Direct Electron Detectors and their Application in (Scanning) Transmission Electron Microscopy

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Overview

- What is a Direct Electron Detector (DED)?
- Overview of DEDs in EPIC-TEM Facility
- Advantages for (S)TEM Applications

Features of the Ideal Detector for (S)TEM

- Respond to every electron (Quantum efficiency)
- Does not respond where the electrons are not (Low Noise)
- Know the position of these electrons (Spatial Resolution)

Detector Quantum Efficiency (DQE (f))

$$\mathrm{DQE(s)} = \frac{\mathrm{SNR_{out}(s)}}{\mathrm{SNR_{in}(s)}}$$

- Knows when the electron was detected (Temporal Resolution)
- Knows how many electrons arrived (Linearity / Dynamic Range)

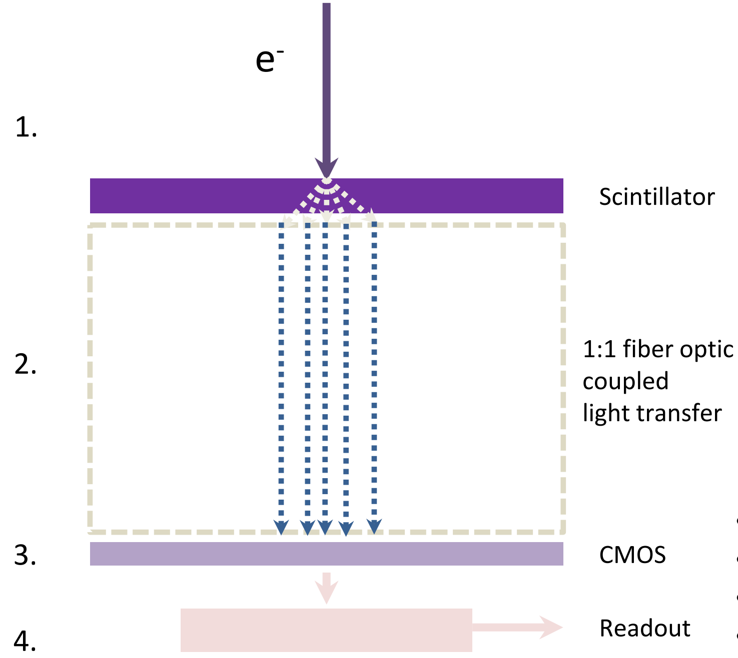
- Energy of every electron
- Momentum of every electron
- Spin of electron
- …

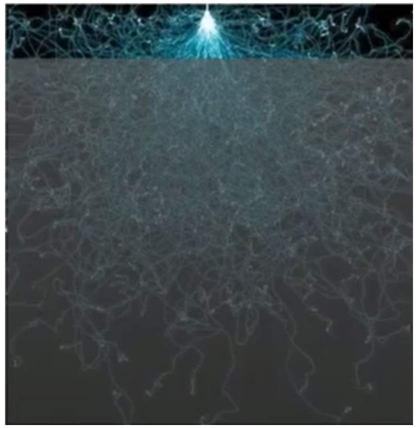
Traditional vs. Direct Electron Detection (DED) Camera

Traditional scintillator camera (CCD or CMOS)

Traditional cameras use a **scintillator** to generate light that is transferred to a sensor and detected

1. Convert electrons to light
2. Transfer light
3. Detect light and convert to signal
4. Electronically transfer signal and read-out to form image



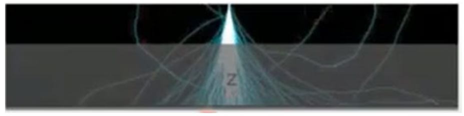


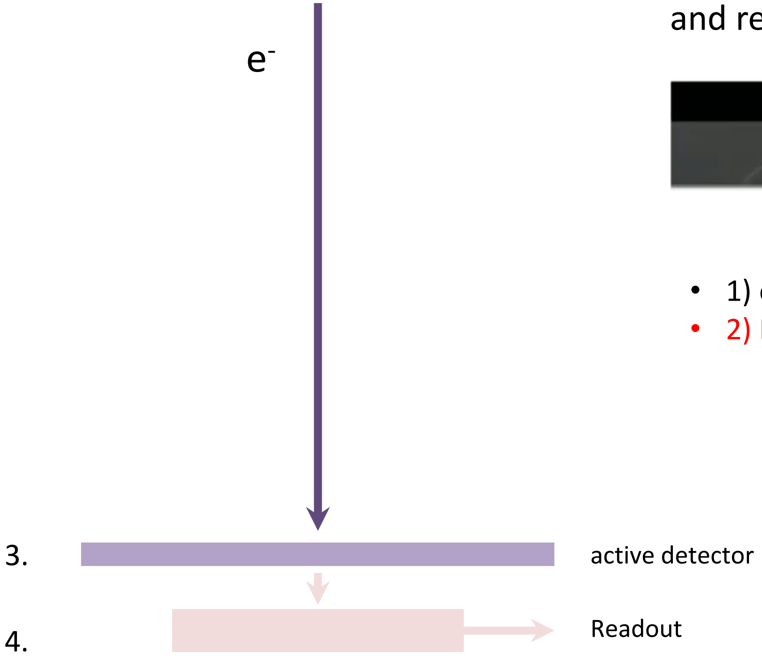
- e^- scatter in high Z scintillator
- e^- backscatter from fiber optic
- Scattering of light in fiber optic
- Distortions from fiber optic

Monolithic Direct Electron Detection camera

Direct detection refers to using a detector which is directly exposed to the e- beam to create a signal

1. ~~Convert electrons to light~~
2. ~~Transfer light~~
3. Detect **electrons** and convert to signal
4. Electronically transfer signal and read-out to form image





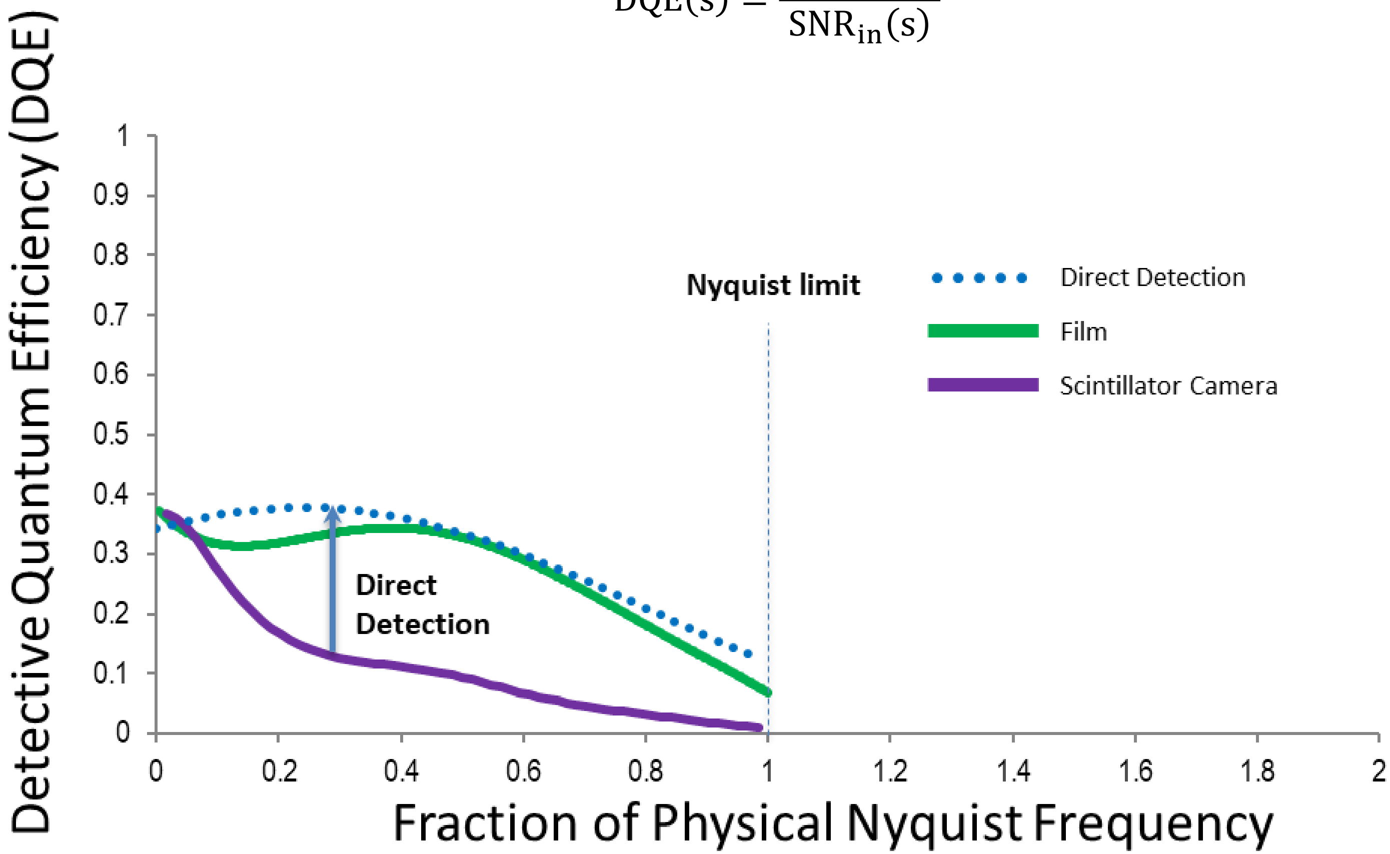
- 1) e^- scatter in low Z sensor
- 2) Damage to pixel

