of box function folded with Gaussian of free width to data points.

Point Spread Function vs Energy and Pixel Size

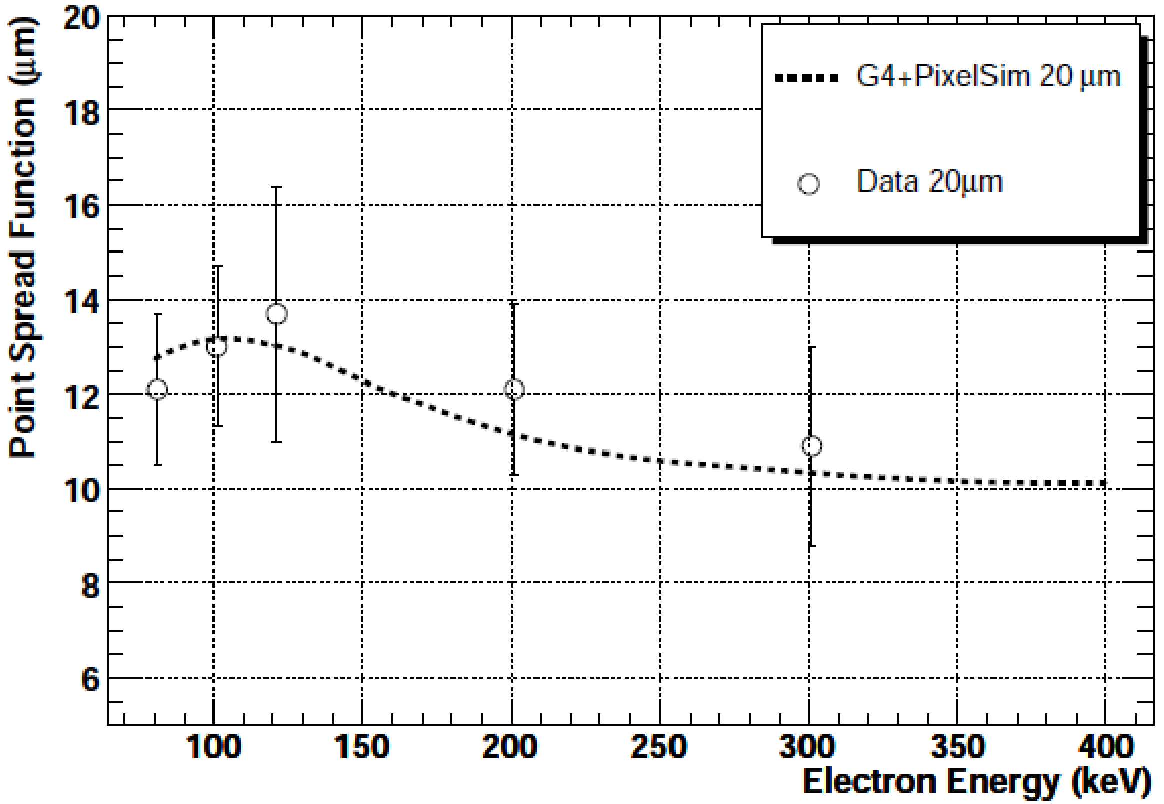


PSF < 10 μm with 10 μm pixels at 200-300 keV

$$\mathrm{DQE} = 0.78 \pm 0.04 \quad \text{and} \quad 0.74 \pm 0.03$$
$$80 \leq E_e \leq 300\,\mathrm{keV}$$

Low Energy Response

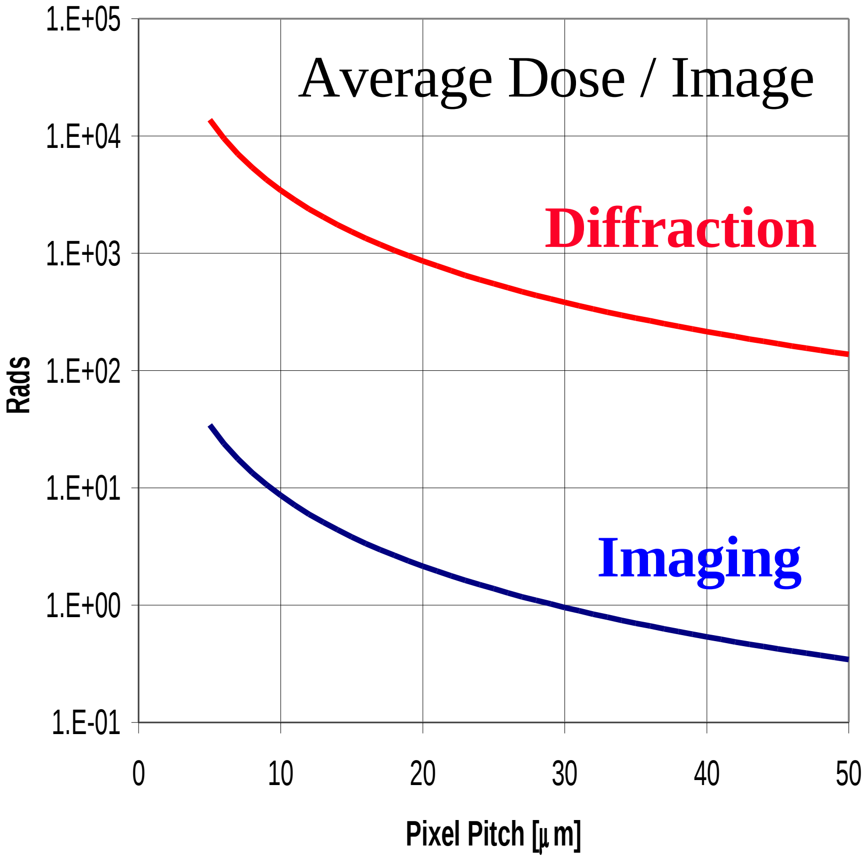
Displacement damage threshold scales as $\sqrt{E}$.
Significant interest in TEM at 80-100 keV for organic samples:
Maximum energy transfer to C atom by 80 keV e^- = 15.6 eV < threshold for knock-on damage to C atom:



Degradation of PSF with decreasing energy reaches plateau around ~120 keV due to decrease of e- range with decreasing energy; prediction consistent with data at 80 keV.

