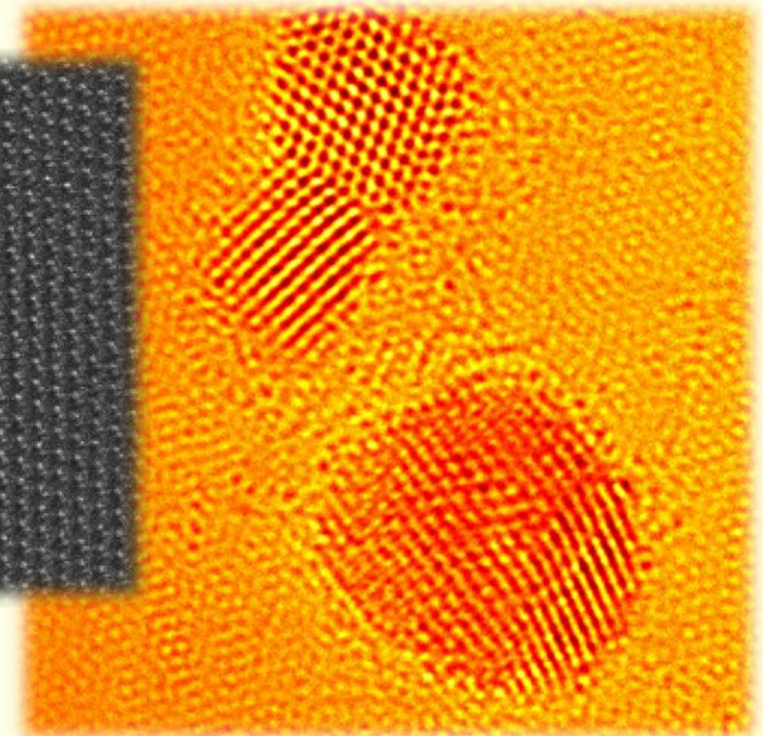
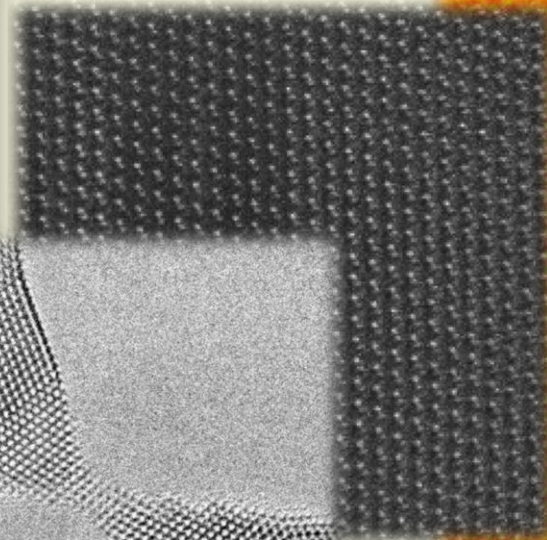


# High speed, radiation hard CMOS imagers for high resolution Transmission Electron Microscopy



**Devis Contarato**

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A. Gautam, P. Giubilato, D. Gnani, C. Ophus,  
V. Radmilovic (LBNL)

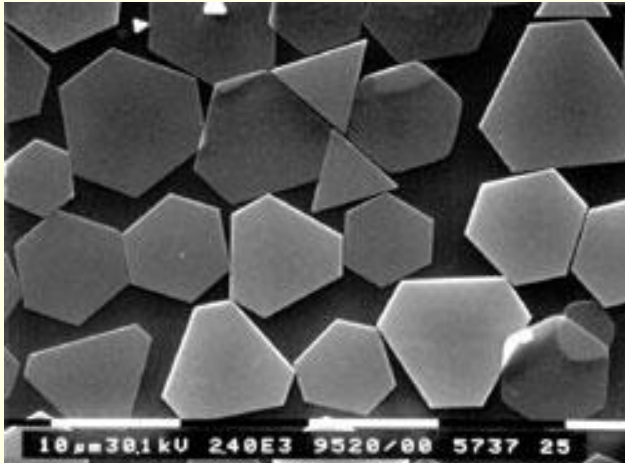


**TIPP 2011**

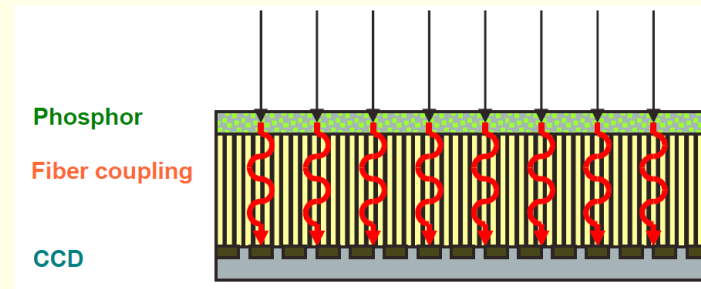
**Technology and  
Instrumentation in  
Particle Physics 2011**

**Chicago, June 9, 2011**

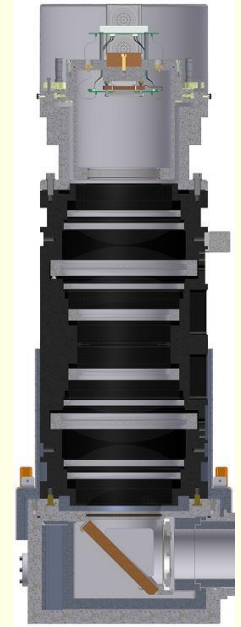
# Introduction: imaging in TEM



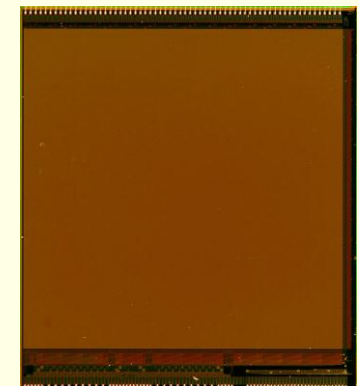
Film



Optically Coupled CCDs



- Traditional direct detectors such as **film** or **image plates** have large area and high granularity, but are slow and do not allow fast dynamic imaging
- **Optically-coupled CCDs** have limited Point Spread Function (PSF) due to the presence of the scintillator and to back-scattering from fiber optics
- **Direct semiconductor detectors** allow higher PSF and Detective Quantum Efficiency (DQE), can be high-speed, high-granularity and include processing capabilities (e.g. back-scatter discrimination) → ideal candidates for applications such as fast in-situ imaging or low-dose biological cryo-EM

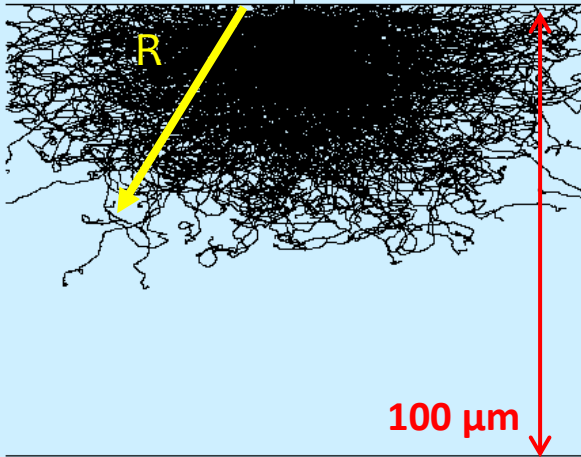


Direct detectors  
(semiconductor)

