CMOS Active Pixel Sensors as Fast, High Resolution Direct Detectors for Electron Microscopy

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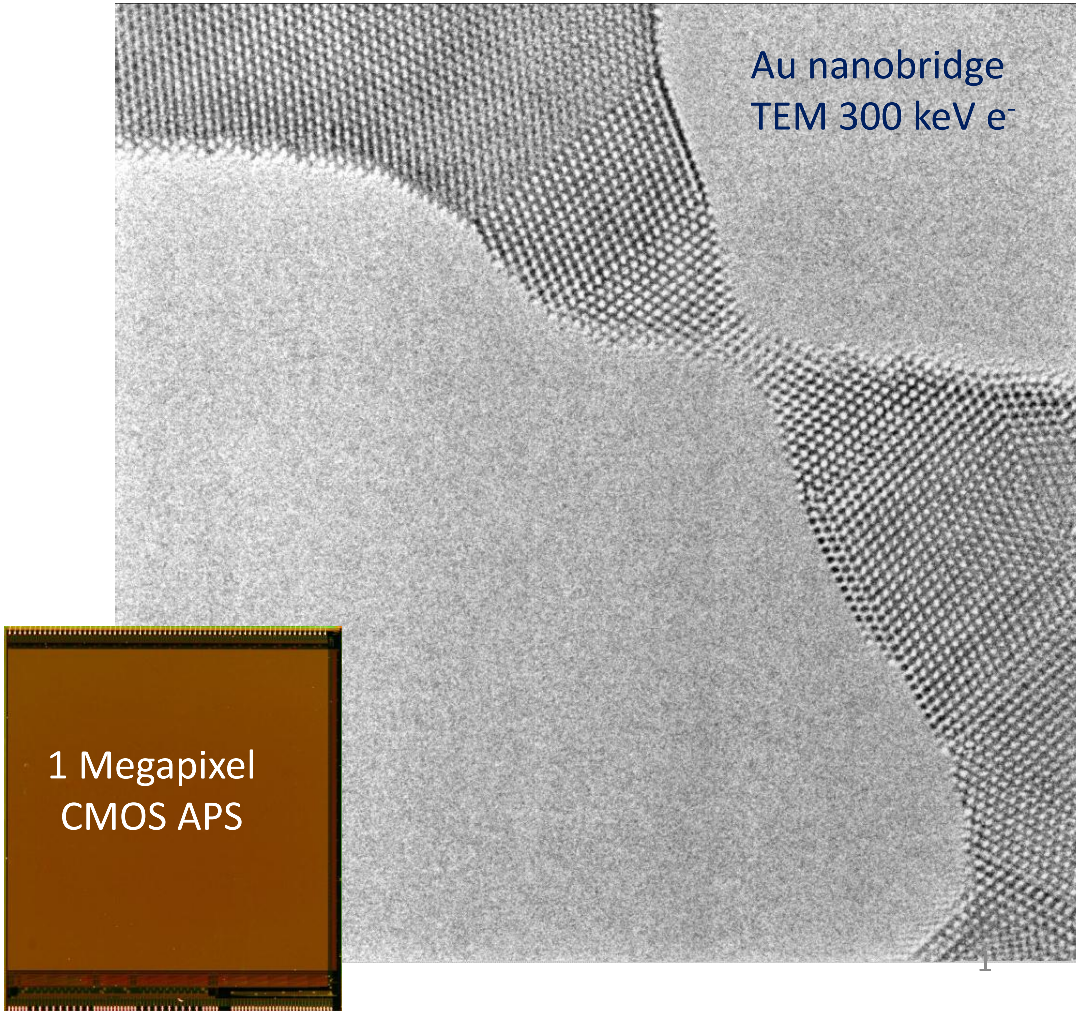
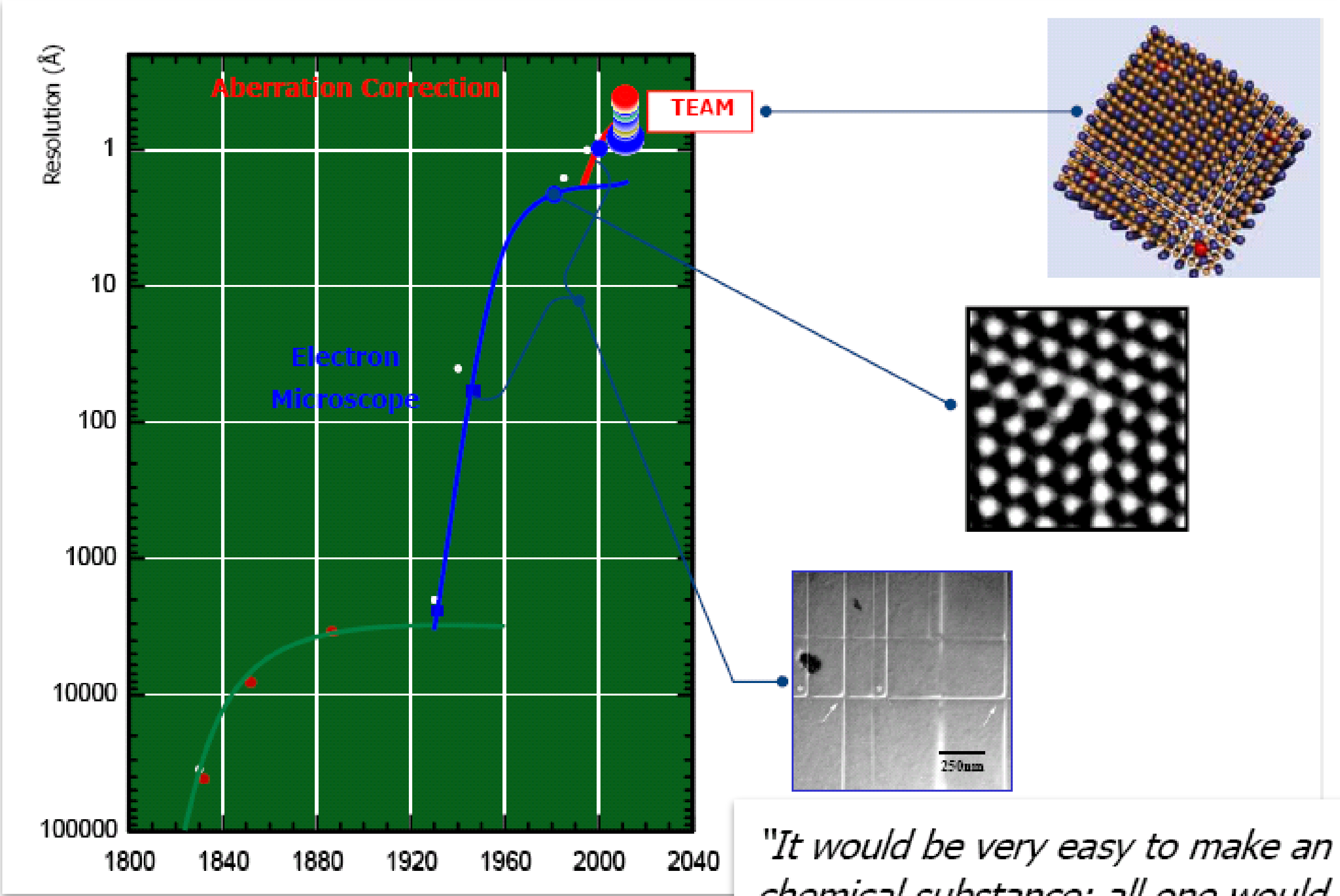
December 5, 2012



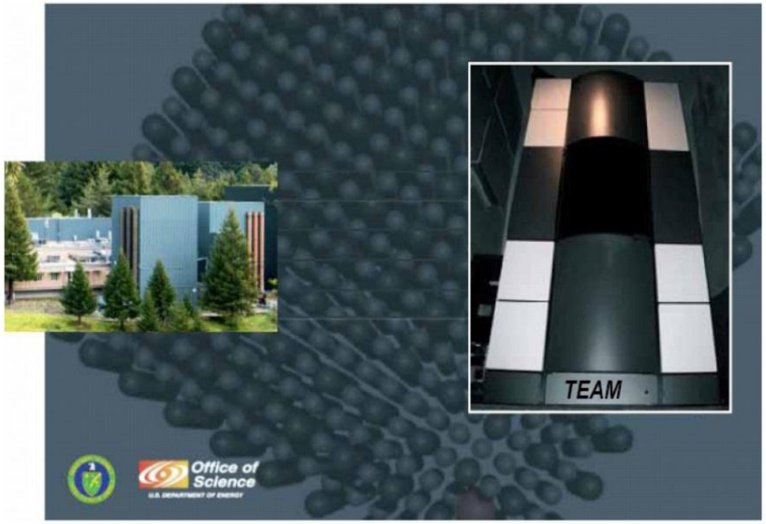
Progress in Electron Microscopy



"It would be very easy to make an analysis of any complicated chemical substance; all one would have to do would be to look at it and see where the atoms are. The only trouble is that the electron microscope is one hundred times too poor … I put this out as a challenge: Is there no way to make the electron microscope more powerful?"

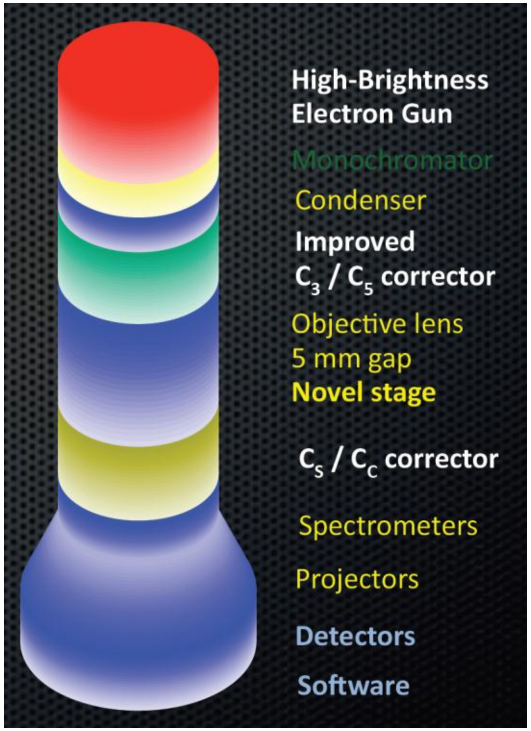
R. Feynman, 1959

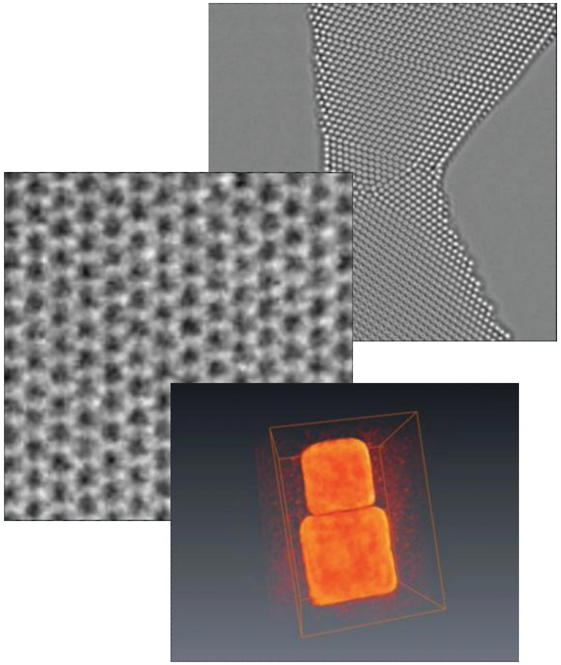
The TEAM Project: Enabling sub-Å Resolution



TEAM: Transmission Electron Aberration-corrected Microscope

http://ncem.lbl.gov/TEAM-project/index.html



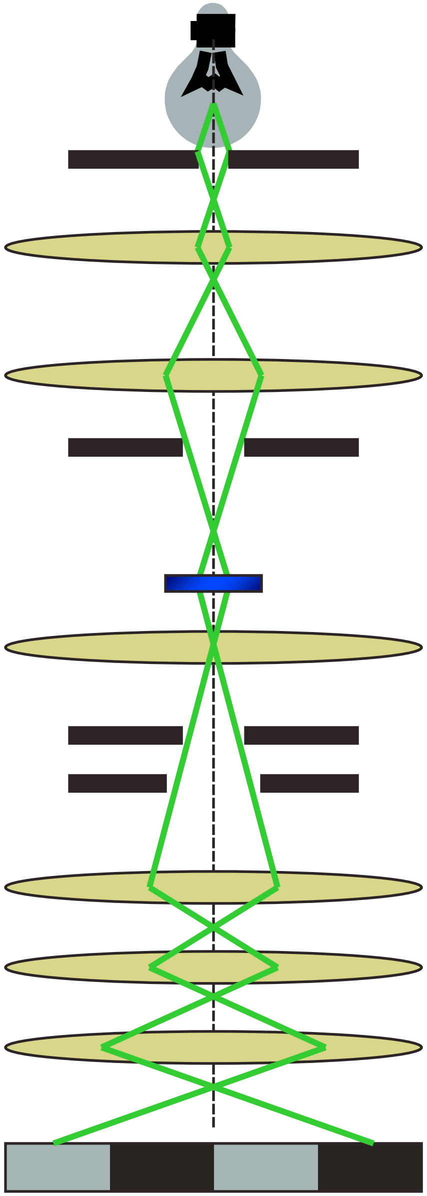


- Funded by the US Department of Energy, Office of Science, Basic Energy Sciences
- 80-300 keV electron energy, 0.5Å spatial resolution TEM/STEM, 0.1 eV δE
- Major advances in electron optics and aberration correction
- Capabilities for 3D atomic-scale tomography and in-situ/dynamics experiments
- Project completed 09/2009, now national user facility

U.S. DEPARTMENT OF ENERGY

Office of Science
U.S. Department of Energy

Imaging in Transmission Electron Microscopy



Electron gun

Anode

Condenser aperture

Sample

Objective lens

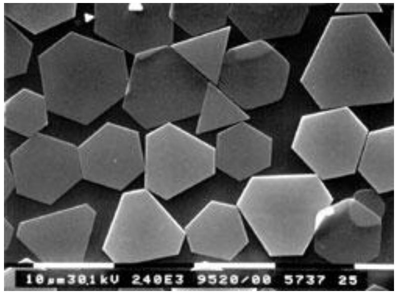
Apertures

Magnification and projection

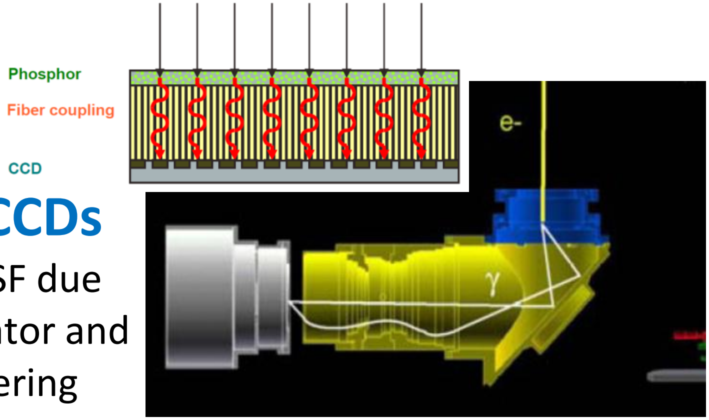
Detector

Film

- Large area
- High granularity
- Slow, no dynamic imaging



Optically Coupled CCDs

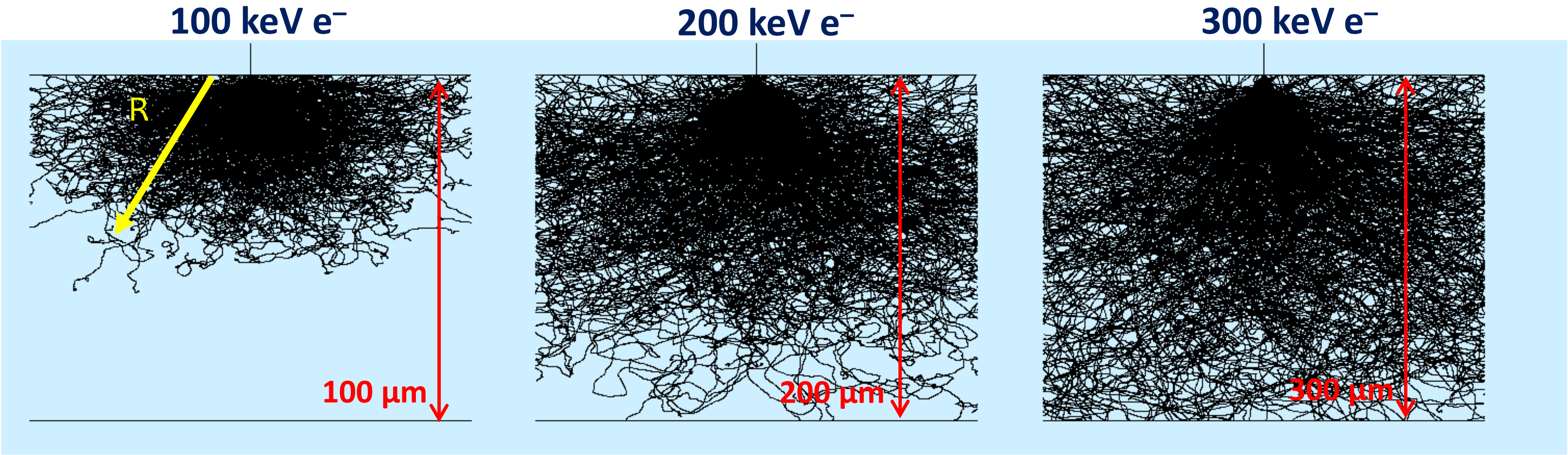


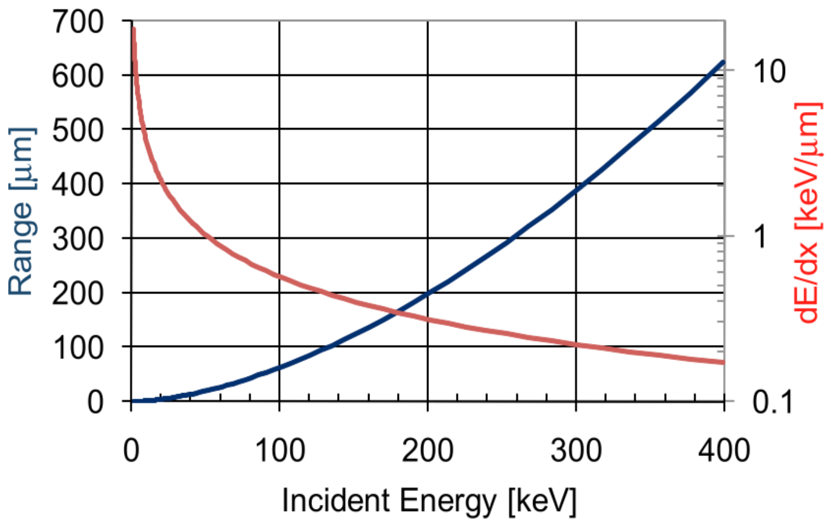
- Limited PSF due to scintillator and backscattering from optics

