



**COVALENT
METROLOGY**

Welcome

covalentmetrology.com

**AGE OF
TRANSMISSION
(SCANNING)
TRANSMISSION
ELECTRON
MICROSCOPY**

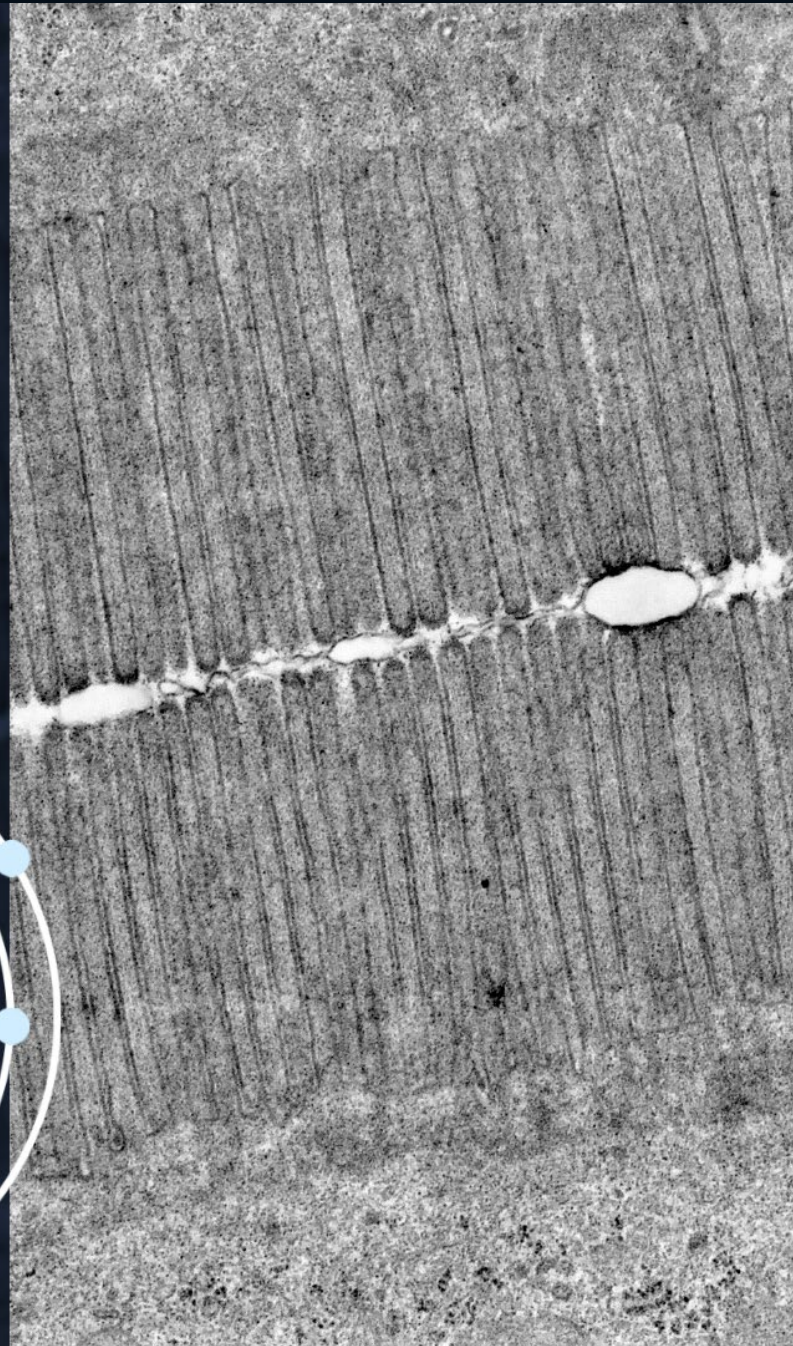
**Jason Donald &
Jeff Sullivan, PhD**

September 3, 2020, 11am PDT



**COVALENT
ACADEMY**

EPISODE 16





- Disruptive New **Platform** for Outsourced Measurement, Test & Characterization
 - 32 team members (2/3 with advanced degrees)
 - 9,500 ft² lab in Sunnyvale (and growing)
 - 35 in-house instruments (and growing); >\$10M in instruments
 - 7 instrument partnerships
(ThermoFisher, Rigaku, JA Woollam, Anton Paar, PVA TePla, Asylum Research / Oxford Instruments, Keyence)
 - 11 partner labs
- **>300 customers** (80% repeat)

hello@covalentmetrology.com

Electron & Scanning Probe Microscopy

*SEM / TEM, AFM,
EDS, EELS,
Nanoindentation,
Acoustic Microscopy*

Surface Analysis

*XPS, Auger,
TOF-SIMS,
d-SIMS*

Optical Microscopy & Spectroscopy

*Laser Confocal, White
Light, Chromatic
Aberration, Spectral
Ellipsometry, UV-Vis-Nir
Spectroscopy*

Materials / PC Board Failure Analysis

*Cross Sections,
Inspections, Dye &
Pry Testing*

X-Ray Characterization

*XRD / XRR,
XRF,
 μ CT*

Chemical Analysis

*ICPMS, GCMS,
FTIR, Raman
Spectroscopy, NMR*

Nanoparticle Analysis

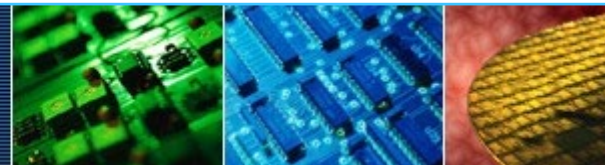
*DLS, PSA,
Particle Zeta
Potential*

Bulk Properties Characterization

*DSC, TGA, DMA,
Rheometry, Tensile
Testing, Pencil Test,
Surface Zeta Potential*

RIGA ANALYTICAL LAB

ANALYZE TO SUCCEED



Covalent is thrilled to announce that the outstanding team at **Riga Analytical Labs** – an independent service lab operating in the heart of Silicon Valley – will be joining our organization, effective immediately.

An excited **message from our CEO, Craig Hunter:**

“ The acquisition advances Covalent’s mission to provide the marketplace with affordable access to cutting-edge data at ever faster turnaround times. We welcome Giorgio and his entire technical team to the Covalent family. ”

Please take a moment to read the full Press Release at:

<https://covalentmetrology.com/covalent-acquires-riga-analytical-lab/>

Introducing Jason Donald

Jason Donald is the Director of TEM and Lamella Prep leading the team focused on sample creation and advanced TEM and STEM analysis. He has 25 years of experience helping in the development and operation of 6 generations of FIB/SEM dual beam systems and cutting-edge automated TEM platforms. In his previous work he led product development teams and was applications leader supporting development of new processes and analytical FIB, SEM and TEM capabilities for the Semiconductor logic, NAND and DRAM memory, MEMS, and data storage markets.

He received his Bachelor of Science in Physics from Humboldt State University.



Introducing

Jeff Sullivan, PhD

Jeff's career has focused on the technology-oriented product life cycle of both materials and capital equipment, from research and development through engineering, new product introduction, and manufacturing to customer adoption. With the General Electric ("GE") Company from 2010 to 2017, Jeff was an Advanced R & D Leader at GE Global Research and Factory Integration Leader at GE Renewables. He held the roles of Director of Engineering, Senior Program Manager and Engineer Manager at Applied Materials, Inc. from 1999 to 2010.

Jeff holds a PhD and Master of Science from the University of Wisconsin – Madison as well as a Bachelor of Arts from Grinnell College. He has been awarded more than fifteen United States patents in disciplines ranging from laser interferometry to photovoltaic manufacturing. Jeff is certified as a Green Belt and trained as a Master Black Belt in GE's Lean/Six Sigma quality system.



Introducing

Roozbeh Nikkhah-Moshaie, PhD

Roozbeh Nikkhah-Moshaie, Ph.D. has joined Covalent Metrology as Senior Member of Technical Staff for S/TEM characterization. Roozbeh brings extensive experience in materials and data analysis and characterization as well as a profound understanding of structure-processing-property and performance correlation in materials at micro and nanoscale. Roozbeh is a Senior Member of IEEE and Fellow of Royal Microscopical Society.

He holds a PhD in Materials Science & Engineering from Florida International University.





TEM History and Basics

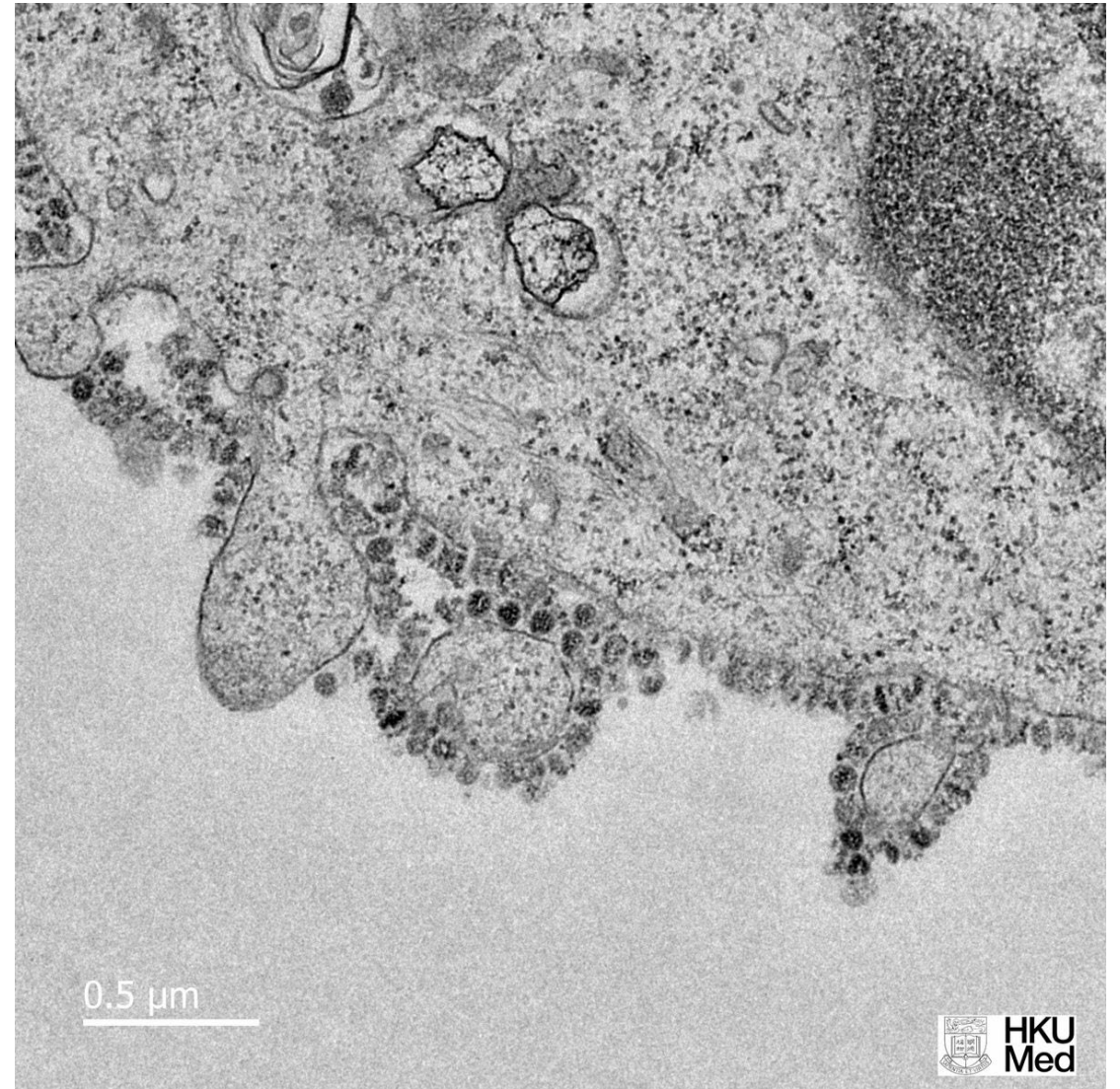
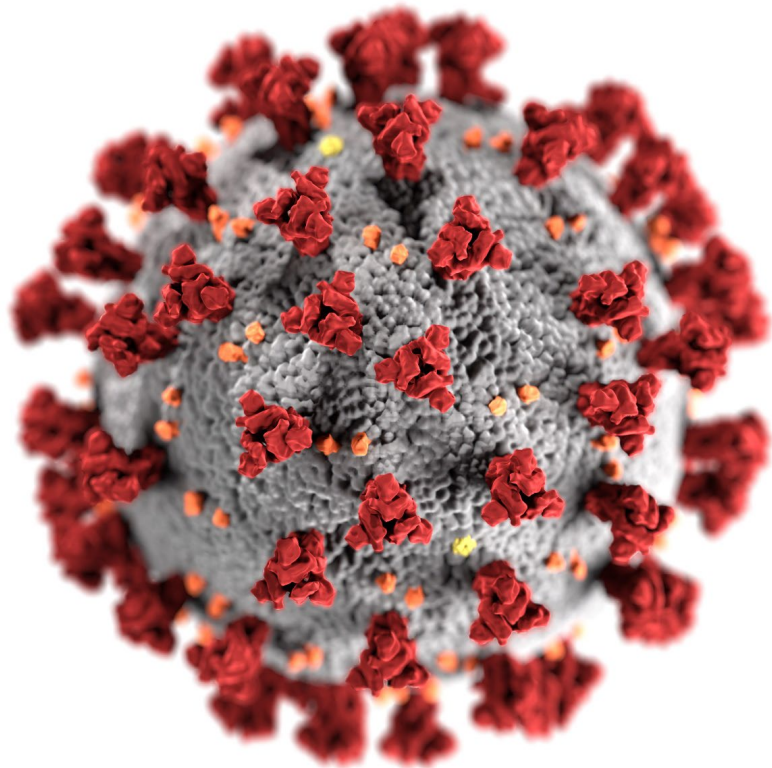
Modern Advancement in TEM