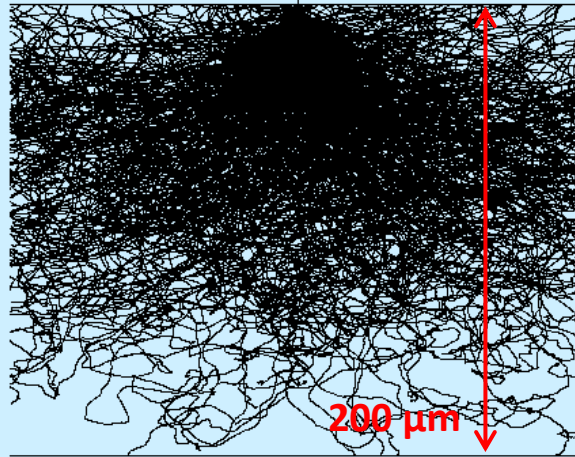


# Driving detector R&D: multiple scattering

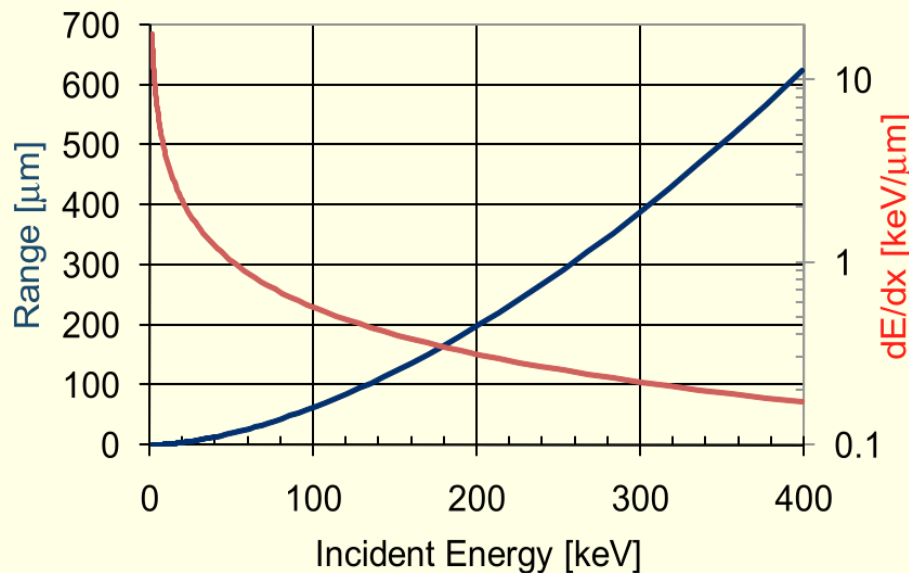
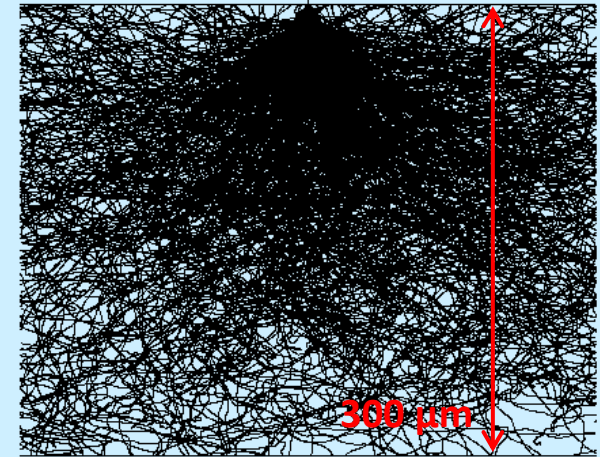
100 keV e<sup>-</sup>



200 keV e<sup>-</sup>

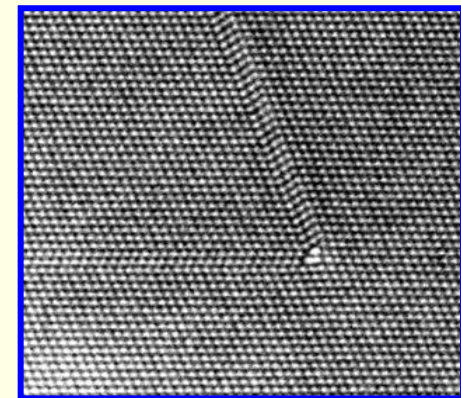
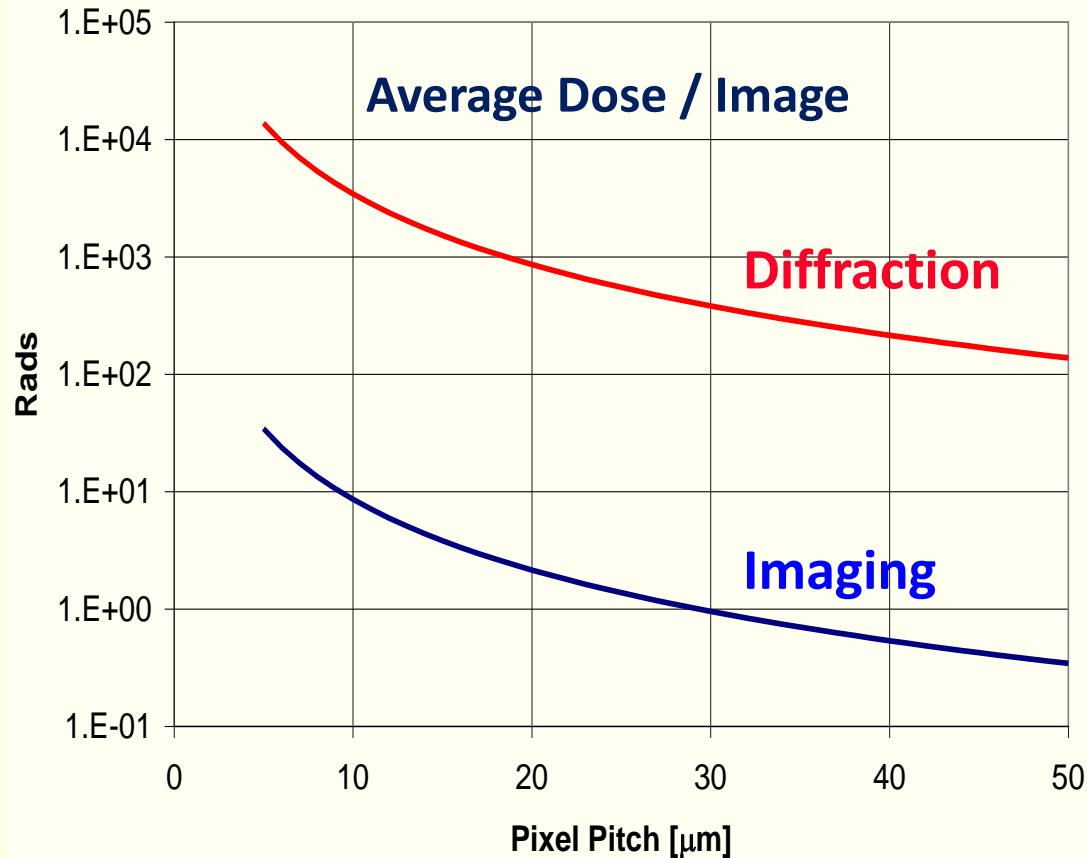


300 keV e<sup>-</sup>



- Energies of interest to TEM: 80-400 keV
- Electron range  $R$  [ $\mu\text{m}$ ]  $\sim E$  [keV]
- Energy loss  $dE/dx \propto 1/E$
- Need for a thin sensitive layer to minimize scattering contribution to Point Spread Function