Direct Semiconductor Pixel Detectors

- High PSF and DQE from direct detection
- High speed readout
- Data processing capabilities

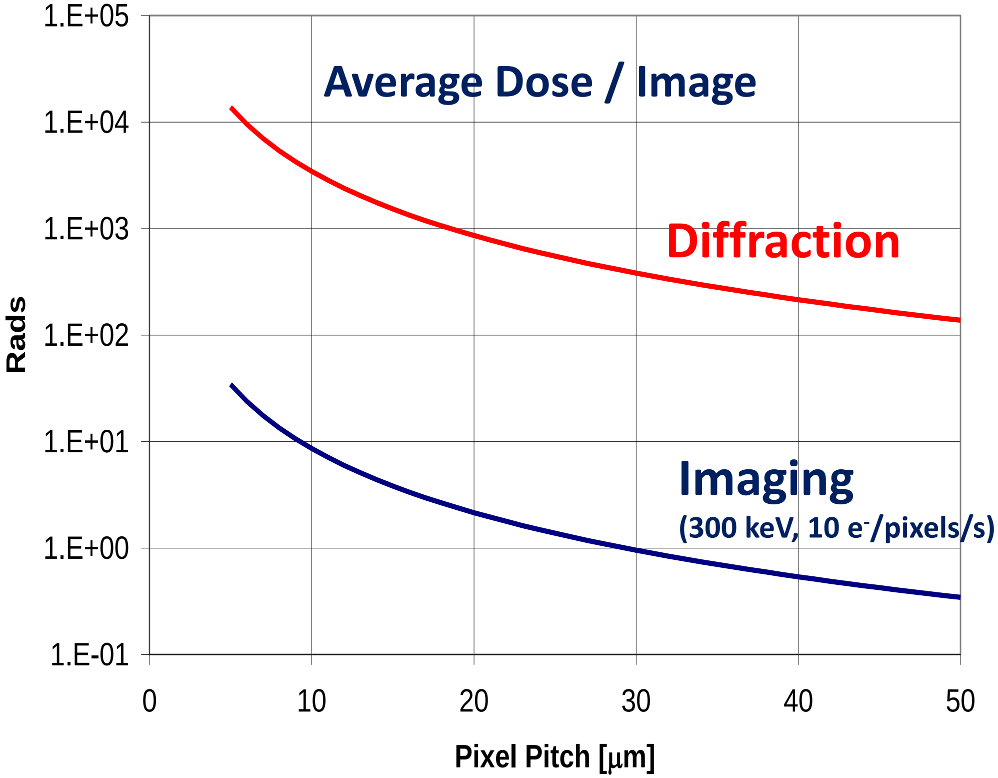
Detector R&D Drivers: Multiple Scattering

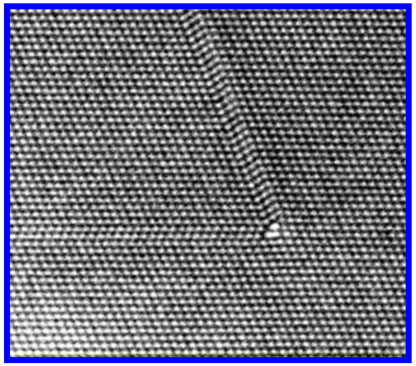




- Energies of interest to TEM: 80-400 keV
- Electron range **R [µm] ~ E [keV]**
- Energy loss **dE/dx ∝ 1/E**
- Need for a thin sensitive layer to minimize scattering contribution to Point Spread Function

Detector R&D Drivers: Radiation Hardness





- **Imaging mode**: O(1-10 Mrad) ionising dose expected for typical yearly usage (low dose conditions)

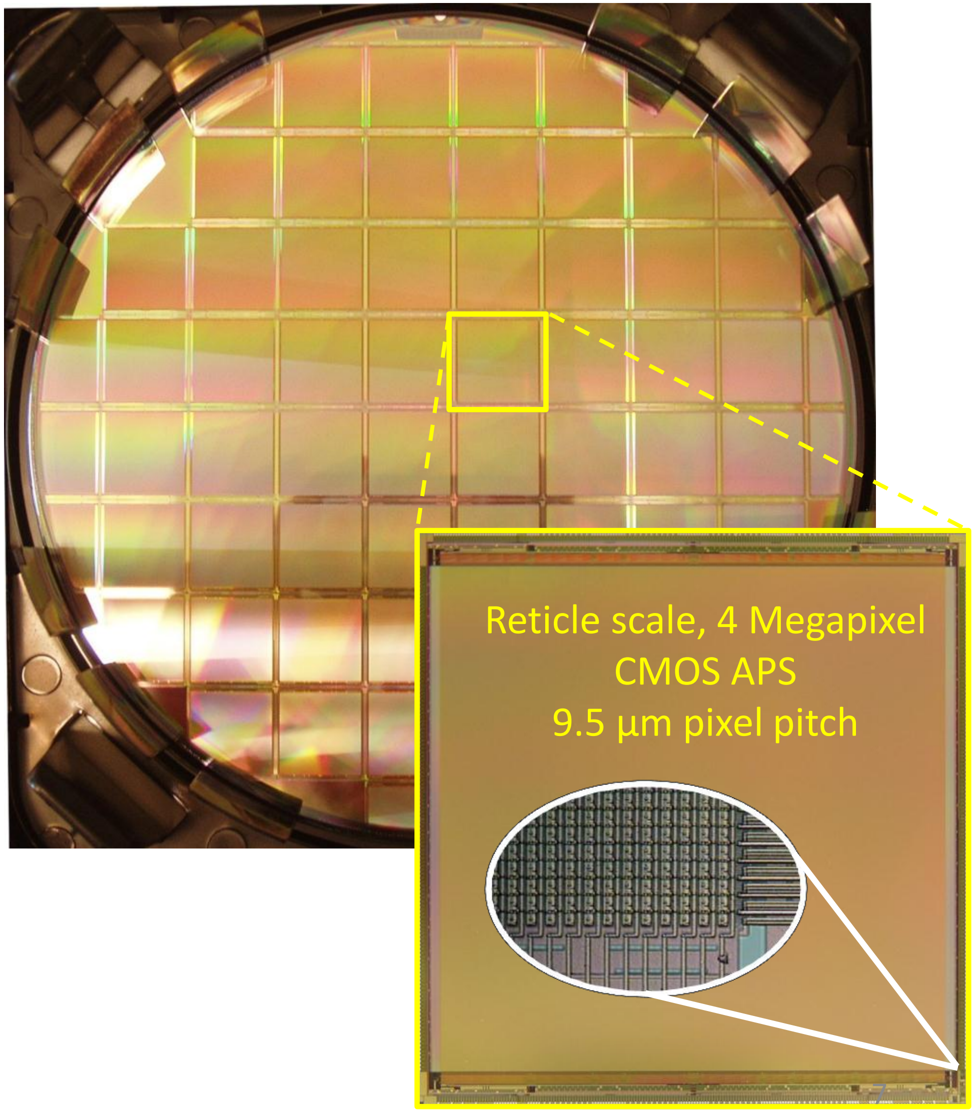
- **Diffraction mode**: very high doses localized in bright spots

- Radiation tolerance requirements comparable or worse than High Energy Physics applications → leverage extensive R&D on radiation tolerant design of sensors and readout electronics

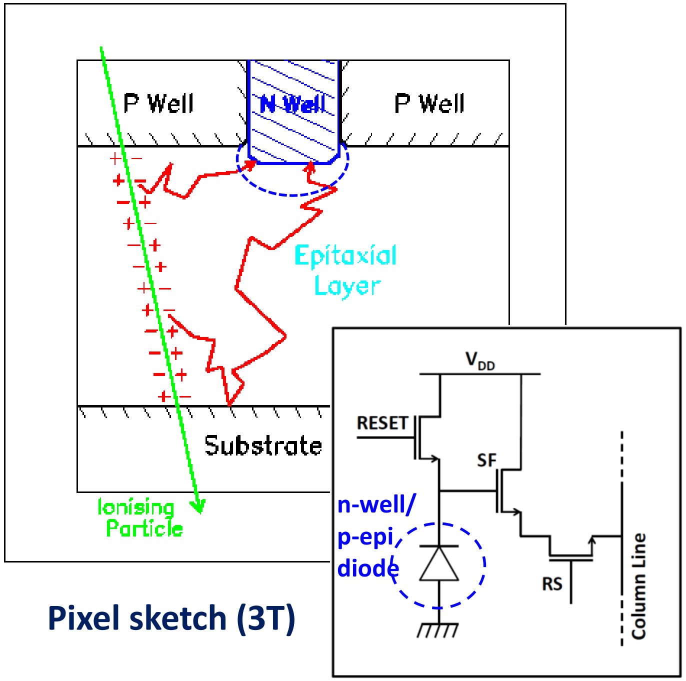
CMOS Imagers as New Eyes for TEM

- CMOS Active Pixel Sensors provide high resolution and low material budget particle detectors at low cost, as they are fabricated in commercial manufacturing processes

- We have developed CMOS APSs as an alternative to conventional, optically-coupled CCD cameras for TEM imaging:
  - Single electron sensitivity via direct detection
  - Excellent Point Spread Function: small pixels and thin sensitive volume
  - High readout speed: O(100) frame/s achievable for Megapixel-scale imagers
  - Improved radiation hardness: lifetime of several years possible

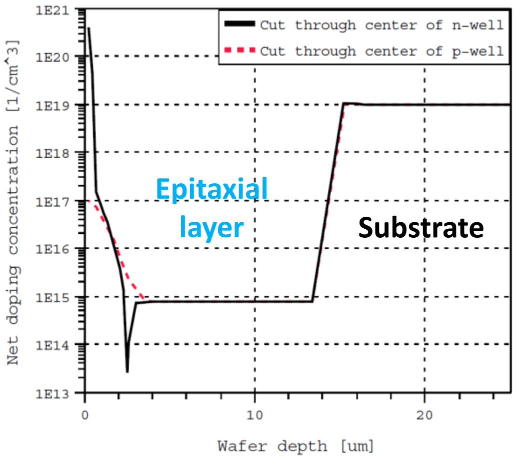

Reticle scale, 4 Megapixel CMOS APS
9.5 µm pixel pitch

CMOS APS Adapted for Radiation Detection

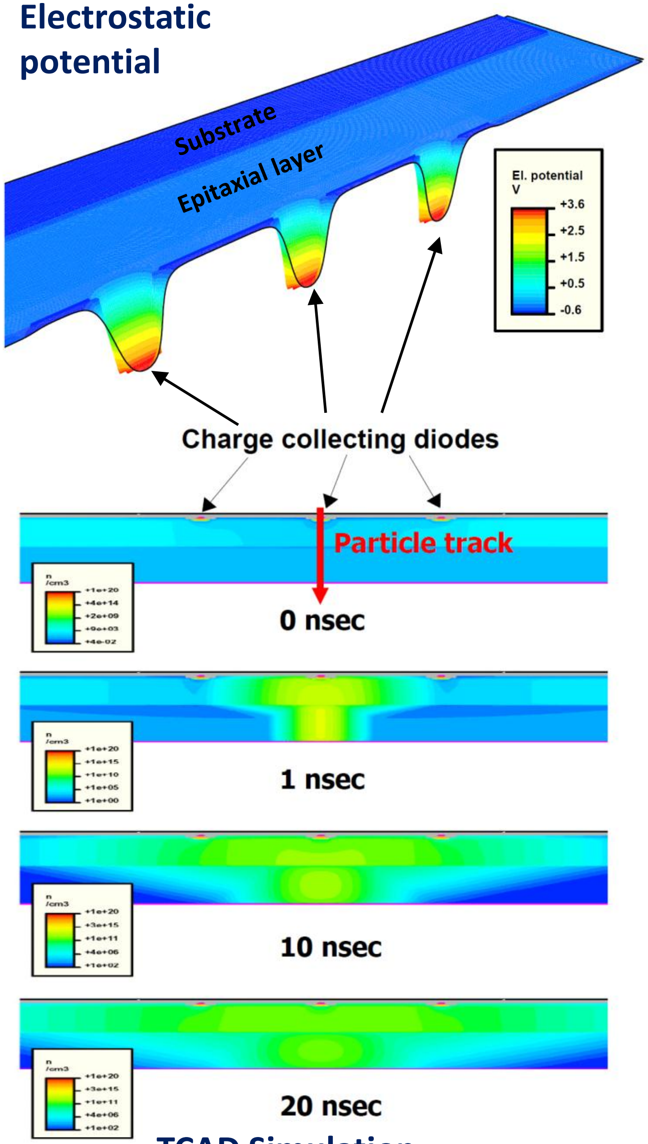


Pixel sketch (3T)

Doping profile



Electrostatic potential



TCAD Simulation of m.i.p. detection

- Proposed for charged particle detection in Turchetta et al., Nuclear Instruments and Methods A 458 (2001) 677
- Achieve 100% fill factor by using twin-well CMOS process: charge collecting diode formed by n-well/p-epi junction, pixel circuitry integrated in complementary p-well
- Charge collected mainly by diffusion in the moderately doped, field-free epitaxial layer

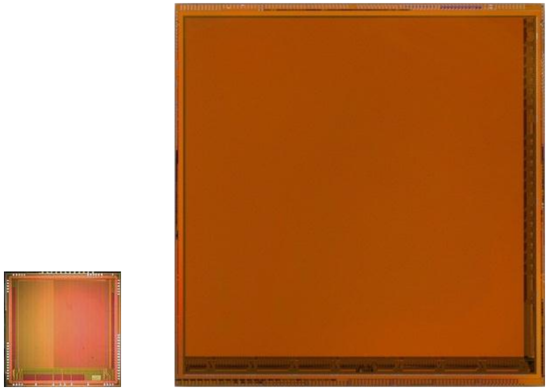
Three Generations of CMOS APS for TEM Imaging

1st Generation (2008-2009)

0.35 µm CMOS
9.5 µm pixels
1 & 4 Mpixels
400 fps → 400 Mpixels/s
Imagers for the TEAM Project at NCEM

2nd Generation (2009-2011)

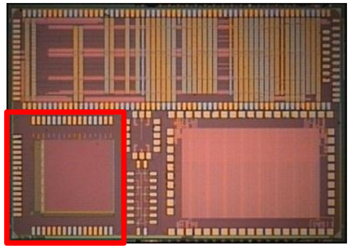
0.18 µm CMOS
5 µm pixels
16 MPixels
400 fps → 6400 Mpixels/s
Commercialized by Gatan, Inc.