Covalent Metrology Talos F200X

Practical Applications of TEM

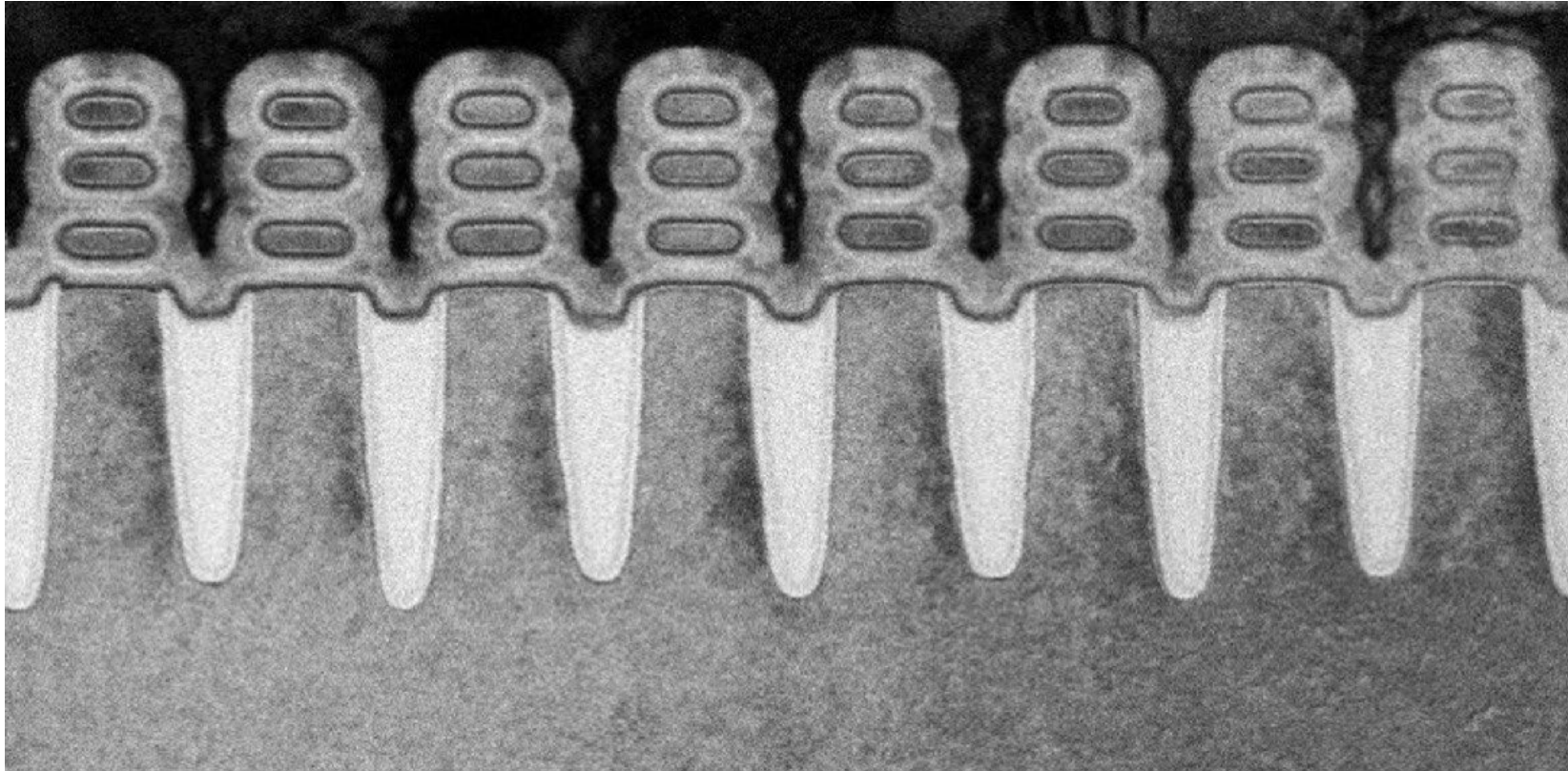
What can you see with TEM

TEM images and data are all around us in the 21st century.
The impact of this data has direct influence on our health, as with TEM reconstructions of the COVID-19 virus

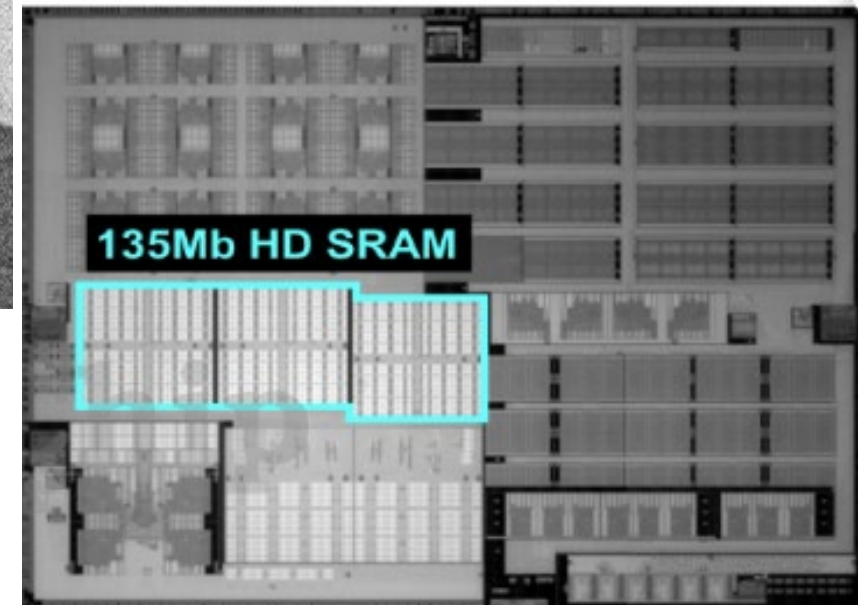


TEM image of Gate all around FET

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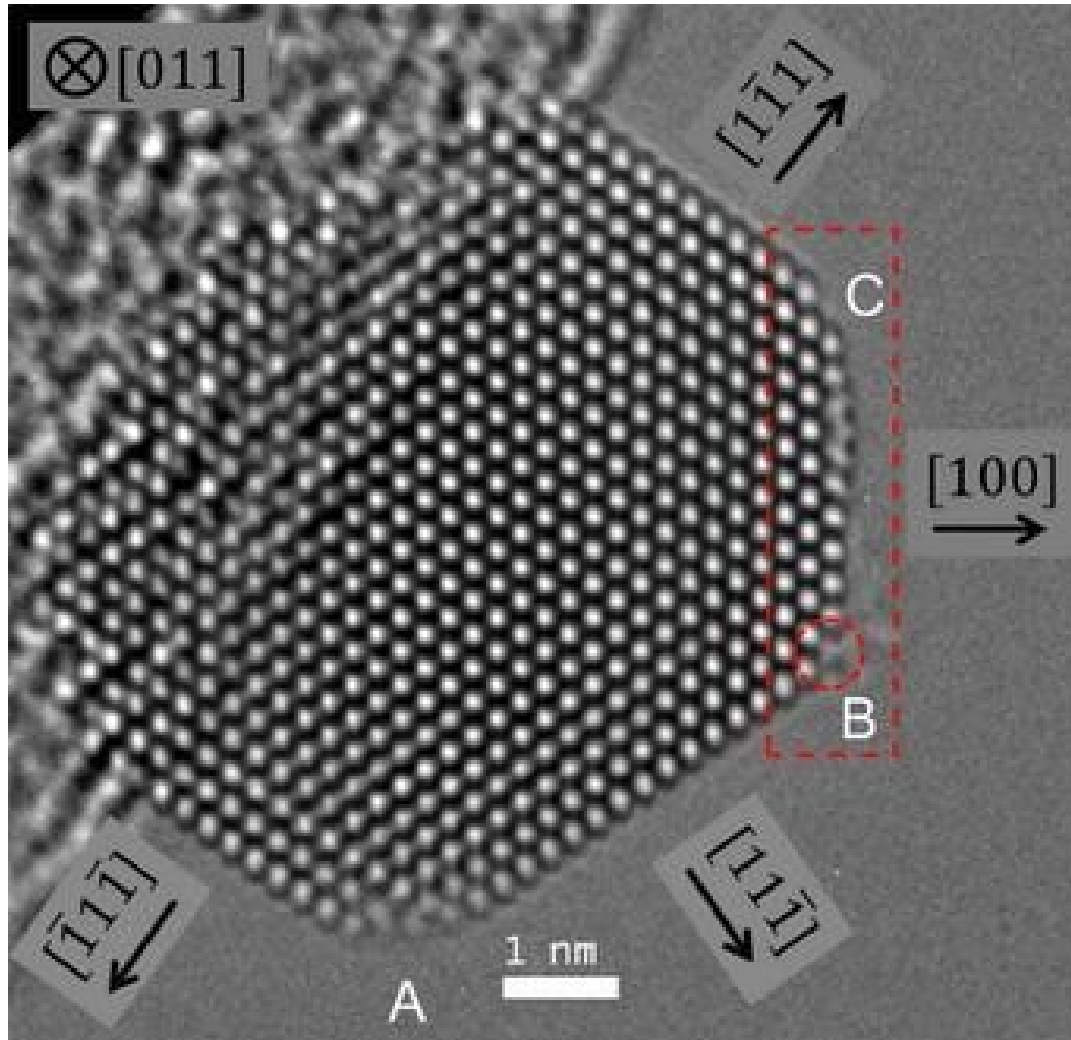


Images of the latest generation of Computer chips, These are Gate all around FET that will enable the 5nm technology node



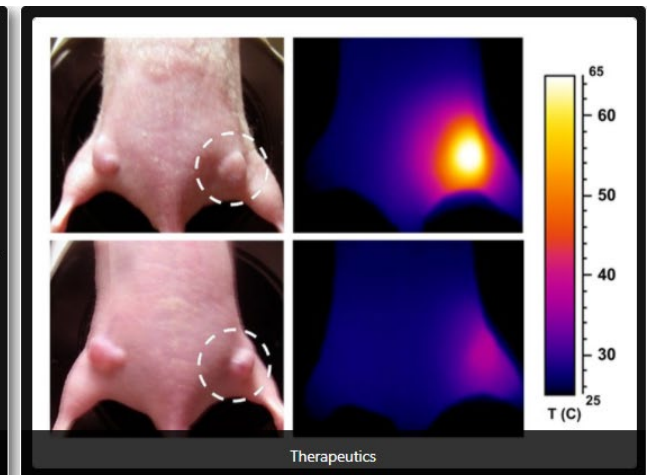
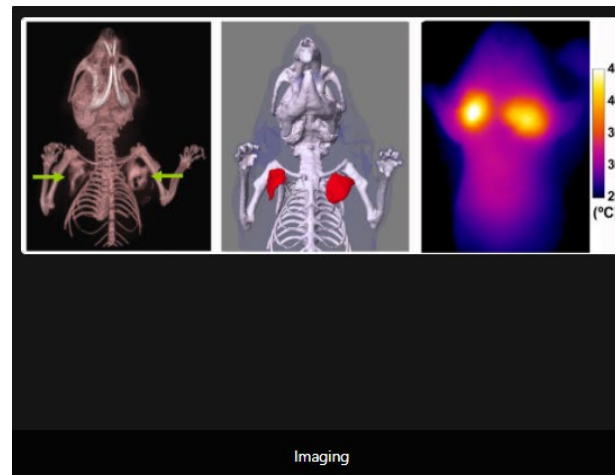
<https://www.atomiclimits.com/2017/07/03/the-dawn-of-atomic-scale-processing-the-growing-importance-of-atomic-layer-deposition-and-etching/>

Atomic level resolution of Au Particle



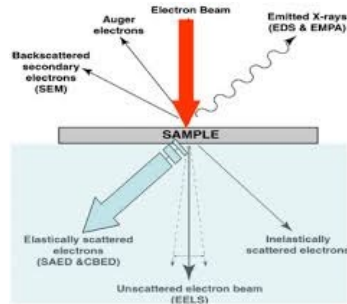
Atomic level resolution of the latest Au Nanoparticles

Au particles are used for medical research and treatment

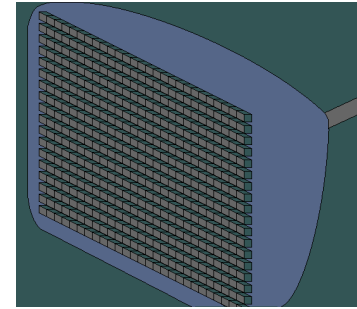


What is TEM (and STEM)

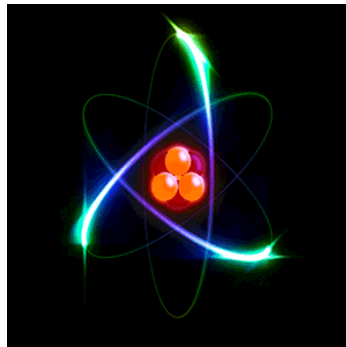
— Transmission



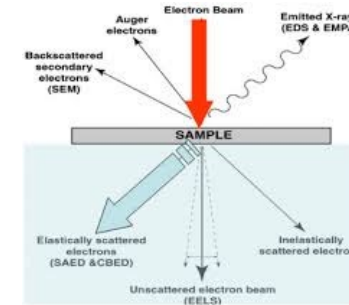
— Scanning



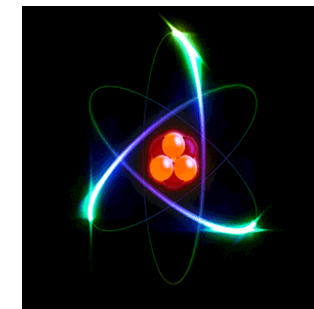
— Electron



— Transmission



— Electron

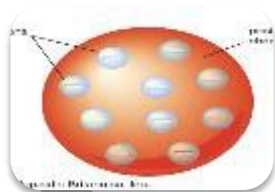


— Microscopy

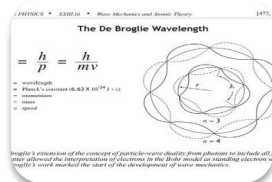


— Microscopy

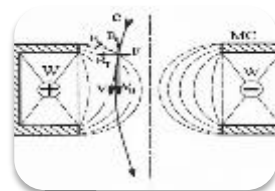




- 1897 J.J. Thomson discovers the electron



- 1924 L.deBroglie identifies the wavelength for electron



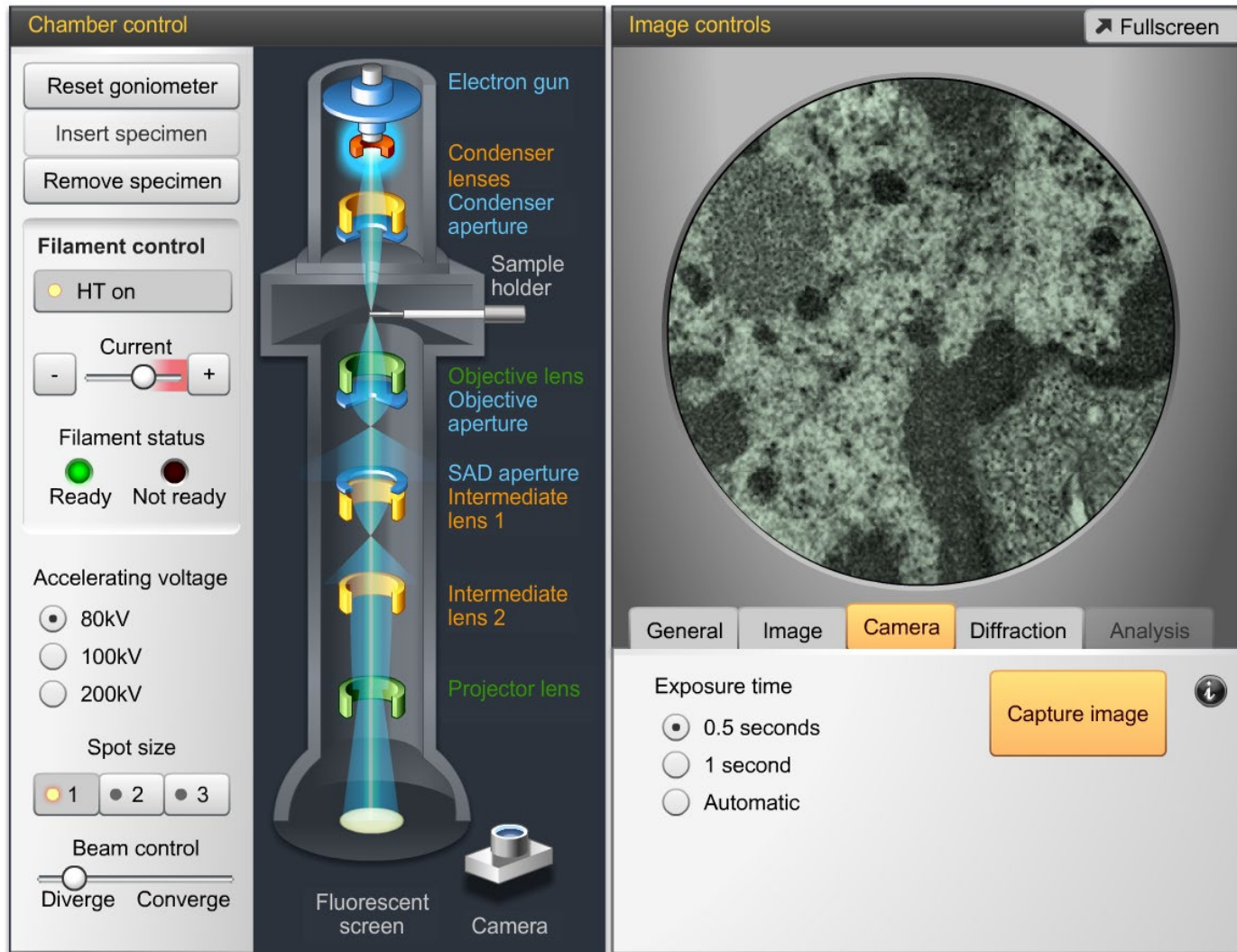
- 1924 E. Ruska publishes thesis on magnetic lenses