Low Point Spread Function (PSF)

Performance of Traditional vs. DED Camera

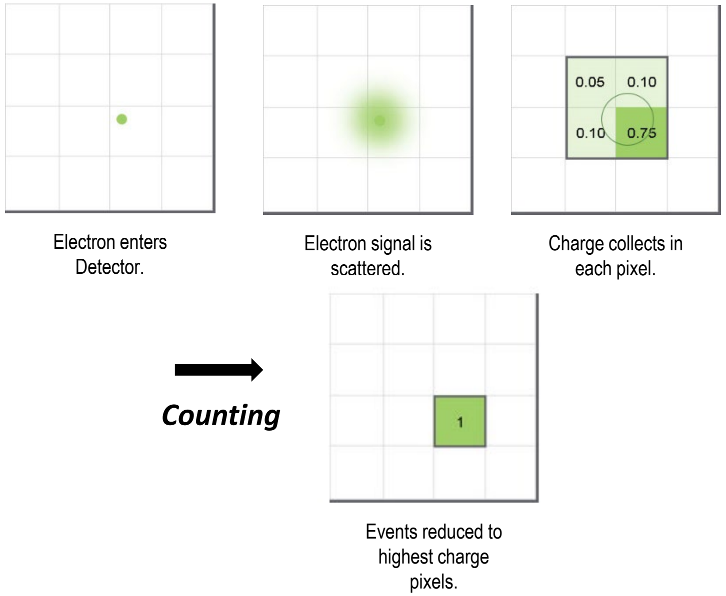
- **DQE** is a function of sampling frequency, and is amongst others dependent on dose rate and accelerating voltage
- Nyquist frequency defines the max. spatial frequency (or minimal resolution) that can be measured by a detector. Minimal resolution = 2 × pixel size
- **For a perfect detector, DQE = 1** for all spatial frequencies

$$\mathrm{DQE(s)} = \frac{\mathrm{SNR_{out}(s)}}{\mathrm{SNR_{in}(s)}}$$

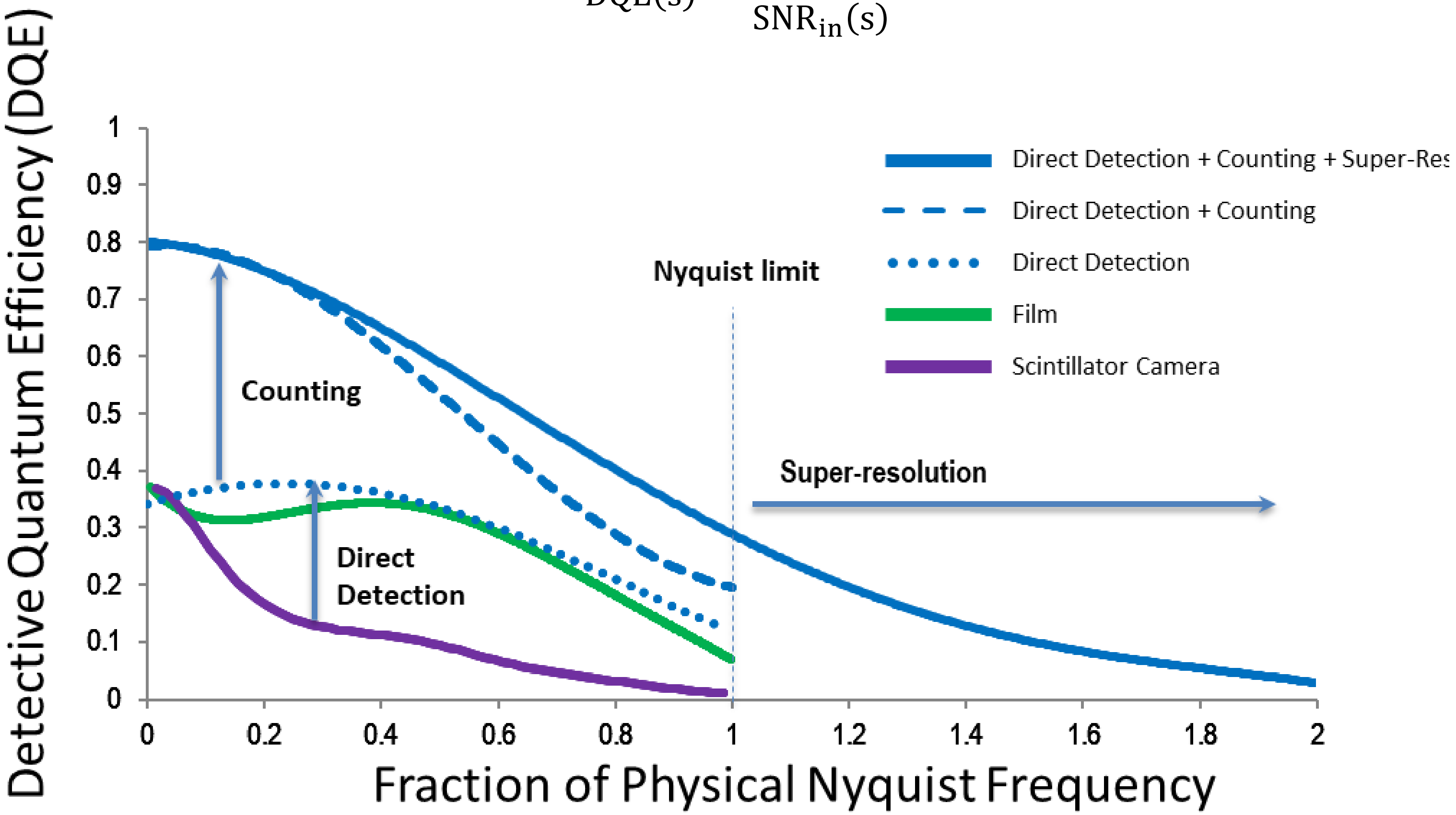


Electron Counting in a Direct Electron Detector

- Each electron strikes the sensor creating a cloud of charge that spans a few pixels
- The charge is collected in each pixel
- a complex “centroiding” algorithm reduces the multi-pixel charge to a single pixel



$$\mathrm{DQE}(s) = \frac{\mathrm{SNR}_{\mathrm{out}}(s)}{\mathrm{SNR}_{\mathrm{in}}(s)}$$



Direct Detectors in the EPIC-TEM Facility

JEOL ARM200CF (S)TEM

- 200 kV/80 kV/60 kV
- Aberration corrected (probe)
- 0.08 nm STEM resolution
- 0.23 nm TEM resolution
- 0.35 eV energy resolution
- Dual SDD EDS detector (1.7sr)
- Simultaneous HAADF/BF/ABF
- Gatan Quantum Dual EELS (Updated with K2 direct camera)
- AXON drift-correction software

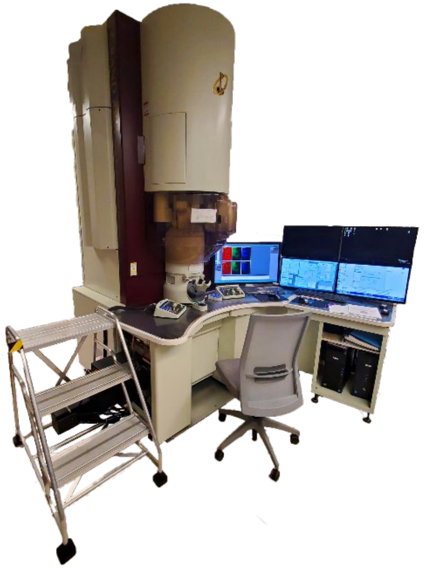
K2 IS

GIF Quantum K2

- 4k by 4k electron counting camera
- max. 1600 frames per second
- K2 direct detection sensor
- Dose fractionation mode for beam sensitive materials
- Quantum GIF + K2 leverages the counting capabilities of the K2 camera (EELS acquisition)

GIF Continuum K3 with Stela

JEOL JEM-ARM300F (S)TEM



- 300 kV/80 kV/60 kV/40 kV
- 0.22 nm TEM / 0.19 nm STEM resolution
- K3 IS DED (Gatan)
- Stela Hybrid-Pixel DED (Gatan)
- Updated with GIF Continuum (Gatan)
- 0.29 eV energy resolution (300 kV)
- Simultaneous imaging by HAADF/BF/ABF
- SDD holder / delivery system
- Wide gap pole-EDS detector
- Hummingbird gas piece allows for wide variety of *in situ* experiments (heating, liquid, biasing, mechanical...)
- AXON drift-correction software

K3 IS

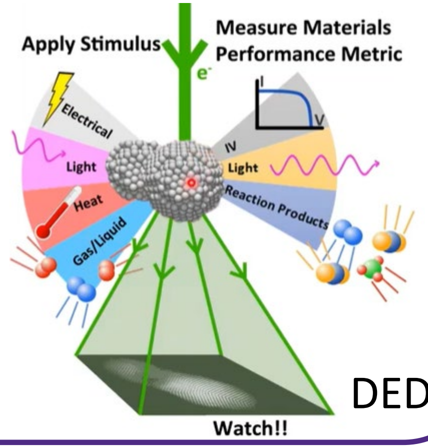
- The world's first counting, high-speed, large format cameras for *in-situ* microscopy
- Large (3456 x 3456) field of view
- max. 3000 frames per second
- Synchronize frames for 4D STEM applications via STEMx