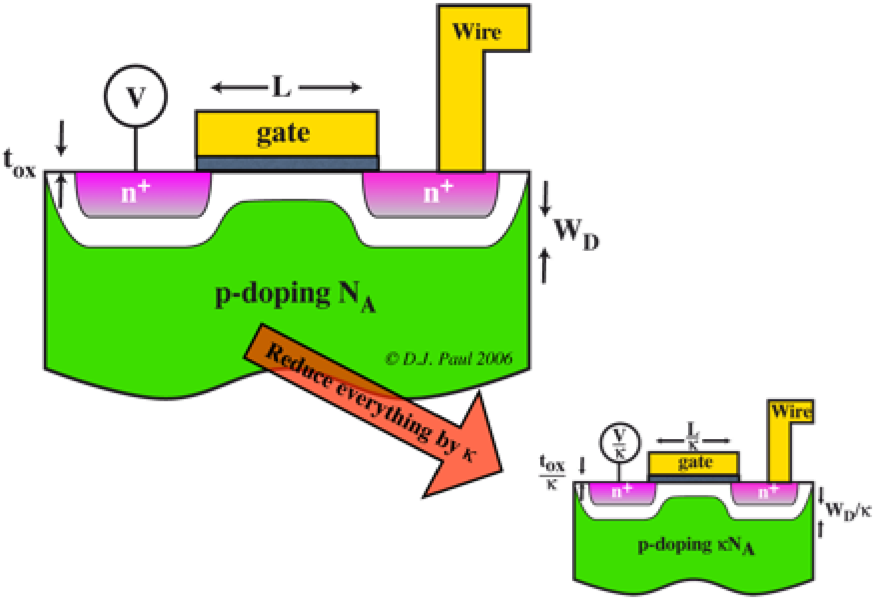
http://www.gatan.com/K2/

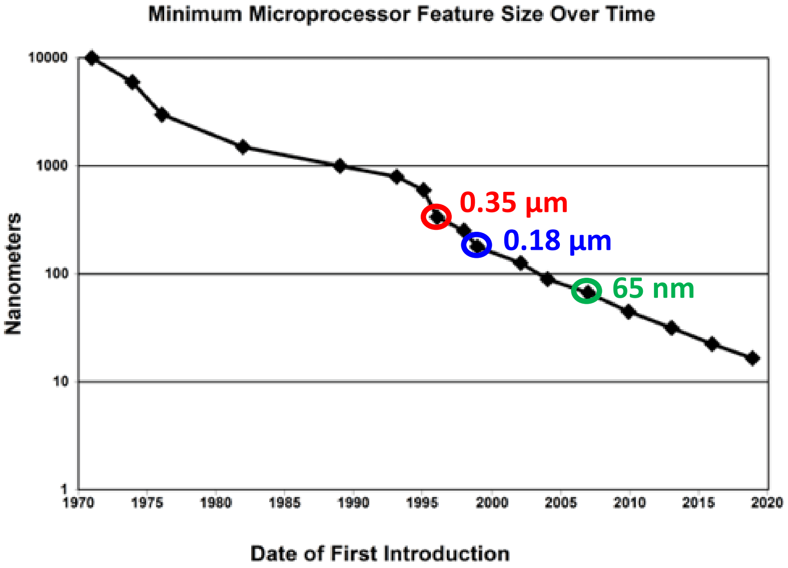
3rd Generation (2011 – in progress)

65 nm CMOS
2.5 µm pixels
Prototype sensor under evaluation

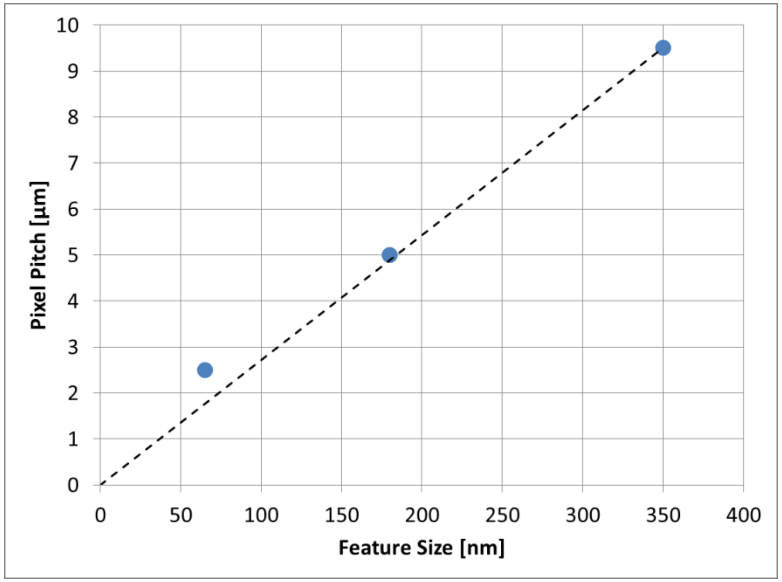


Footnote: Scaling the Feature Size

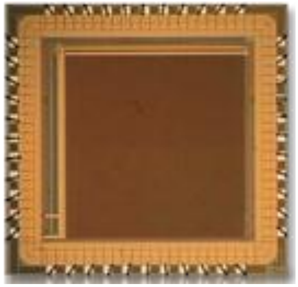




- The reduction of feature size fueled by Moore's Law enables:
  - ➢ Higher integration capabilities: smaller pixels, more pixels/chip
  - ➢ Improved radiation hardness (thinner oxides)
- But also:
  - ➢ Higher prototyping costs
  - ➢ Lower dynamic range (lower V_{DD}, and lower C → higher µV/e^-)

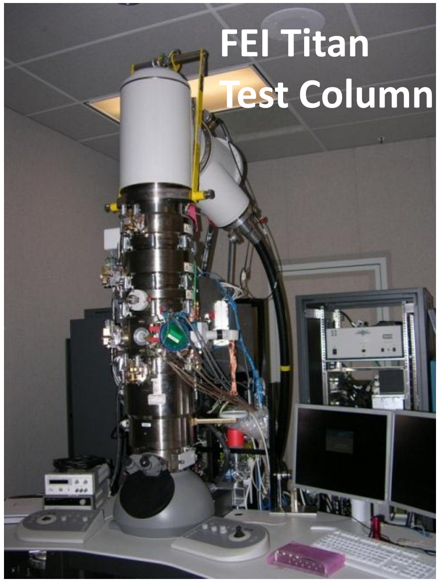


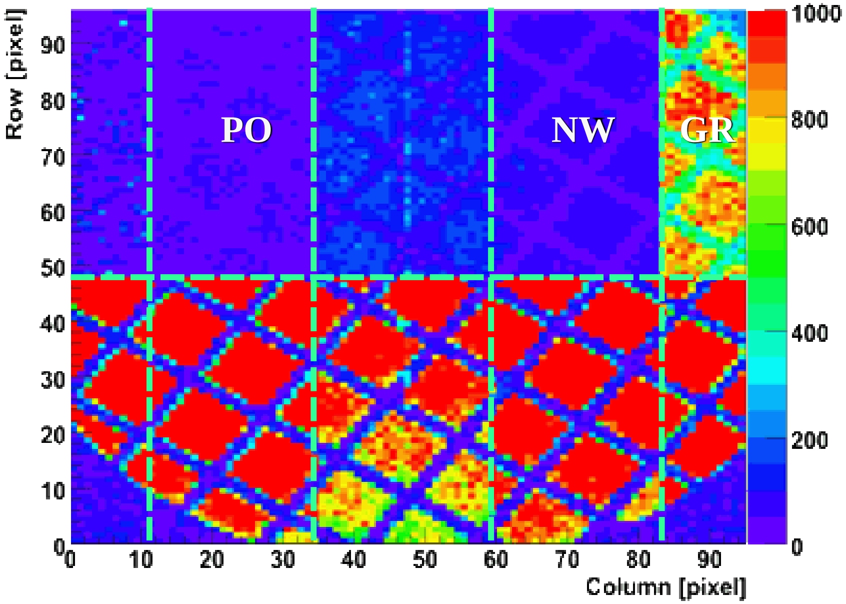
The Pixel Selection Process



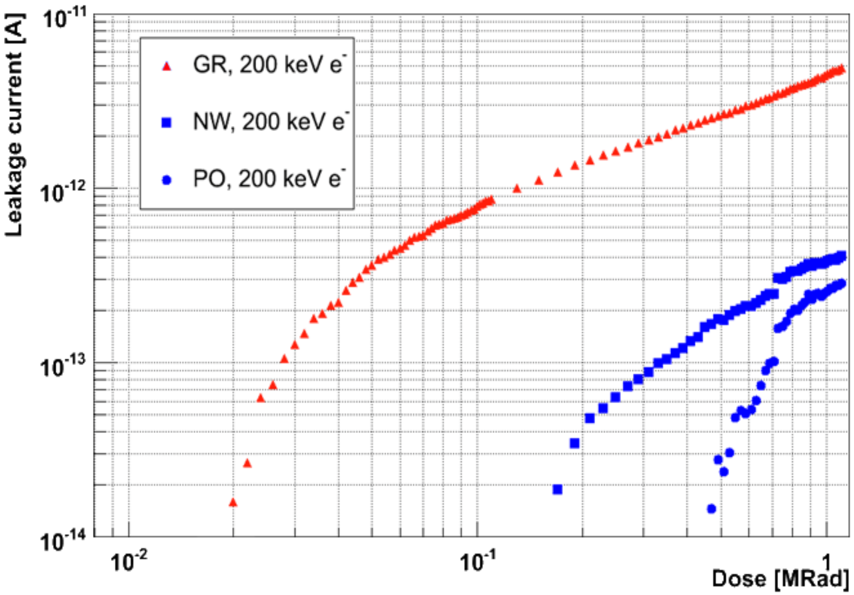
0.35 μm CMOS test chip
96×96 pixels
10 design options

- Design and manufacture small scale prototype in target process, implementing various pixel architectures and radiation-tolerant layout options
- Test comparatively electron detection capabilities and radiation tolerance in TEM
- Port best-performing design to large-scale imager





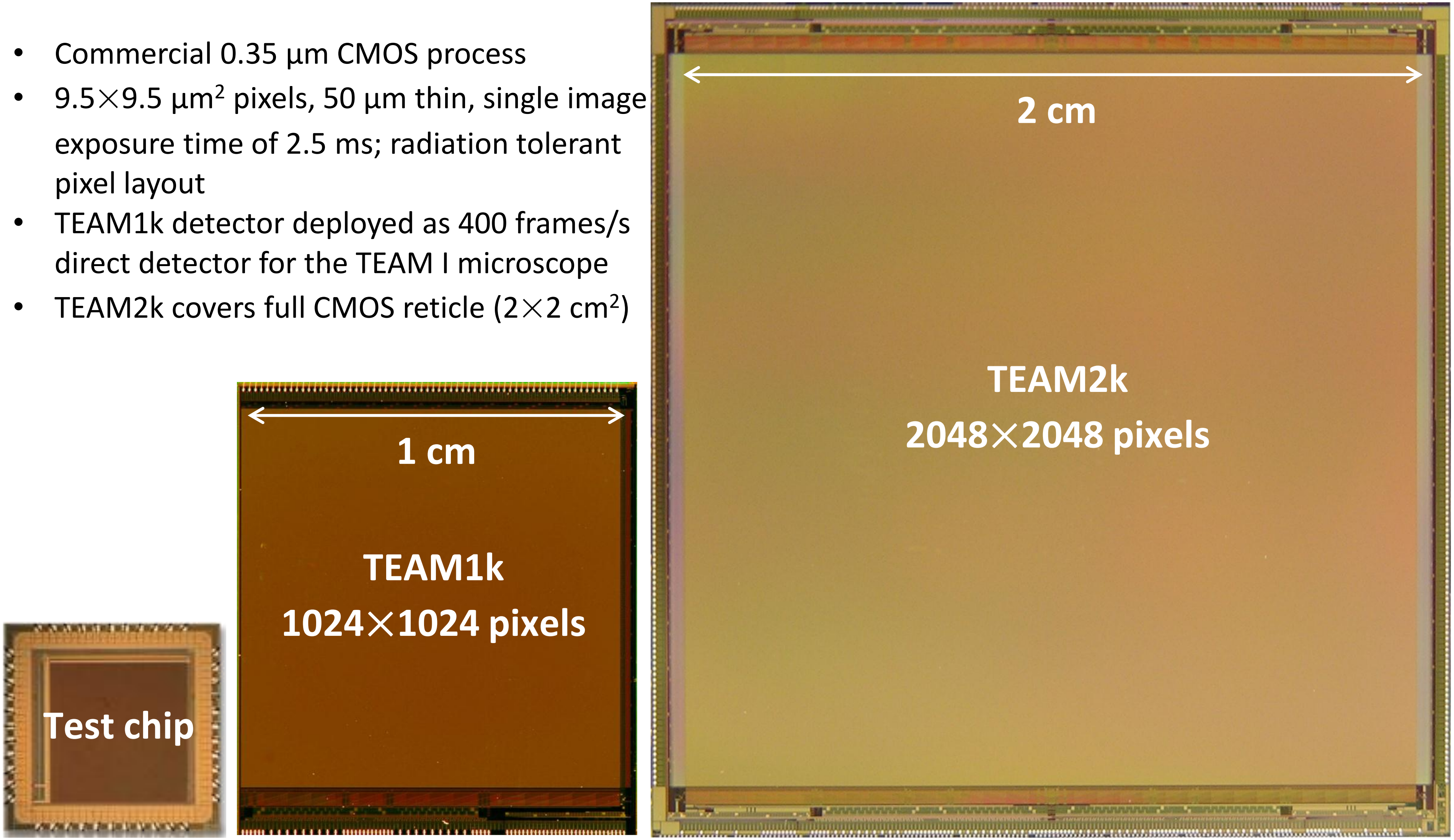
Latent image due to leakage current on irradiated sensor (chip covered with gold mesh during irradiation)



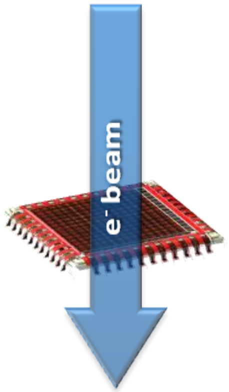
[Nuclear Instruments and Methods A 598 (2009) 642]

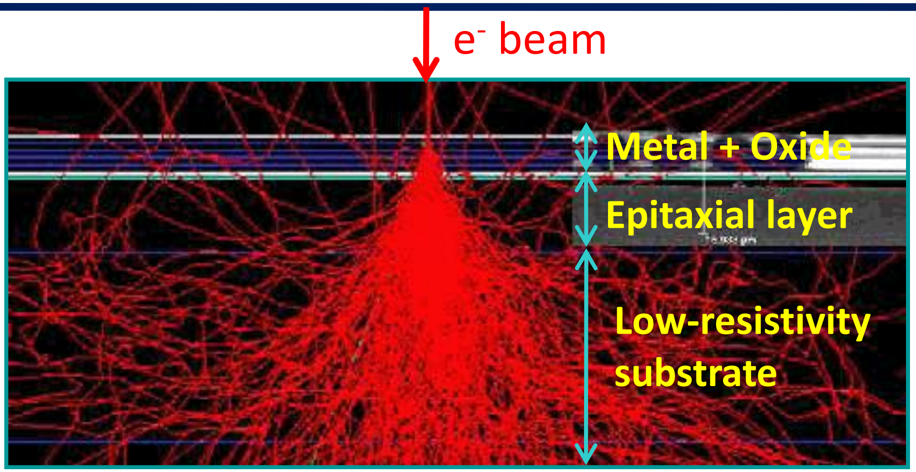
Detectors for the TEAM Project

- Commercial 0.35 µm CMOS process
- 9.5×9.5 μm^2 pixels, 50 µm thin, single image exposure time of 2.5 ms; radiation tolerant pixel layout
- TEAM1k detector deployed as 400 frames/s direct detector for the TEAM I microscope
- TEAM2k covers full CMOS reticle (2×2 cm^2)



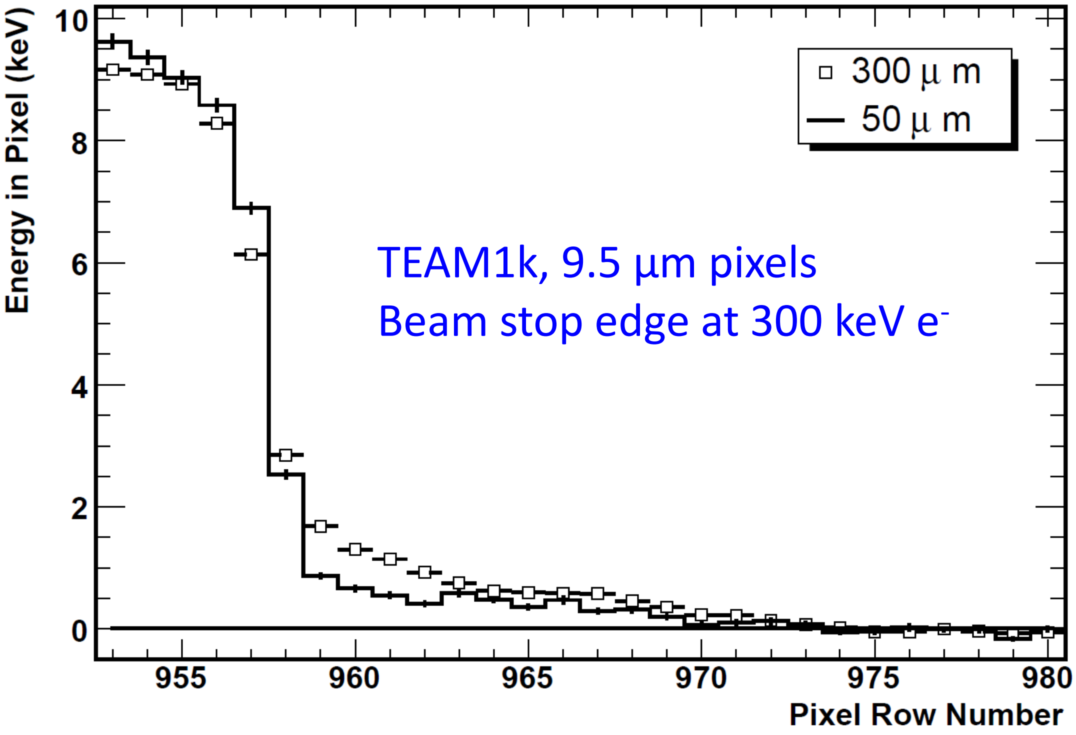
Improving Resolution by Thinning





- Geant-4 simulation of 200 keV e^- in CMOS Active Pixel Sensor (0.35 µm process)