Radiation Tolerance for HEP and TEM

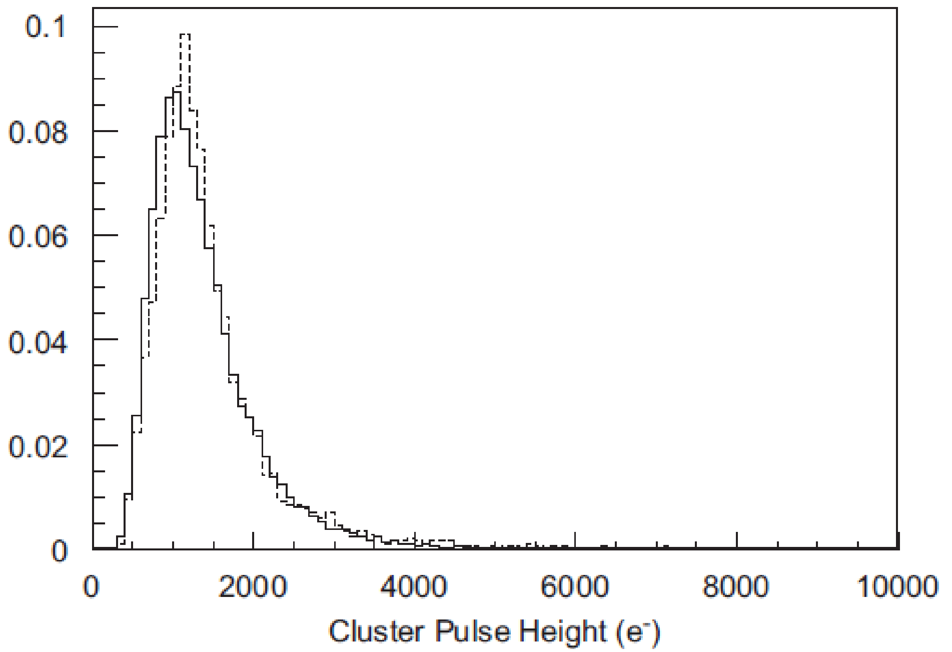
Machine	non-Ionising $n_{eq} cm^{-2} yr^{-1}$	Ionising MRad yr^{-1}
LHC	$1.4\ 10^{14}$	11.3
SLHC	$1.4\ 10^{15}$	71.4
ILC 0.5 TeV	10^{11}	0.05
CLIC 3 TeV	10^{11}	0.07
SuperBelle	$6\ 10^{11}$	0.50
SuperB	$6\ 10^{12}$	~1
RHIC	$3\ 10^{12}$	0.08