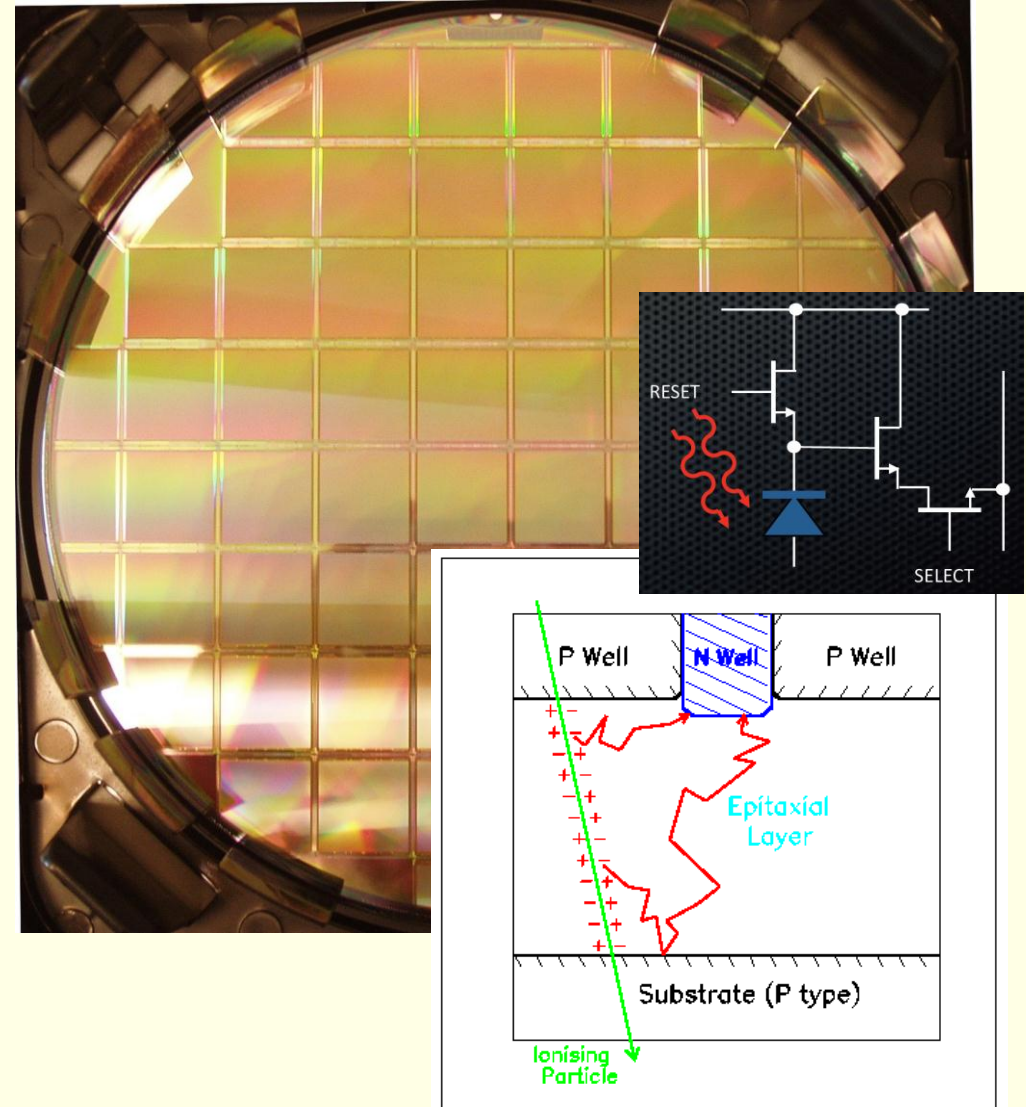
# Driving detector R&D: radiation hardness



- **Imaging mode:** O(Mrad) ionising dose expected for typical yearly usage
- **Diffraction mode:** very high doses localized in bright spots
- Radiation tolerance requirements comparable with HEP applications
- Highly sensitive detectors require low node capacitance  $\rightarrow$  high sensitivity to radiation  $\rightarrow$  need for careful radiation tolerant design of pixel cell

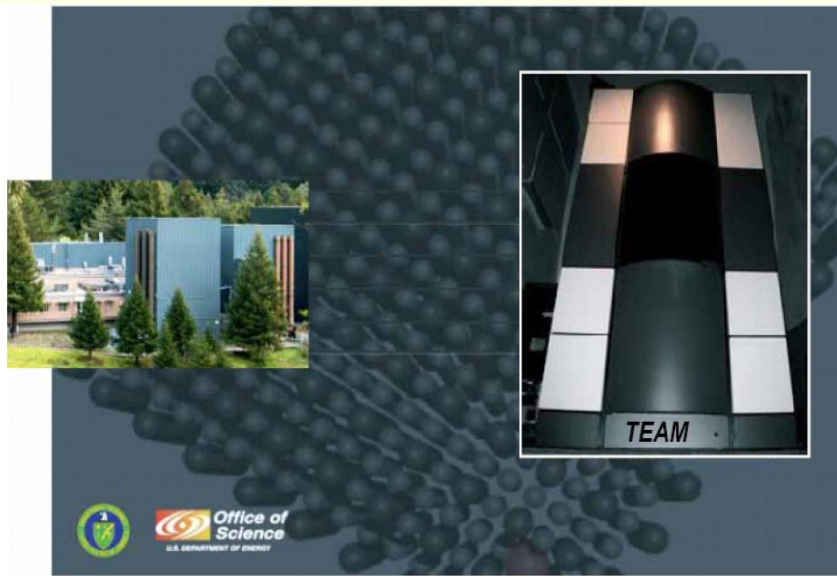
# CMOS imagers: new eyes for TEM

- CMOS Monolithic Active Pixel Sensors provide high resolution and low material budget particle detectors at low cost, as they are fabricated in commercial manufacturing processes
- CMOS pixels are being developed as an alternative to conventional, optically-coupled CCD cameras for TEM imaging. Advantages:
  - **Single electron sensitivity** from direct electron detection
  - **Excellent Point Spread Function**: small pixels and thin sensitive volume
  - **High readout speed**: several 100 frame/s achievable for Megapixel-scale imagers
  - **Improved radiation hardness**: lifetime of several years possible



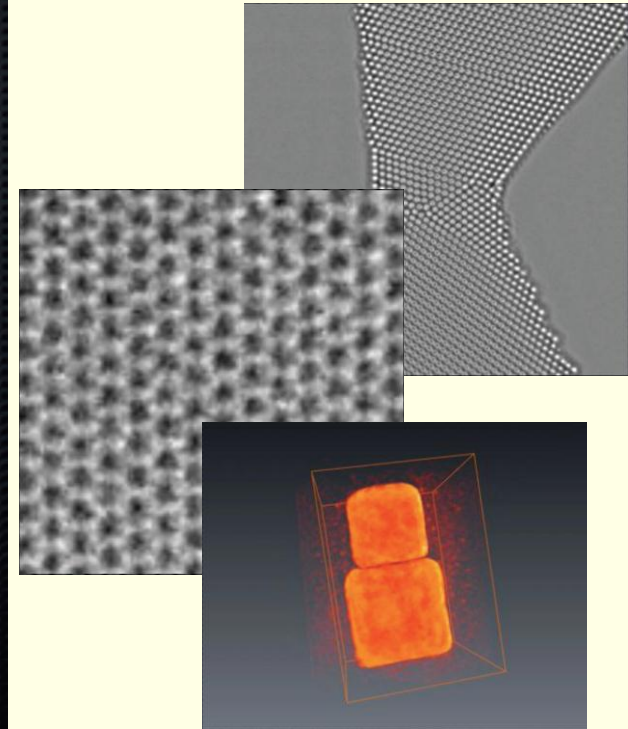
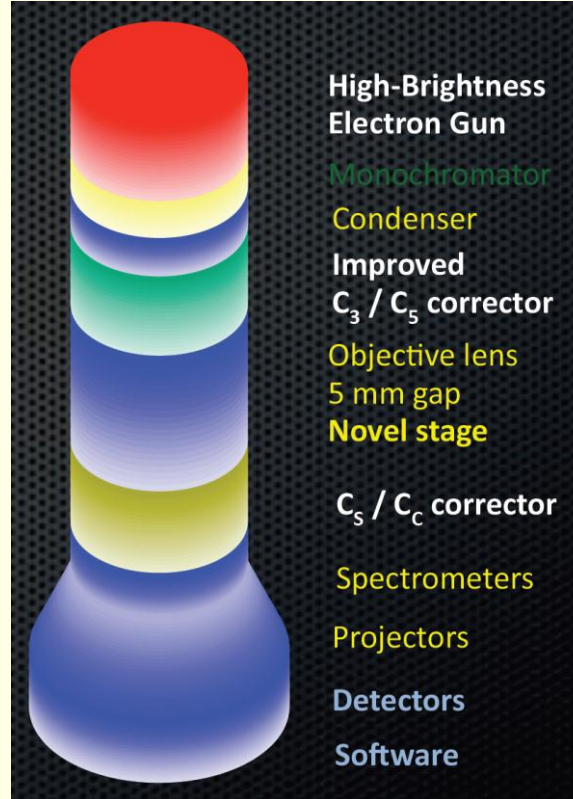