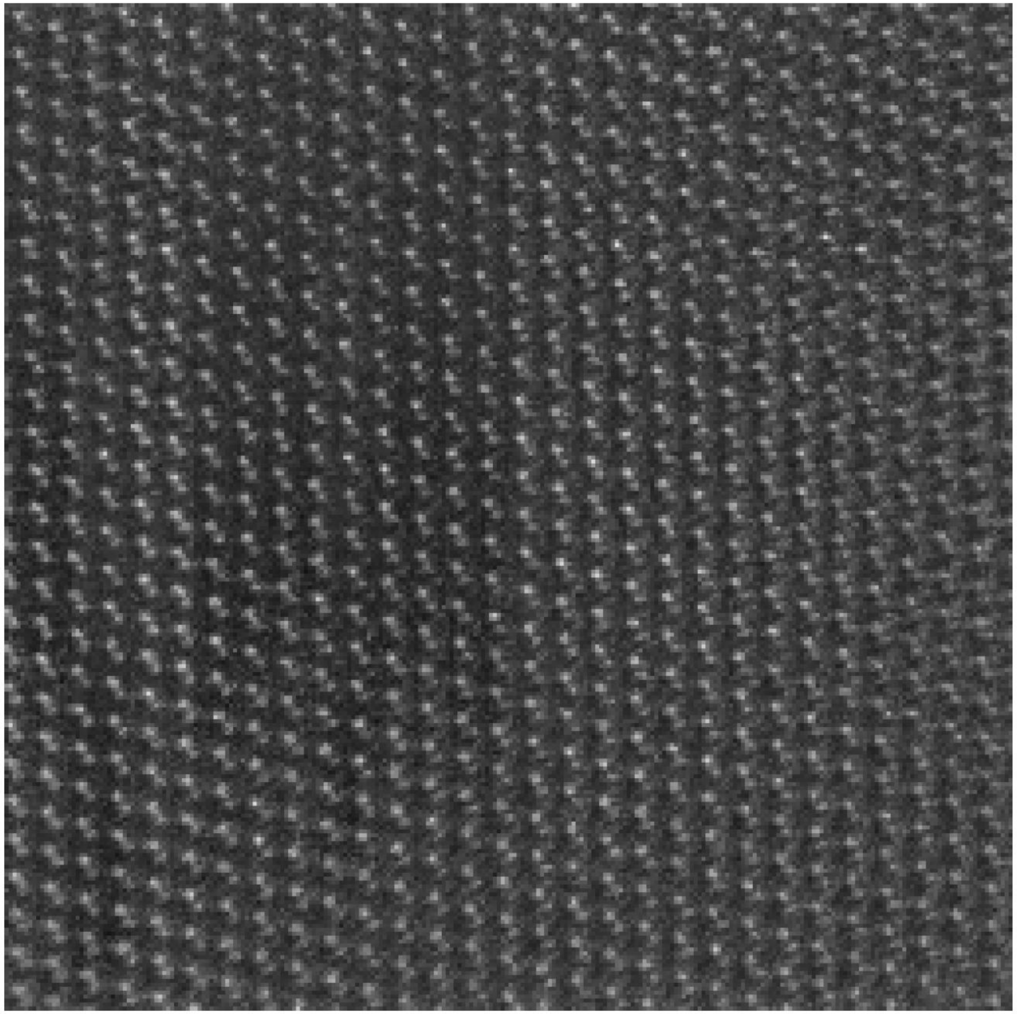
- Sensor thinning to e.g. 50 µm improves Point Spread Function, thanks to reduction of back-scattering from detector substrate

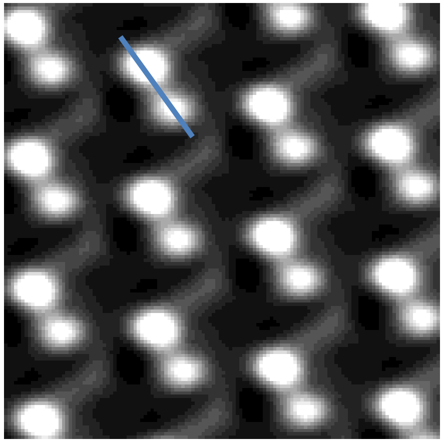


TEAM1k, 9.5 µm pixels
Beam stop edge at 300 keV e^-

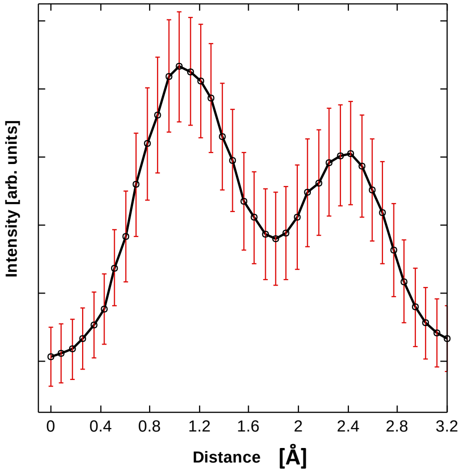
[Nuclear Instruments and Methods A 598 (2009) 642]
[Nuclear Instruments and Methods A 622 (2010) 669]

Imaging Si[110] dumbbells in 2.5 ms





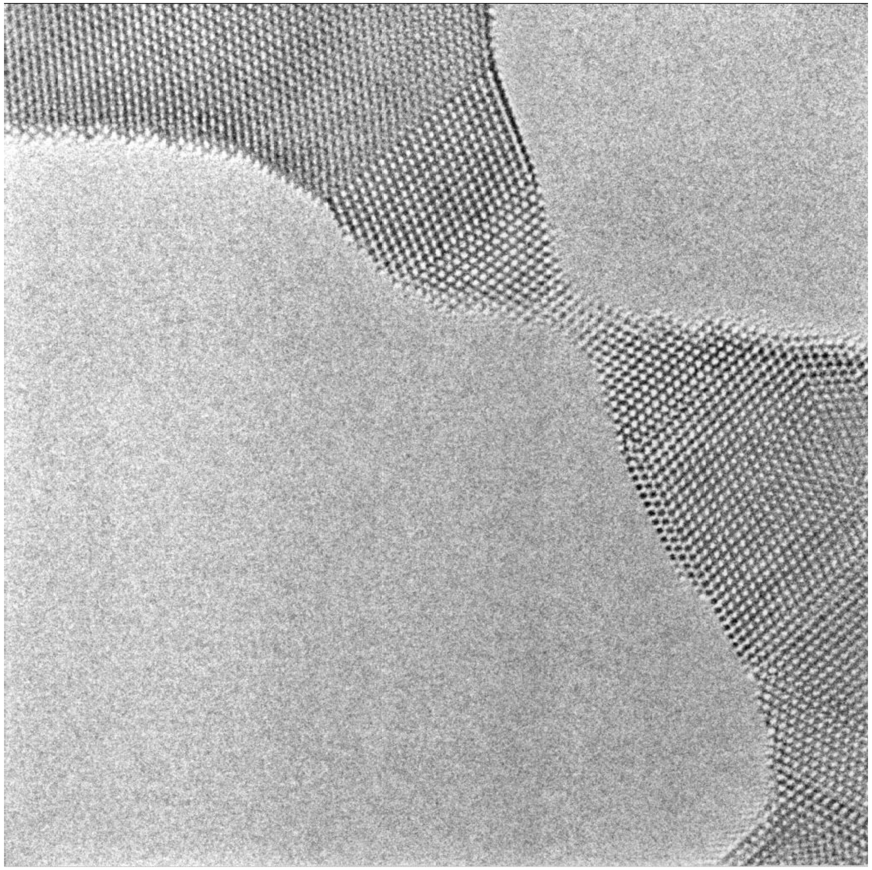
Line profile across dumbbell in averaged image



300 keV e^-, 1024×1024 pixels (TEAM1k chip); single, raw, unprocessed 2.5 ms exposure
→ obtain resolution comparable to film at 1000x shorter exposure time

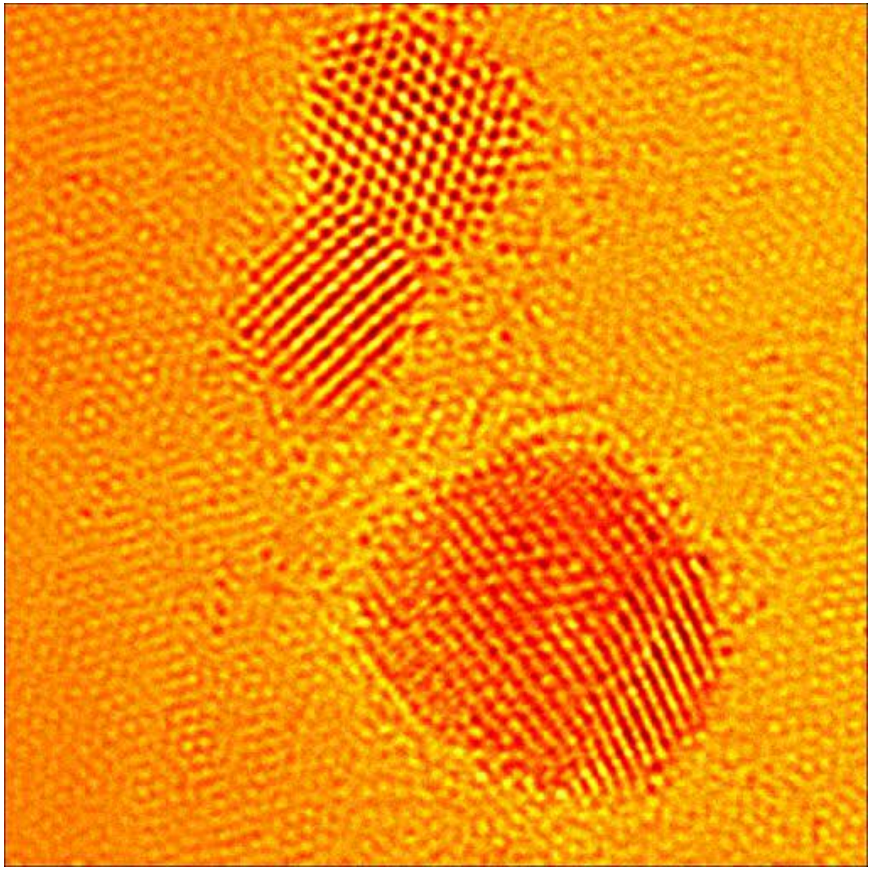
Enabling New Imaging Capabilities

Au nanobridge



TEAM I, 300 keV
Single image, 20 ms exposure
(raw data, 2010)

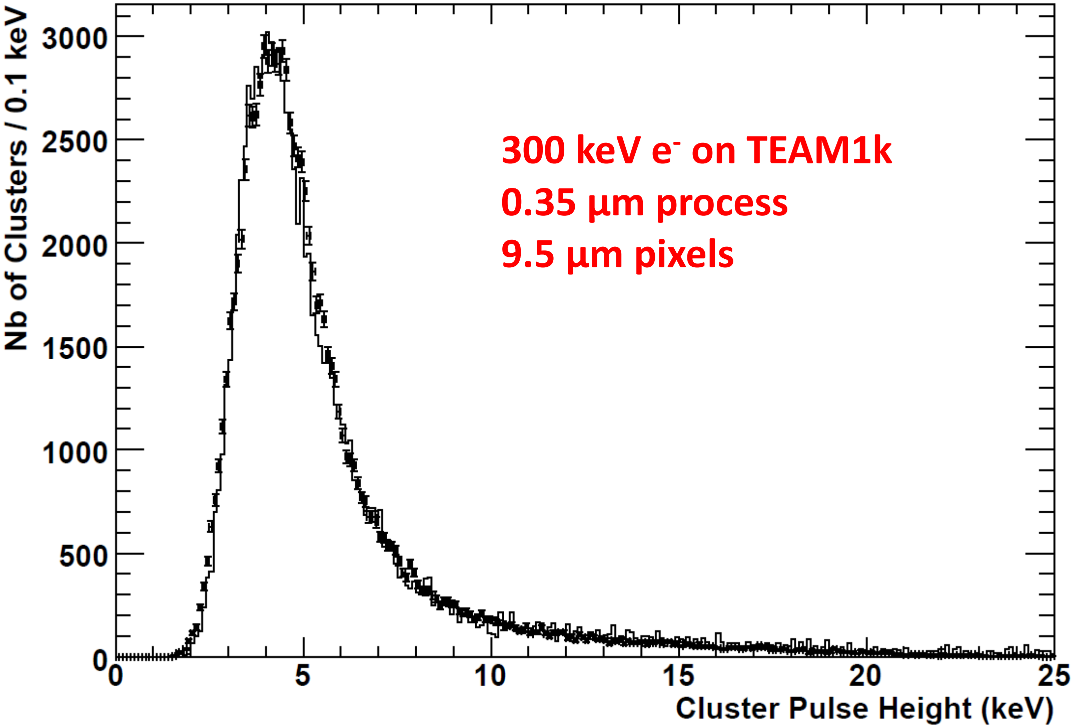
Au/FeO on graphene nanobridge



TEAM I, 80 keV
Average of 40 2.5 ms images
(raw data, 2010)

[images courtesy A. Gautam, P. Ercius, C. Ophus, V. Radmilovic, NCEM]

Direct Detection of Single Electrons

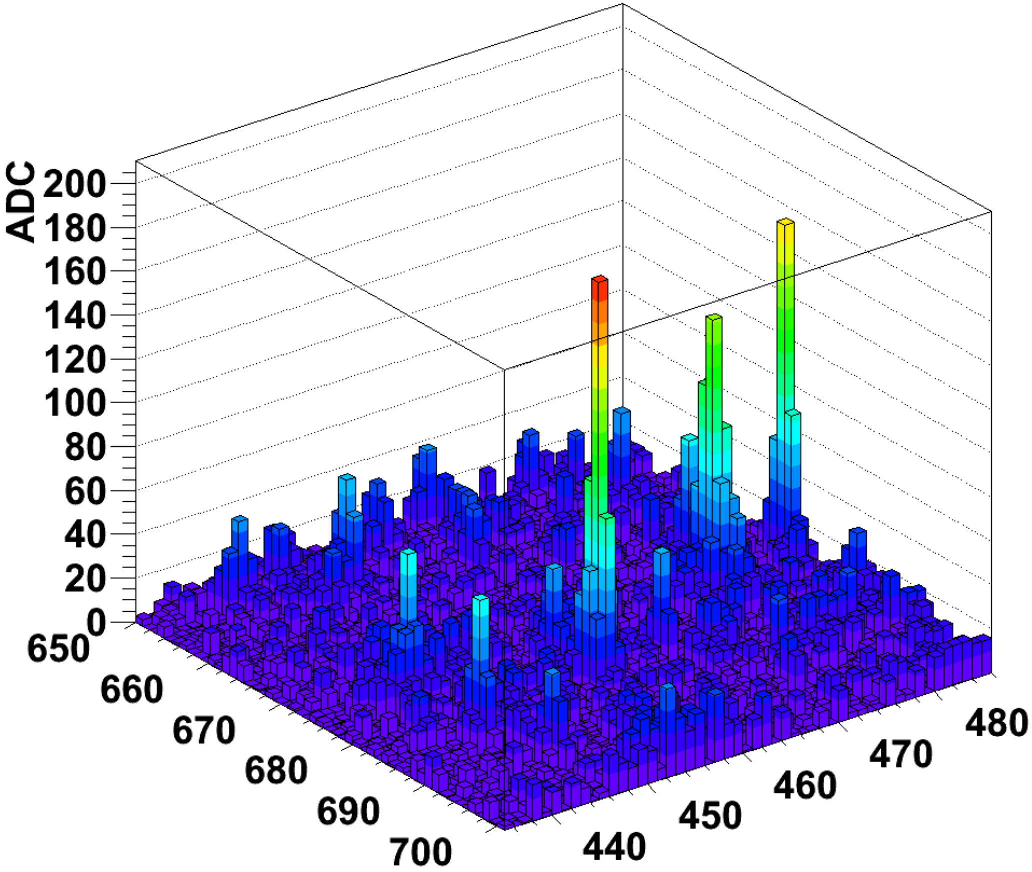


300 keV e^- on TEAM1k
0.35 μm process
9.5 μm pixels

- In low dose conditions, single electron events can be detected by the direct detector with good S/N performance (15-20)
- Energy deposition for single 300 keV e^- follows a Landau distribution
- Can we extract more information from the reconstruction of single electron events?

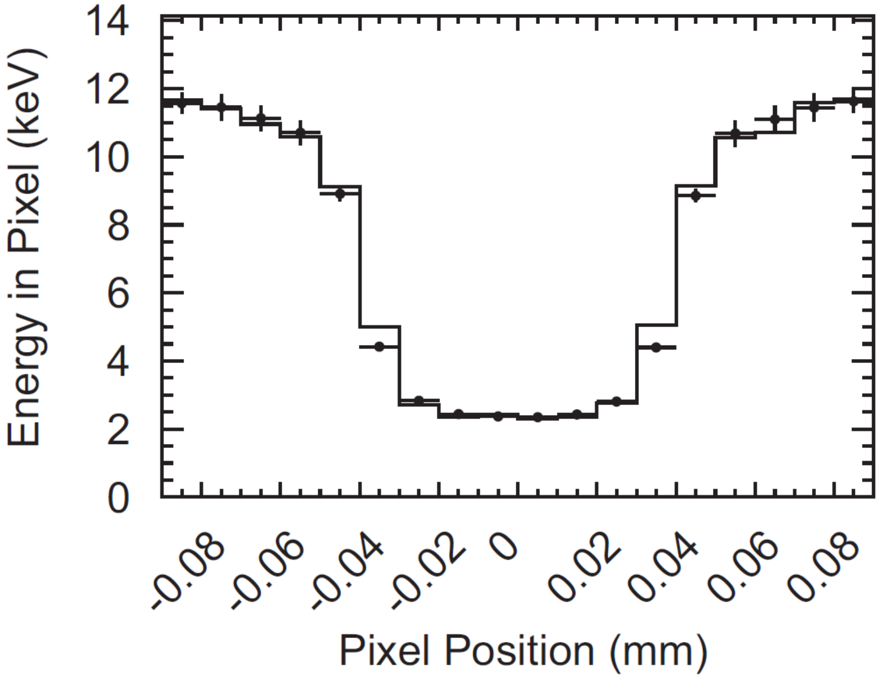
Improving Resolution by Single Electron Detection

- For low electron rates (< 0.05 e^-/pixel), the signal generated by single electron events will be detected by “clusters” of pixels
- The electron impact positions can be reconstructed with larger accuracy w.r.t. pixel pitch, e.g. by interpolating the charge distributed among the pixels in the cluster
- The “**cluster imaging**” technique composes higher resolution images at lower total electron doses from the superimposition of many frames with sparse electron hits