Stela Hybrid-Pixel camera

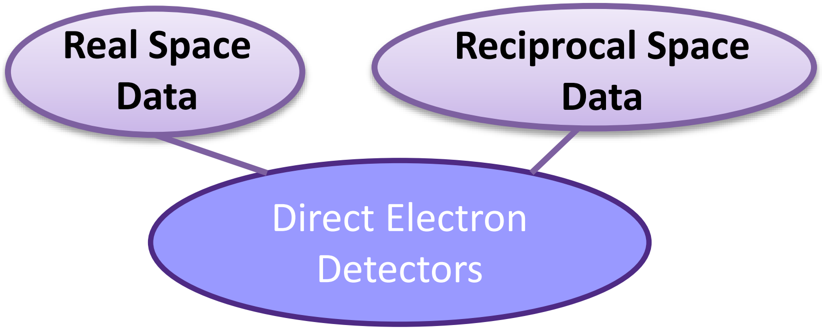
- **Hybrid-Pixel counting camera**
- 4D-STEM & diffraction imaging at low kV
- 512 x 512 pixels
- > 16,000 fps
- High dynamic range for weak reflections

(S)TEM Techniques Benefiting from Direct Electron Detectors

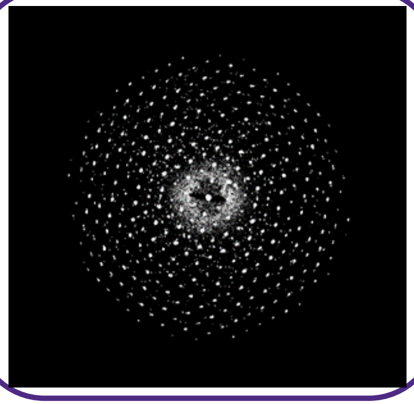
(*in situ*) TEM Imaging



Applications: direct visualization of biasing, heating, liquid, liquid He effects

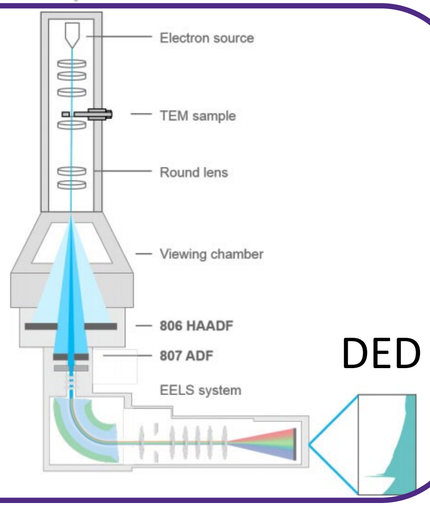


(Selected Area) Diffraction, CBED & MicroED



Applications: (3D) crystal structure, Single CBED to measure specimen thickness, lattice strain measurements, point and space group determination, phonon structure, structure factor and charge density, etc.

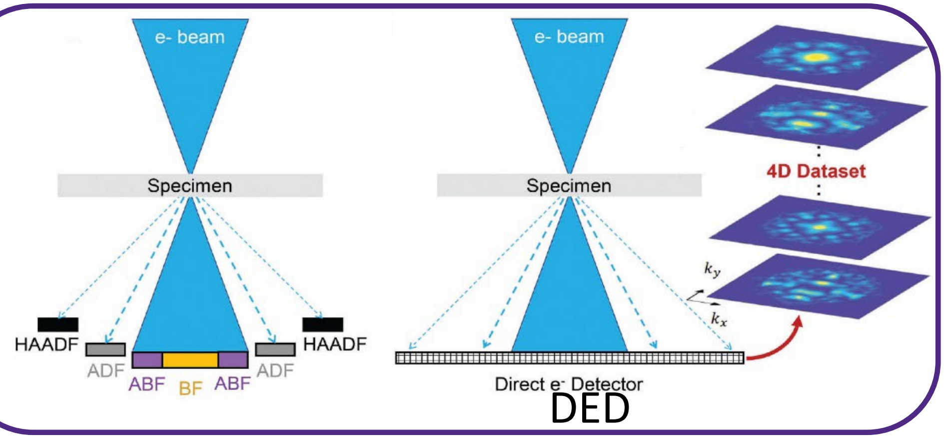
(*in situ*) EELS and EFTEM



Applications:

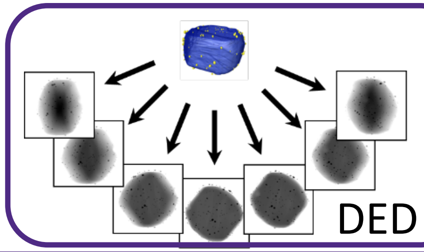
EELS measurement	Information obtainable
Low-loss intensity	Local thickness, mass thickness
Plasmon energy	Valence-electron density
Plasmon peak shift	Alloy composition
Low-loss fine structure	Dielectric function, JDOS
Low-loss fingerprinting	Phase identification
Core-loss intensities	Elemental analysis
Orientation dependence	Atomic site location
Near-edge fine structure	Bonding information
Chemical shift of edges	Oxidation state, valency
L or *M* white-line ratio	Valency, magnetic properties
Extended fine structure	Interatomic distances
Bethe ridge (ECOSS)	Bonding information

(*in situ*) 4D-STEM



Applications: Virtual imaging, Orientation mapping, Strain mapping, Electric/magnetic field mapping, Phase Contrast Imaging (DPC, Ptychography), Fluctuation electron microscopy (medium-range ordering), Pair Distribution Function Mapping (short-medium range ordering etc.)

Electron Tomography, SPA, Holography, etc.



Applications: 3D visualization

Electric/magnetic field imaging, thickness, etc.

R.F. Egerton, EELS in the Electron Microscope, 2011

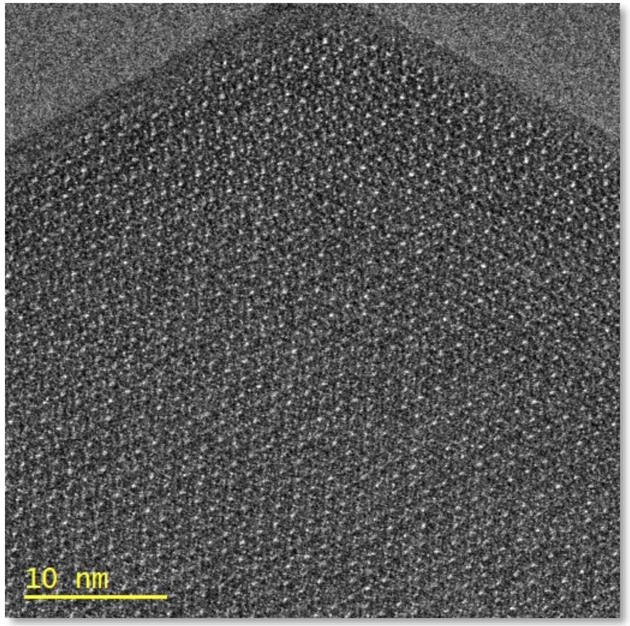
Specimen: Tylenol crystal
Diffraction image size: 512 x 512 pixels
Exposure per frame: 5 ms
Tilt range: ±70°
Total acquisition time: 3 s

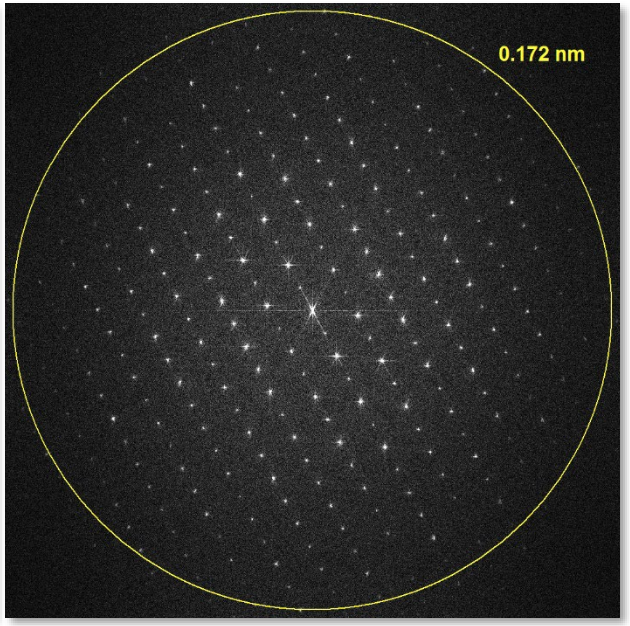
DED advantage for Real Space Data Acquisition