# The TEAM project



## 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  $\delta E$
- Major advances in electron optics and aberration correction to obtain excellent spatial resolution
- Capabilities for 3D tomography and in-situ experiments; development of novel stage for atomic-scale tomography and novel detector for in-situ/dynamics studies
- Project completed 09/2009, now national user facility



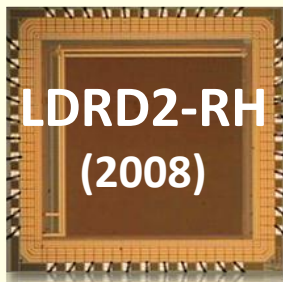
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CMOS imagers for high-resolution TEM

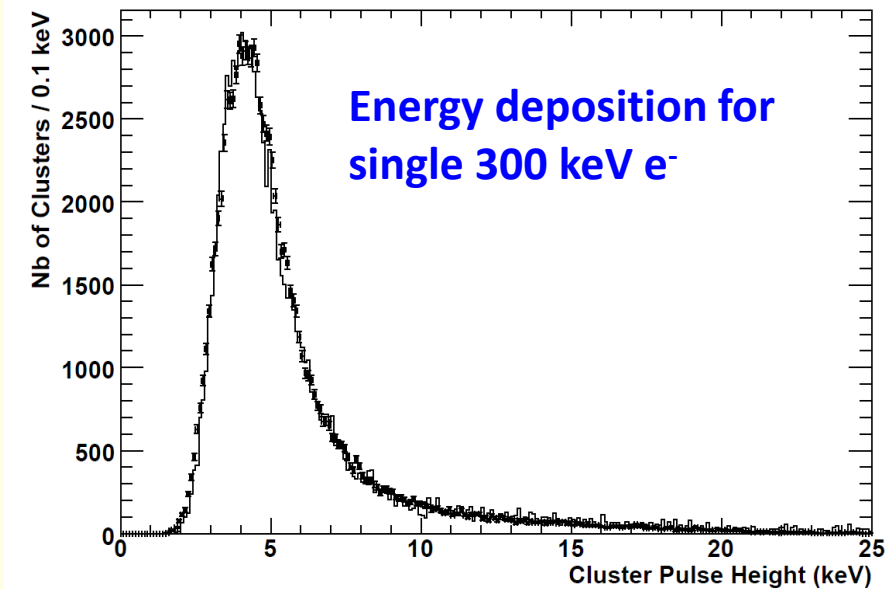


# The TEAM detectors

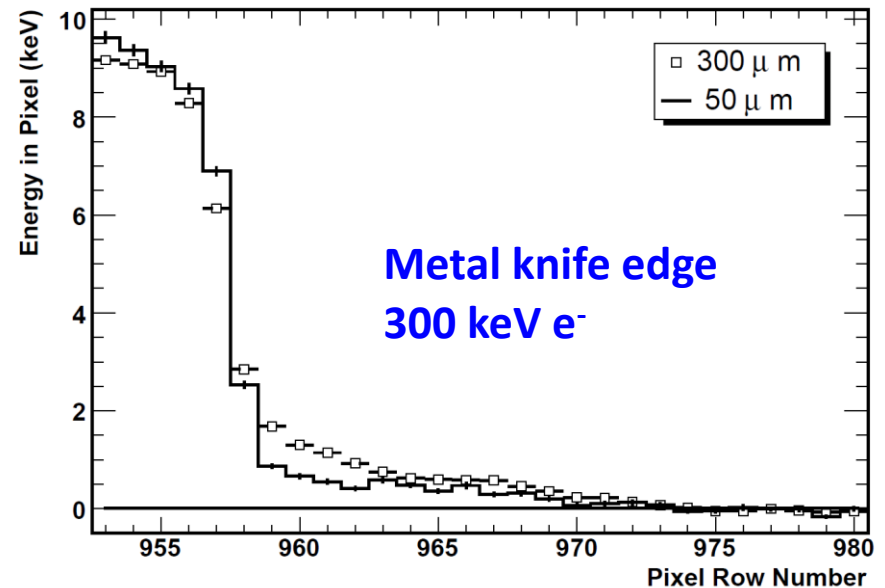
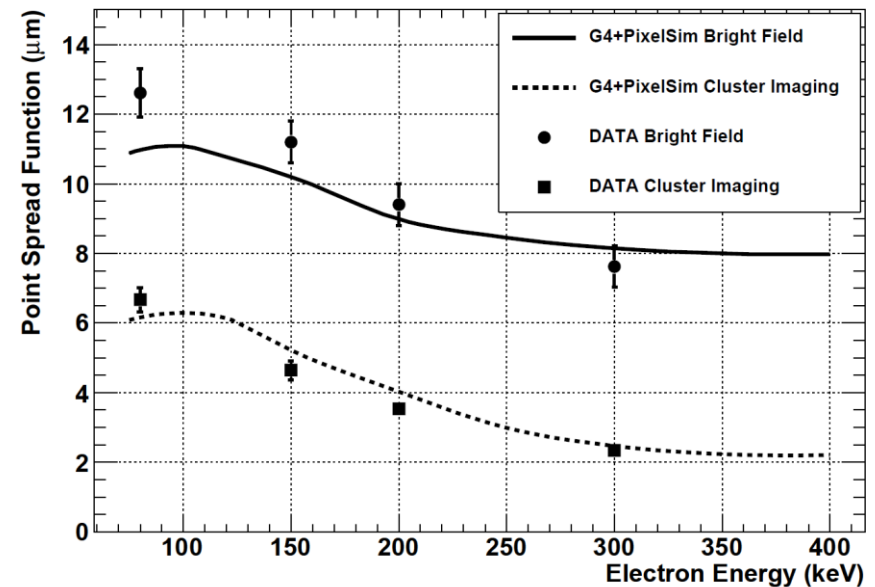
- CMOS imagers manufactured in commercial 0.35  $\mu\text{m}$  CMOS process
- $9.5 \times 9.5 \mu\text{m}^2$  pixels, 50  $\mu\text{m}$  thin, single image exposure time of 2.5 ms; rad-hard pixel layout selected from small scale prototype
- TEAM1k detector deployed as 400 frames/s direct detector for the TEAM I microscope
- TEAM2k covers full CMOS reticle ( $2 \times 2 \text{ cm}^2$ ), to be commissioned on TEAM I in 2011