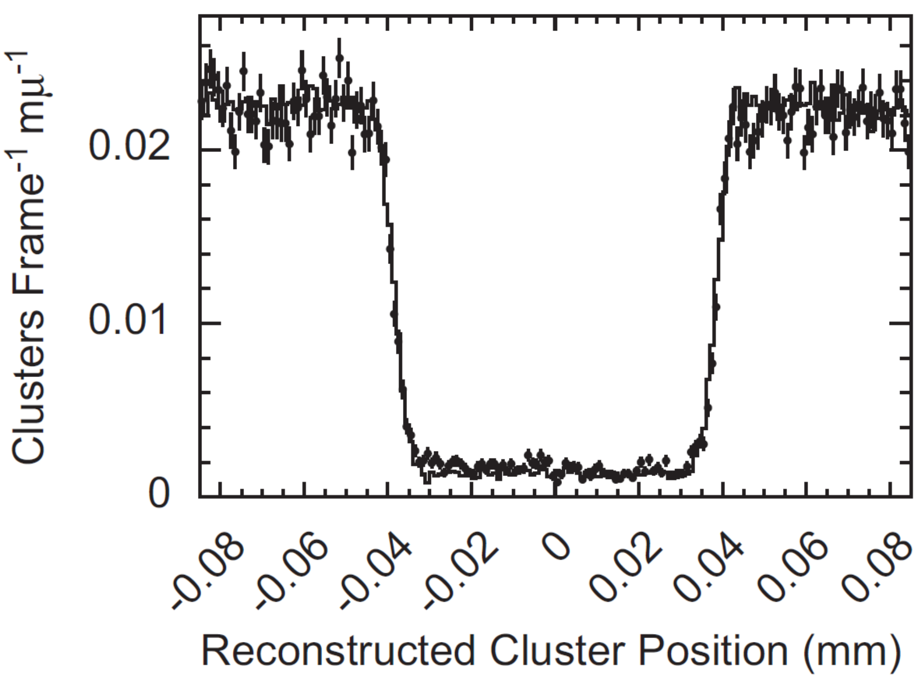


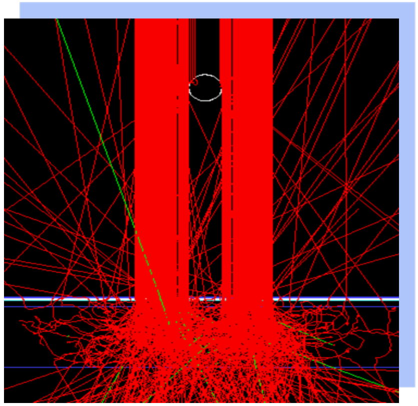
The Cluster Imaging Technique: Demonstration

Bright field



Cluster imaging

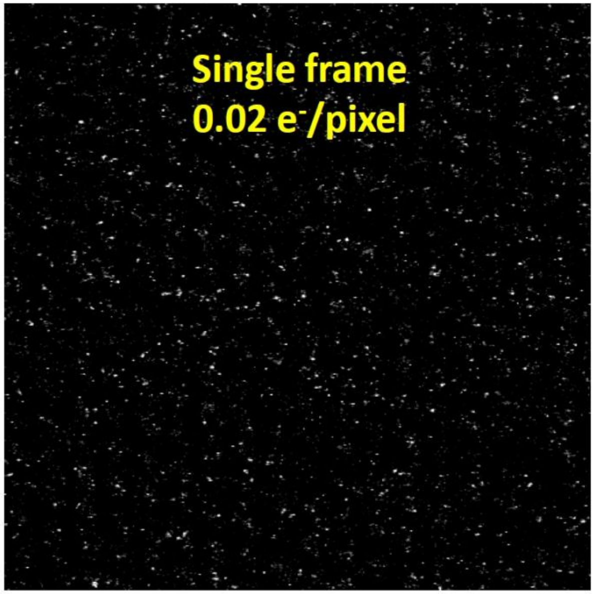




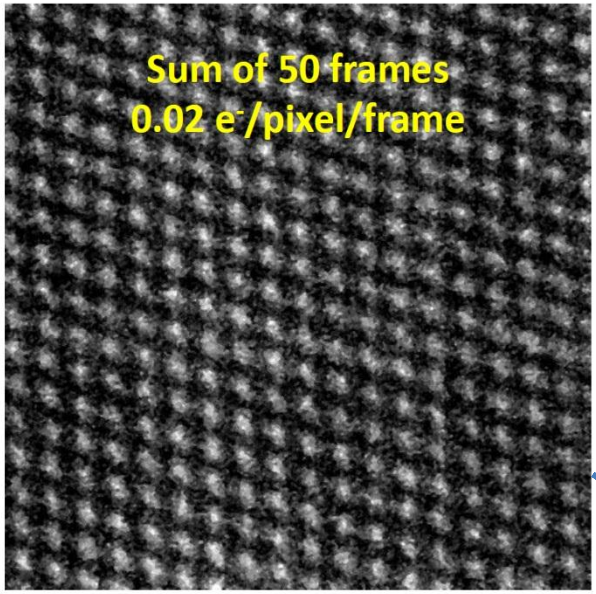
- Image of 60 µm thin gold wire (sharp edges) with 300 keV electrons in two imaging modes on TEAM1k chip (9.5 µm pixel pitch)
- PSF originates from diffusion and scattering

[Nuclear Instruments and Methods A 608 (2009) 363]

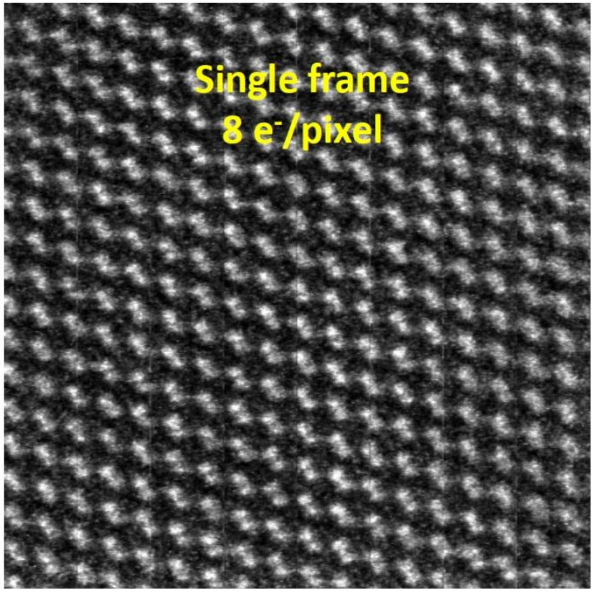
Enabling Low Dose, High Resolution Imaging



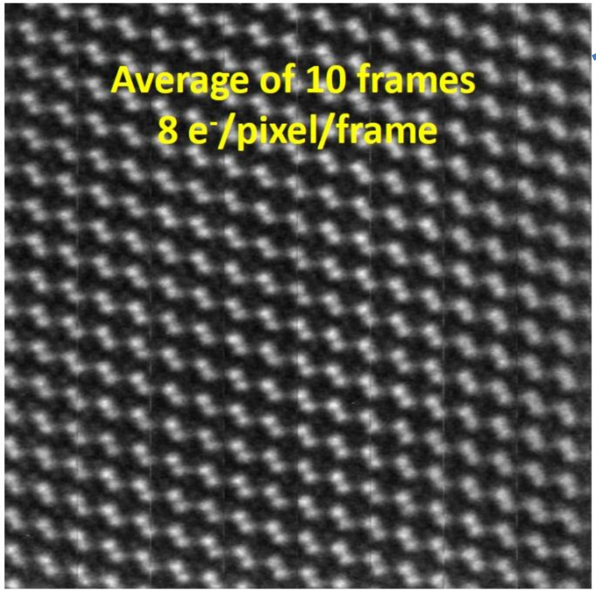
Single frame
0.02 e^-/pixel



Sum of 50 frames
0.02 e^-/pixel/frame



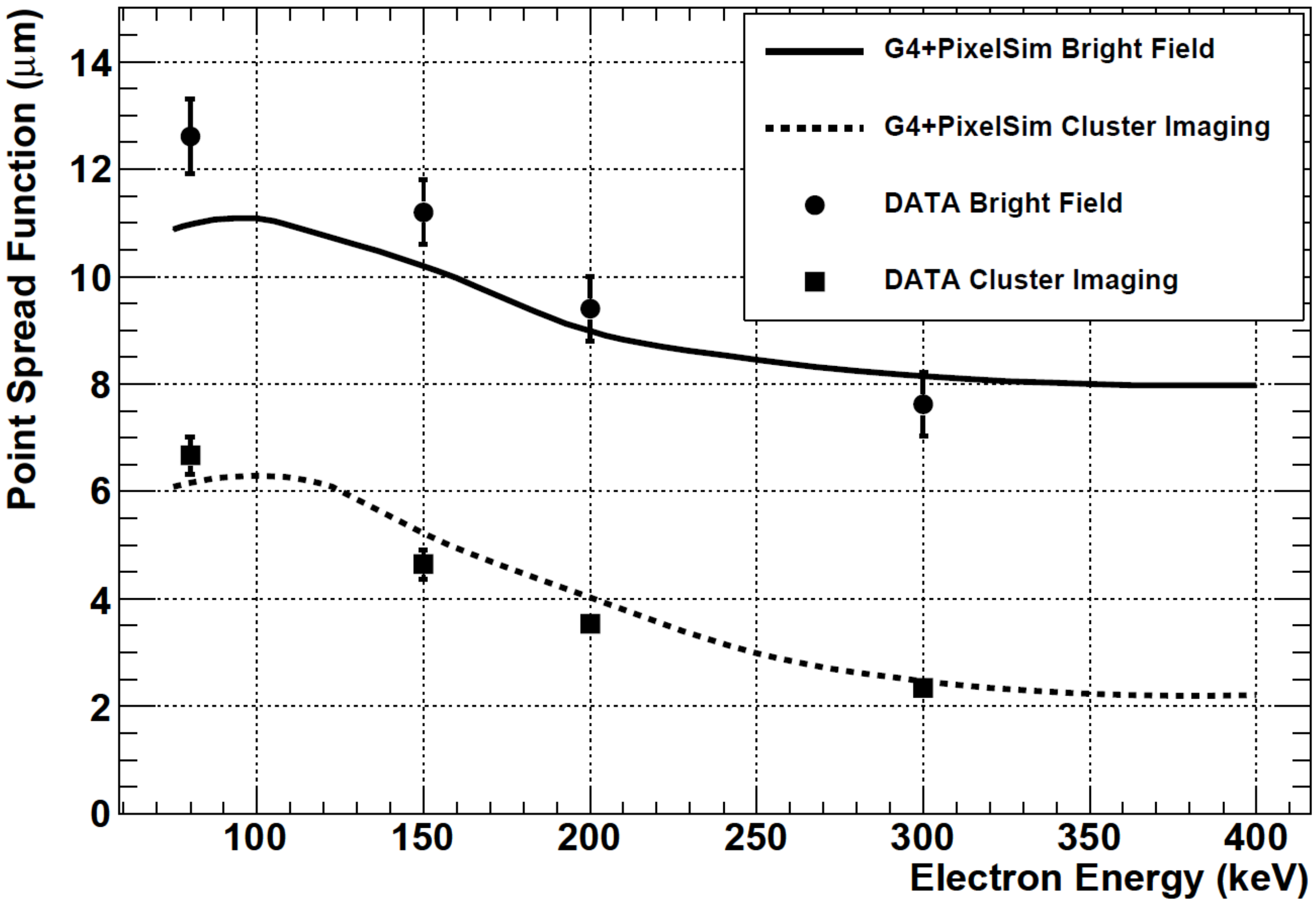
Single frame
8 e^-/pixel



Average of 10 frames
8 e^-/pixel/frame

80x dose difference

Point Spread Function Improvement by Clustering

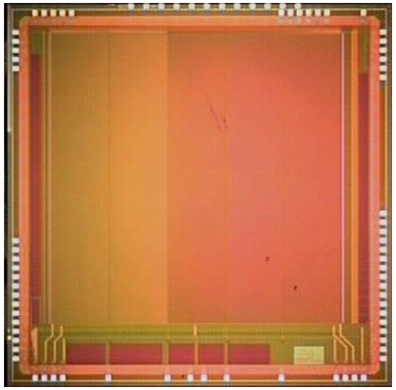


- A factor 2-3 improvement in position resolution can be achieved with cluster imaging with respect to conventional bright field imaging

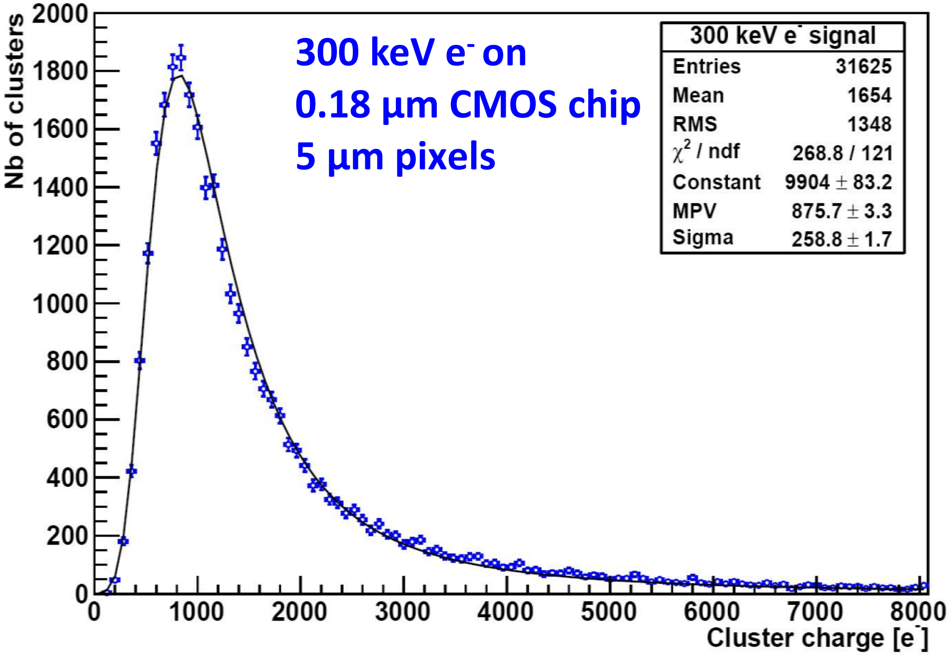
[Nuclear Instruments and Methods A 622 (2010) 669]

2^{nd} Generation Development in 0.18 µm CMOS

0.18 µm CMOS test chip
760×768 pixels
12 design options



HHMI
HOWARD HUGHES
MEDICAL INSTITUTE



- Prototype chip funded by Howard Hughes Medical Institute (HHMI)
- 0.18 µm CMOS process, 5 µm pixel pitch
- 12 pixel design options: best architecture selected from radiation hardness tests
- Detection performances comparable with TEAM sensors
- 0.18 µm w.r.t 0.35 µm process shows higher gain, lower leakage but reduced dynamic range

Fabrication process [µm]	Conversion Gain [µV/e-]	Noise (@ RT) [e-]	Leakage current [fA]	Well depth [e-]
0.35	9.4	30	10	90000
0.18	15.5	35-40	4	23000

[Nuclear Instruments and Methods A 635 (2011) 69]

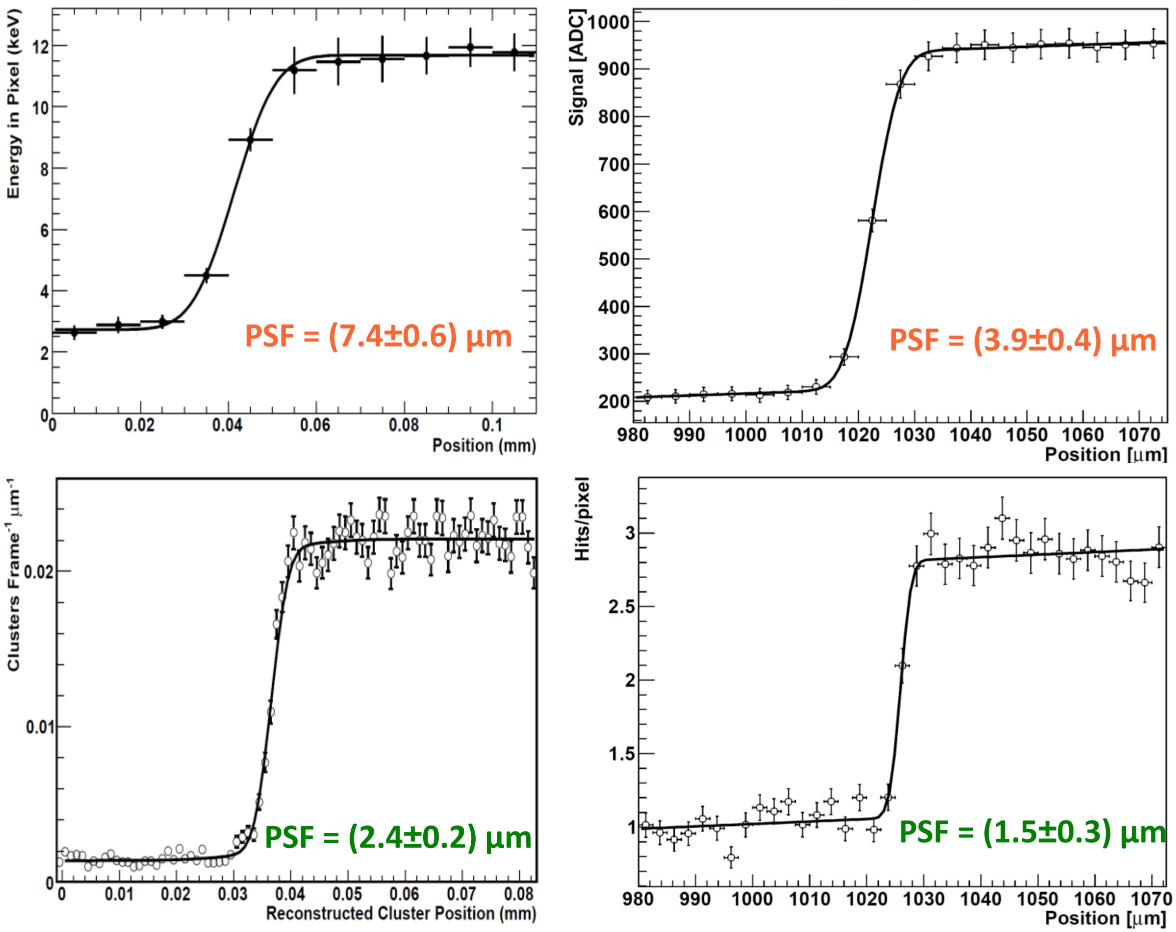
Compare Point Spread Function Performance