- Examples using K3 IS

TEM Imaging

Zeolite

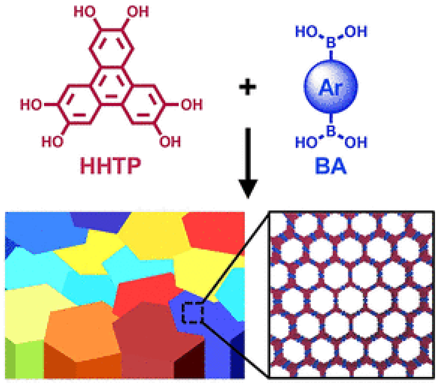




$0.74\ e^{-}\ Å^{-2}\ s^{-1}$ Dose Rate
$25\ e^{-}\ Å^{-2}$ Total Dose

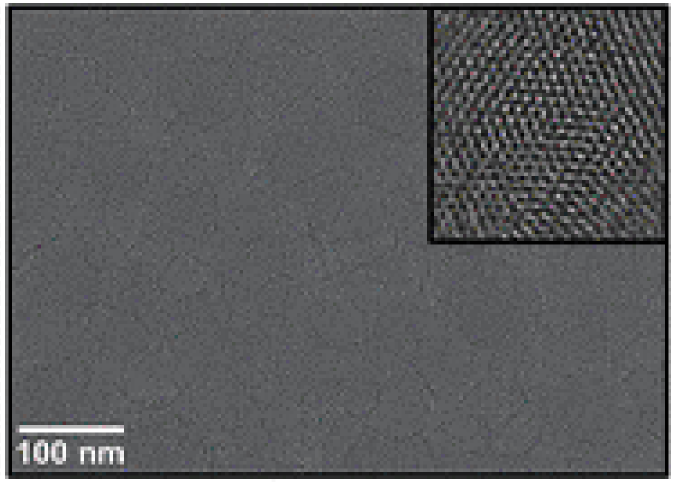
Counting individual electrons allows you to collect high-quality images at even lower doses and dose rates

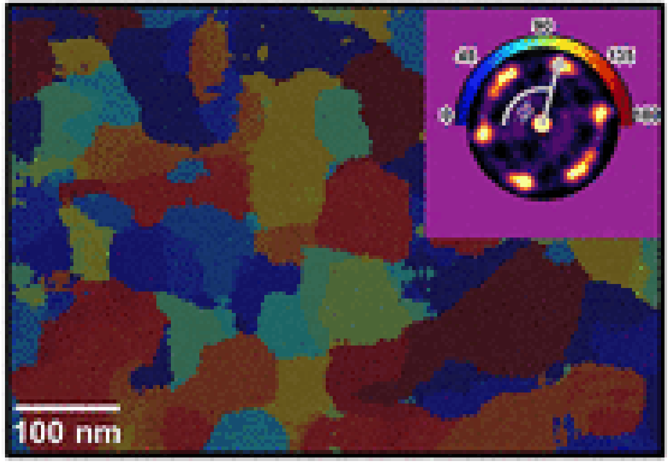
Covalent Organic Frameworks (COFs)



Polycrystalline COF Film

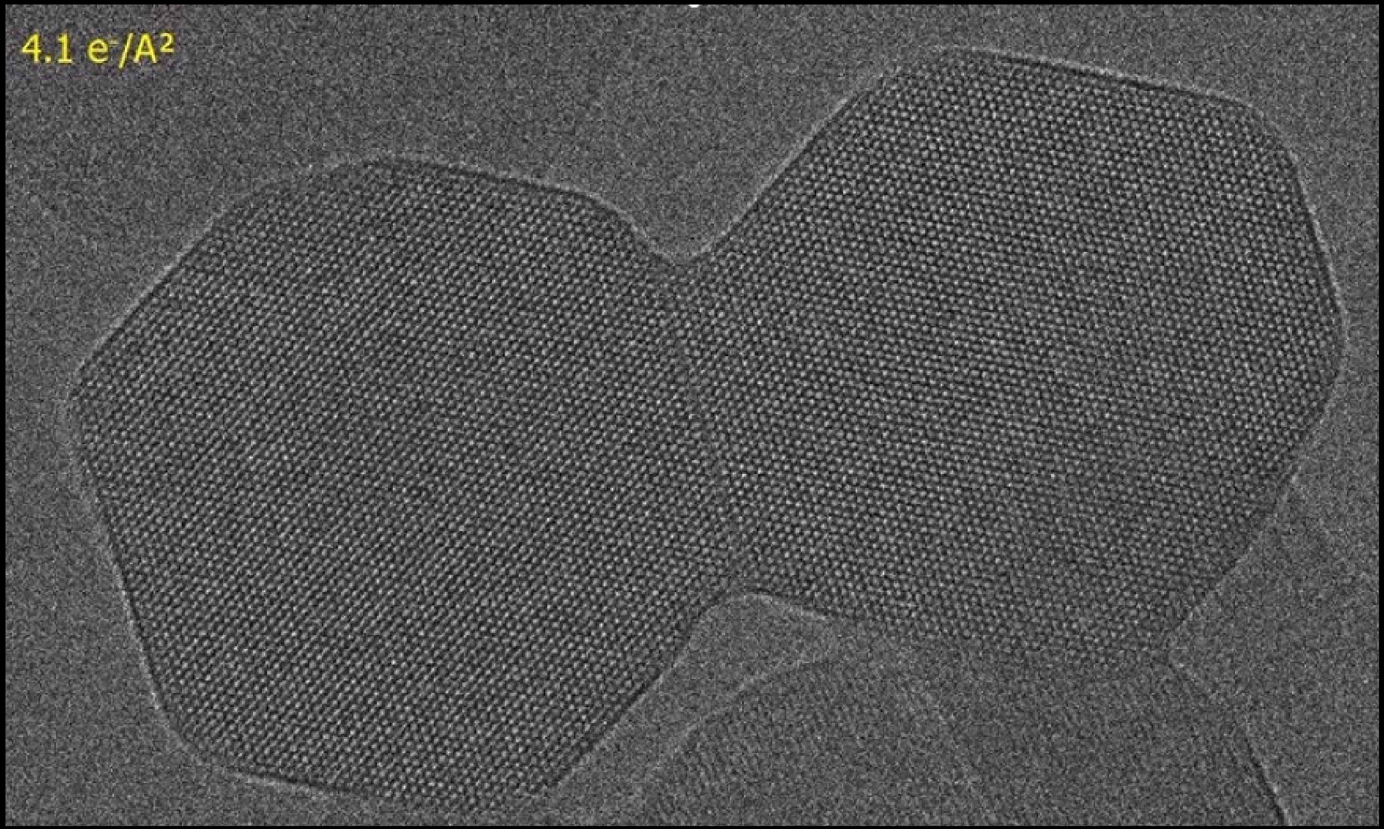
$2.1\ e^{-}\ Å^{-2}\ s^{-1}$ Dose Rate
$\mathbf{14\ e^{-}\ Å^{-2}}$ Total Dose

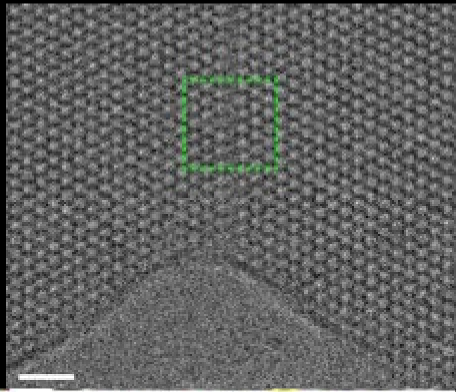


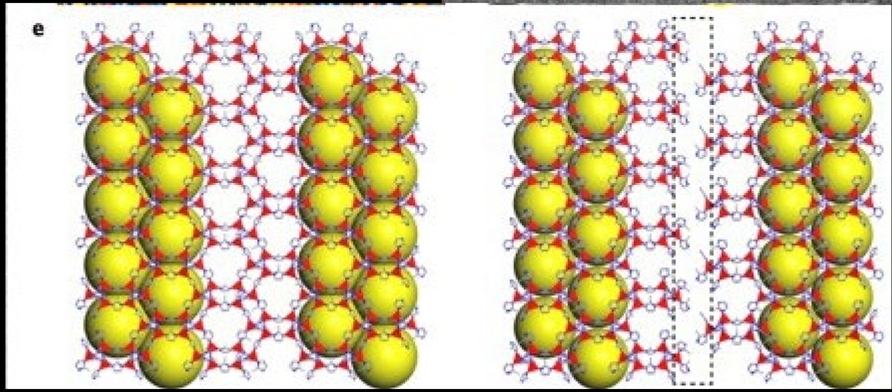


I. Castano, A. M. Evans, R. dos Reis, V. P. Dravid, N. C. Gianneschi, and W. R. Dichtel. Chemistry of Materials ***2021*** *33 (4), 1341-1352*