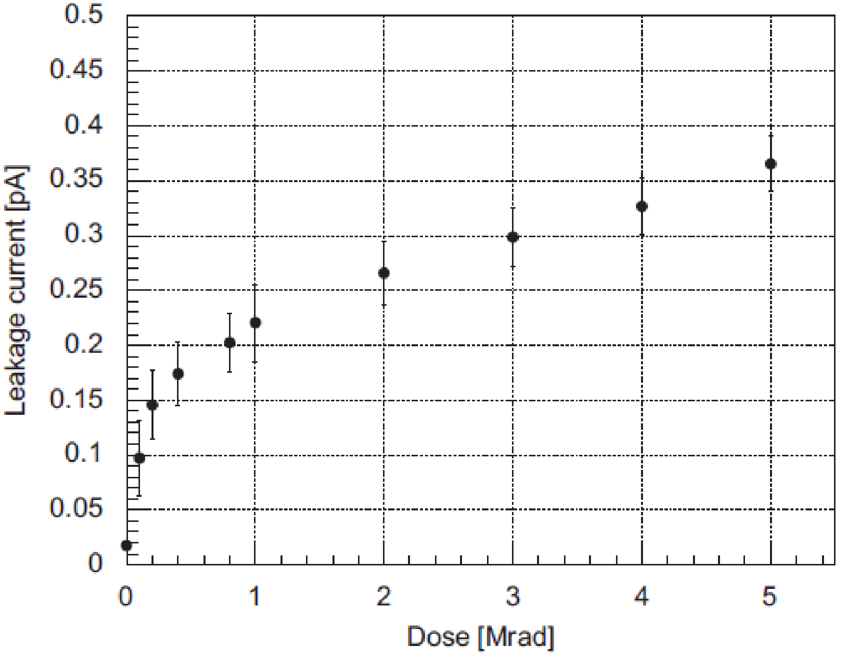


10 e^- $Angstrom^{-1}$

10 rad $pixel^{-1}$ s^{-1} → ~1 MRad yr^{-1}

Response after Irradiation

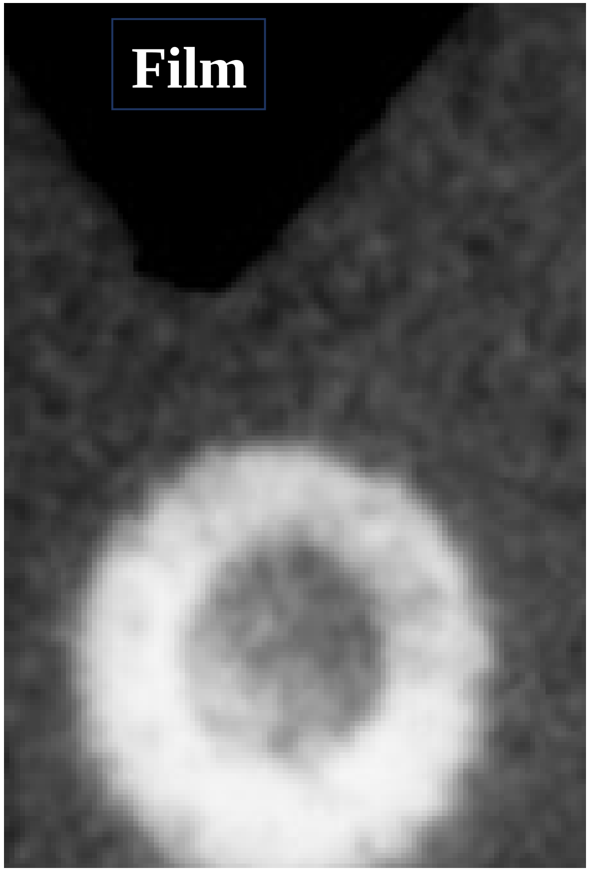


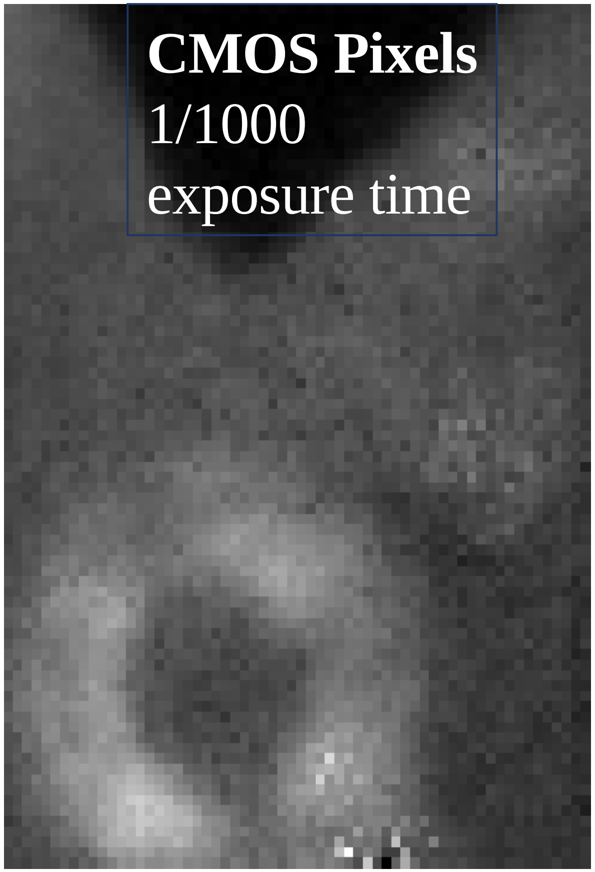


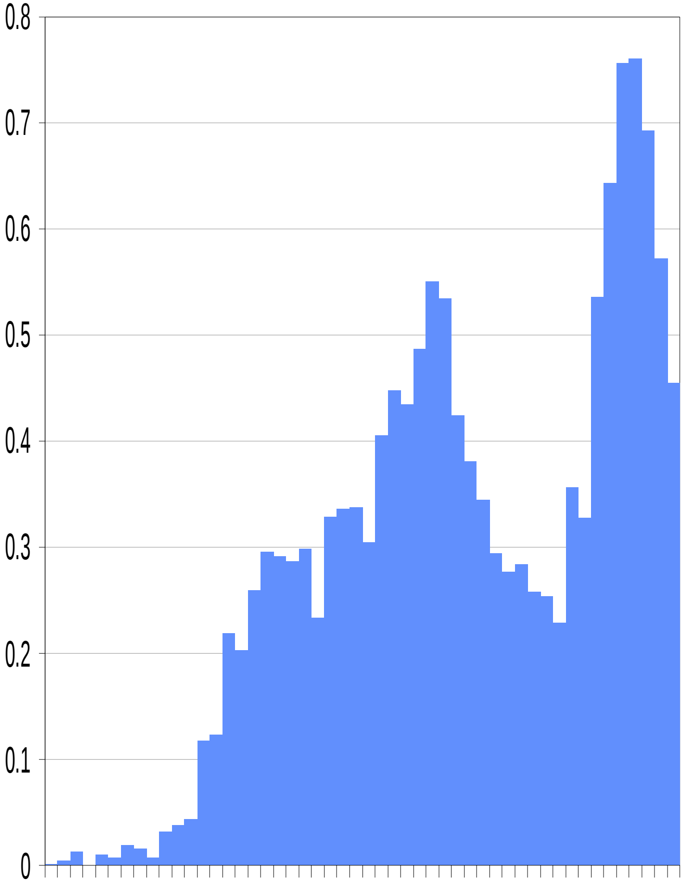
	0 MRad	5 MRad
LSF	$(7.4 \pm 0.6)\,\mu m$	$(7.2 \pm 0.6)\,\mu m$
S/N	12.3	10.9
Dynamic Range		-30%

CMOS Pixel Imaging and Exposure Time

Core-shell precipitates







CMOS imager (10 μm pixels), 200 keV e^-
dark field image, magnification 20000

Cluster Imaging and Single Particle Microscopy

In traditional bright field illumination electron flux large enough that each pixel is illuminated by at least one electron per frame;
Due to charge diffusion signal recorded superposition of direct charge on pixel and diffusion from neighours;

Build images from sum of multiple exposure at low enough flux that single clusters can be reconstructed; sampling frequency pixel pitch → cluster resolution;

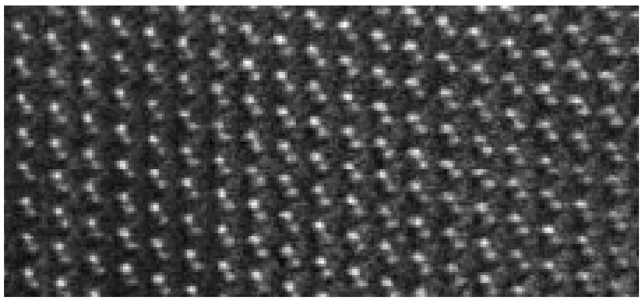
Imaging of biological samples with high resolution is problematic because:
Low threshold for C atom knock-on: sample suffers radiation damage;
Low contrast due to small Z of sample atoms:

Single-particle EM imaging:
averaging, sample motion correction, MVA to get 3D images;

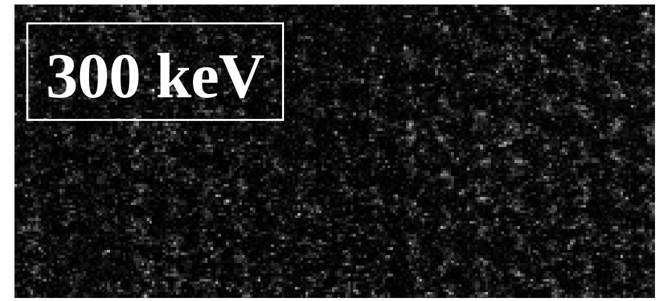
Boekema et al., Photosynt. Res. 102 (2009) 189
Liao et al, Nature Methods, 10 (2013) 584