0.35 µm CMOS
9.5 µm pixels

0.18 µm CMOS
5 µm pixels

Bright field

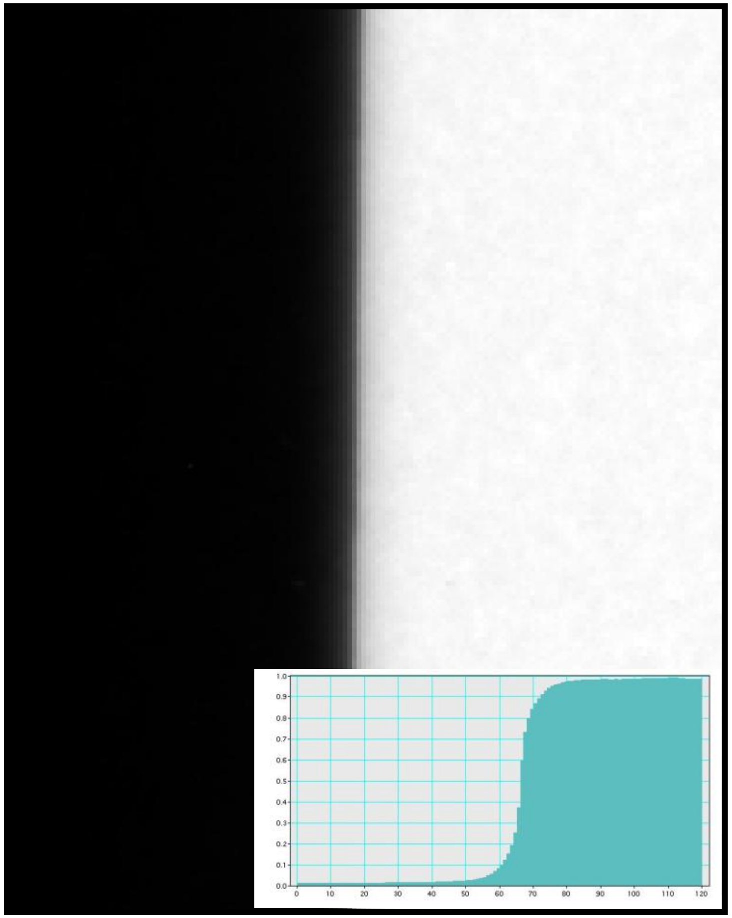
Cluster imaging



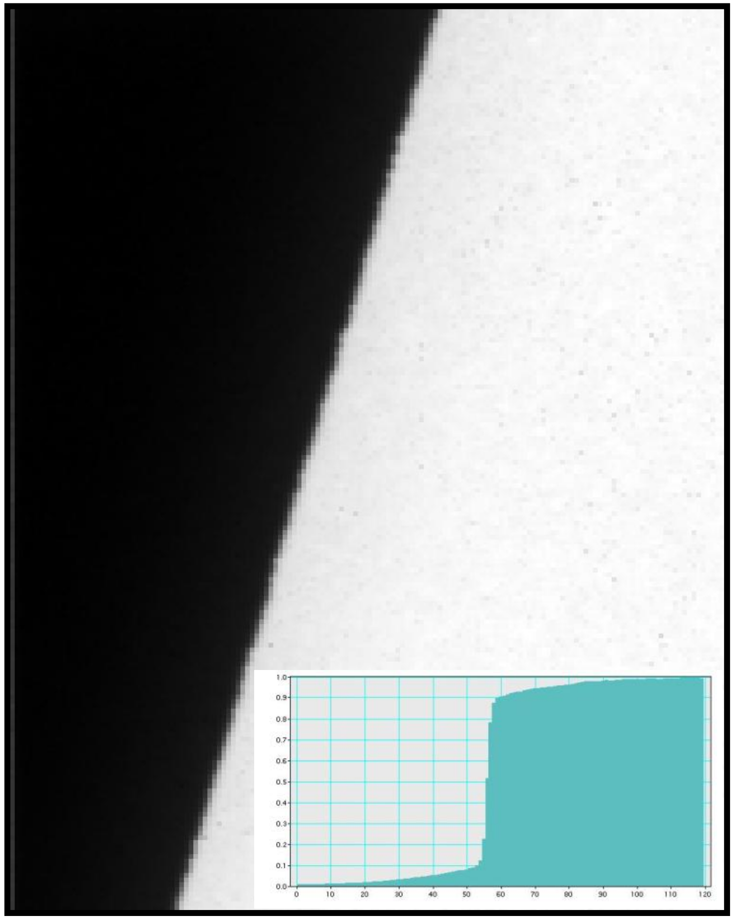
Beam profile of Au wire edge, 300 keV e$^-$

[Nuclear Instruments and Methods A 622 (2010) 669]
[Nuclear Instruments and Methods A 635 (2011) 69]

Improving Resolution with Direct Detection



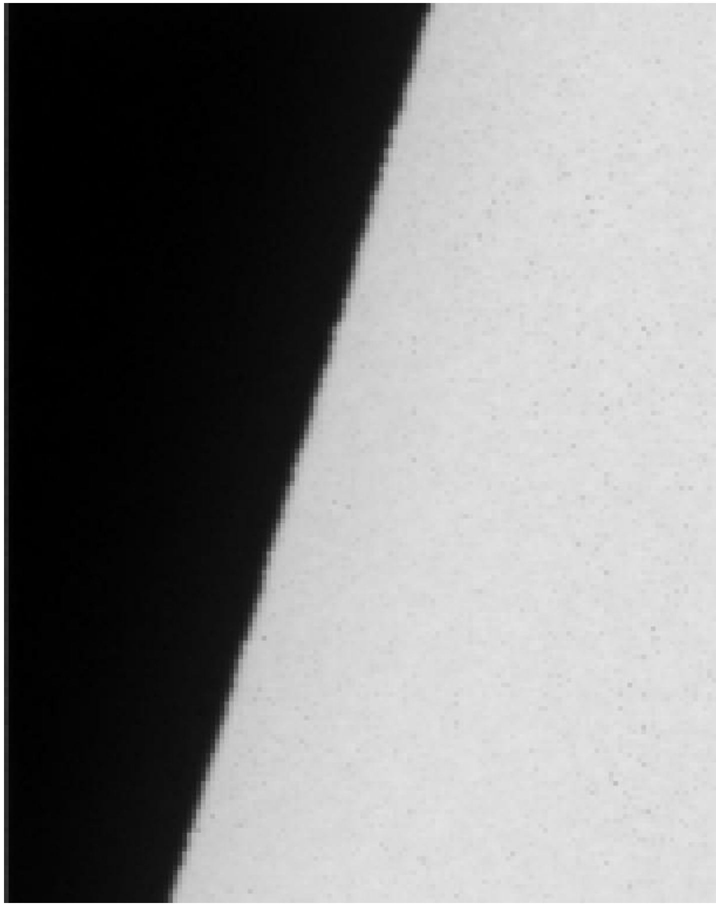
Commercial CCD camera
14 µm pitch



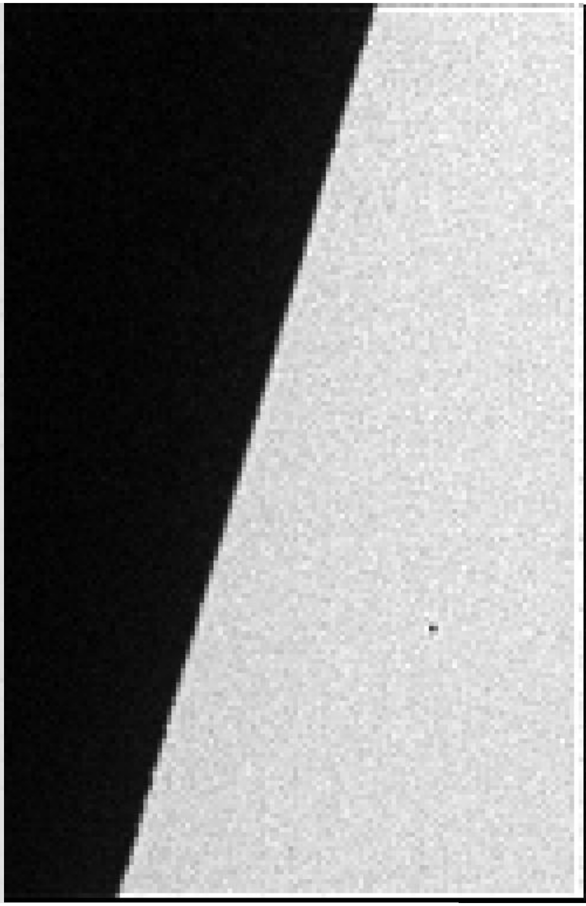
0.18 µm CMOS test chip
5 µm pitch

- Beam stop edge imaged with 300 keV e$^-$, bright field conditions
- Compare optically coupled CCD camera with direct CMOS detector

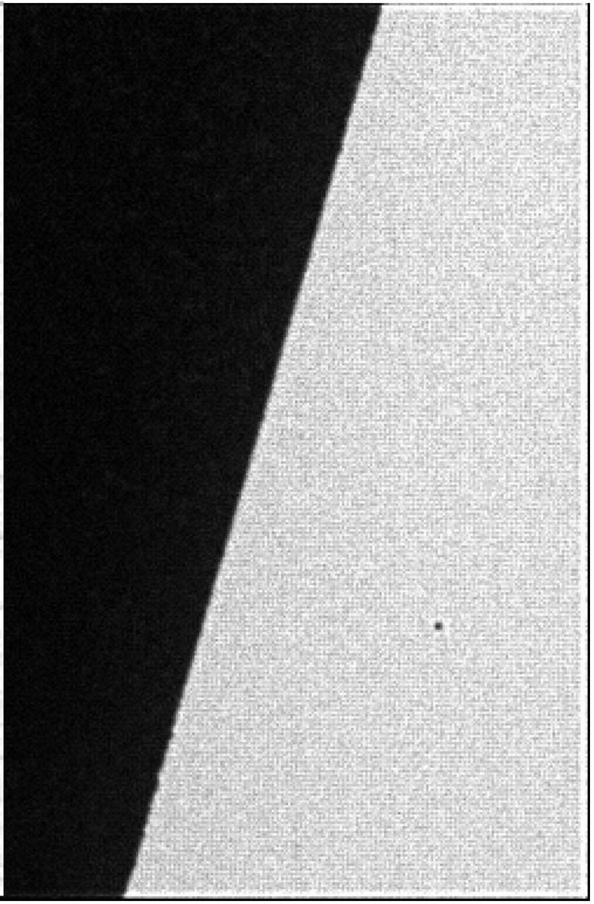
Improving Resolution with Clustering



Bright field



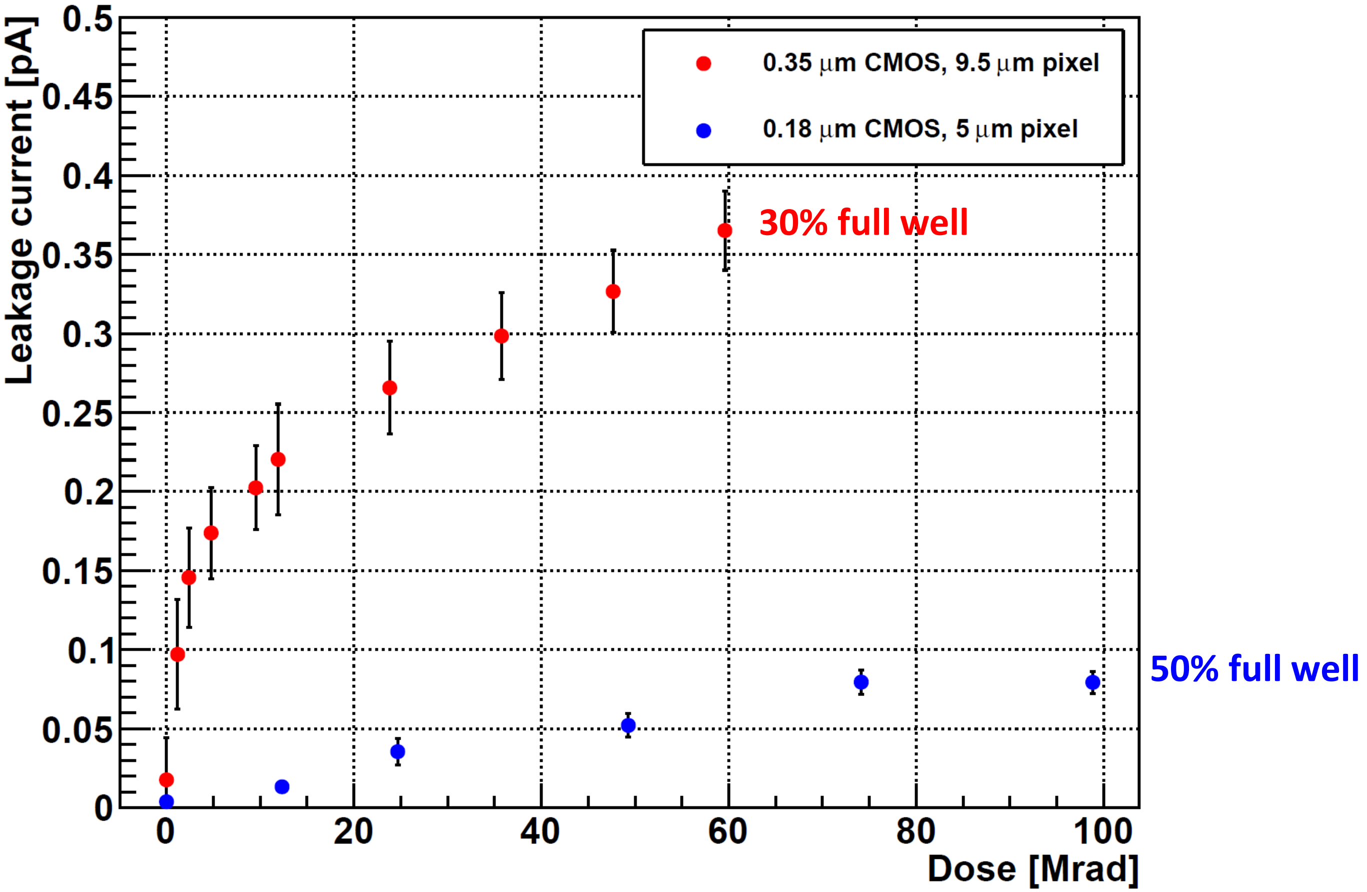
**Counting,
no centroiding**



**Centroiding with
2x upsampling**

- All data from 0.18 µm CMOS test chip, 5 µm pixel

Radiation Tolerance

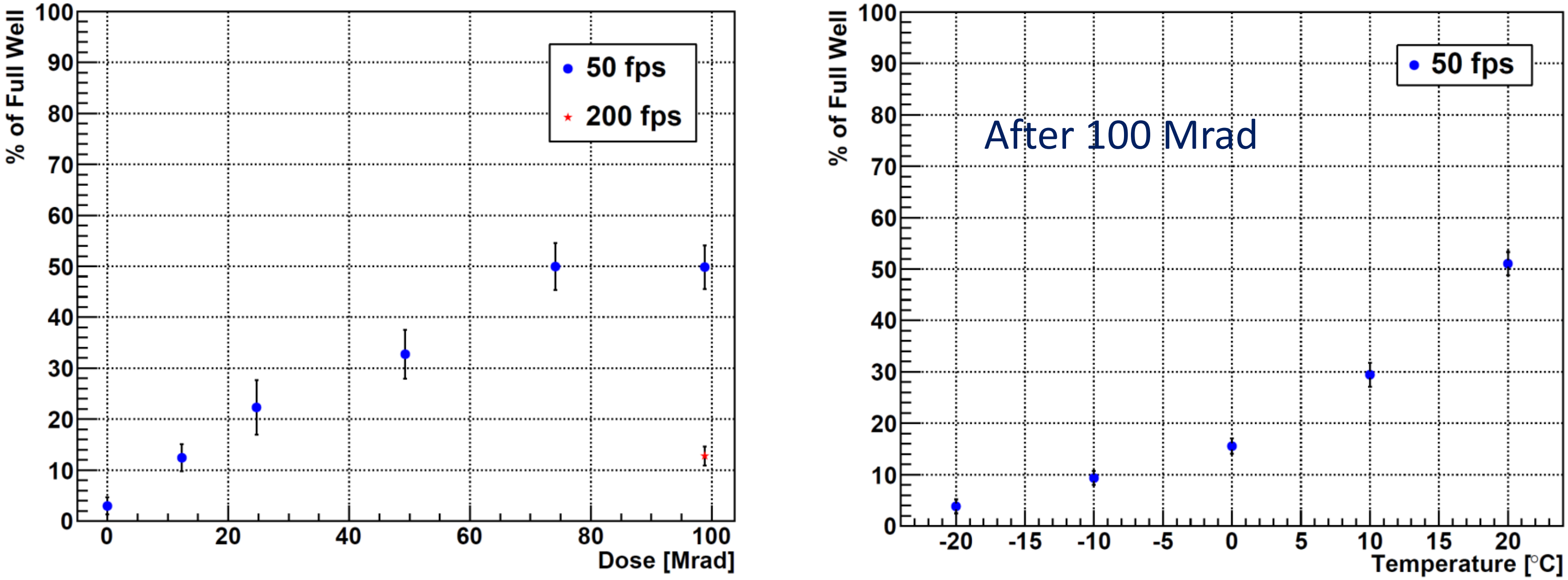


- Leakage current measured as a function of 300 keV electron dose
- Irradiation causes increase of leakage current that results in increased noise and loss of dynamic range
- Finer feature size process is more radiation tolerant, although dynamic range is lower due to lower operation voltage

[Nuclear Instruments and Methods A 635 (2011) 69]

[2011 IEEE Nuclear Science Symposium Conference Record N39-4, 1946-1949]