# Performance of the TEAM detectors



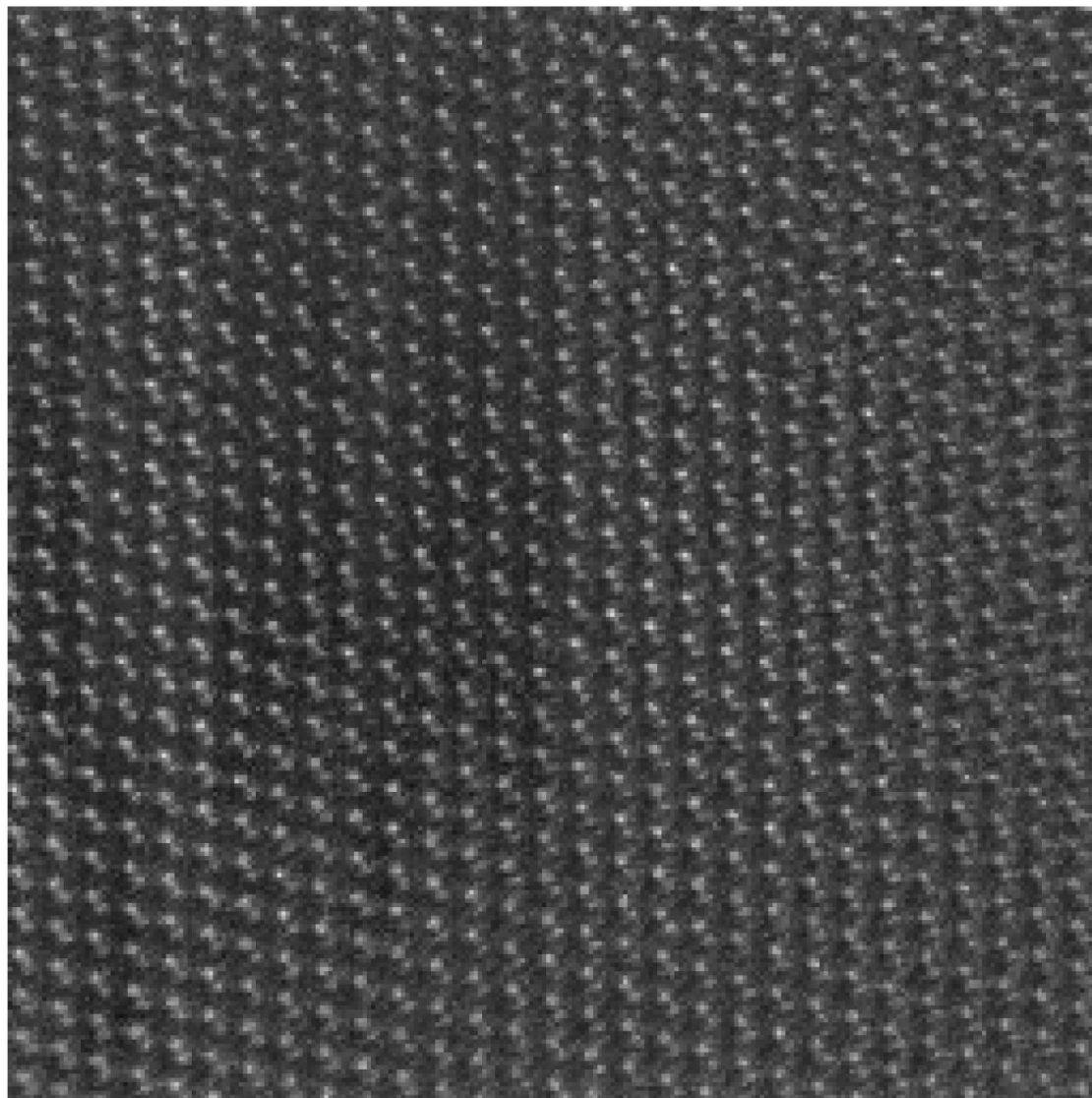
- Single electron sensitivity with low pixel noise (30 electrons) and  $\langle S/N \rangle \sim 15-20$
- Sensor thinning to 50  $\mu\text{m}$  improves Line Spread Function (LSF), thanks to reduction of back-scattering from detector substrate
- LSF can be dramatically improved by reconstructing the impact position of each single electron and reconstructing the image from the superimposition (“cluster imaging”)
- Very good agreement of data with simulation



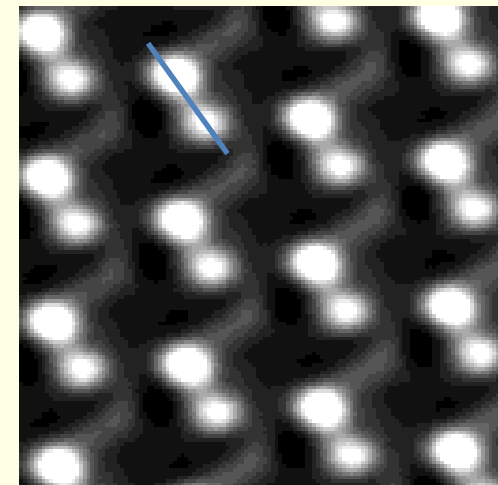
[M. Battaglia et al., Nuclear Instruments and Methods A 622 (2010) 669]