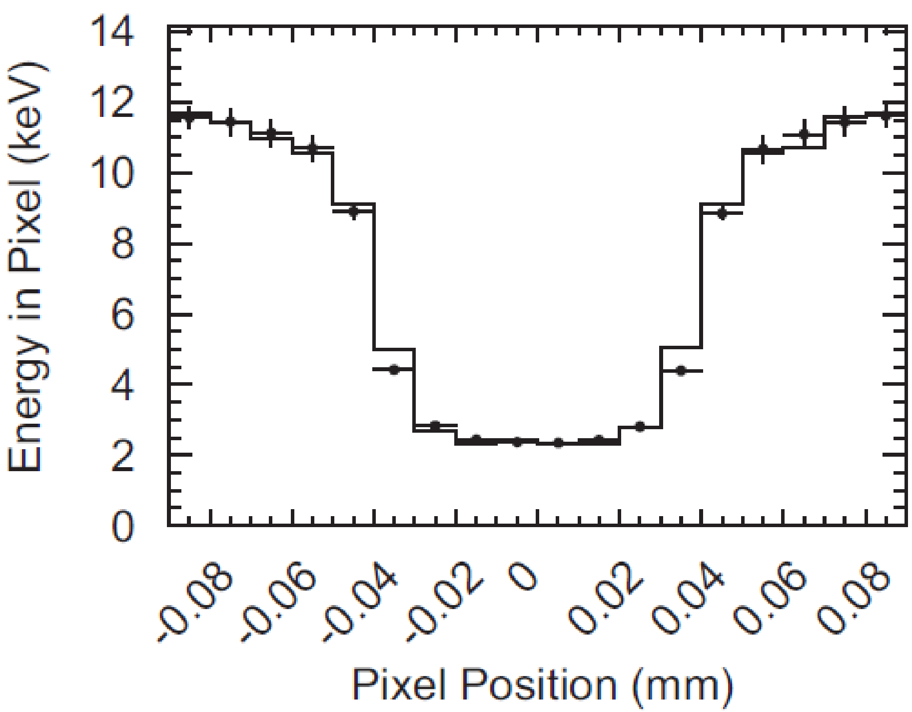
Cluster Imaging

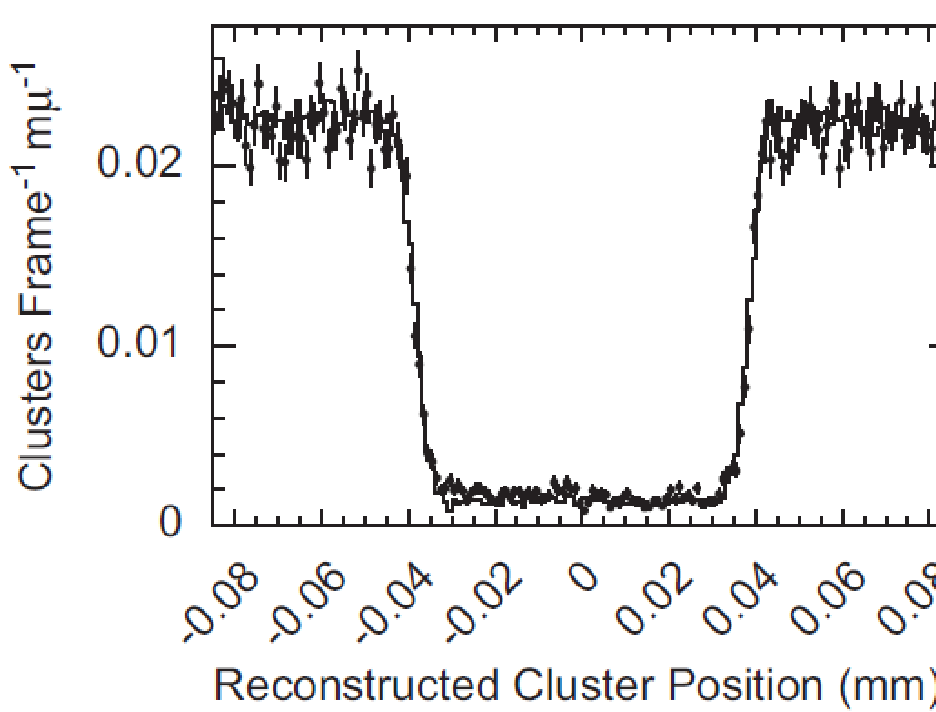
5 x 10^3 e^- mm^{-2} $frame^{-1}$