TEM Imaging – Ultralow Dose MOF



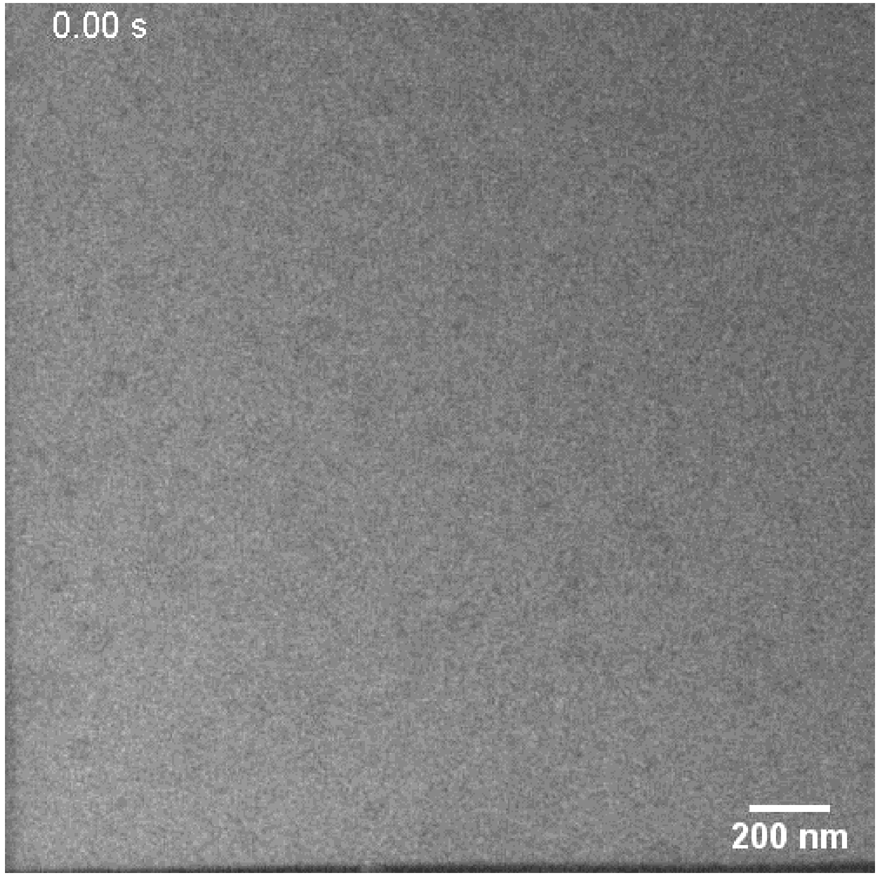




TEM reveals important local structural features of ZIF-8 crystals that cannot be identified by diffraction techniques, including **armchair-type surface terminations** and **coherent interfaces** between assembled crystals

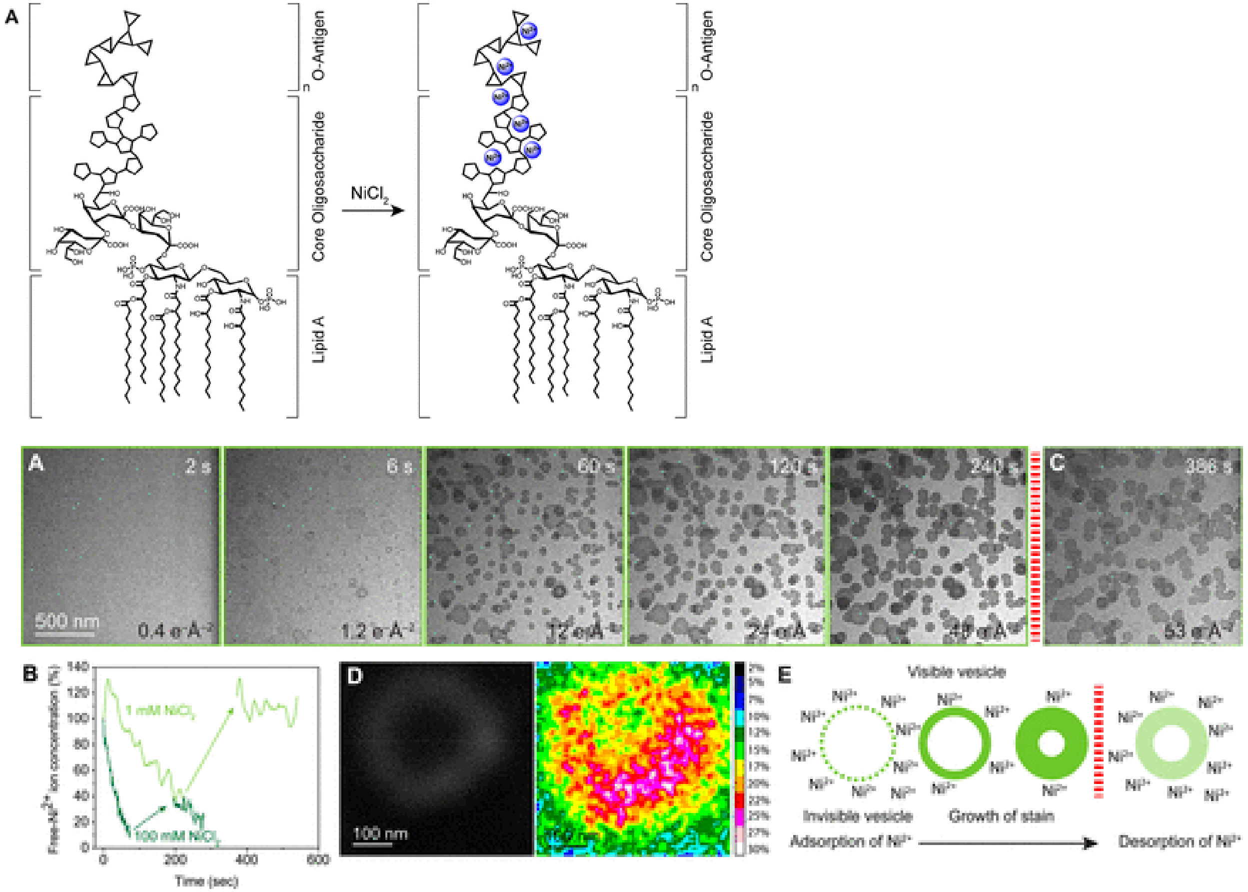
ZIF-8 MOF Dose Rate: 1.02 e/pix/s; pixel 0.86 nm; 120 frames
Zhu, Y. *et al. Nature Materials* **16**, 532–536 (2017)

In situ TEM – *In Situ* Staining Liposome



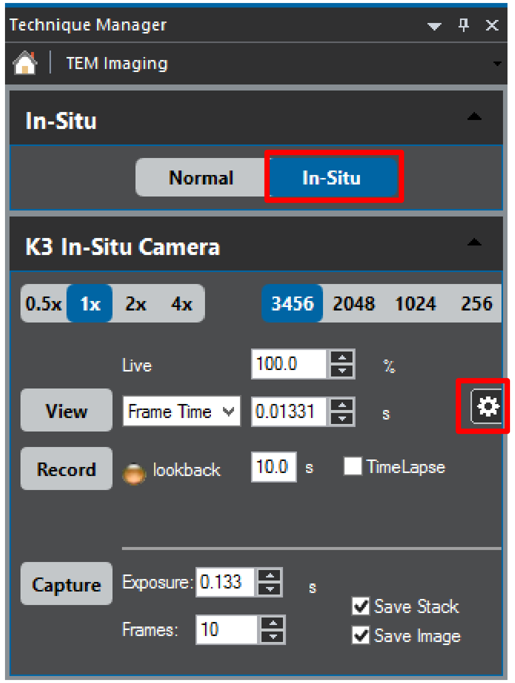
K3 IS – 300kV

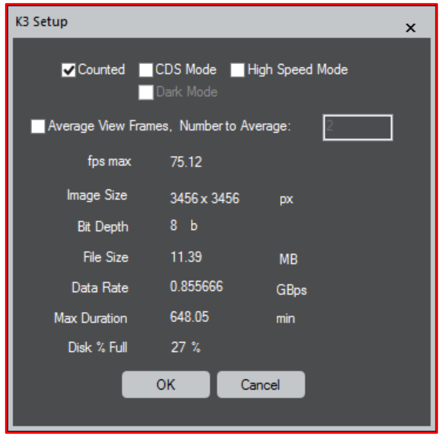
Dose rate 0.2 e^{-} $Å^{-2}$ s^{-1}



K. Gnanasekaran, B. Chang, **P. Smeets**, N. Gianneschi, *Nano Lett.* **2020**, 20, 6, 4292–4297

In situ TEM – ARM300F (K3 IS)





In situ TEM Data Acquisition

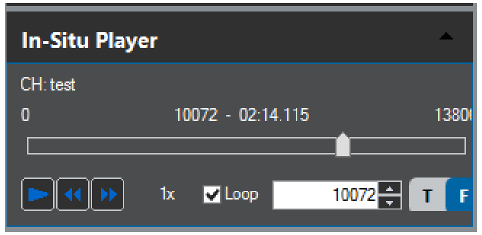
- Binning, camera size
- Frame time (max. 75 fps full-frame)
- **Lookback**: save to disk x s before any dynamic event

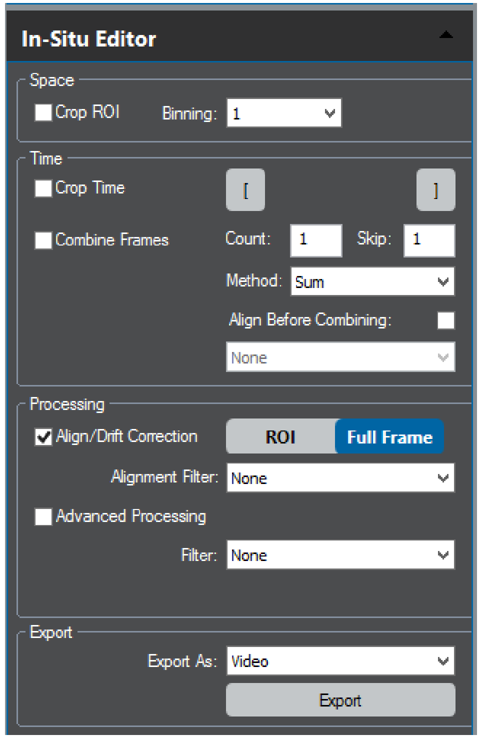
In situ TEM Data Player

- Amount of frames / time
- Change playback speed

In situ TEM Data Editor

- Cropping / frame averaging
- Drift correction using image registration (using imaging filters)
- Imaging filters
- Exporting video (adding scalebar, timestamp)