- 1931 Knoll and Ruska build the first electron microscope

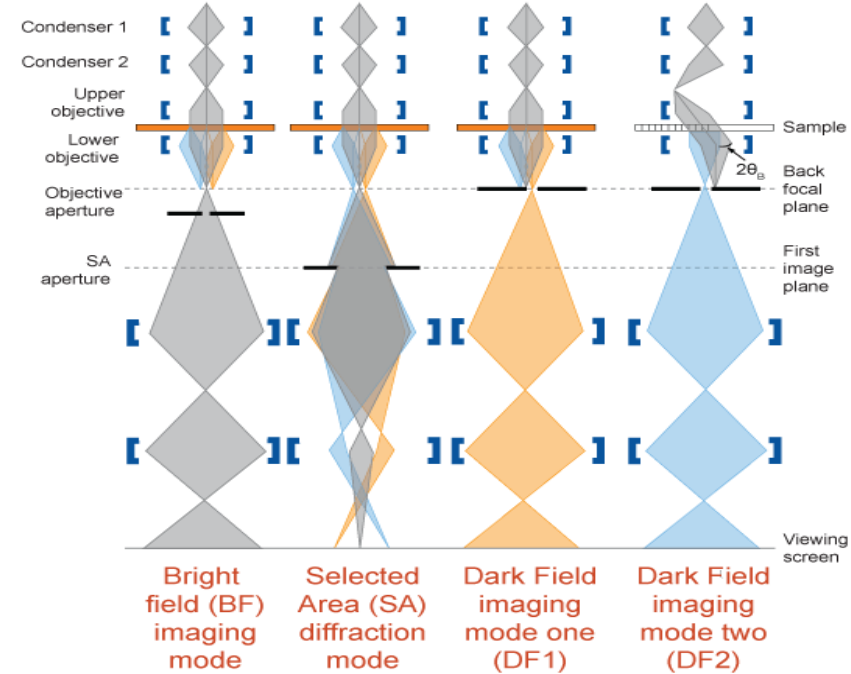
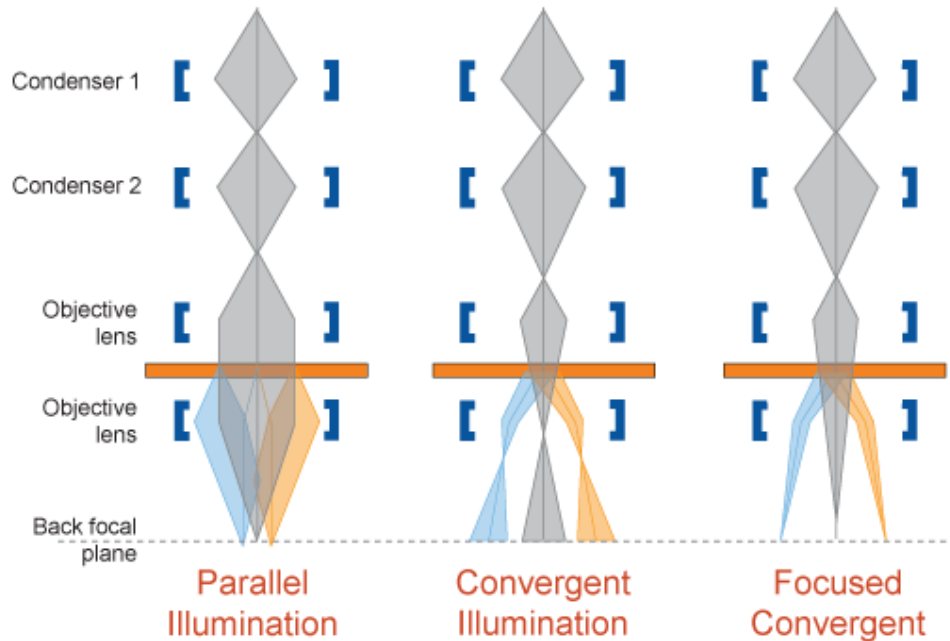


- 1939 Knoll and Ruska build the first commercial TEM



- **Electron Gun**
 - Creates the probe electron and accelerates it to the target
- **Condenser Lens and Aperture** (can have two)
 - Gather the electrons and focus them on the specimen
- **Sample Holder**
 - Holds the sample in the beam path and positions it along the sample orientation
- **Objective Lens and aperture**
 - Focuses and magnifies the image and selects the relevant electrons for the image
- **SAD aperture**
 - Provides Selected area diffraction for the sample
- **Intermediate lenses**
 - Magnify the image from the Objective Lens
- **Projector lens**
 - Magnify the image from the Intermediate lens
- **Camera**
 - Collect the image or wavelength of the electrons

- The beam in the TEM can be adjusted for a Variety of illuminations
 - Parallel Illumination
 - Convergent Illumination
 - Focused Convergent Illumination

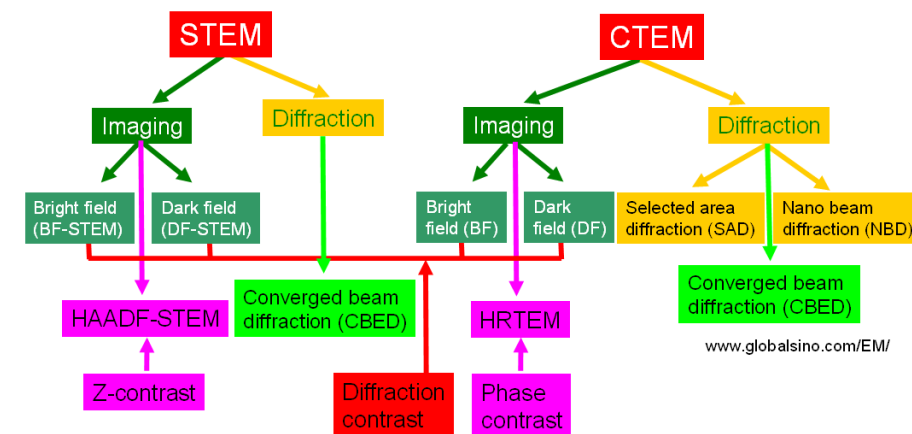
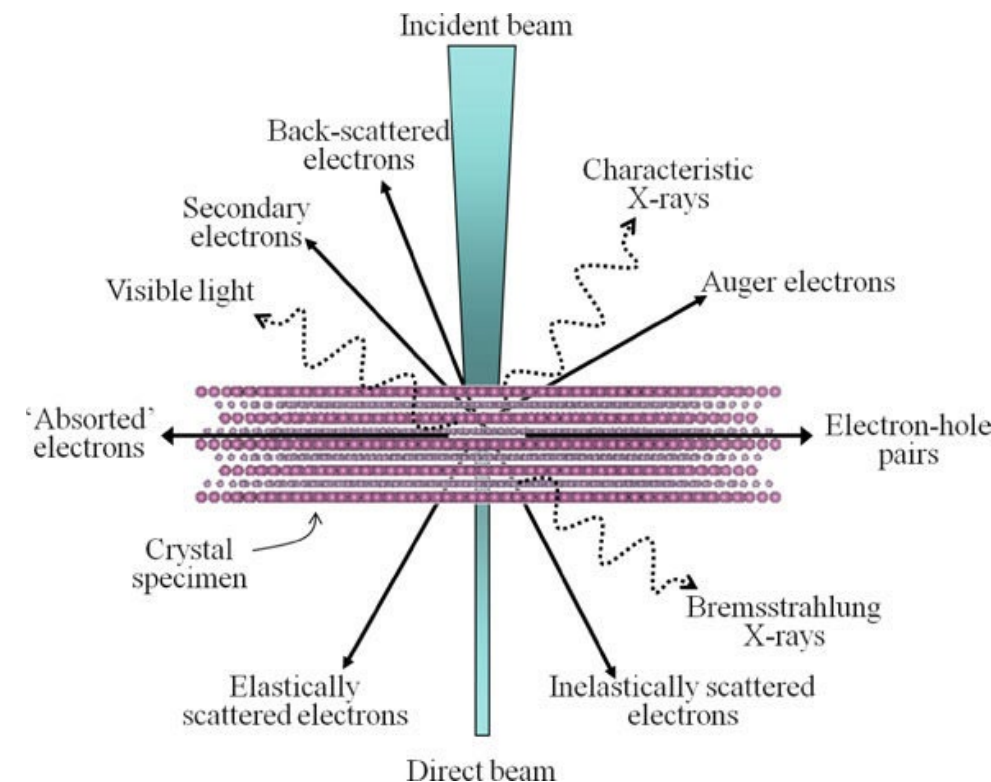


- With different illumination modes we can create different classes of images
 - Bright Field
 - Selected Area Diffraction
 - Dark Field mode one
 - Dark Field mode two

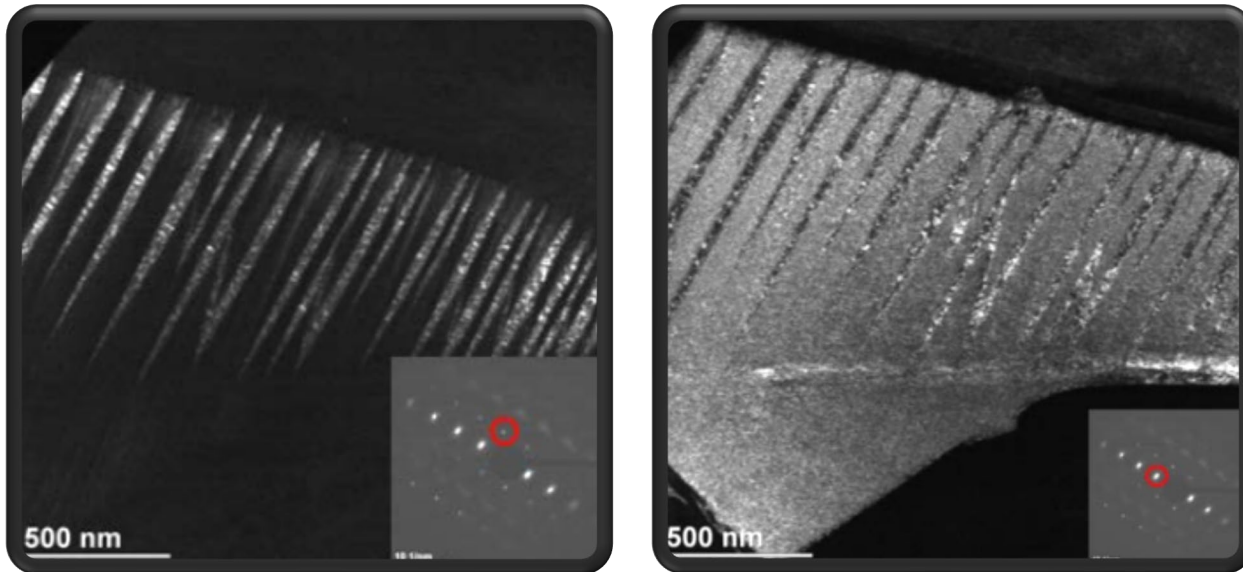
TEM contrast formation

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- The contrast between two adjacent areas in a TEM image can be defined as the difference in the electron densities in image plane. Due to the scattering of the incident beam by the sample, the amplitude and phase of the electron wave change, which results in **amplitude contrast** and **phase contrast**, correspondingly. Most of images have both contrast components.
- Amplitude-contrast** is obtained due to removal of some electrons before the image plane. During their interaction with the specimen some of electrons will be lost due to absorption, or due to scattering at very high angles beyond the physical limitation of microscope or are blocked by the objective aperture.
 - Mass-thickness contrast.** When the beam illuminates two neighboring areas with low mass (or thickness) and high mass (or thickness), the heavier region scatters electrons at bigger angles. Mass-thickness contrast is most important for non-crystalline, amorphous materials
 - Diffraction contrast** occurs due to a specific crystallographic orientation of a grain. In such a case the crystal is in a so-called Bragg condition, whereby atomic planes are oriented in a way that there is a high probability of scattering.
- Phase Contrast**
 - Crystal structure can also be investigated by high-resolution transmission electron microscopy (HRTEM), also known as phase contrast. When using a field emission source and a specimen of uniform thickness, the images are formed due to differences in phase of electron waves, which is caused by specimen interaction.



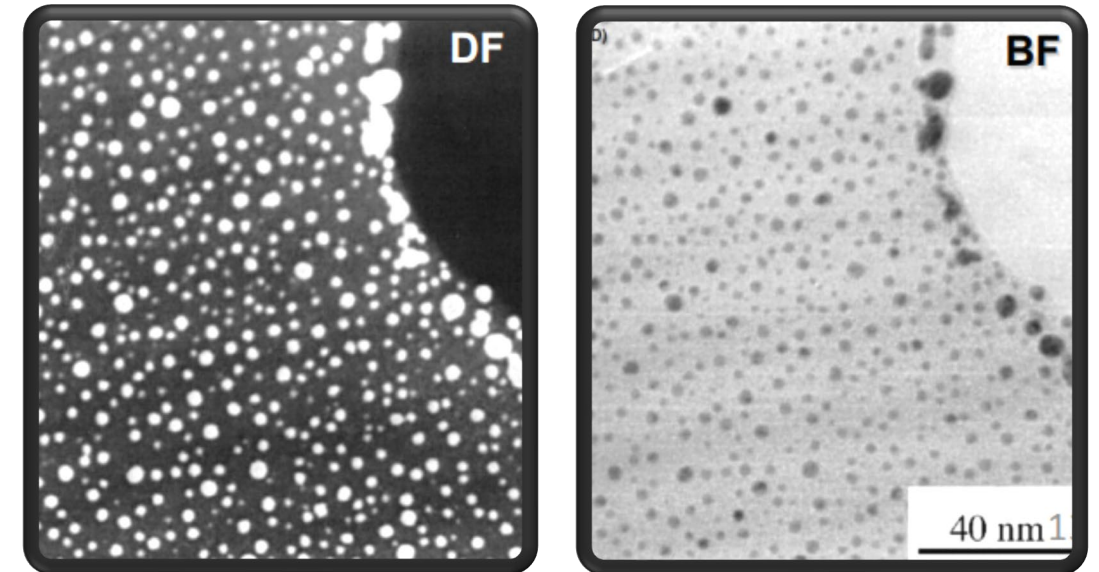
TEM Dark Field and Bright Field



TEM Imaging is useful for imaging the bulk structure, allowing better observations of crystal defects.

Dark Field imaging allows one to highlight lattice defects, like dislocations or precipitates

STEM Dark Field and Bright Field



STEM imaging in HAADF imaging allows for enhanced contrast, especially at lower atomic numbers, compared to TEM.