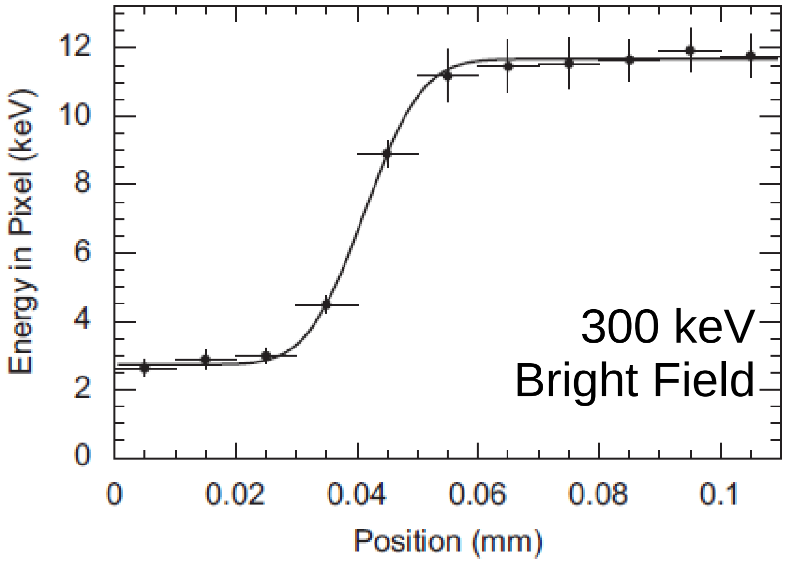


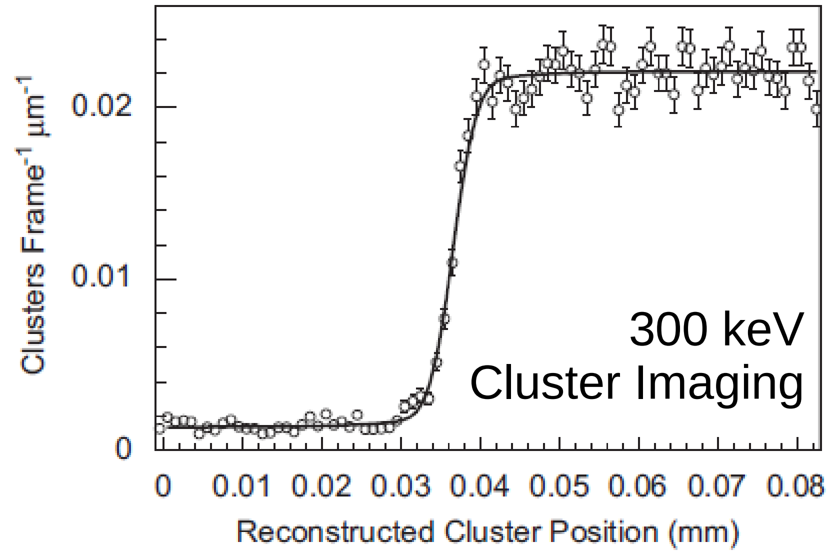
5 x 10^1 e^- mm^{-2} $frame^{-1}$

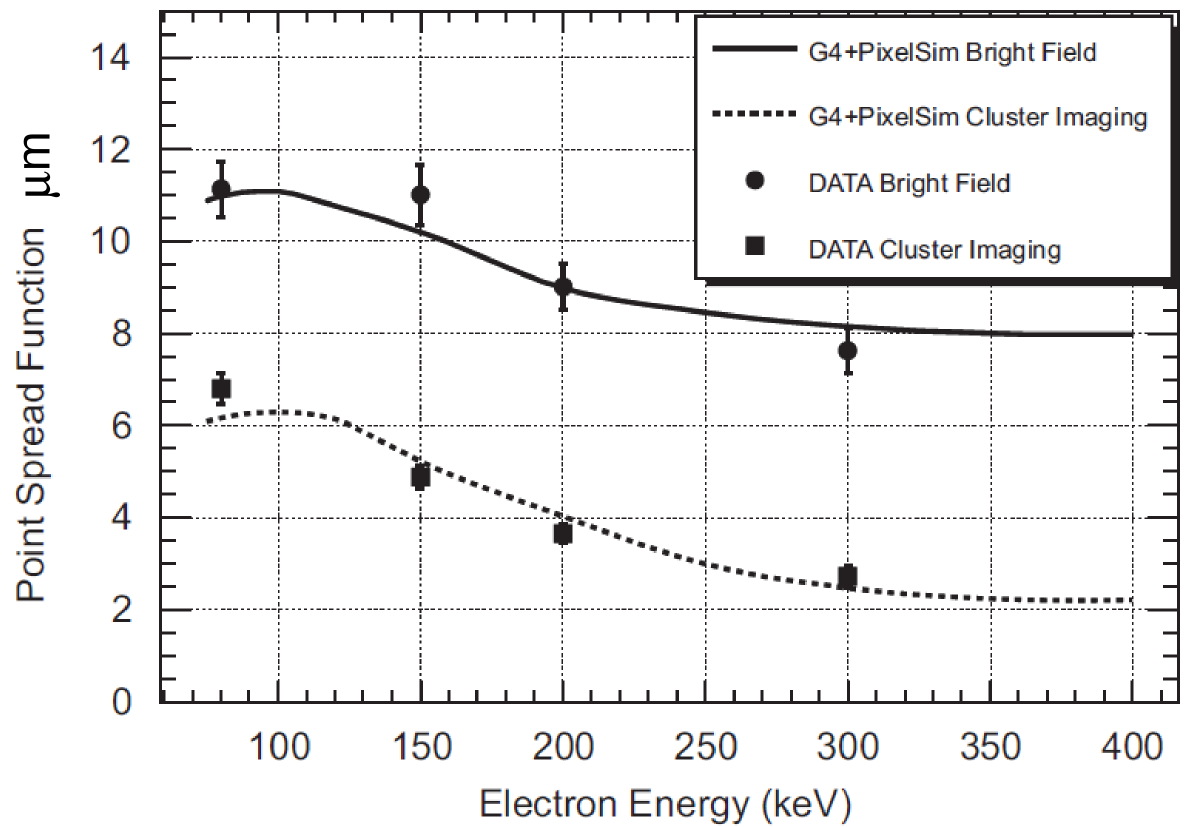