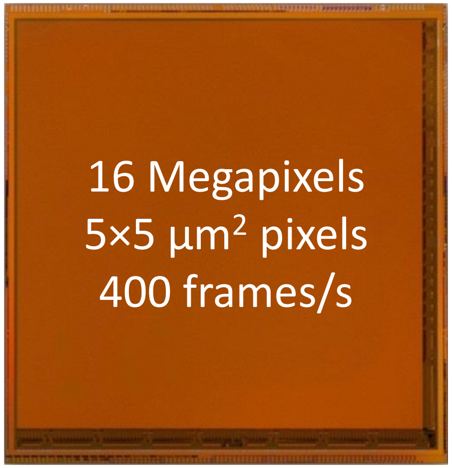
Recovering Performance with Speed and Cooling



- Pixel leakage current (in % of Full Well) measured on 0.18 µm test chip as a function of 300 keV electron dose, and as a function of temperature after the irradiation experiment
- Leakage current decreases linearly with integration time: can be reduced 4x by operating the sensor at 4x faster frame rate
- Leakage current strongly depends on temperature: pre-irradiation dynamic range can be recovered by cooling to e.g. -20°C

[Nuclear Instruments and Methods A 635 (2011) 69]

K2: a next generation CMOS camera for TEM



16 Megapixels
$5\times5\ \mu m^2$ pixels
400 frames/s

http://www.gatan.com/K2/

Collaboration:

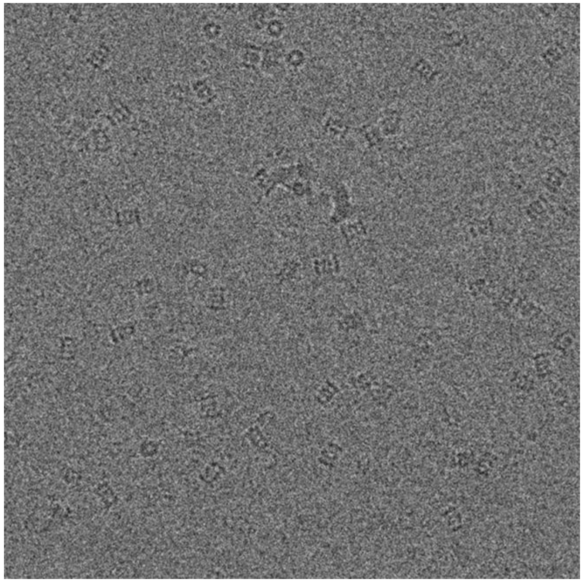
BERKELEY LAB
Lawrence Berkeley National Laboratory

gatan

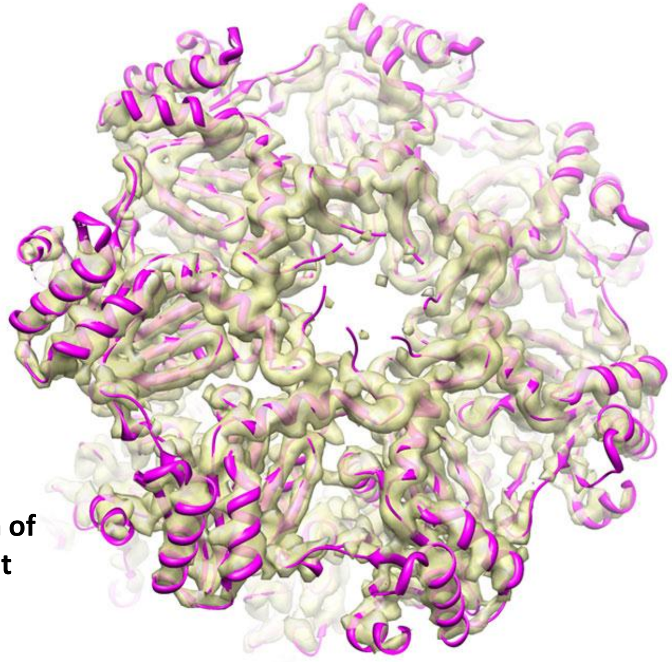
NSF

UCSF
University of California
San Francisco

Cryo-image of 20S proteasome

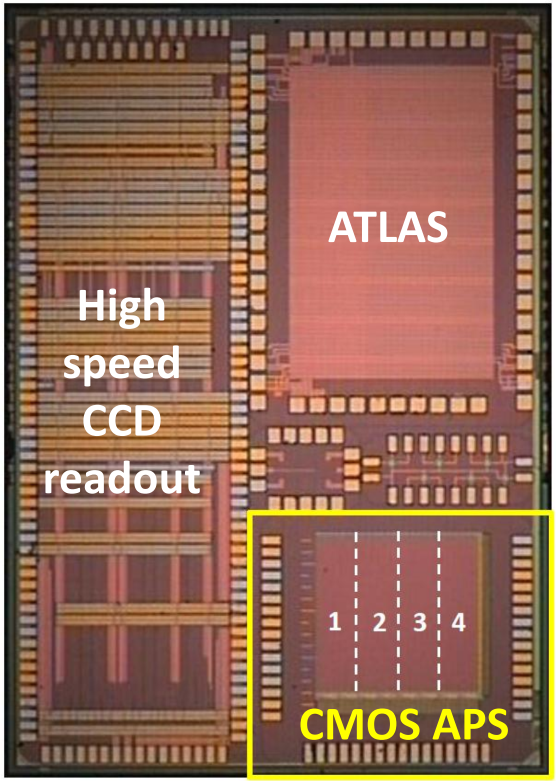


3D reconstruction of 20S proteasome at 4.4 Å resolution



[images courtesy of Dr. Yifan Cheng, Dr. Xueming Li, UCSF, from www.gatan.com/K2/]

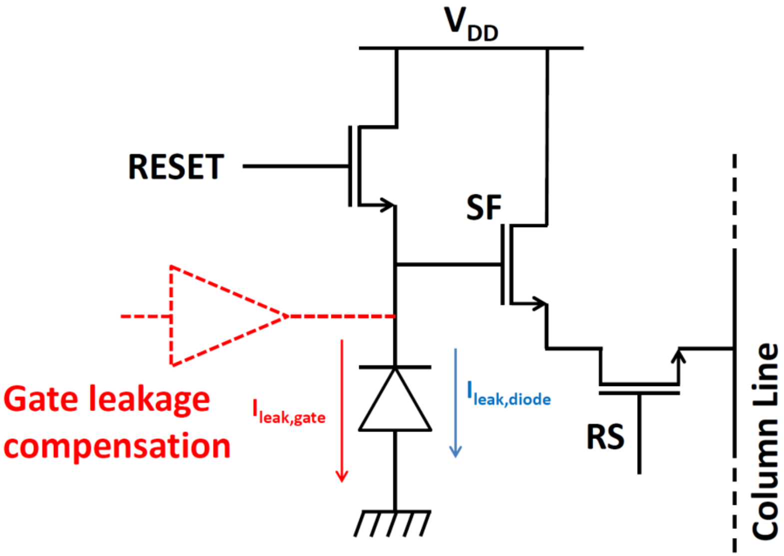
3rd Generation Development in 65 nm CMOS



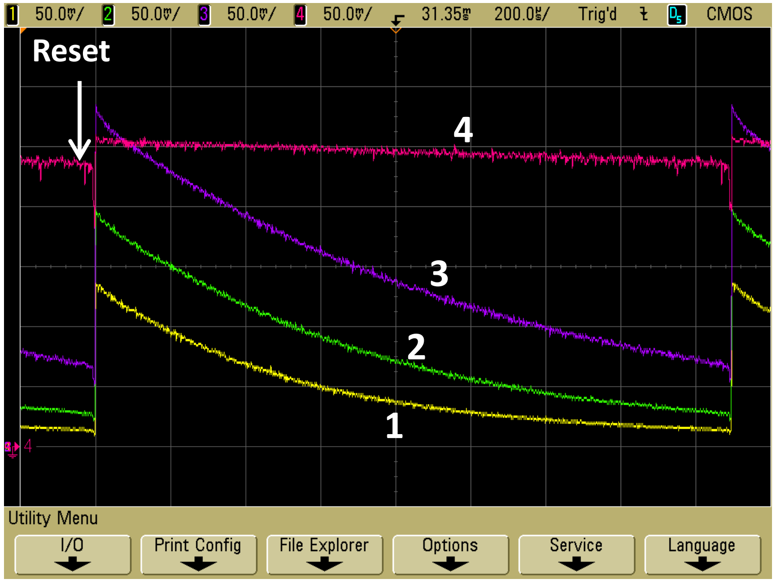
- Commercial 65 nm CMOS mixed-signal/logic process (not imaging process)
- Submission shared with BES and HEP projects (2011)
- CMOS APS: 400×400 pixels, 2.5 µm pitch, 1×1 mm^2 active area
- Implement 4 sectors with various pixel layouts

	Sector 1	Sector 2	Sector 3	Sector 4
Diode layout	TEAM-like	TEAM-like	TEAM-like	New “pseudo-pinned”
Gate Leakage Compensation	Yes	Yes	Yes	No
MOSFET layout	Enclosed Layout	Standard	Standard	Standard
MOSFET V_{th}	Standard (0.3 V)	Standard (0.3 V)	Low (0.2 V)	Low (0.2 V)

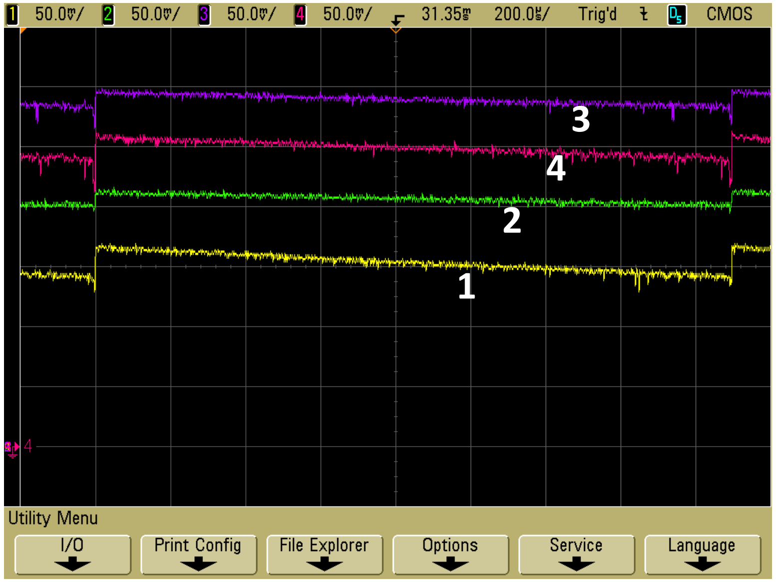
Features of 65 nm CMOS Process: Gate Leakage



- Thin oxides are good for radiation hardness but also subject to conduction via tunneling → "gate leakage" (temperature independent)
- Observe strong leakage current effect in Sectors 1-2-3 (thin oxides around diode)
- Gate leakage compensation performed via on-pixel biasing structure



Un-compensated



Compensated

Features of 65 nm CMOS Process: Doping Profile

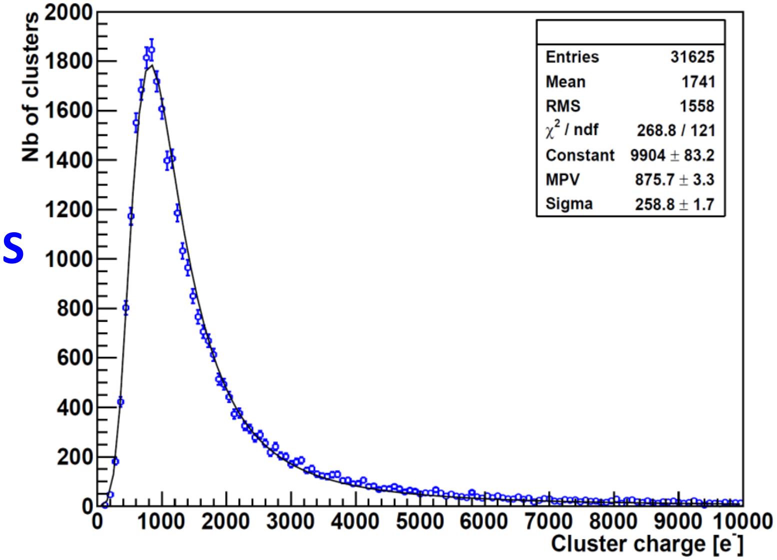
- The 65 nm CMOS process used in our test chip has a non-epitaxial substrate
- Expect large signals (good) but also large diffusion (bad) → expect Point Spread Function performance to be compromised by large diffusion in the substrate

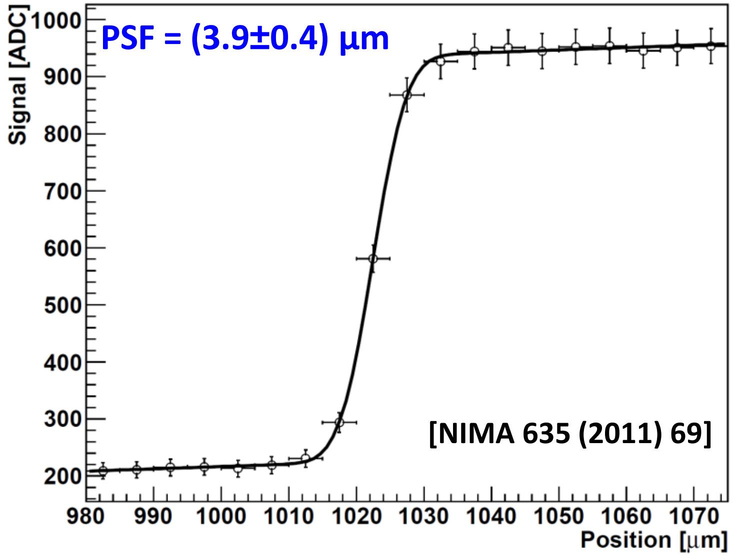
Electron Detection and PSF: 65 nm vs. 0.18 µm

300 keV electrons, pulse height

Point Spread Function

0.18 µm CMOS
5 µm pixel

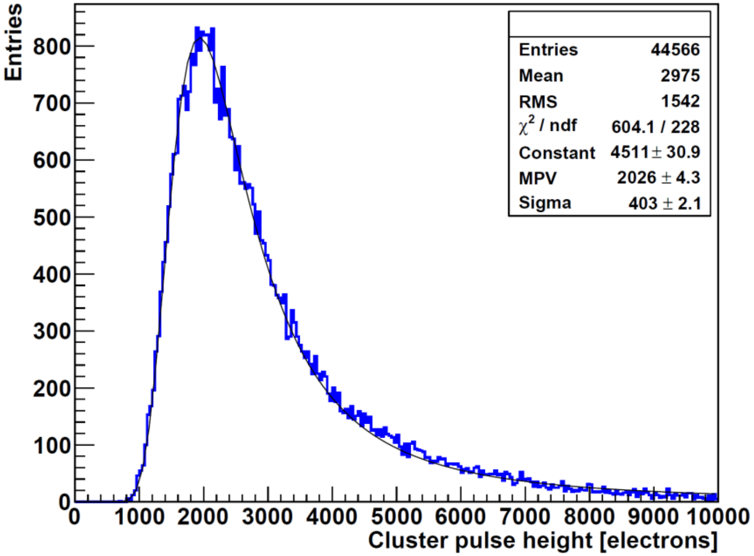


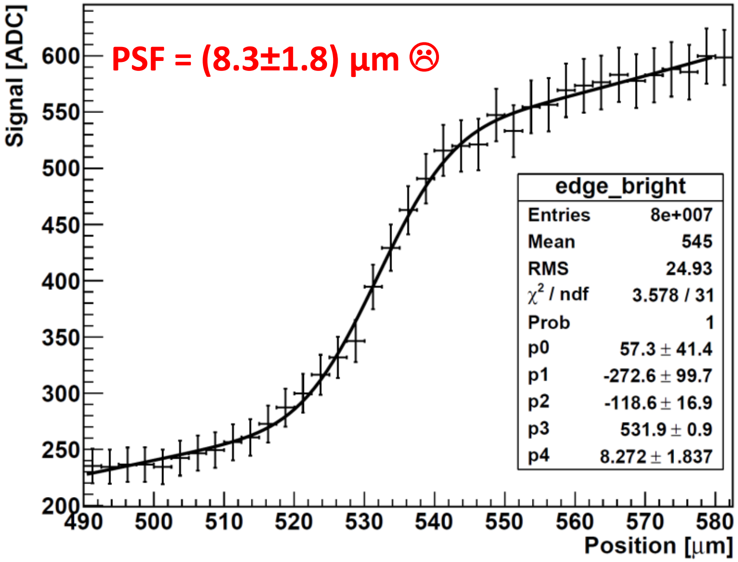


PSF = (3.9±0.4) µm

[NIMA 635 (2011) 69]