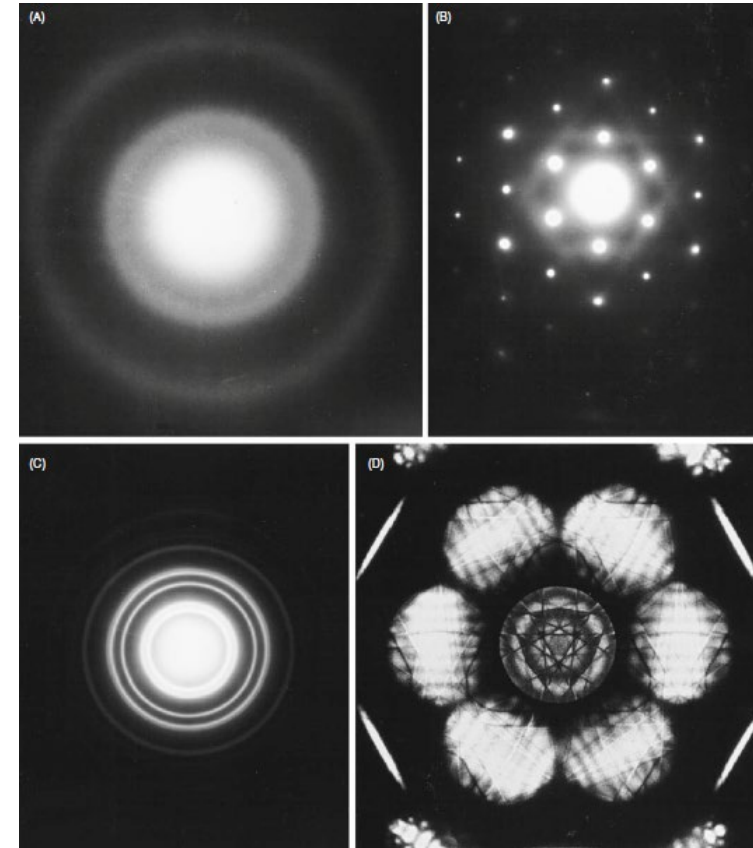
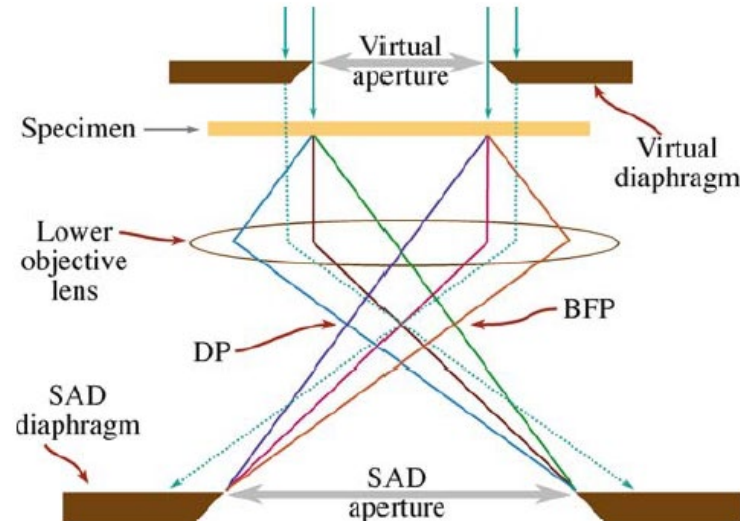
Bright-field advantages include:

- Enhanced contrast of the image
- The common and widely accepted technique

Each point in the diffraction pattern corresponds to an atomic plane

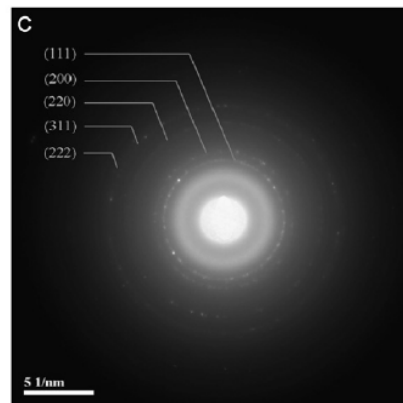
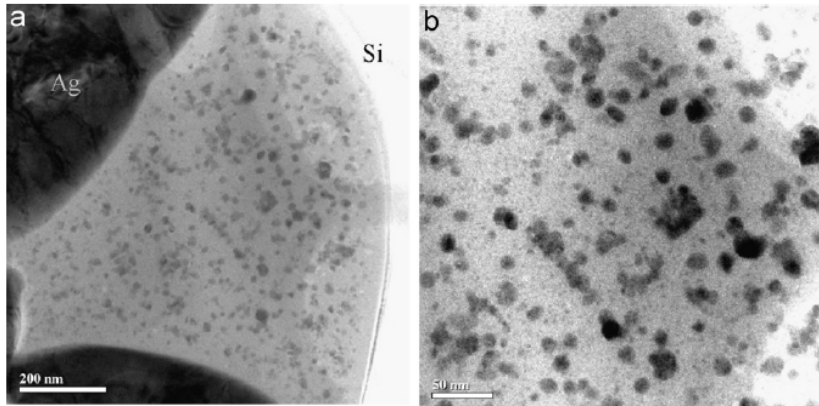
Diffraction Pattern (DP) and data can give:

1. Crystallinity of the sample
2. Amorphous sample (diffuse ring)
3. Single crystal materials (spot pattern)+ Orientation and zone axis
4. Indexing of atomic planes
5. Polycrystalline materials (ring pattern) + Indexing
6. Identification of materials/compounds by extracting the interplanar spacing (fingerprint)
7. Identification of matrix and second phase (precipitate)
8. Help to identify various materials at the interface



Ray diagram showing SADP formation: the insertion of an aperture in the image plane results in the creation of a virtual aperture in the plane of the specimen. Only electrons falling inside the dimensions of the virtual aperture at the entrance surface of the specimen will be allowed through into the imaging system to contribute to the SAD pattern.
(Transmission Electron Microscopy, Williams and Carter).

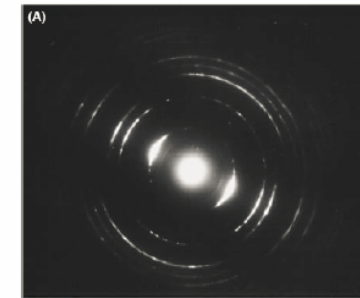
Ag-bulk/glassy-phase/Si heterostructures of printed Ag contacts on crystalline Si solar cells



- (a) **TEM bright field image.** The large glassy-phase enclosed with Si and Ag-bulk.
- (b) **Ag precipitates** in the large solidified glassy-phase region.
- (c) **Selected area diffraction pattern** of the glassy-phase region shown in (b). Only Ag crystallites exist.

(Ching-Hsi Lin et al, Solar Energy Materials & Solar Cells)

Textured Diffraction Pattern



Incomplete rings indicates non-random orientation distribution – Preferred orientation of the grains

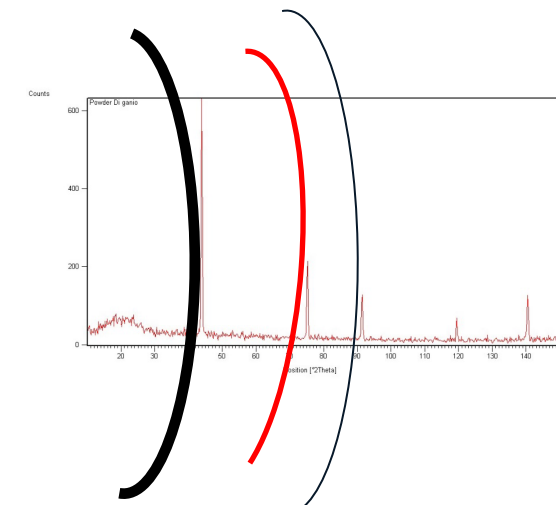
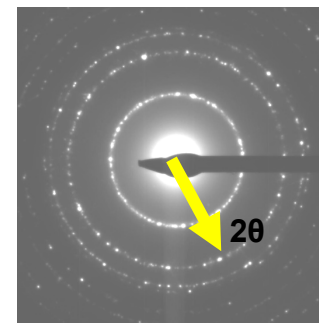


Weakly textured



Strongly textured

Electron vs X-ray diffraction pattern



TEM Advancements

And how they relate to your sample

7 myths about modern TEM

22



TEM Takes a long time

Modern TEM are much quicker than previous generations. Samples can be prepared and imaged in a few hours

TEM is the best resolution

STEM can be a better imaging choice for some samples as it allows for enhanced contrast

EELS is needed for elemental analysis

EDX concentration is much more accurate and has been used for elements as low as B

TEM is expensive

Due to higher speed and new systems TEM can be cost comparable to cross sectional SEM

TEM requires 300kV

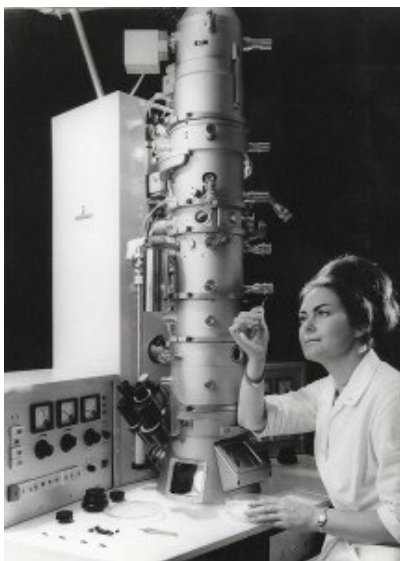
Thinner samples and lower kV TEM can provide better information with less damage

Only a PHD can understand TEM data

TEM images are as clear as other Microscopy techniques. EDX maps are easy to interpret

Corrected TEM is better

Correctors can increase resolution but additional resolution often is not helpful



Modern advancements in TEM

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Digital TEM

Computer driven operation of TEM increases speed and control

1980s



1980s



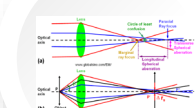
CCD Camera

Introduction of CCD and CMOS detectors have allowed for digital recording of data

Corrected TEM

Image and probe correctors have increased resolution

1990s



2000s



Sample preparation

FIB sample preparation and other techniques can create thinner and cleaner TEM samples

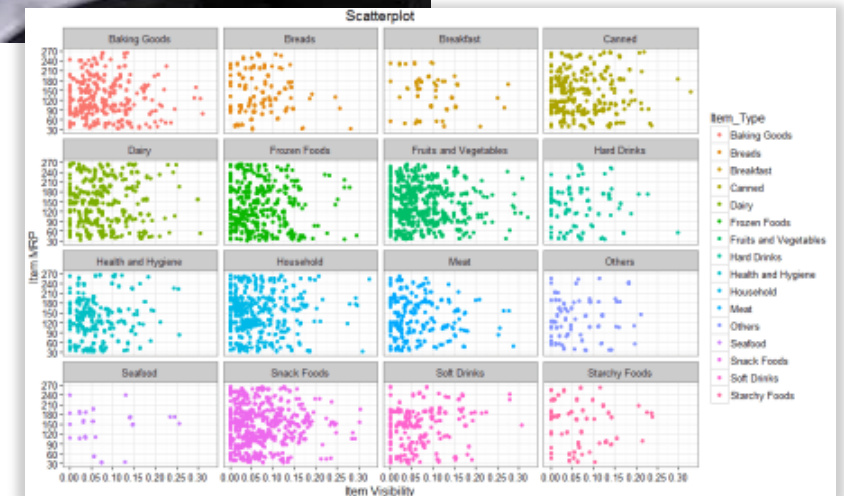
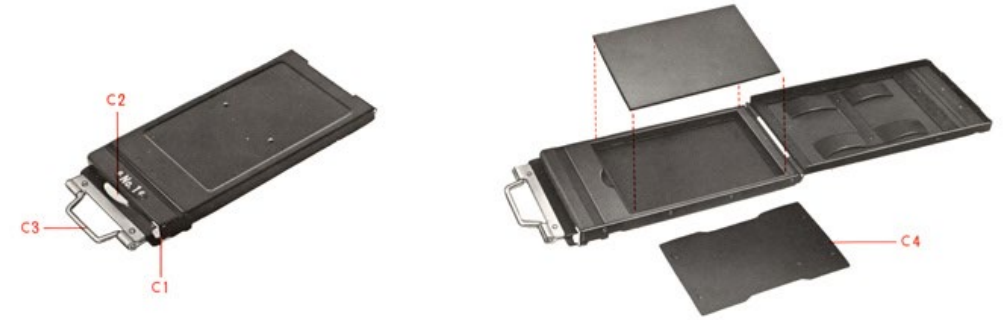
Automation

Advancements in automation have increased TEM consistency

2010s

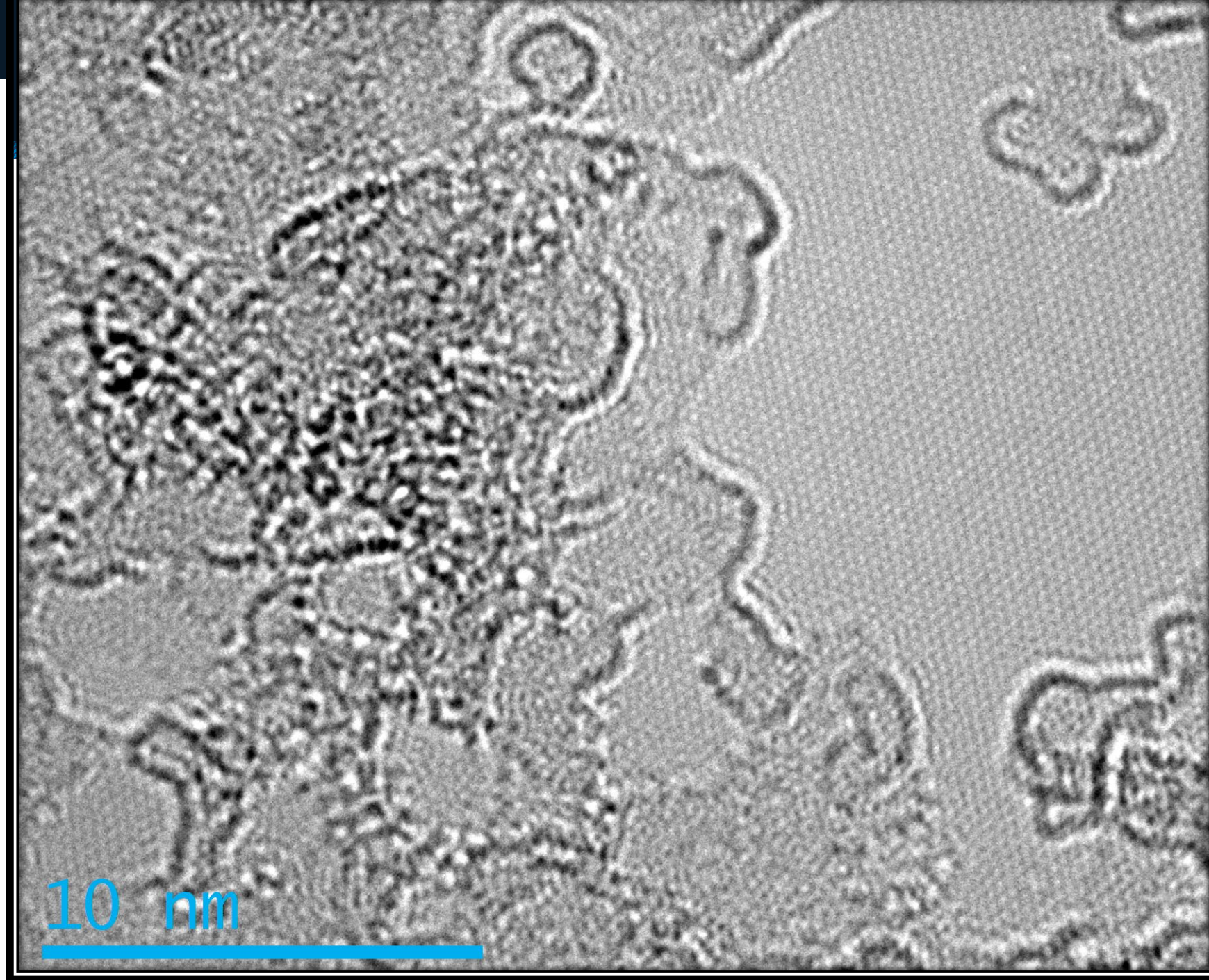
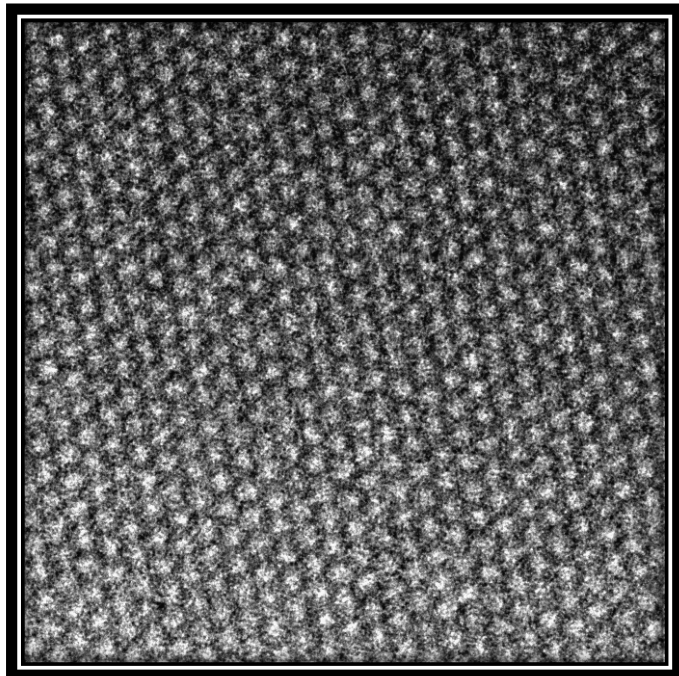