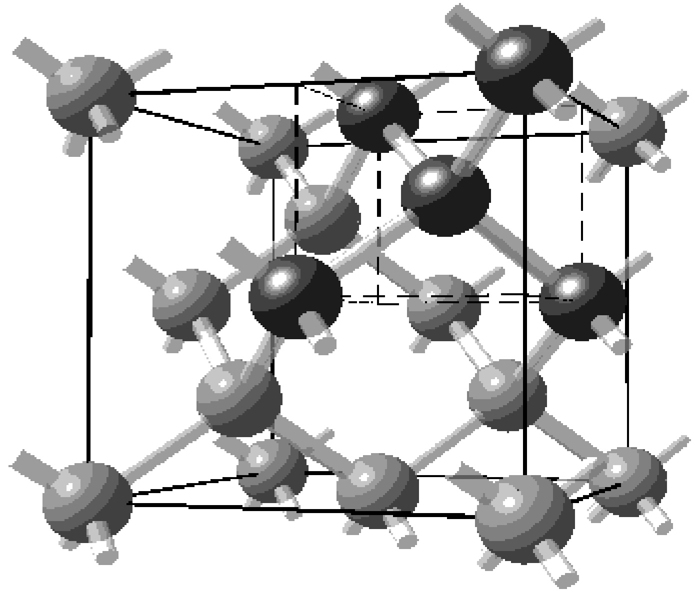


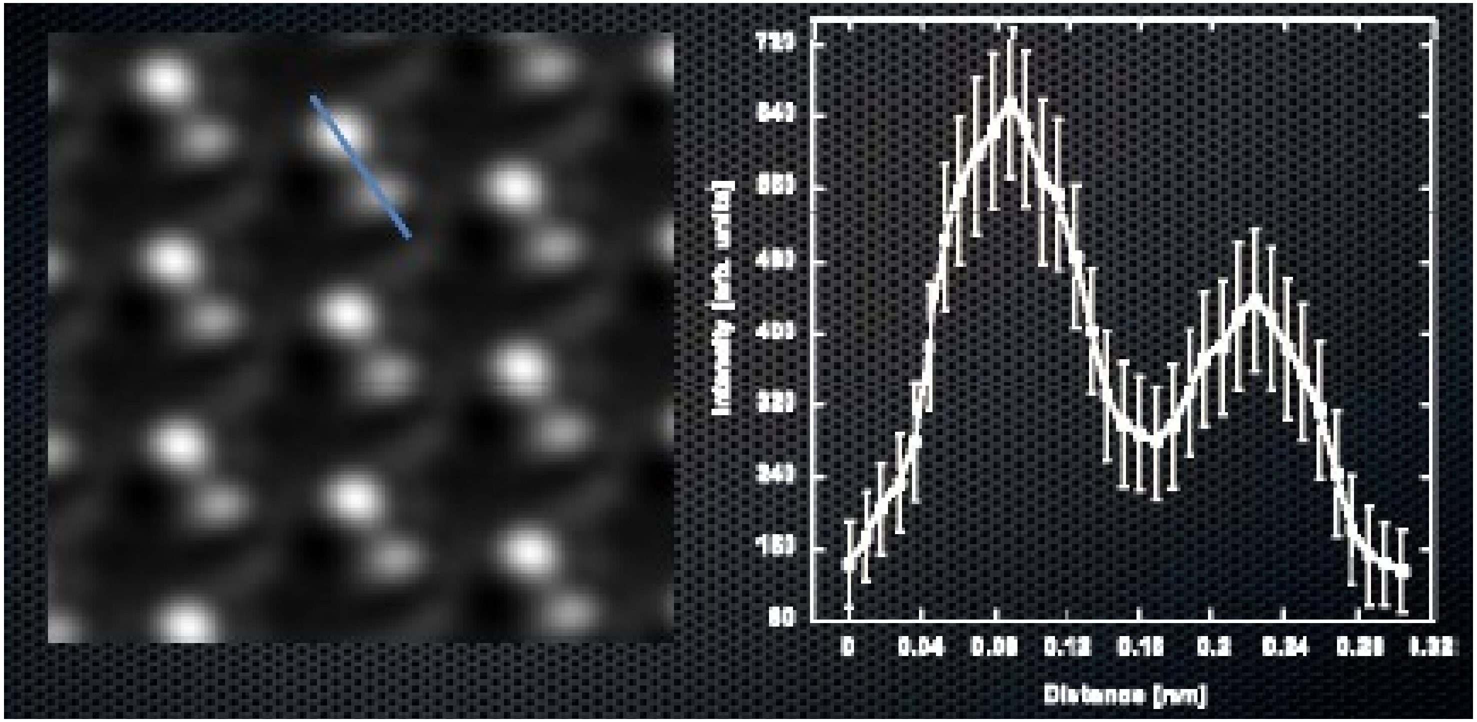


Bright Field

Cluster imaging

Si(111) Dumbbells at TEAM-1