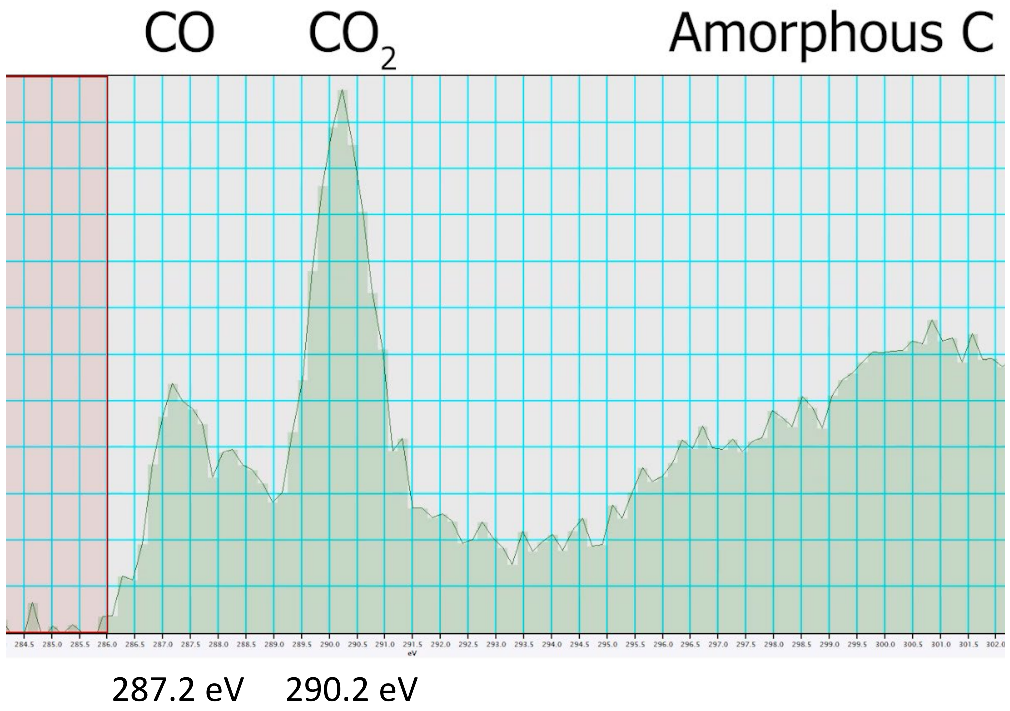


In Situ EELS

- *In situ* EELS showing the interaction between electron beam and gas molecules

Protochips Atmosphere 210 System attached on ARM 200CF

- Pressure range: 760 Torr (101.3 kPa) to 1 Torr (0.13 kPa);
- Flow range: 0.005 sccm to 1 sccm (ml/min);
- Temperature range: RT to 1000 °C with max 10 ºC/s heating rate;
- Gas mixing: 0.01%-99.99% mixture of up to 3 non-corrosive gases;
- Vapor allowable: water, methanol, ethanol, hexane, naphtha, etc;
- Reaction gas Analyzer (RGA) attached

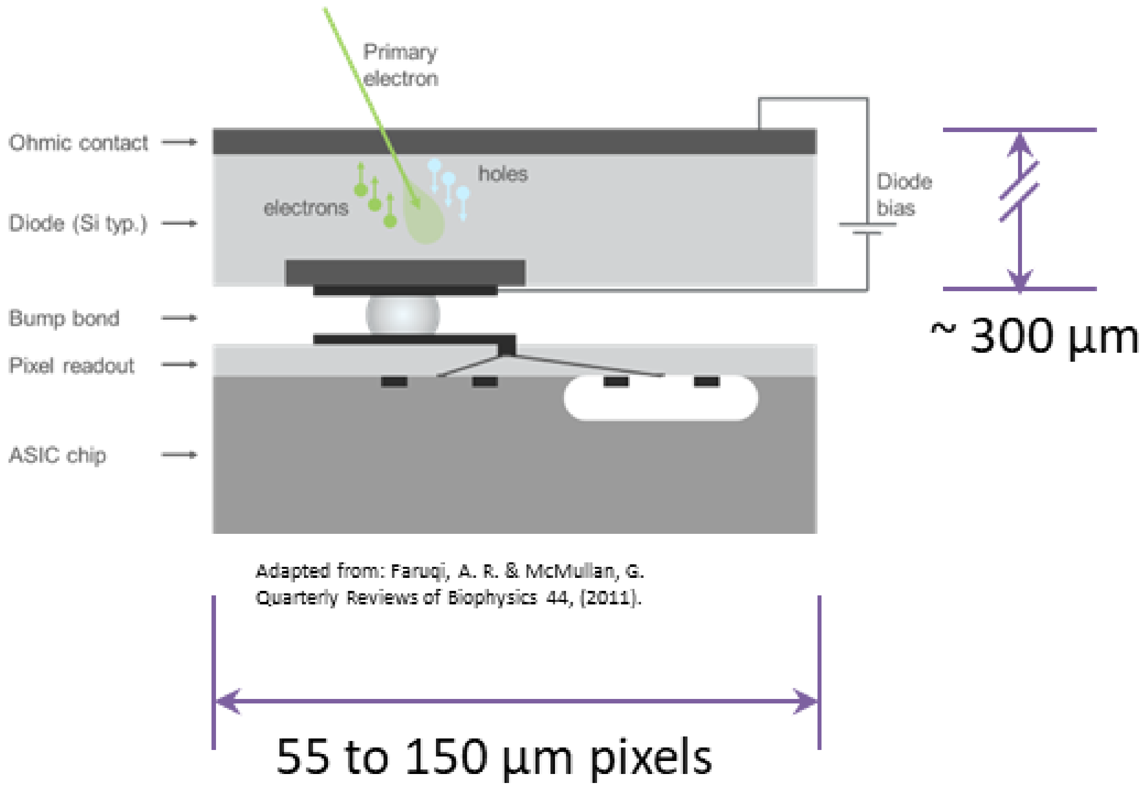


Drs. Kunmo Koo & Xiaobing Hu, in prep.

DED advantage for Reciprocal Space Data Acquisition

- Examples using Stela Hybrid-Pixel Detector

Stela Hybrid-Pixel Direct Detector (ARM300F)



Adapted from: Faruqi, A. R. & McMullan, G. Quarterly Reviews of Biophysics 44, (2011).

Advantages

- Fast readout, with direct digitization
- Near zero read noise enables multi-pass frame summing
- Broad range of input count rates (high dynamic range; works also well at higher dose rates)
- **Very sharp PSF at lower kVs**

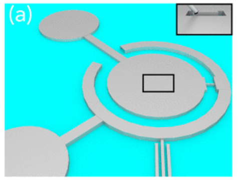
Limitations

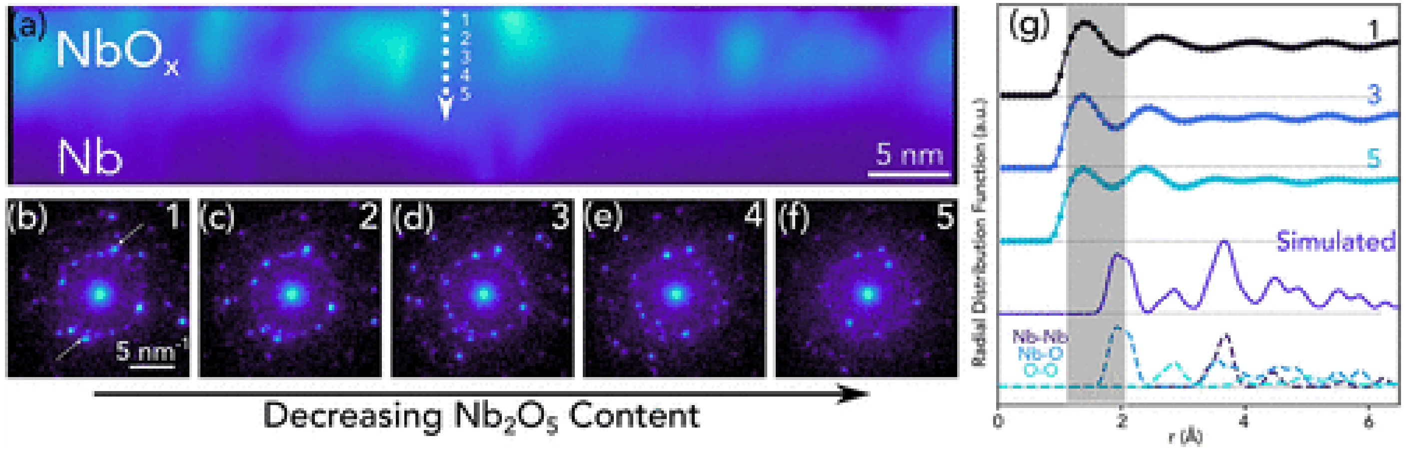
- Physically large, small number of pixels
- Poor PSF at high kVs

Ideal for diffraction applications!