- Digital imaging has allowed live imaging and collection of data without the need for Film stock
- System operation is much faster and much more accurate with a computer interface
- Computer controller adjustment of the tool allows for saved column alignment files and quick adjustment of key image chain features
- Digital interface simplifies operational focus
 - Software programming allows for Automation
- Digital data recording allows for all components of the imaging process to be tracked and recorded



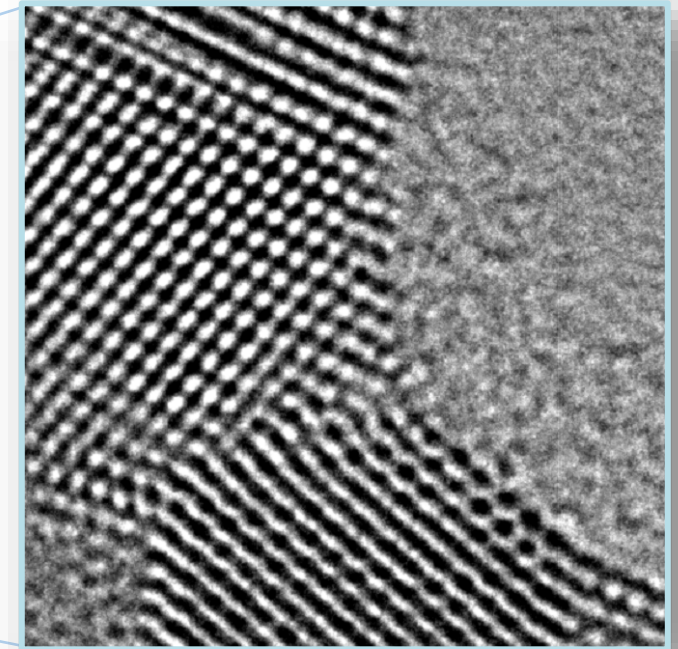
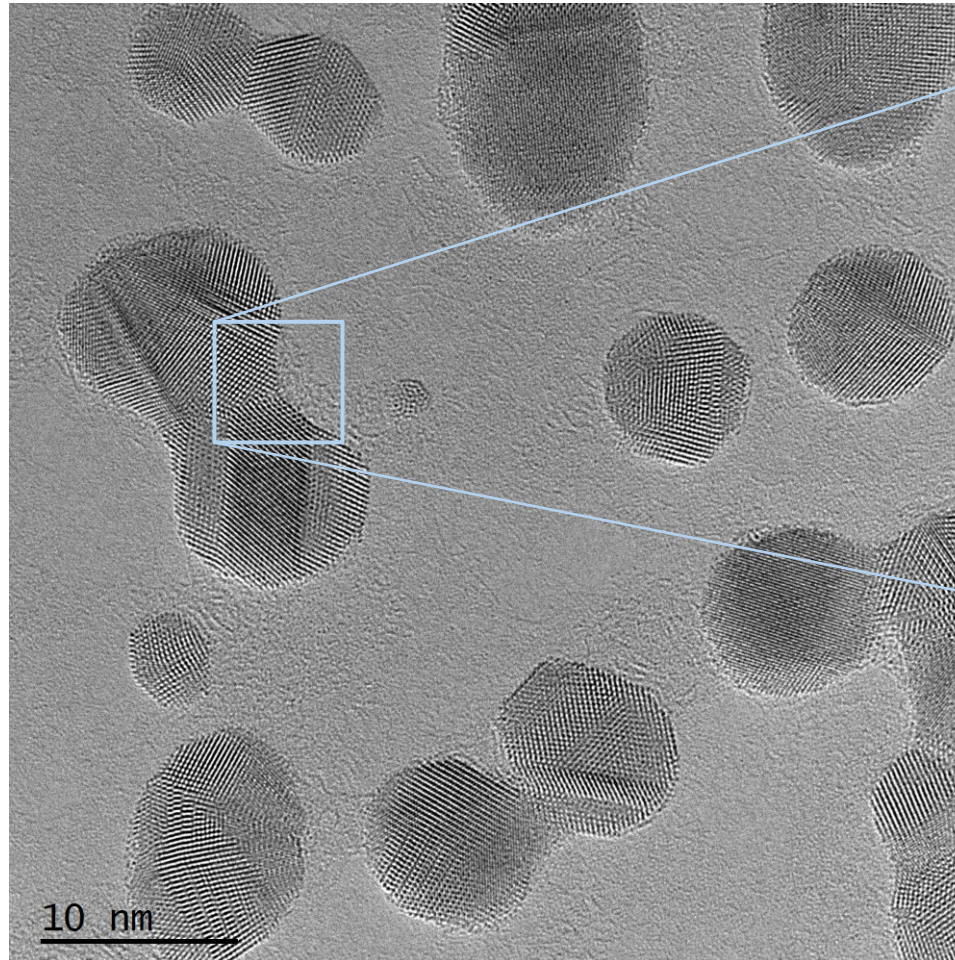
Impact of 4K Imaging

Relative comparison of
1k by 1k and 4k by 4k



High Resolution Lattice Imaging

Modern CMOS detectors allow for quick collection of detailed images while maintaining resolution

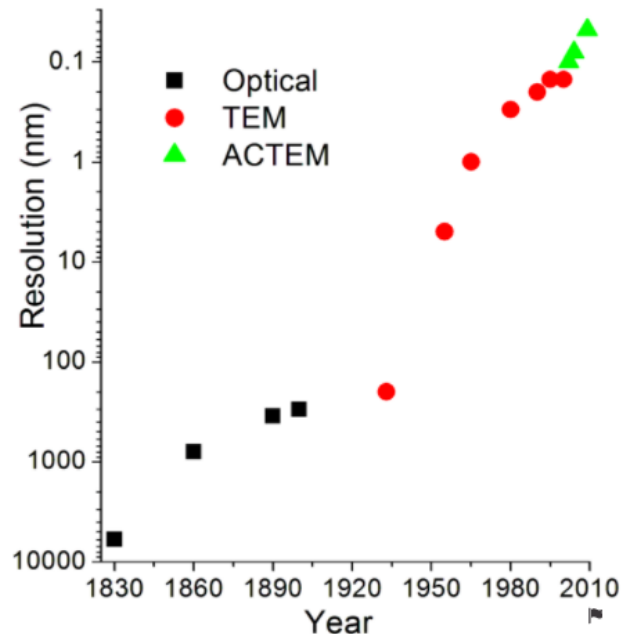


Because of OneView camera's **high resolution** and **high sensitivity**, lattice imaging with a large field of view is effortless.

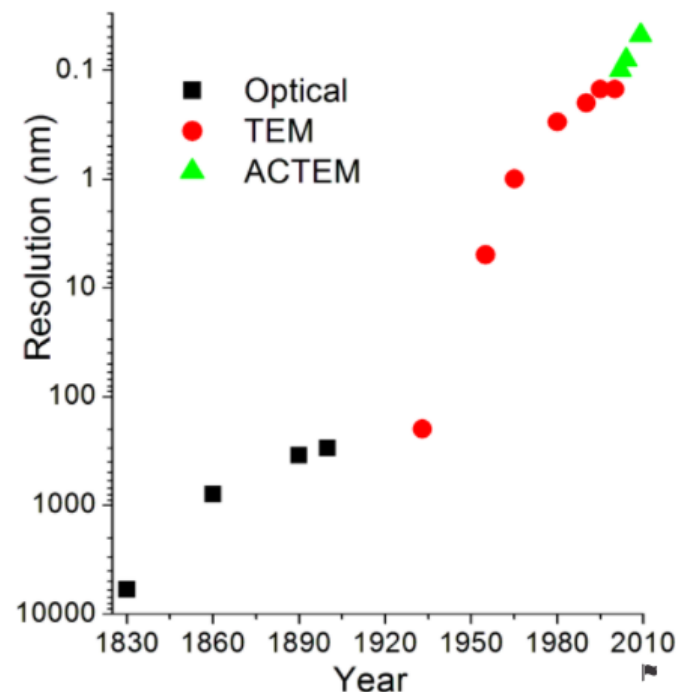
How TEM compares to other high “Magnification”

- Magnification comparison:
 - Magnification of Magnifying glass 3X
 - Magnification of Cell phone 100X
 - Magnification of Table top Microscope 800X
 - Magnification of Best Telescope 24000X
 - Magnification of SEM 250000X
 - Talos 2M X
 - Magnification of TEM (*corrected) 10M X (0.5A)

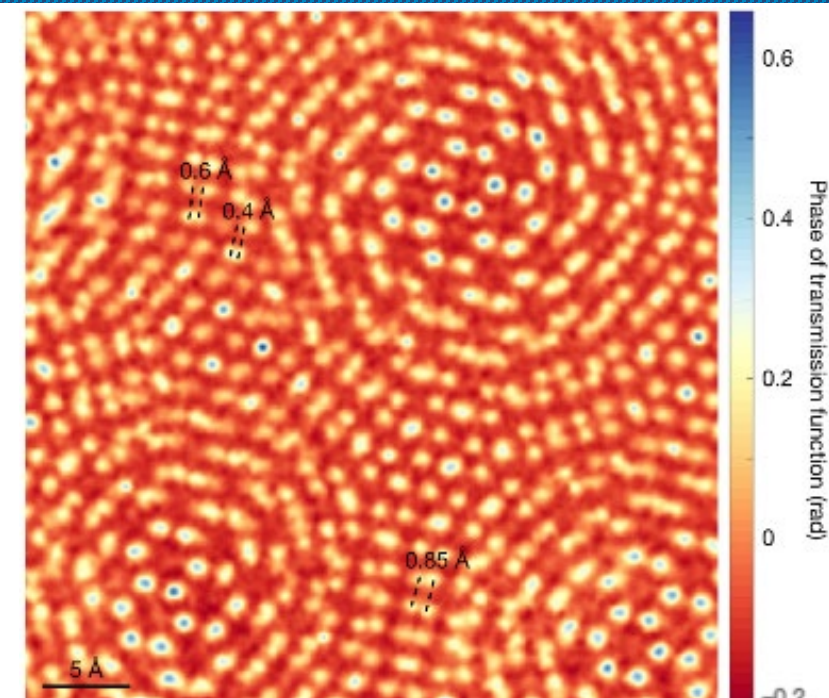
Why Magnification is an outdated concept!



- A Cs-corrector can be located in your condenser or below your objective lens.
 - The corrector in the condenser forms a finer beam (**probe corrected**) that provides higher spatial resolution in STEM mode and/or a better performance for spectrum imaging
 - A corrector below the objective lens results in a higher resolution in imaging mode (**image corrected**).
 - A monochromator reduces the energy spread of the electron beam by the cost of intensity.
- Development and use of corrected TEM and STEM has driven new resolution performance and opened up significant imaging capability with the technology
- **Current record resolution is 0.39 Å**
 - Size of a Cu Atom is 1.40 Å
 - Size of a Pb Atom 2.02 Å
 - Size of O Atom is 1.52 Å
 - Size of Si Atom 2.10 Å



[1] Jiang Y, Chen Z, Han Y, Deb P, Gao H, Xie S, Purohit P, Tate M W, Park J, Gruner S M, Elser V and Muller D A 2018 Electron ptychography of 2D materials to deep sub-ångström resolution *Nature* **559** 343–9



Real-space resolution test of full-field ptychography using twisted bilayer MoS₂. The two sheets are rotated by 6.8° with respect to each other, and the misregistration of the molybdenum atoms provides a range of projected distances that vary from a full bond length down to complete overlap. Atoms are still cleanly resolved at a separation of 0.85 ± 0.02 Å, with a small dip still present between atoms separated by about 0.61 ± 0.02 Å, similar to the contrast expected for the Rayleigh criterion for conventional imaging. Atom-pair peaks at 0.42 ± 0.02 Å show a 6% dip at the midpoint, suggesting that the Sparrow limit lies just below 0.4 Å. The Raleigh resolution for ADF STEM is 1.2 Å for these imaging conditions