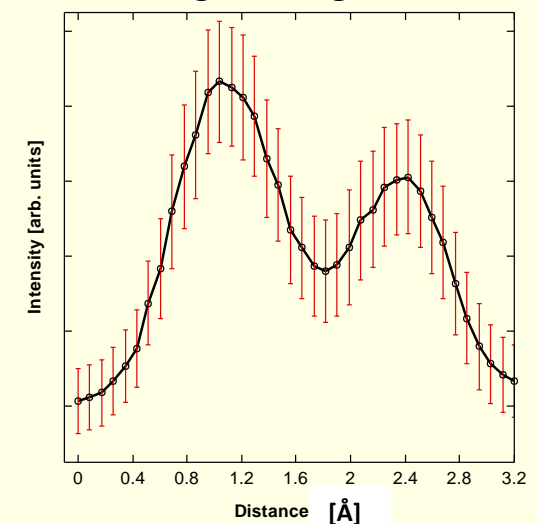
# Imaging Si[110] dumbbells in 2.5 ms



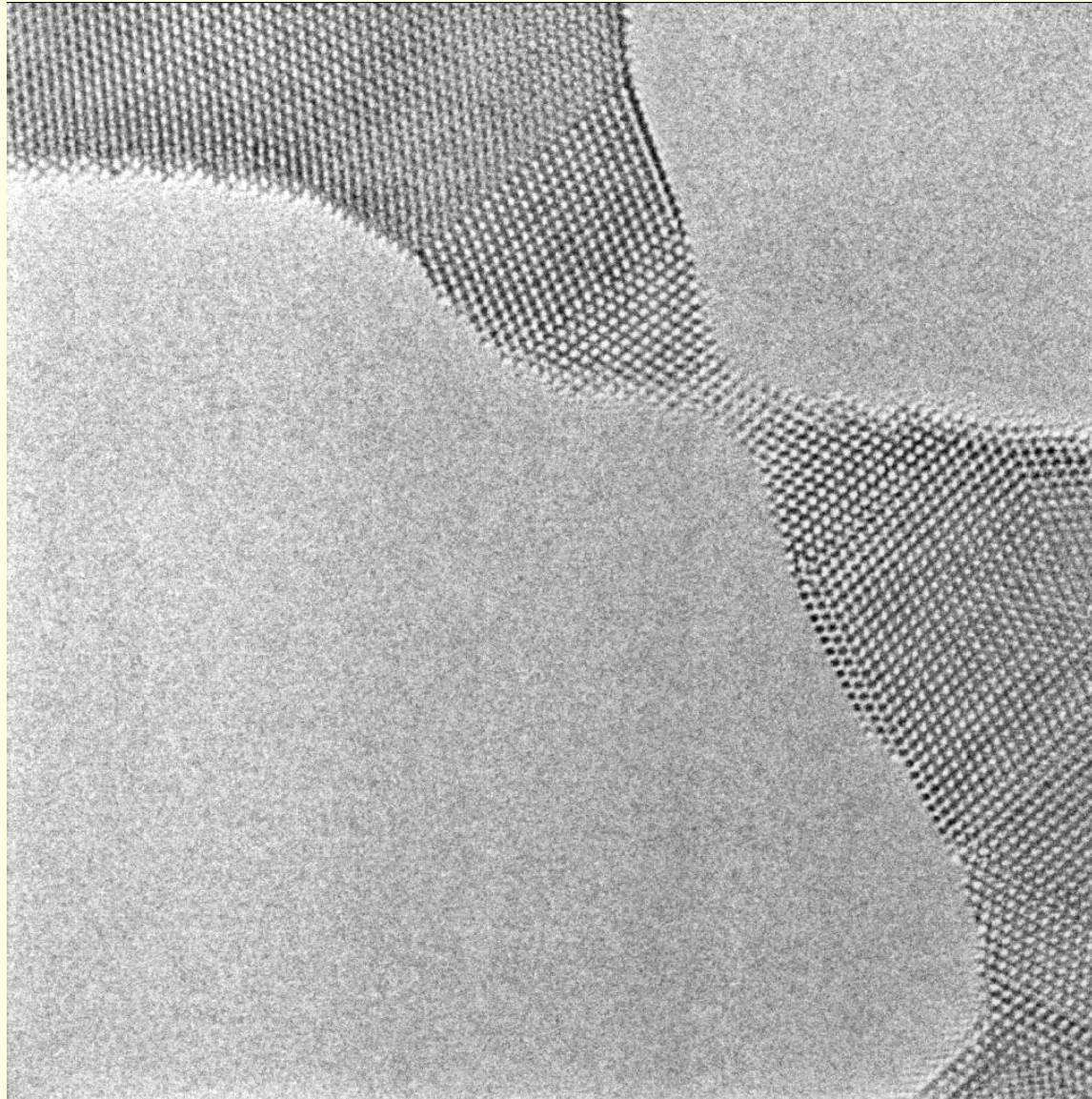
300 keV e<sup>-</sup>, 1024×1024 pixels (TEAM1k chip); single, raw, unprocessed 2.5 ms exposure



Line profile across dumbbell in averaged image



# Au nanobridge (300 keV)

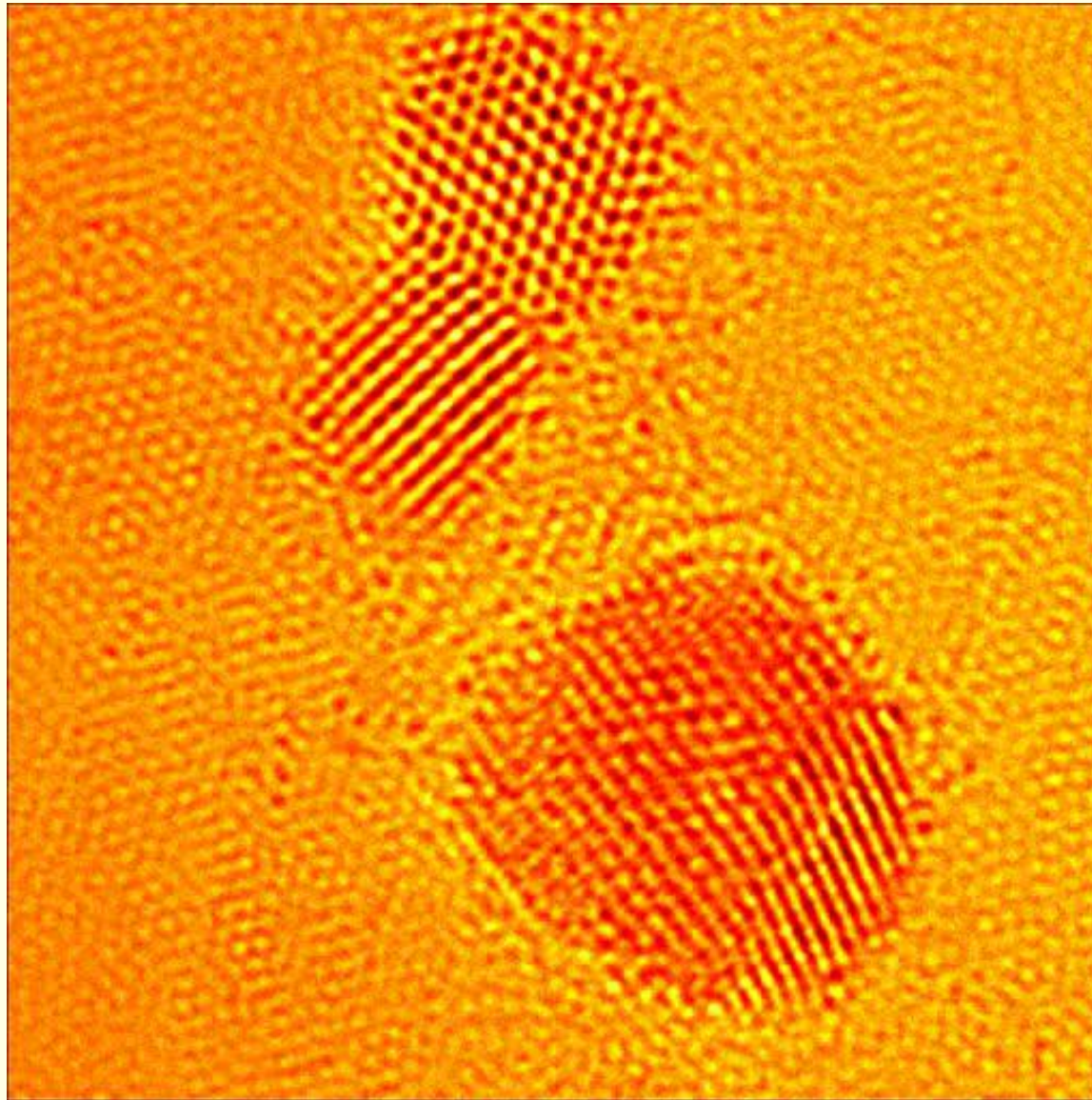


TEAM I  
300 keV  
Single image  
20 ms exposure  
(raw data, 2010)

[A. Gautam, NCEM]



# Au/FeO on graphene nanobridge (80 keV)

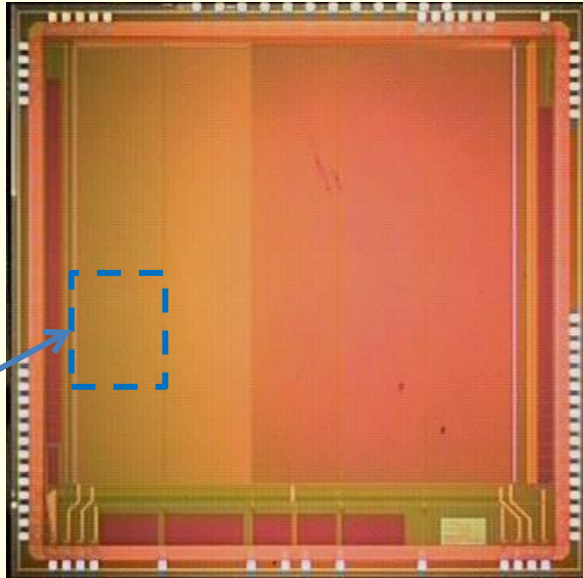


TEAM I  
80 keV  
Average of 40  
2.5 ms images  
(raw data, 2010)