4D-STEM

Fluctuation Electron Microscopy (FEM) on niobium (Nb)-superconducting transmon qubits to assess medium-range ordering





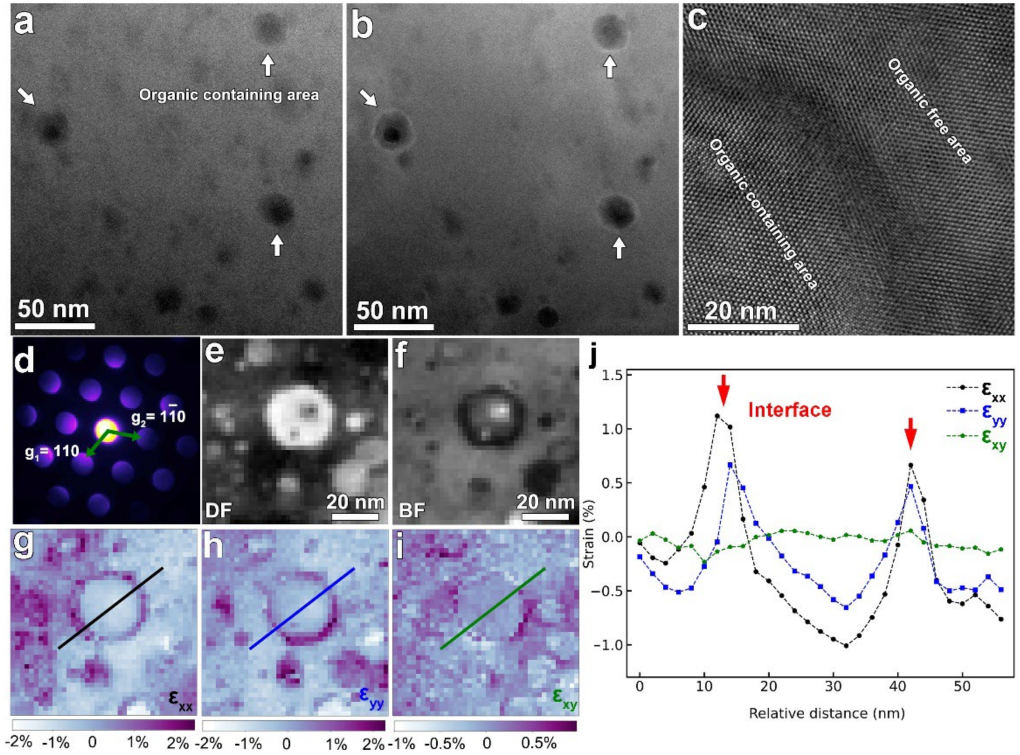
Structure factor S(Q)

$$S(Q) - 1 = \frac{I(Q) - A(Q)}{B(Q)}$$

RDF

$$g(r) = 1 + \frac{1}{2\pi^2 \rho_0 r} \int_0^\infty Q\,[S(Q) - 1] \times \sin(Qr)\, dQ$$

Strain Analysis across soft-hard interface in nacre



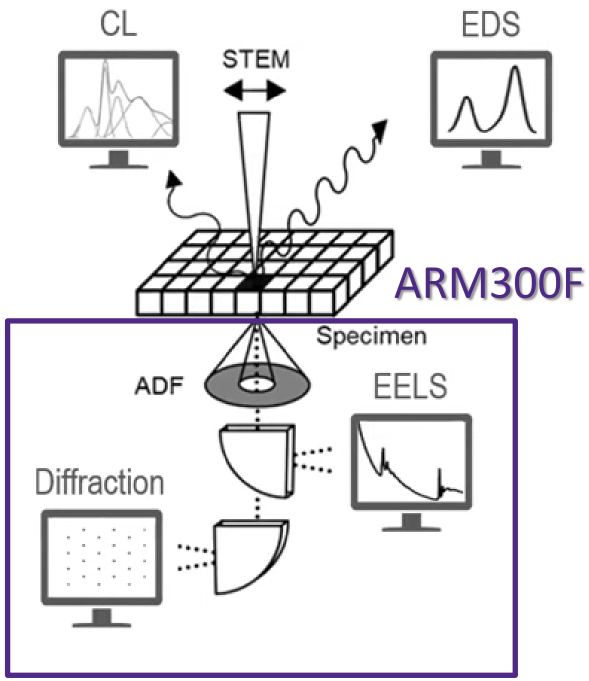
Drs. Xiaobing Hu & Paul Smeets, in prep.

Elucidating the Role of Nanoscale Organics in Natural Nanocomposite Materials, **M&M 2023**, Tuesday, 7/25 3-5pm

Akshay A. Murthy, Paul Masih Das, Stephanie M. Ribet, Cameron Kopas, Jaeyel Lee, Matthew J. Reagor, Lin Zhou, Matthew J. Kramer, Mark C. Hersam, Mattia Checchin, Anna Grassellino, Roberto dos Reis, Vinayak P. Dravid, and Alexander Romanenko, *ACS Nano* **2022** *16* (10), 17257-17262

(*In Situ*) 4D-STEM & EELS (Multimodal)

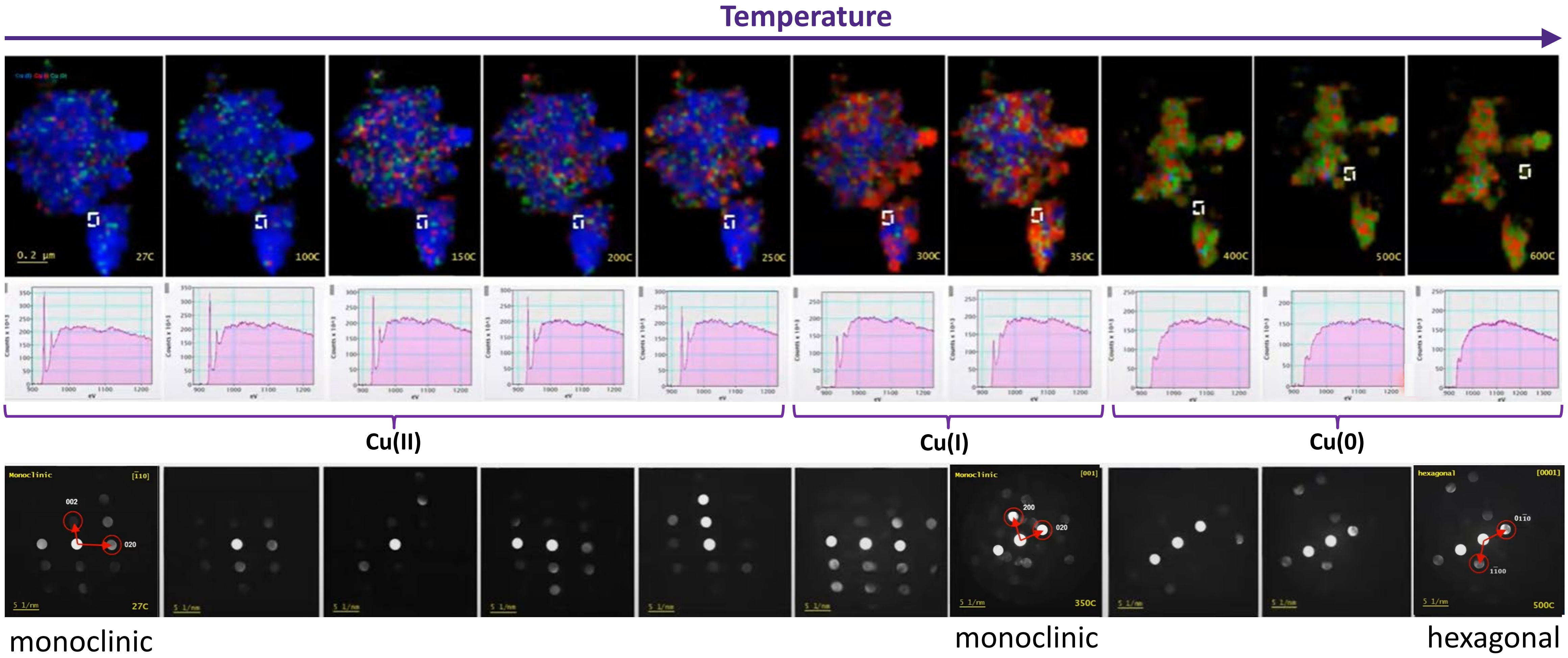
- On the **ARM300F** → EELS and 4D-STEM datasets from the same region can be obtained (Multimodal)
- Various SI Acquisition within the same software platform
- One sample, one microscope session, one data format