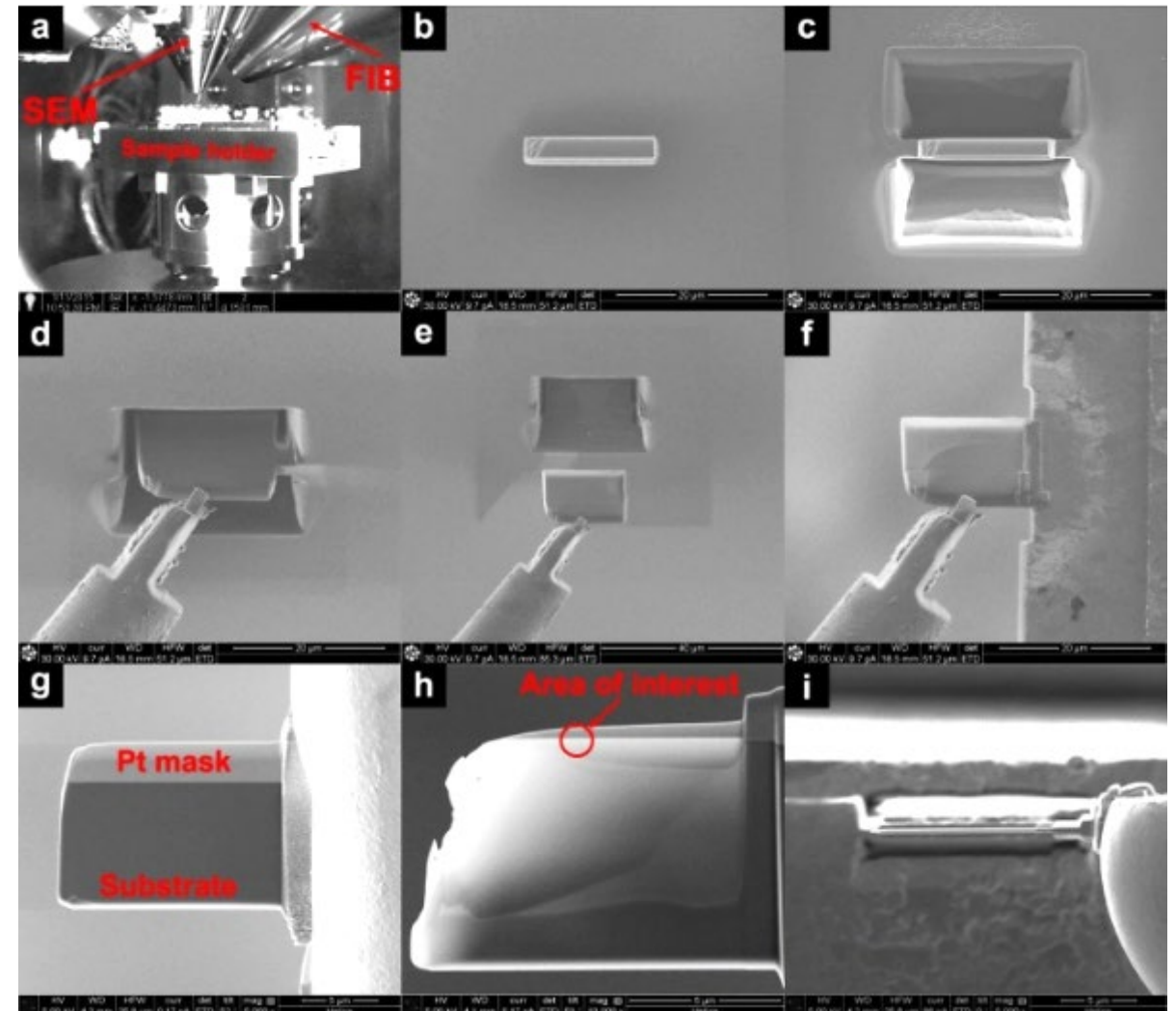
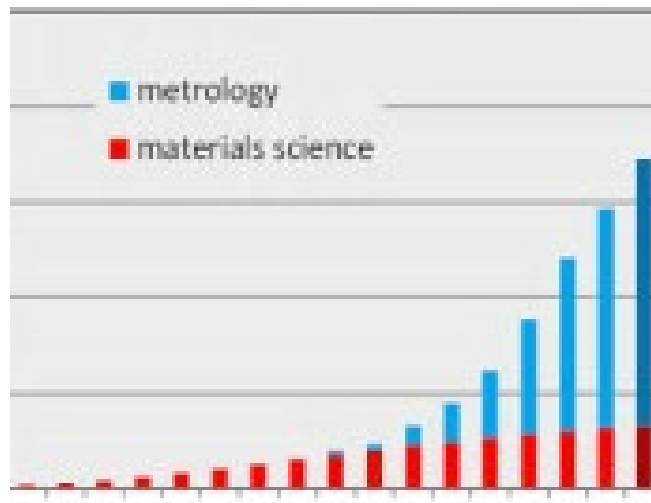
- Thermo-Fisher(FEI) Scios FIB-SEM
- Thermo-Fisher(FEI) Helios UC FIB-SEM
- Lamella down to 20nm thickness can be created
- Lamella can be created in 1 to 4 hours, depending on complexity
- Lamella can be created automatically for repetitive jobs



FIB TEM sample prep

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- FIB sample prep and exploded the use of TEM imaging for modern material science:
- Specifically **semiconductor** and **solid state devices**
 - greatly decreased sample costs and
 - faster time to data

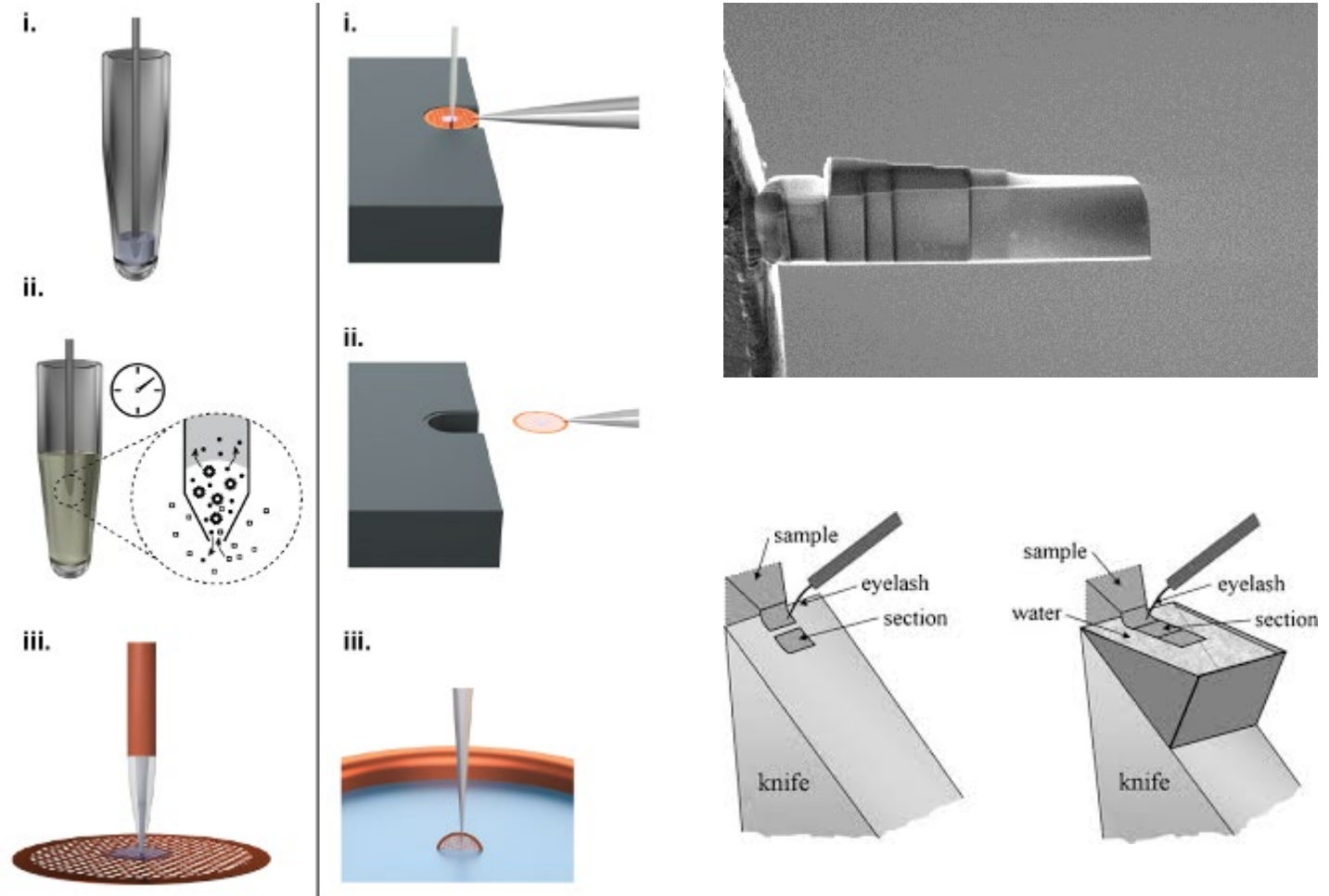


- 2 to 3 million Lamella created per year
 - 500,000 capacity added each year

Alternative Sample Prep for TEM

Advances in aqueous particles, biological samples large area samples have also greatly increased

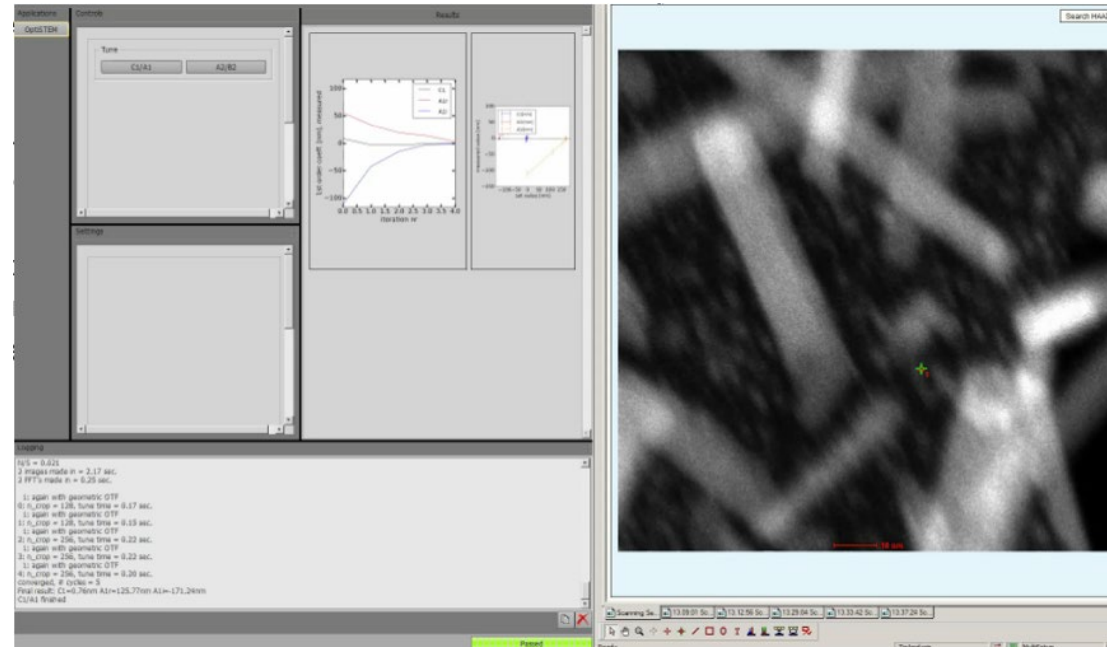
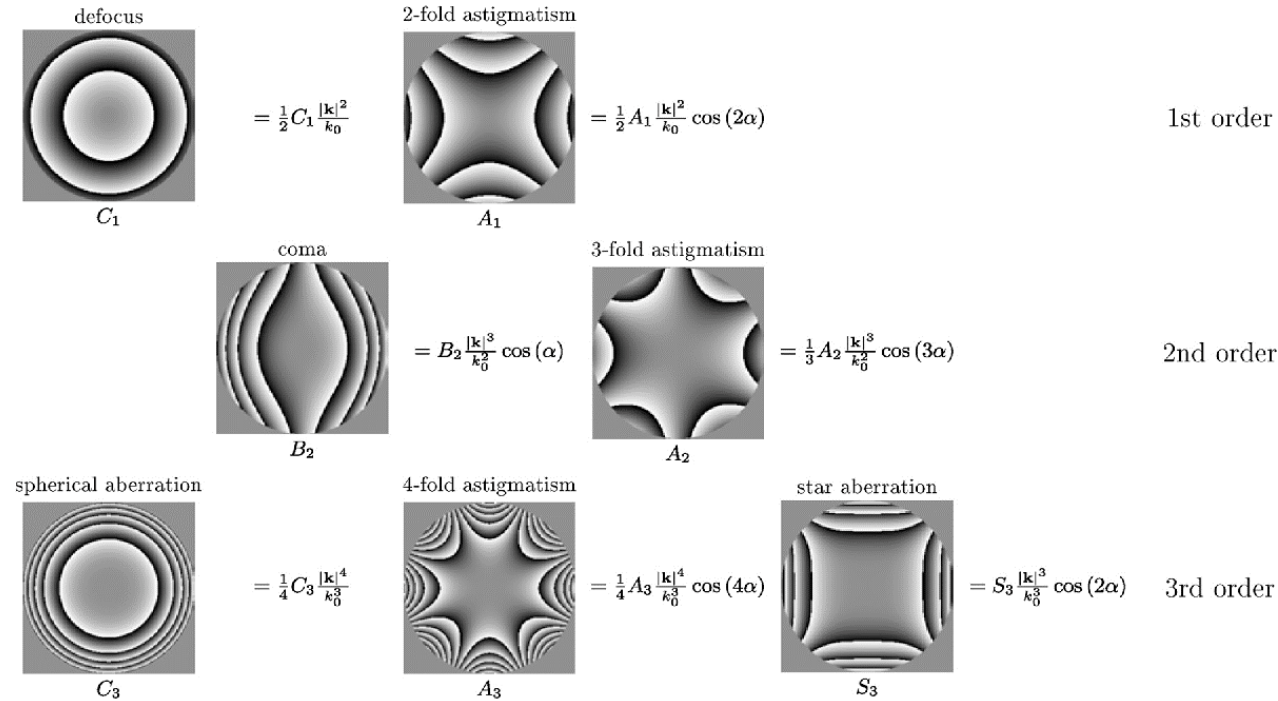
- Micro Droplet quickly isolate soluble samples to carbon grids
- Cryo sample prep allows for visualization of portions and viruses impacted modern health and pharma development
- Advanced Microtomes and Plasma FIB allow for large area TEM sample prep



Auto Alignment of image aberration

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- Automated correction
 - Focus
 - Astigmatism
 - Advanced Auto Alignments can adjust 2nd order aberrations
- From 5kx - 910kx mag.
 - Fast and reproducible
 - Best foundation for HRSTEM
- All samples

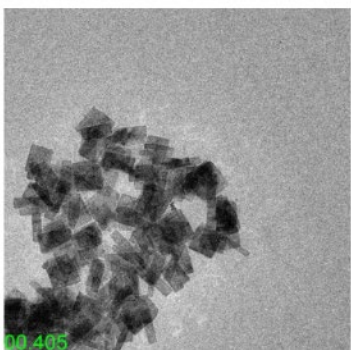


Drift correction (and low frequency vibration)

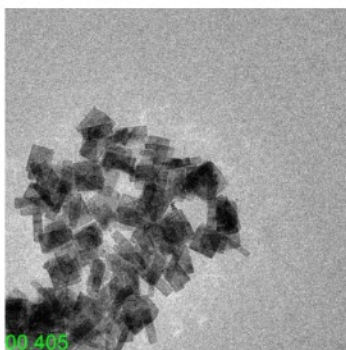
33

- Auto correction integrates image frames to keep the sample in the FOV
- Specific particles can also be tracked to maintain focus

Recursive averaging and frame alignment

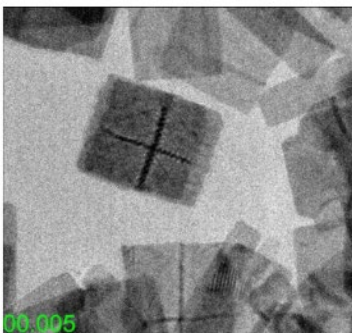


Average 5 frames without frame alignment, as the sample drifts, averaged frames look fuzzy

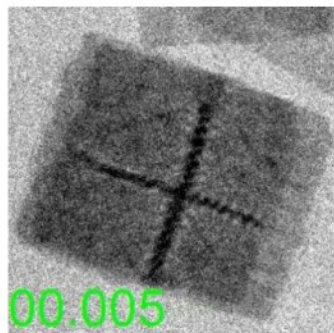


Average 5 frames with frame alignment, as the sample drifts, averaged frames stay sharp