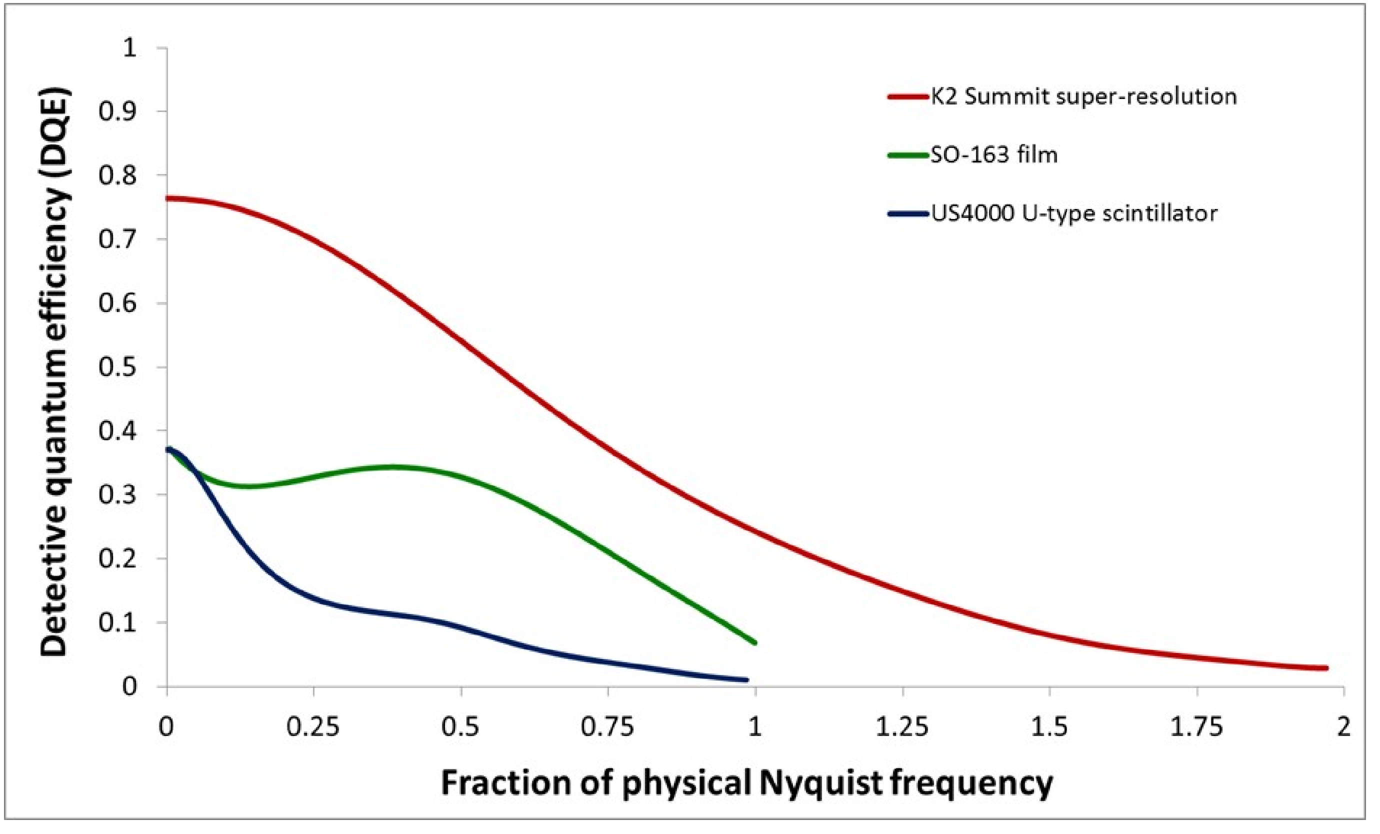


Commercial Gatan K2 Camera

4k x 4k pixels;

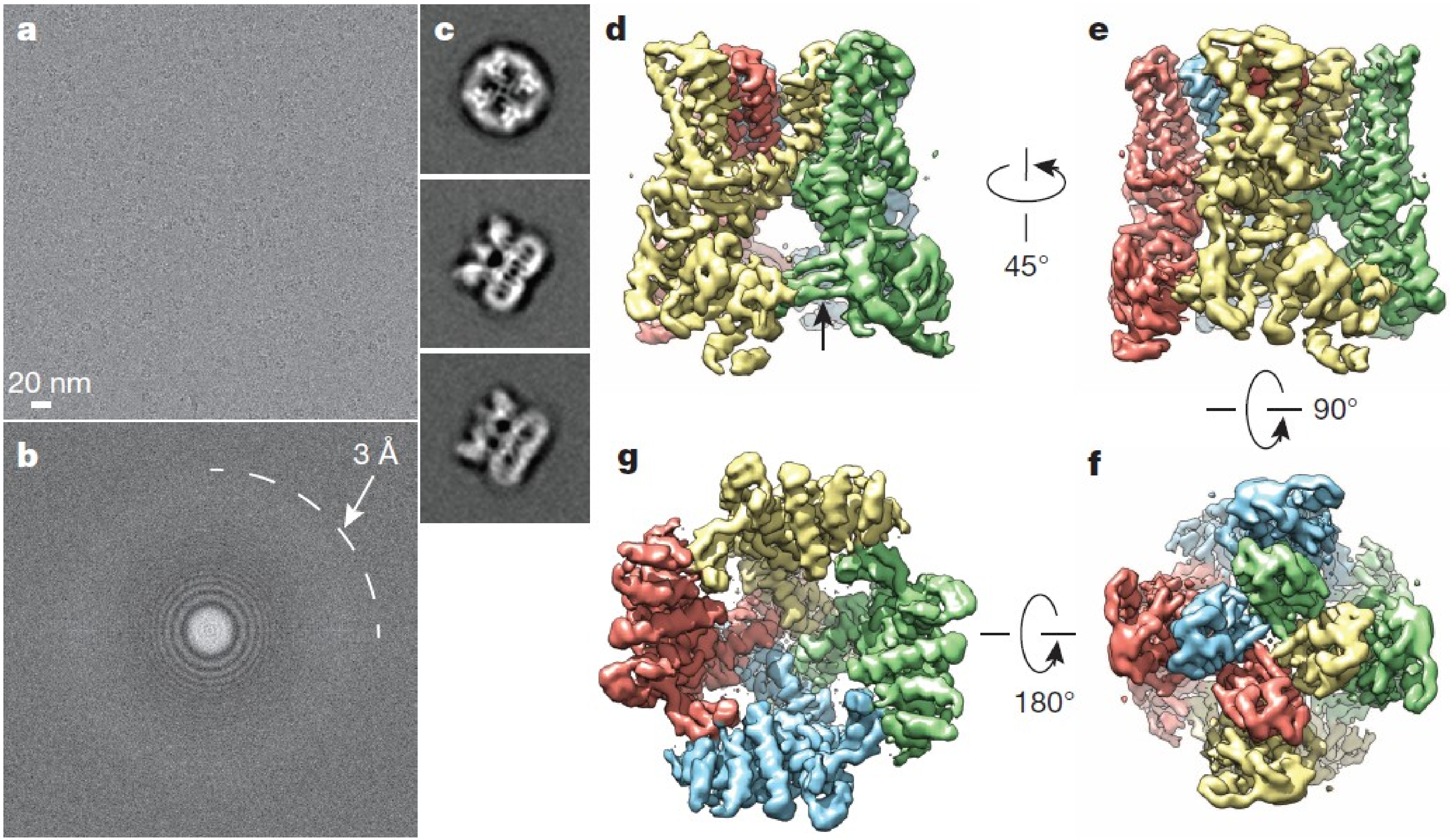
5 μm pixel pitch;