[P. Ercius, C. Ophus, V. Radmilovic, NCEM]

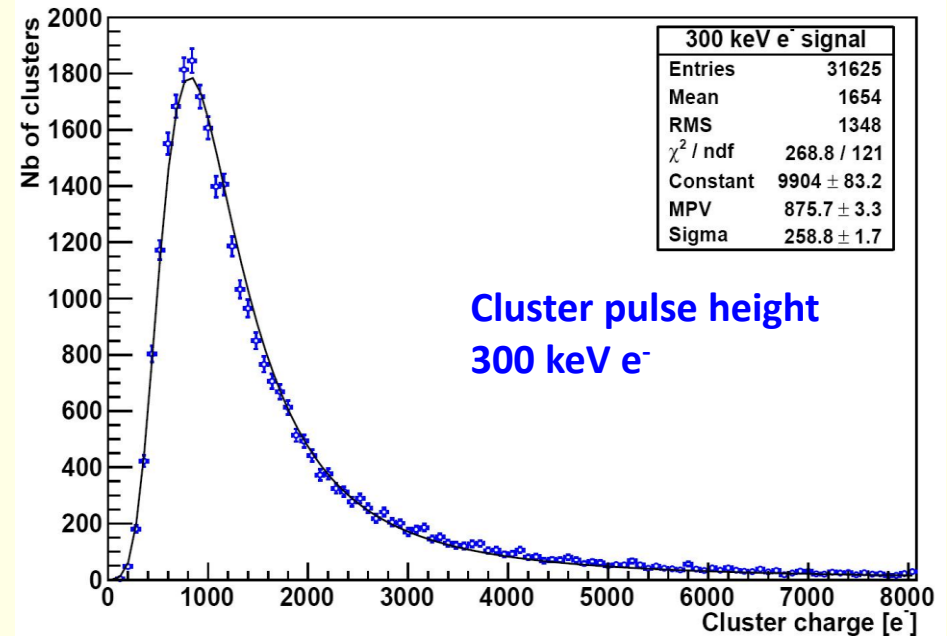


# Scaling down to 0.18 $\mu\text{m}$ process



152 x 192 pixel section with best layout (0.76 x 0.96 mm<sup>2</sup>)

- Prototype chip funded by Howard Hughes Medical Institute (HHMI)
- 0.18  $\mu\text{m}$  commercial CMOS process
- 760x768 pixels of 5  $\mu\text{m}$  pixel pitch, 50  $\mu\text{m}$  thin
- 12 pixels layouts, best architecture selected from radiation hardness performance as baseline for reticle size camera
- Detection performances comparable with TEAM sensors
- 0.18  $\mu\text{m}$  w.r.t 0.35  $\mu\text{m}$  process shows higher gain, lower leakage but reduced dynamic range



Fabrication process [ $\mu\text{m}$ ]	Conversion Gain [ $\mu\text{V}/\text{e}^-$ ]	Noise (@ RT) [ $\text{e}^-$ ]	Leakage current [fA]	Well depth [ $\text{e}^-$ ]
0.35	9.4	30	10	90000
0.18	15.5	35-40	4	23000

[D. Contarato et al., NIMA A 635 (2011) 69]



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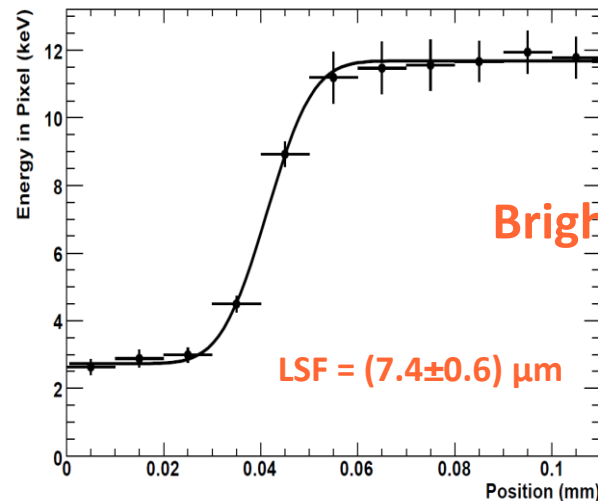
CMOS imagers for high-resolution TEM



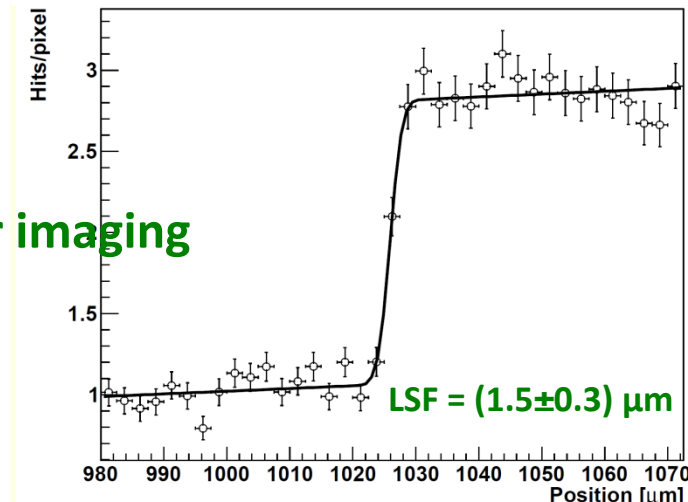
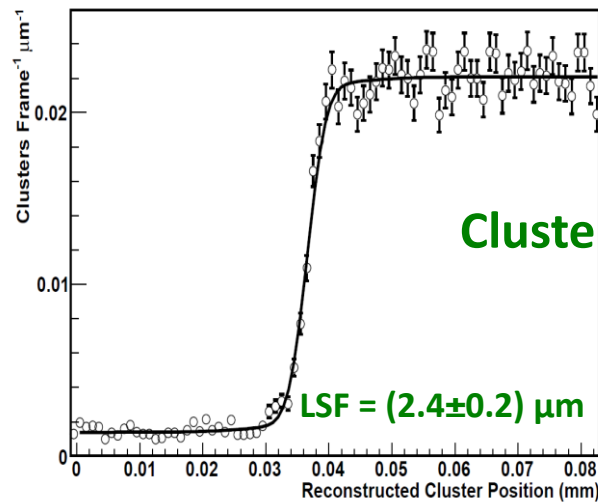
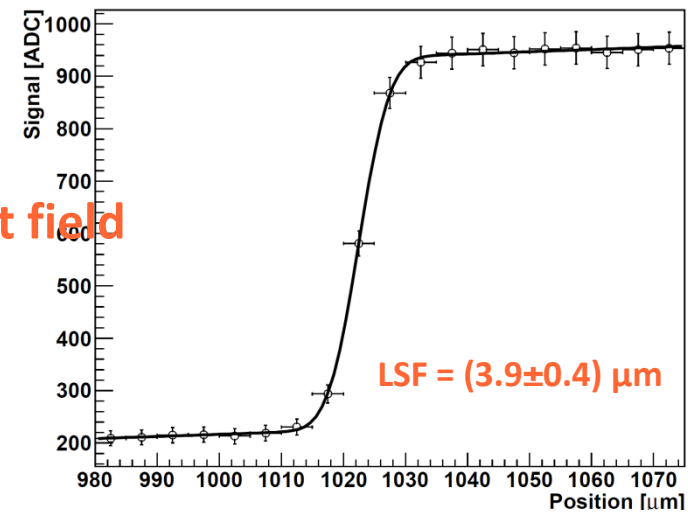
# Point spread function improvement by clustering