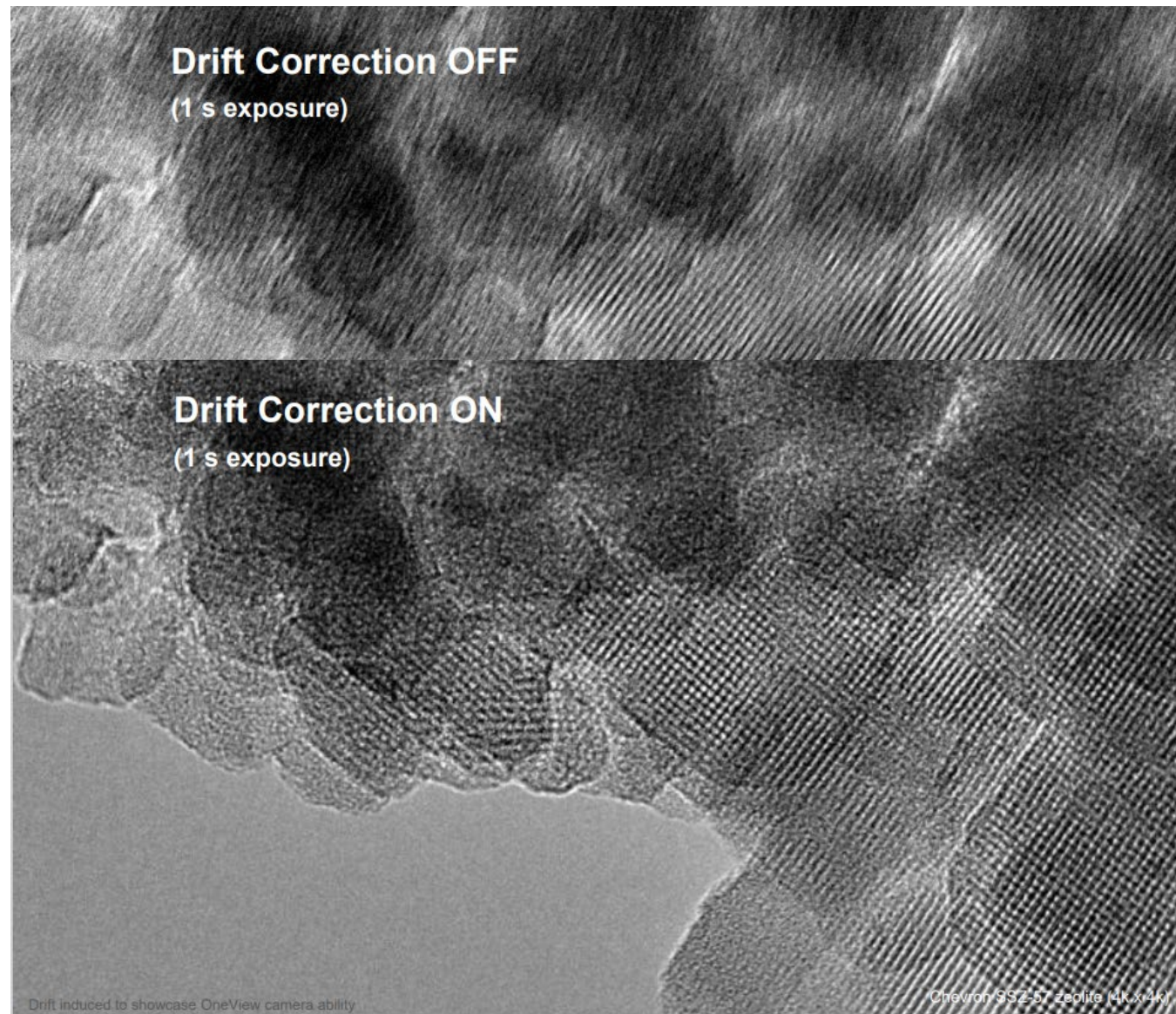
Particle tracking



Original dataset, particle of interest is moving in the FOV



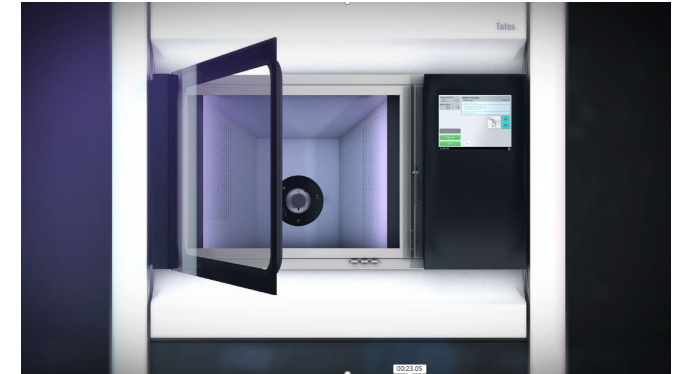
Movie saved with Track Particle on, the particle of interest stays stationary in the center of the FOV



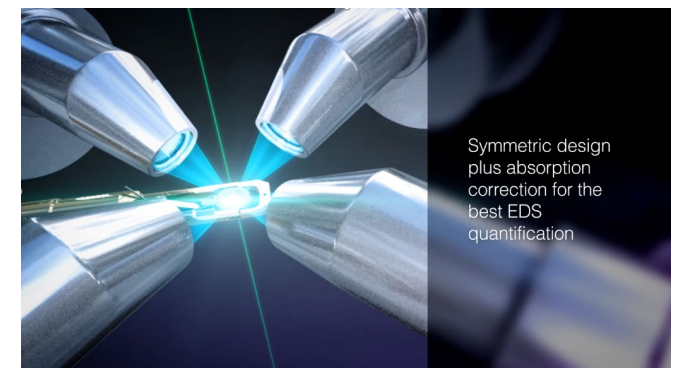
Covalent Metrology

Talos F200X Overview

- Smart Box
 - Reduces environmental sensitivity
 - Touch-screen info panel built-in
- Highest Brightness (X-FEG)
 - More electrons in the smallest spots
 - Better S/N in HRSTEM images
 - More X-rays gives higher sensitivity
- Reduced acquisition time; largest and cleanest EDS
 - Super-X G2 detector with 4 independent symmetrical SDD's
 - Enables EDS tomography for 3D chemical info
 - Unique absorption correction for the most accurate quantification
 - No matter what your sample orientation is



Sample courtesy of Louisiana State University,
Professor Rongying Jin funded by NSF



- Gatan OneView Camera is the most powerful general purpose detector commercially available

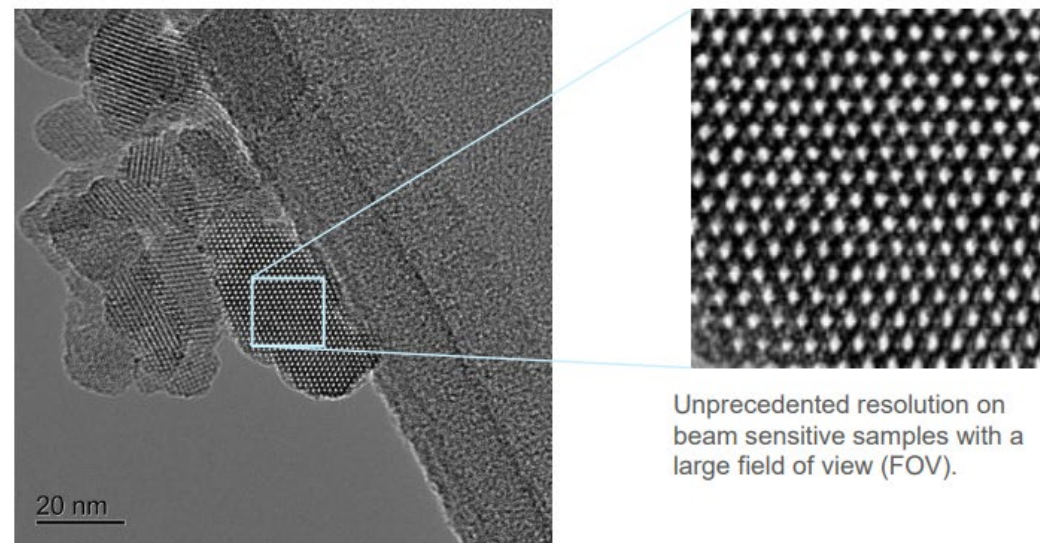


OneView™ Camera

Speed vs. Resolution

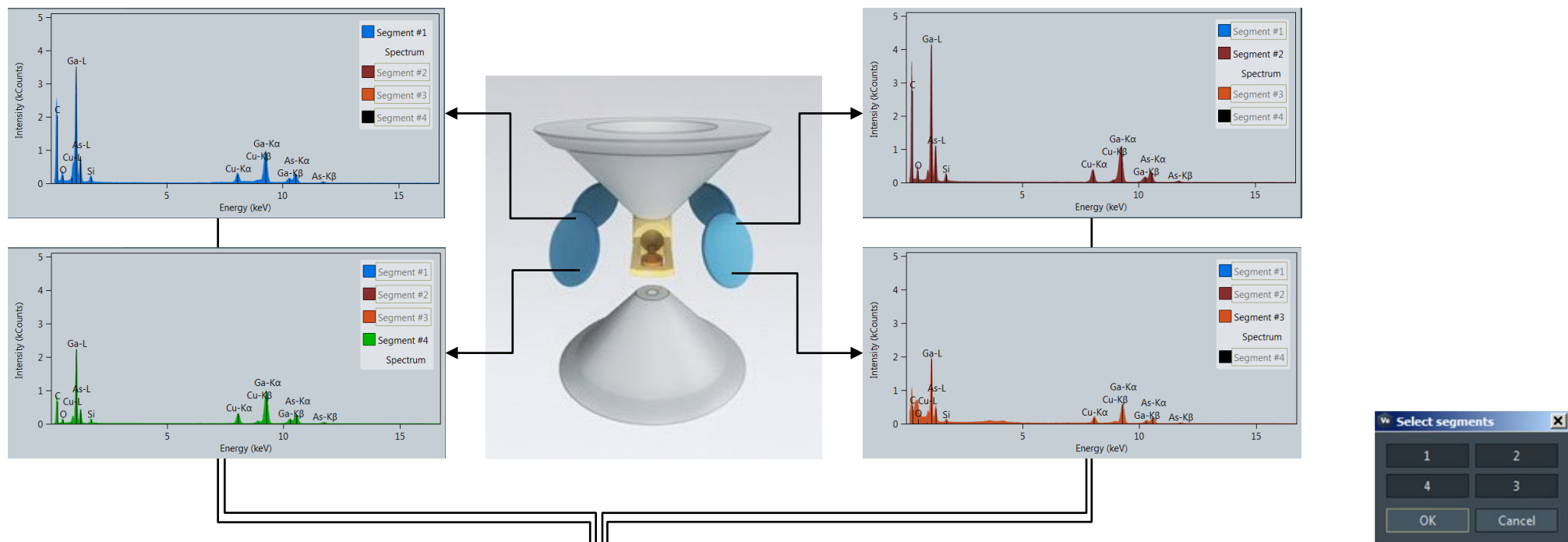
Live Drift Correction

High Resolution on Beam Sensitive Samples

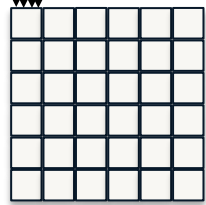


Unprecedented resolution on beam sensitive samples with a large field of view (FOV).

Image information: sample: Zeolites; magnification: 255kx; beam energy: 200 keV; image size: 4k x 4k; exposure: 2 s; number of frames: 50
Image courtesy of Chevron SSZ-57.



- Critical for correcting for shadowing
 - Due to sample geometry and roughness
- For most accurate quantification

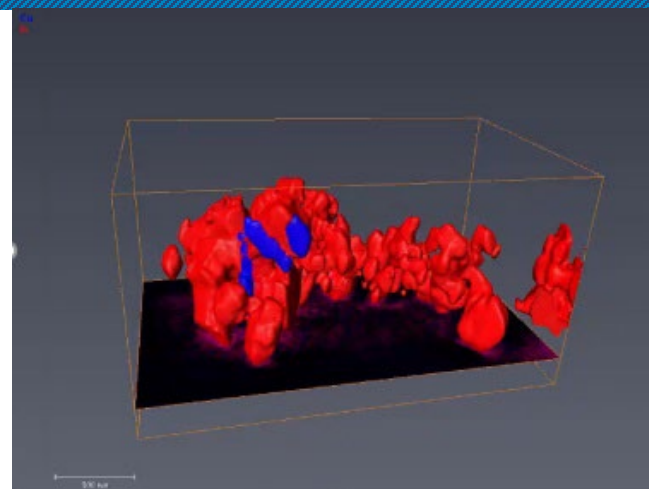


Optimized for Automated 3D-EDS



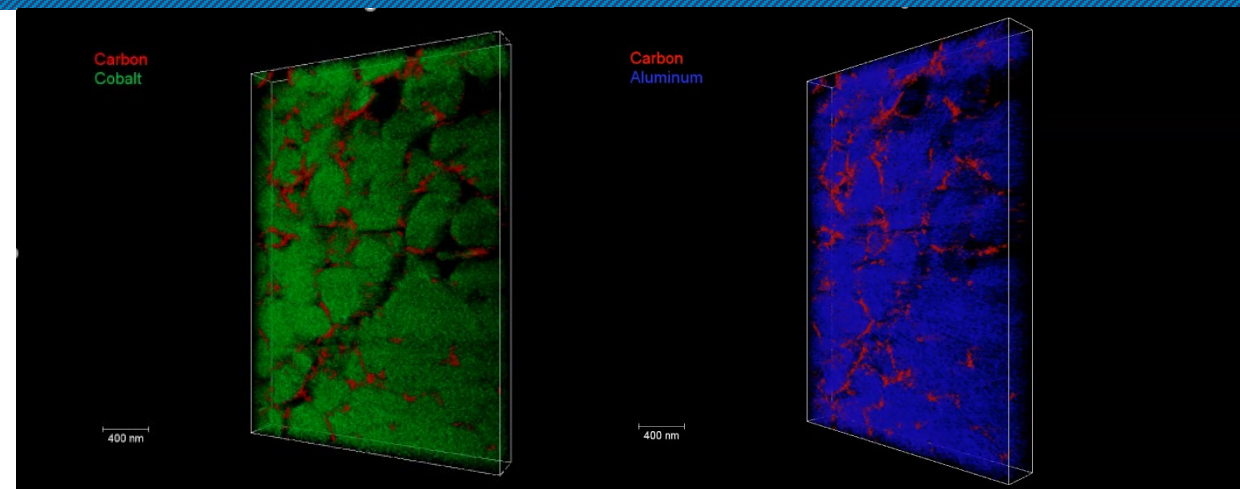
Sample Courtesy Michigan Tech University, Dr Reza Shahbazian Yassar

Zn-P-In tubes - electrode material for Na-ion & Li-ion batteries

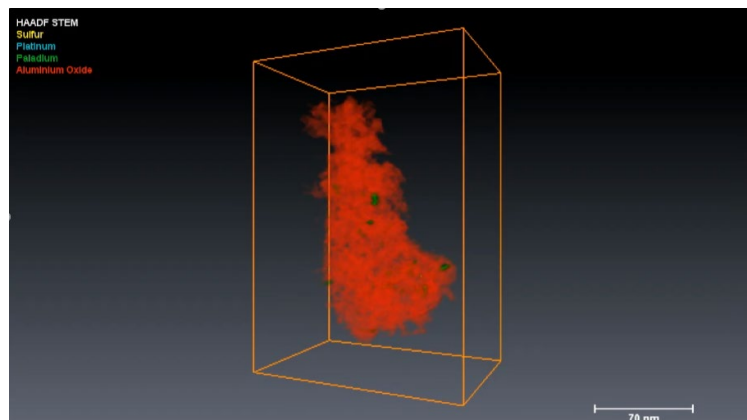


Sample courtesy: Prof. Neerish Revaprasadu, University of Zululand

CuInS nanostructure Cu-In

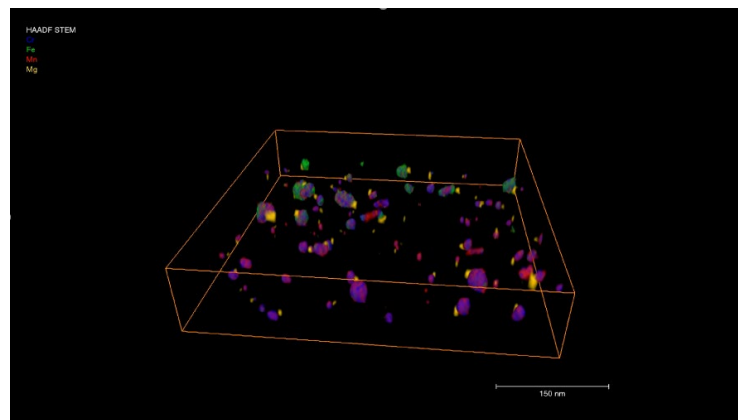


Al-Ni-Co-C battery anode material



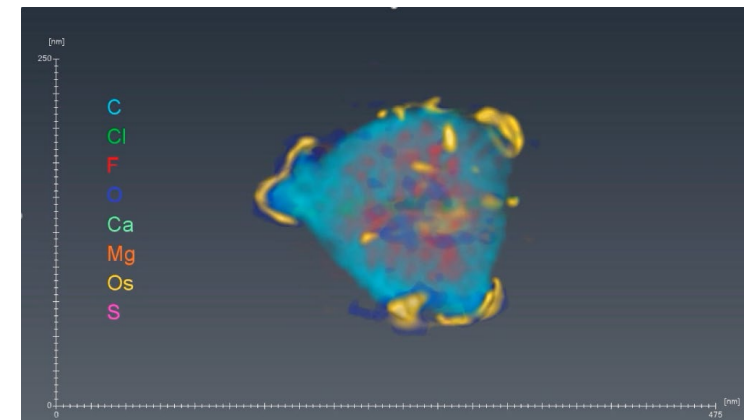
Sample courtesy: Tampere University of Technology