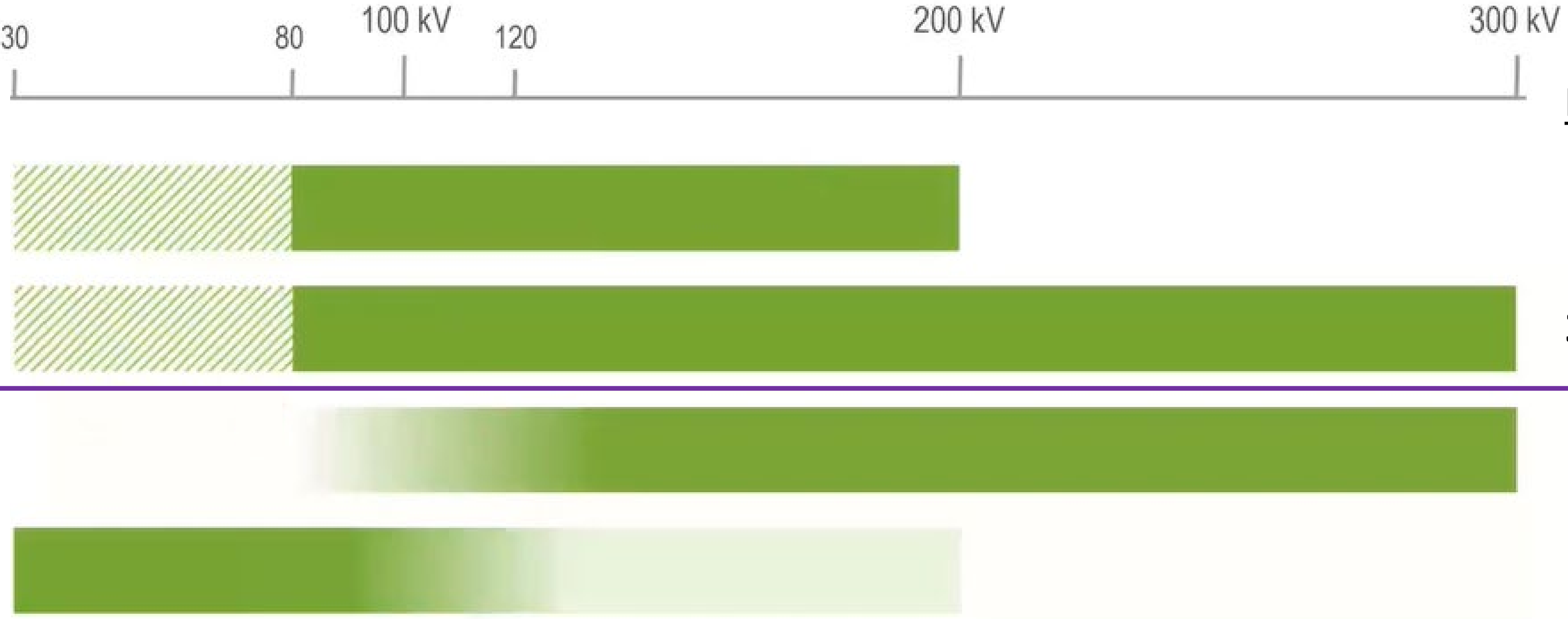
Multidimensional Electron Microscopy in the STEM: Scanning Diffraction and EELS Workshop
https://www.youtube.com/watch?v=I2G1-t6bKKo

In Situ 4D-STEM & EELS (Multimodal)

- Heating experiment of $Fe_2O_3 \cdot H_2O$ nanoparticles
- Cu(II) reduces to Cu(0) with temperature (EELS), crystal structure change from monoclinic to hexagonal



Detector Performance

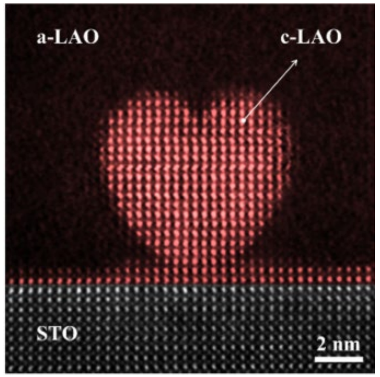


Camera	Max. Framerate
Rio 2100F	**160 fps**
OneView ARM200CF/ARM300F	**300 fps**
K2/K3 IS ARM200CF/ARM300F	**1600/3500 fps**
Stela ARM300F	**16000 fps**

DED Matchmaker for the ARM300F

(S)TEM Technique	K3	Stela
EELS Mapping		
Low-kV EELS		
EFTEM imaging		
Low-dose imaging		
In-situ EFTEM		
In-situ EELS		
4D STEM / micro-ED		

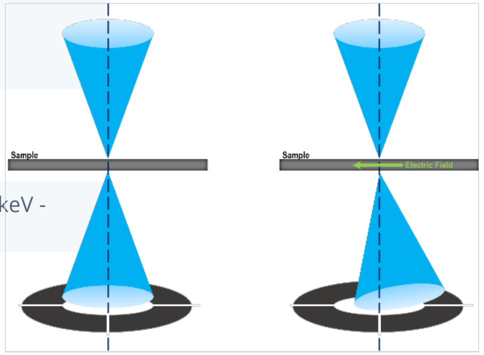
- **Stela** – High speed and high dynamic range ideal for diffraction imaging at low kV
- **K3** – High sensitivity, large pixel count ideal for low-dose imaging and *in-situ* studies

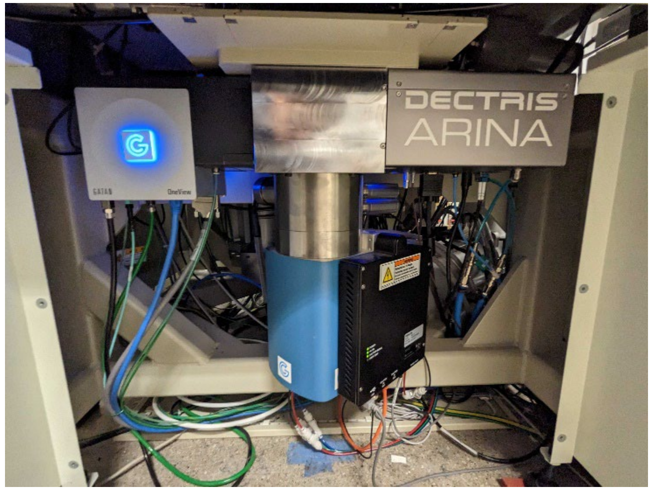


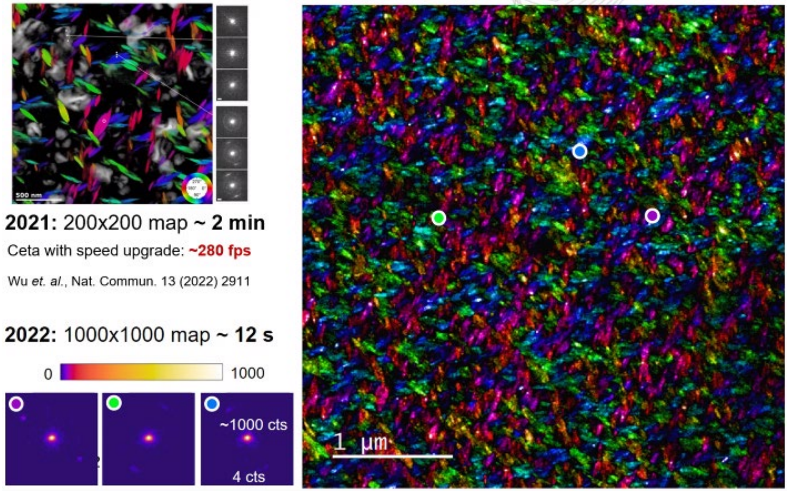
Just Installed on the JEOL ARM200CF…

Ideal for 4D-STEM applications

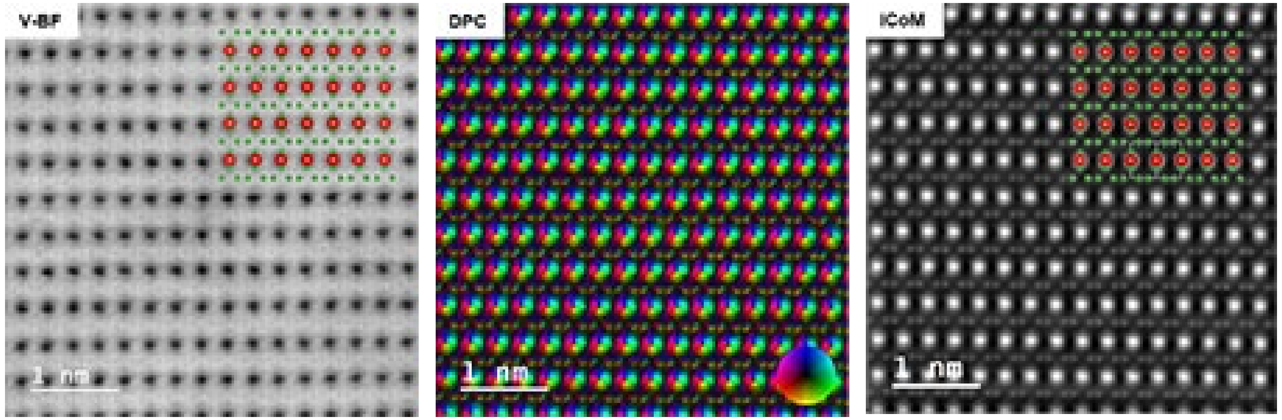
Number of pixels (W x H)	192 x 192
Active area (W x H) [mm²]	20 x 20
Pixel size (W x H) [µm²]	100 x 100
Sensor material	Silicon (Si) or high-Z
Energy range [keV]	30 - 300
Frame rate (max.) [Hz]	120,000
Count rate (max.) [el/s/pixel]	10^8
Detective Quantum Efficiency, DQE(0)	at 80 keV - 0.82 | at 200 keV - 0.75 | at 300 keV - 0.75
Detector mounting	Retractable







SmB_6



Contact Info

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