- For low electron rates ( $< 0.05 \text{ e}^-/\text{pixel}$ ), clusters of pixels interested by signals generating from single electron interactions can be distinguished
- 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 (technique widely used in HEP)
- The “cluster imaging” technique composes higher resolution images from the superimposition of many frames with sparsified electron hits
- Point spread function of  $O(1 \text{ }\mu\text{m})$  possible with pixel pitches of  $O(10 \text{ }\mu\text{m})$

TEAM1k, 9.5  $\mu\text{m}$  pixels



HHMI, 5  $\mu\text{m}$  pixels



Gold wire edge, 300 keV  $\text{e}^-$

[M. Battaglia et al., NIM A 608 (2009) 363]

[M. Battaglia et al., NIMA A 622 (2010) 669]

[D. Contarato et al., NIMA A 635 (2011) 69]

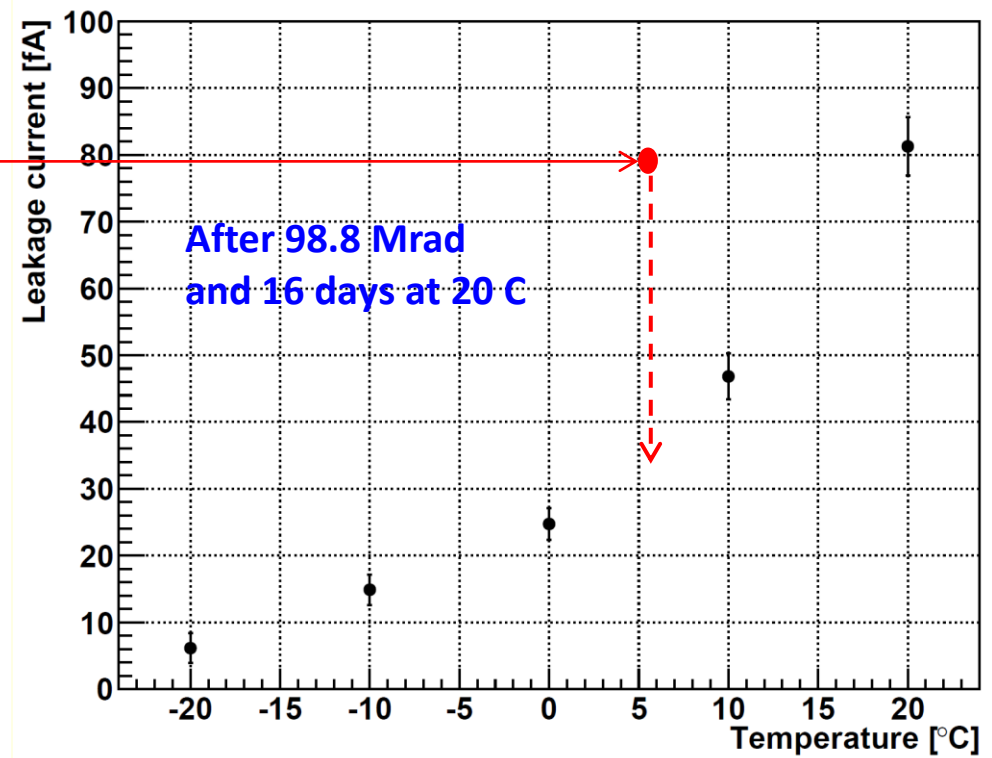
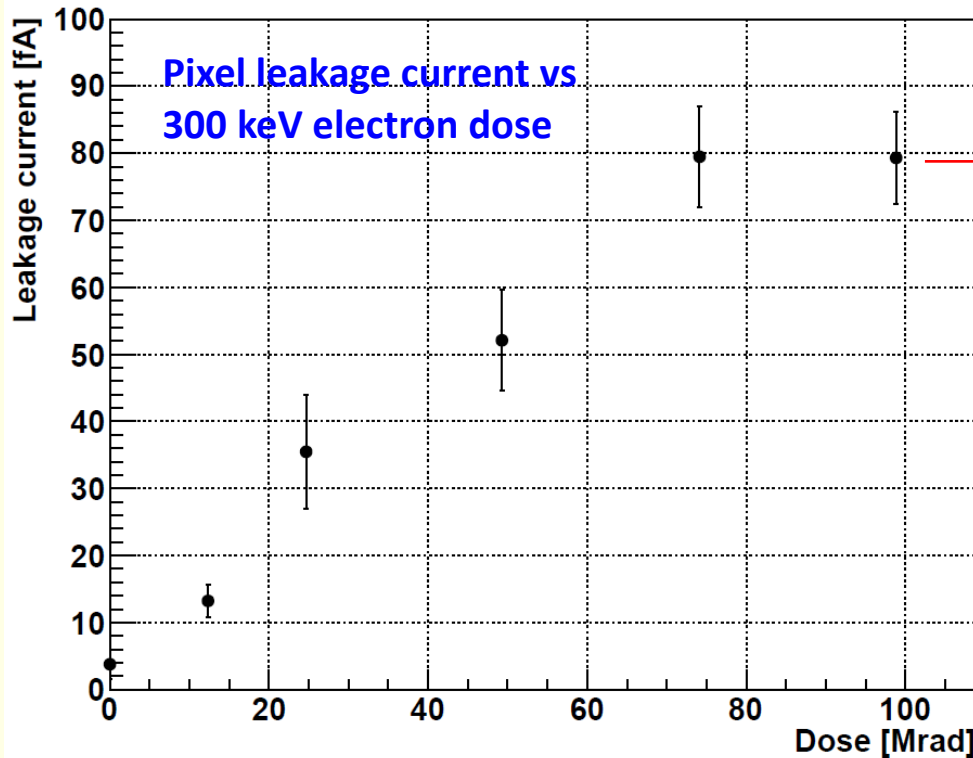


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CMOS imagers for high-resolution TEM



# Radiation hardness: leakage current



- Irradiations performed with 300 keV electrons,  $T = +5^{\circ}\text{C}$
- Dose rate 4300 rad/s, 6.25 MHz clock frequency (integration time  $\sim 10\text{-}20$  ms)
- Moderate leakage current increase with dose that results in increased noise and loss of dynamic range (50% full well at 100 Mrad); leakage current can be reduced 4x by running the sensor at 25 MHz
- Strong dependence of radiation-induced leakage current on temperature: **pre-irradiation dynamic range can however be recovered by cooling to  $-20^{\circ}\text{C}$**

[D. Contarato et al., NIMA A 635 (2011) 69]



# Summary & Conclusions

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- CMOS pixel sensors have been successfully developed and deployed as direct detectors for high resolution and fast imaging in Transmission Electron Microscopy; direct detection on a thin sensor layer benefits both position resolution and detective quantum efficiency
- Sub-pixel pitch Line Spread Function demonstrated on 10  $\mu\text{m}$  pitch pixels; the successful architecture demonstrated in the TEAM chips (0.35  $\mu\text{m}$  process) has been ported to a 0.18  $\mu\text{m}$  process with 5  $\mu\text{m}$  pixel pitch, yielding  $\mu\text{m}$ -level PSF and radiation hardness to electron doses close to 100 Mrad
- The reconstruction of single electron hit positions and the build-up of images from the superimposition of centroids yields a dramatic improvement in resolution and quantum efficiency; high quality images can thus be obtained from electron counting at high frame rate and reduced electron dose
- Stemming from this development, a camera system with a 16 Mpixel, 400 frames/s, 5  $\mu\text{m}$  pitch direct CMOS sensor and integrated hardware processing for cluster imaging will be commercialized in 2011