65 nm CMOS
2.5 µm pixel



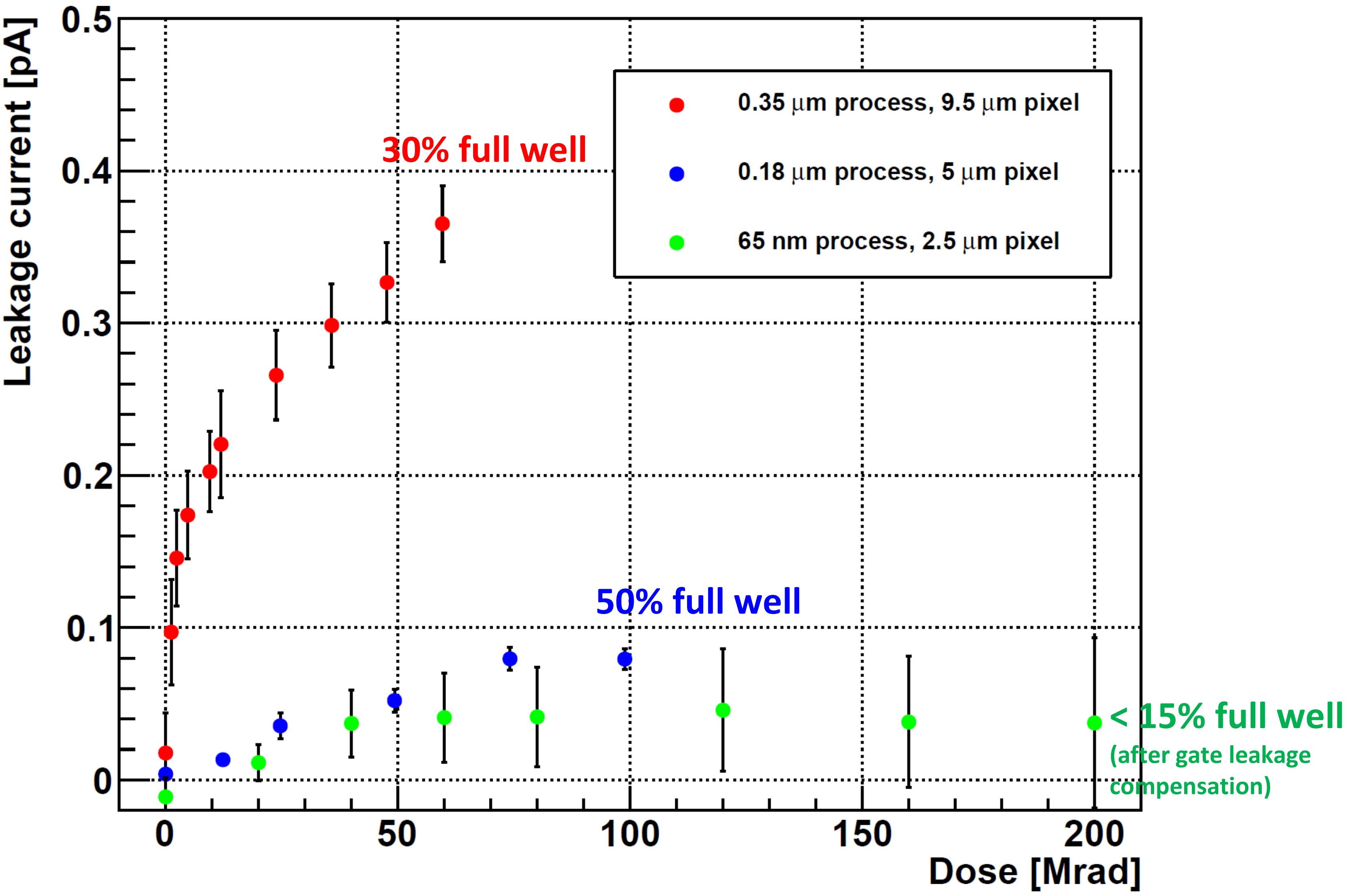


PSF = (8.3±1.8) µm ☹

Pixel Performance Comparison

Process	Pixel pitch [μm]	Gain [μV/e$^-$]	Noise @ RT [e$^-$]	I_{leak} [fA]
0.35 μm (TEAM)	9.5	9.4	30	10
0.18 μm (TEAM-like)	5.0	15.5	35-40	4
0.18 μm (4T/photogate)	5.0	23.3	12	< 1
65 nm (TEAM-like)	2.5	7-10	70-80	< 1 **Gate leakage compensated**
65 nm (pseudo-pinned)	2.5	21	50	8

Radiation Hardness: Compare Three Generations



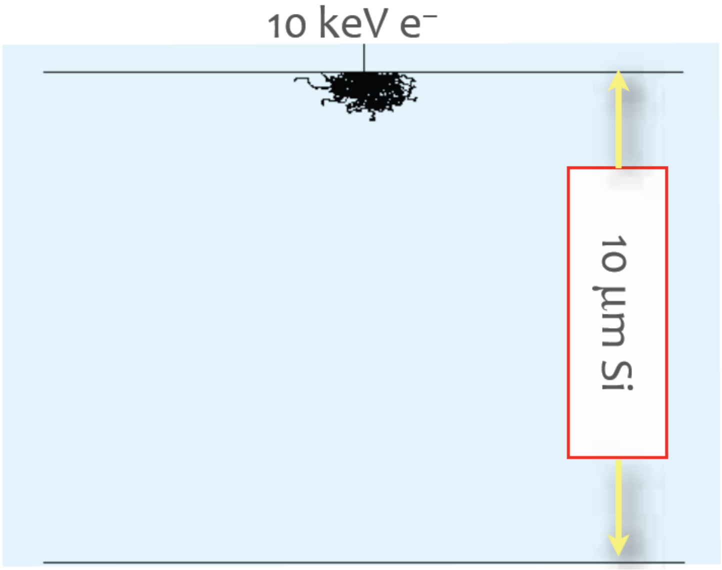
- All data for 300 keV e^-, 20 ms integration time, +5°C

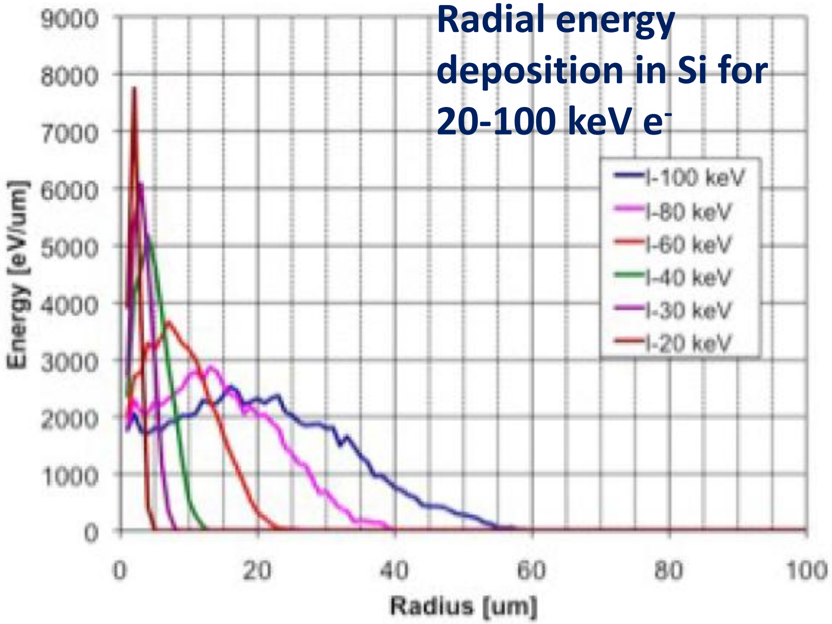
Outlook: Towards a Low-Energy Electron Detector

- Significant interest in the TEM community for low-energy electrons (e.g. to limit radiation damage on sample)
- For E ≤ 20 keV a front illuminated detector is not efficient due to the inactive layers on the sensor surface (metal, passivation)
- Idea for a low energy TEM detector:
  - ➢ Thin the sensor to the few µm thick epitaxial layer
  - ➢ Implant a thin conductive entrance window on the backside
  - ➢ Operate in back-illumination

→ Thin contact may allow full depletion of epilayer (with high enough resistivity), improving charge collection and PSF performance

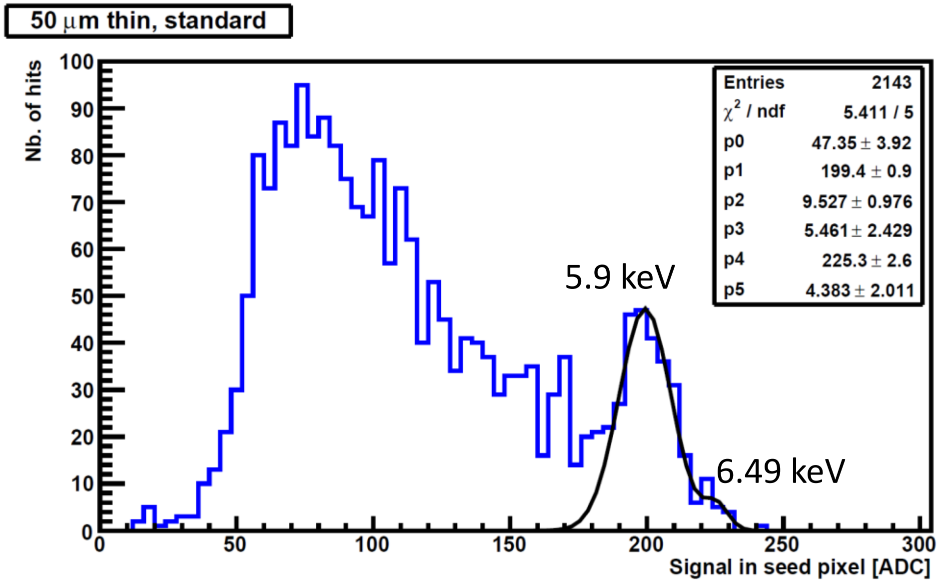
[Footnote: this works also for a soft (≤1 keV) X-ray detector…]

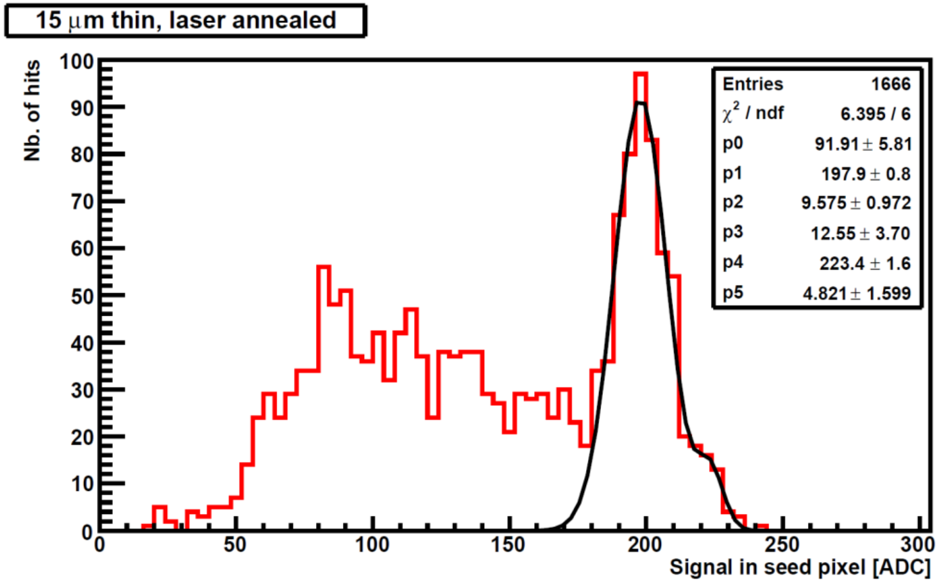


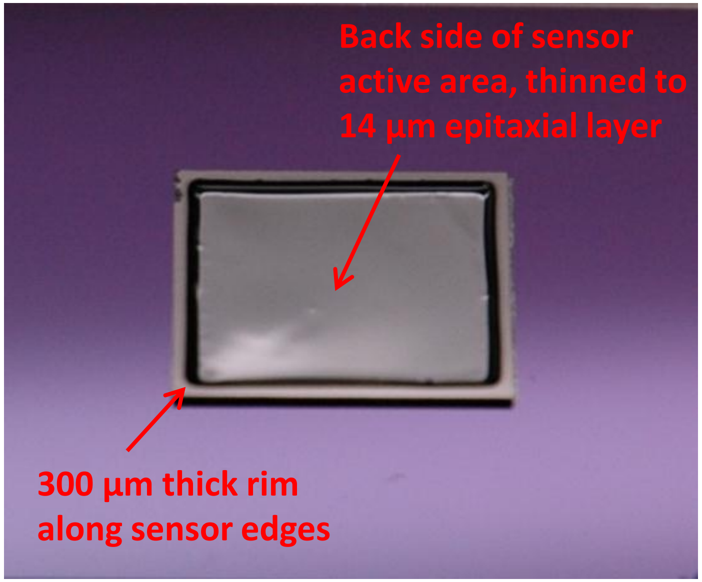


Radial energy deposition in Si for 20-100 keV e⁻

Thinning to the Epitaxial Layer





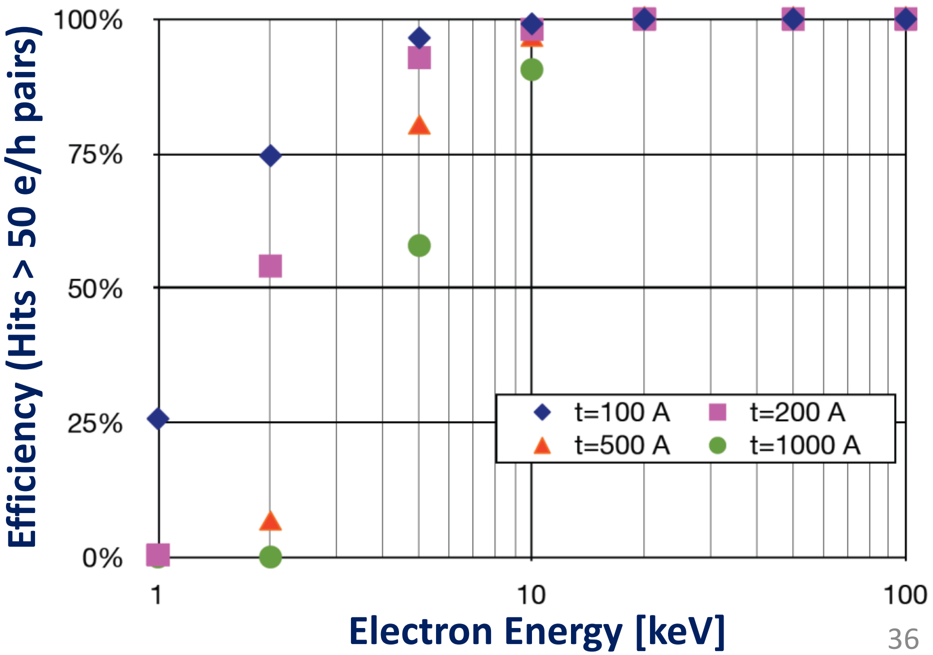


- TEAM1k sensor (0.35 µm CMOS) thinned to the epitaxial layer by chemical etching (Mike Lesser, U. Arizona)
- Backside of epilayer implanted at LBNL
- Post-processing functionality demonstrated with ^{55}Fe in the lab
- Work in progress!

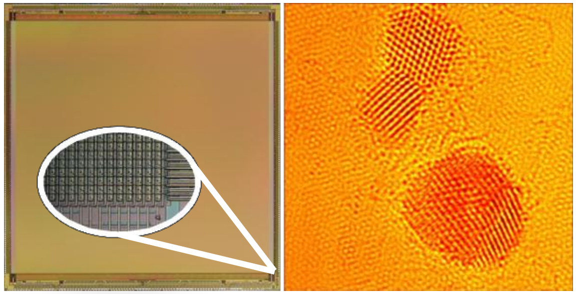
Thin Entrance Window Development

Process	Window thickness	Availability	Status	
Low energy implantation + 500°C annealing	1000-2000 Å	In-house	Successfully applied to SOI, CCD and CMOS devices	Low T
Low energy implantation + laser annealing	400-700 Å	Commercial vendor	Several SOI prototypes functional after processing	
a-Si contact deposition by sputtering	300 Å	In-house	Prototypes functional, high leakage	
In-Situ Doped Polysilicon (ISDP)	100-200 Å	In-house	Standard MSL process	High T
Molecular Beam Epitaxy (MBE)	50-75 Å	NASA/JPL	Developing in-house capability (2013)	Low T

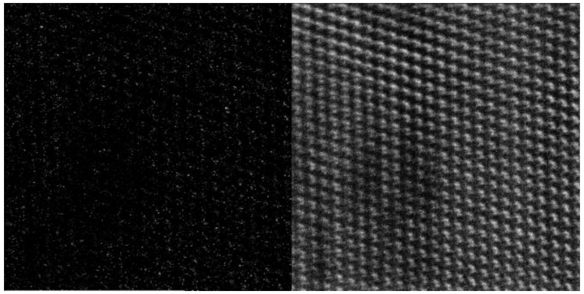
- On-going R&D on entrance window processes, driven by soft X-ray detector applications (e.g. ALS, NGLS)
- Need low-temperature process (< 500°C) to be applicable to metalized, fully processed devices
- In-house development of ~100 nm thin implant enables close to 100% efficiency for ~10 keV electron detection



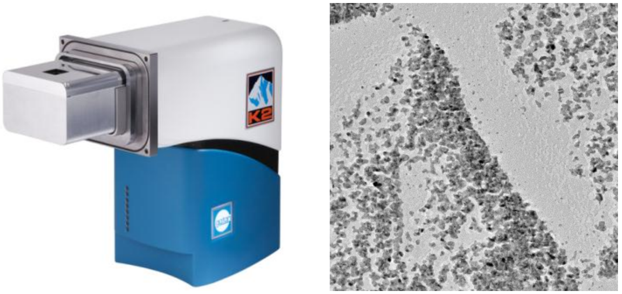
Summary



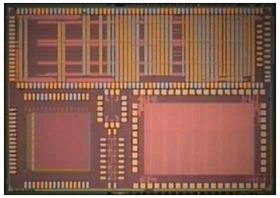
We have successfully developed CMOS Active Pixel Sensors as high-speed, radiation-hard detectors for high resolution and fast imaging in Transmission Electron Microscopy



We have demonstrated an imaging technique based on single electron detection at high frame rate, yielding a dramatic improvement in resolution and quantum efficiency at a reduced electron dose



Our 2nd Generation technology has reached the market in a camera system with an LBNL-designed 16 Megapixel, 400 frames/s CMOS sensor and integrated hardware processing for electron counting and cluster imaging



We continue to push the limits of the technology by investigating advanced manufacturing processes, opening opportunities for higher integration and complexity (e.g. on-chip ADC, data processing)

Thank You!

Acknowledgements

P. Denes (P.I.)

N. Andresen, D. Doering, D. Gnani, J. Joseph, B. Krieger, C. Tindall,
LBNL, Engineering Division

T. Duden, P. Ercius, A. Gautam, C. Ophus, V. Radmilovic,
LBNL, National Center for Electron Microscopy

M. Battaglia, ***University of California Santa Cruz and LBNL***

P. Giubilato, ***University of Padova, Italy***

S. Gubbens, P. Mooney, ***Gatan, Inc.***

D. Agard, ***University of California San Francisco***