Sensor read-out at 400 f/s



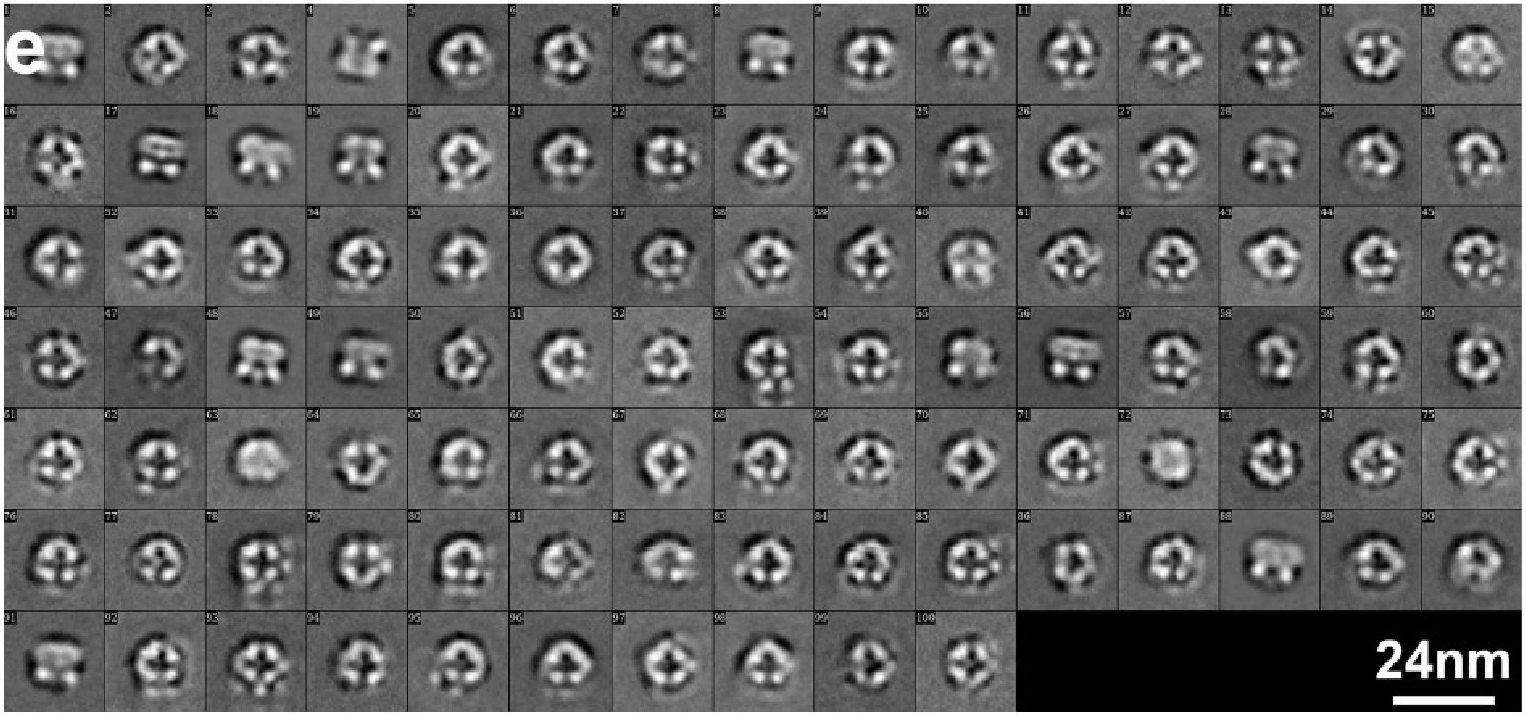
Gatan data

Cellular receptor channel structure studied at atomic level using Cryo-EM and direct detection CMOS pixels

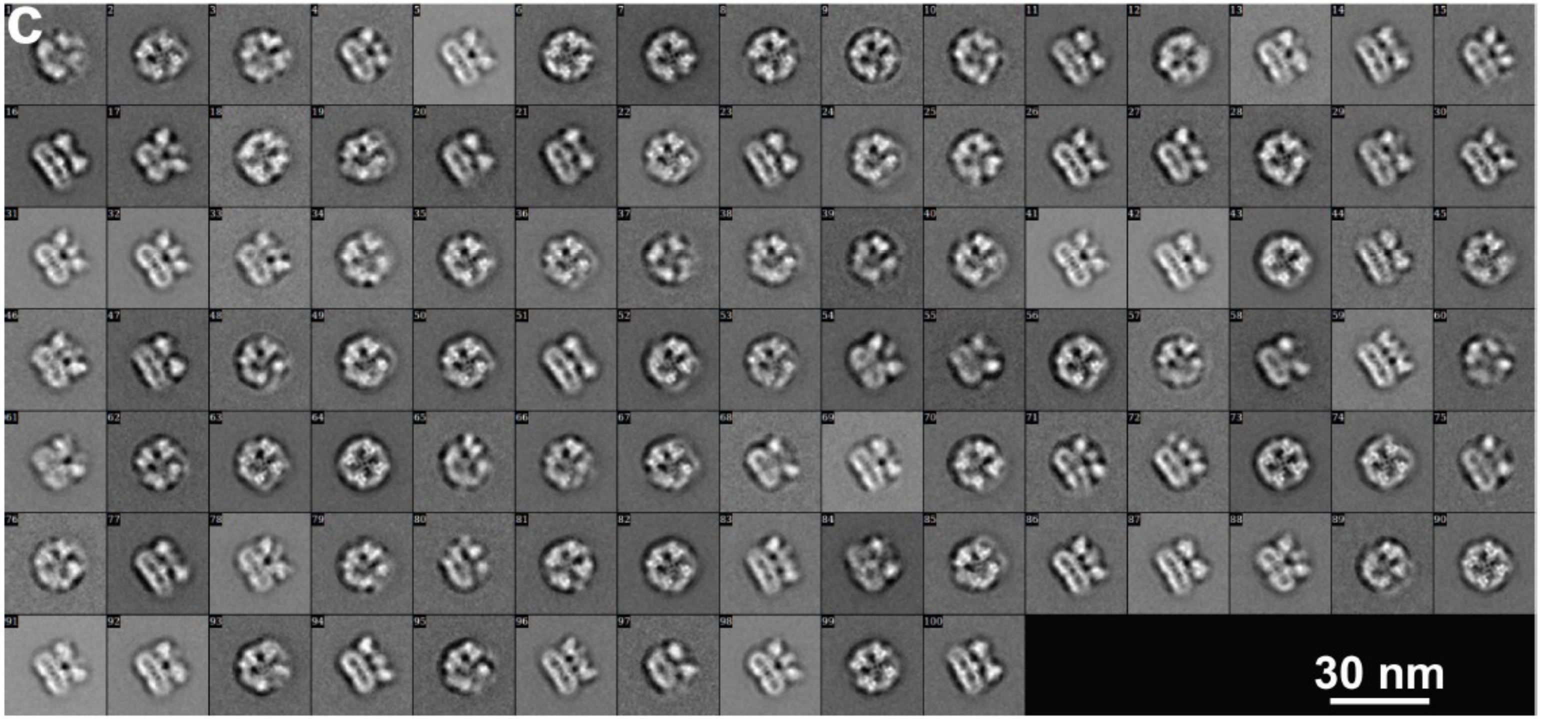


Liao et al.
Nature 504 (2013)

Fiber-coupled CMOS



Direct detection CMOS



Liao et al.
Nature 504 (2013)