Catalyst powder



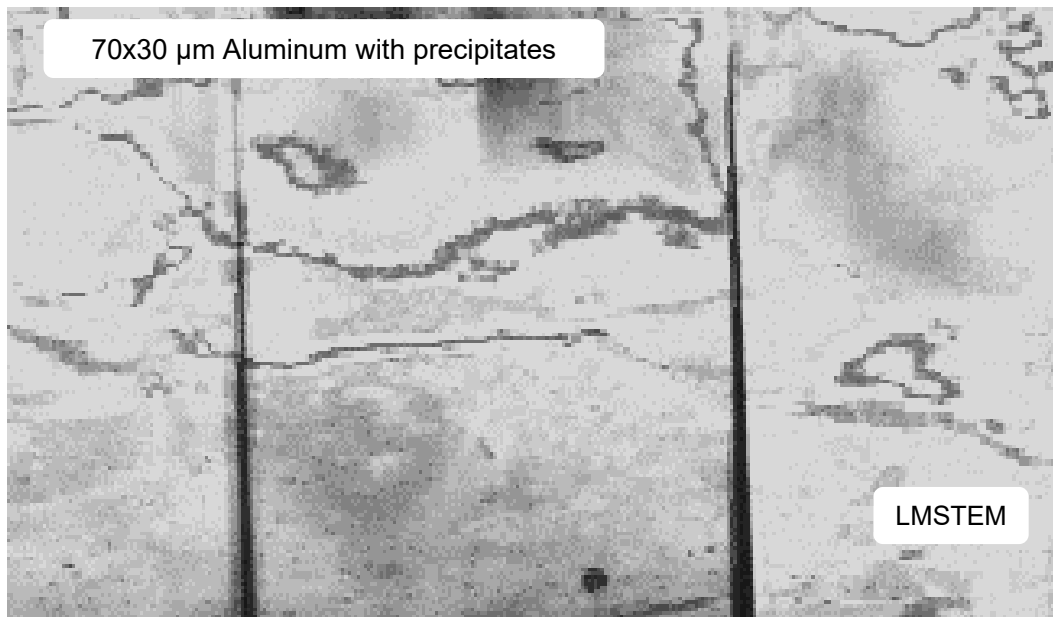
Sample courtesy: Montanuniversitaet Leoben; Thomas Kremmer, Stefan Pogatscher.

AlMgSi alloy; inclusions in Aluminium

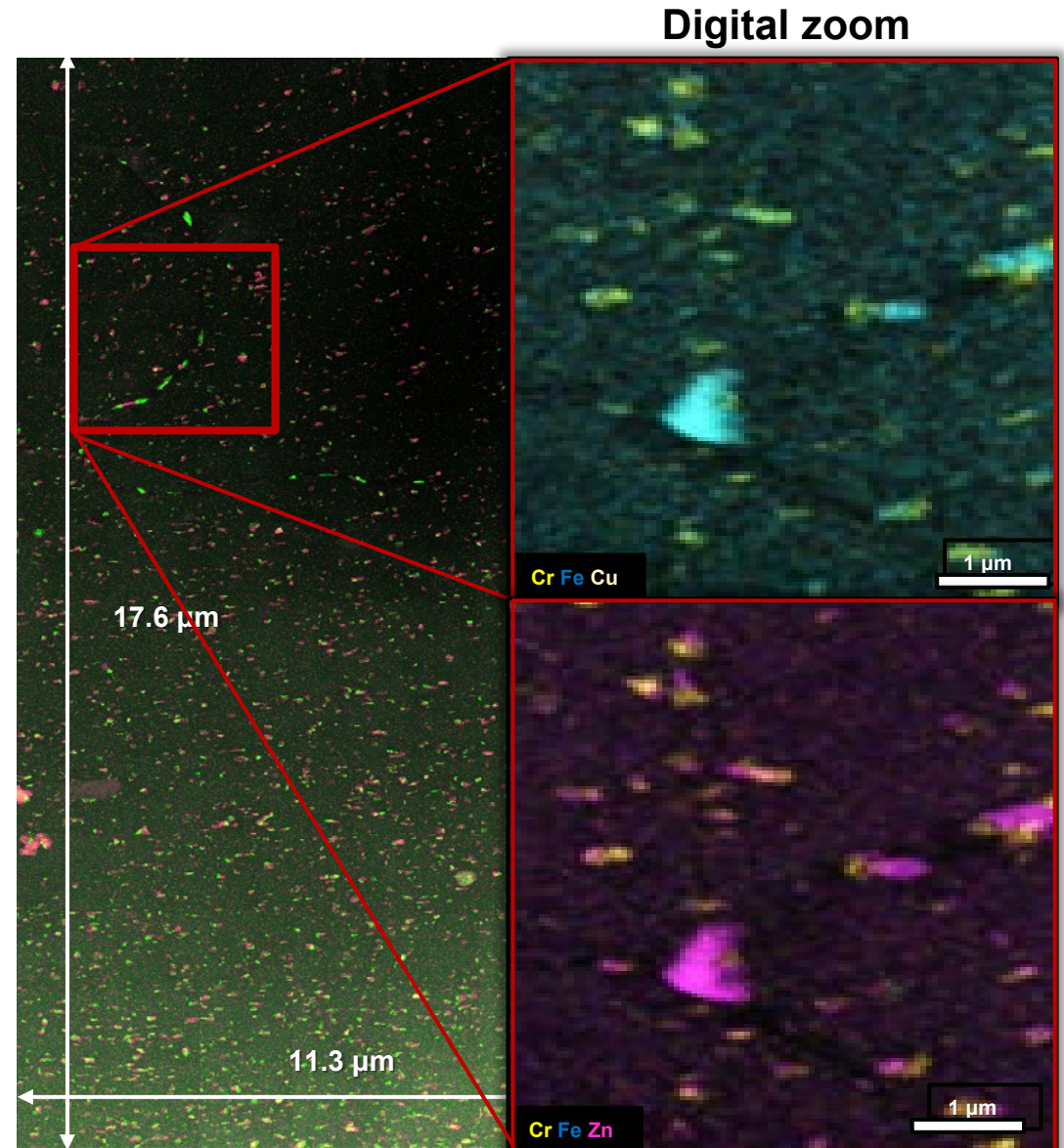


Organic Nanoparticles/Polymer Sheet

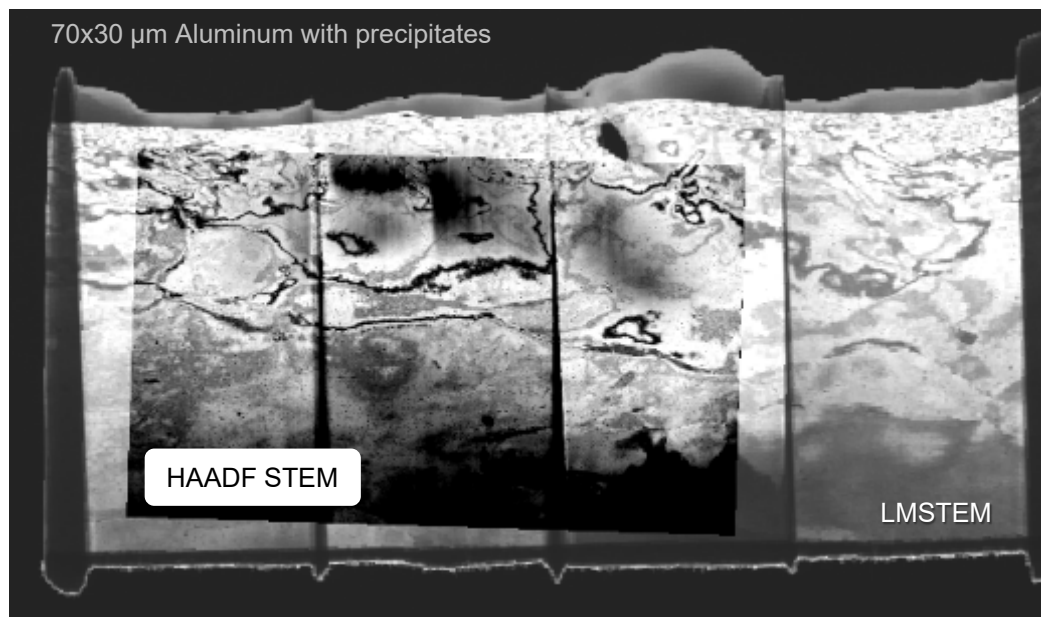
- Analysis with Super-X & Dual-X EDS
 - Automated large area EDS at high resolution
- Aerospace grade aluminum alloy (PFIB prep.)
 - Cr rich particles mainly in Al matrix
 - Fe/Mn inclusions
 - Zn-Cu-Mg rich particles on grain boundaries



Sample courtesy: University of Manchester / University of Trento

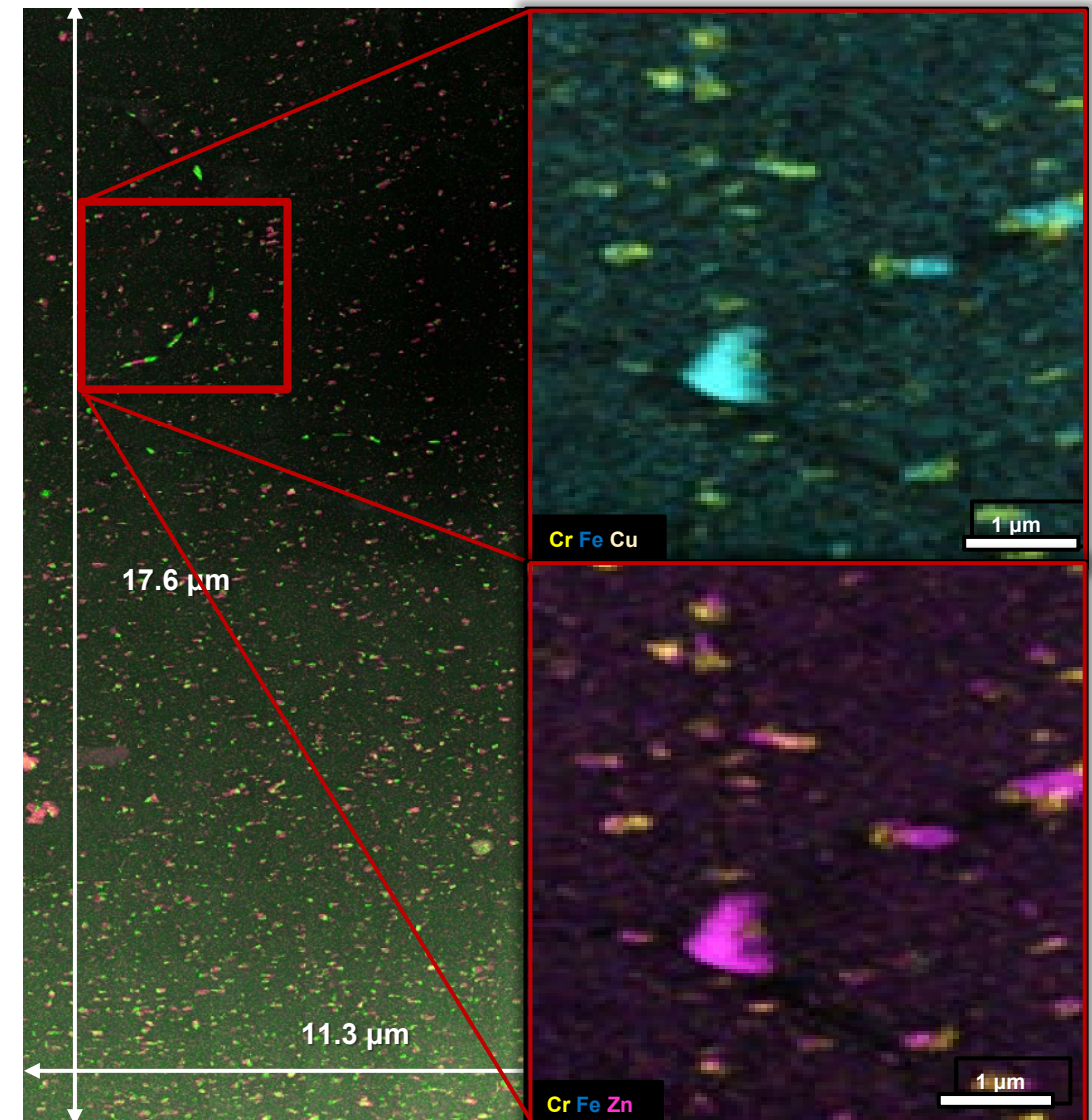


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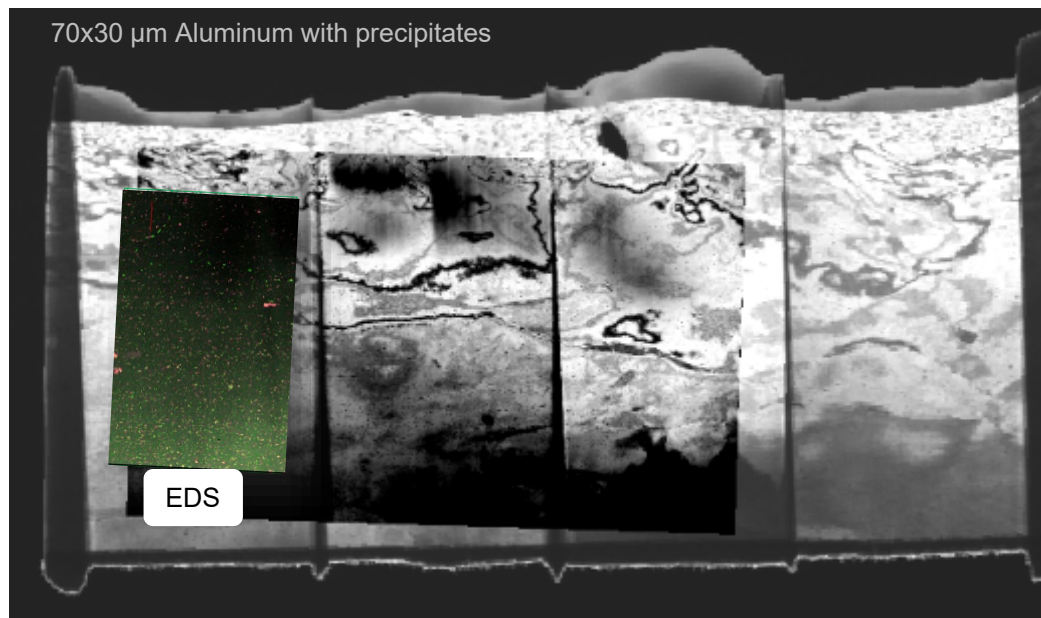


Sample courtesy: University of Manchester / University of Trento

Digital zoom

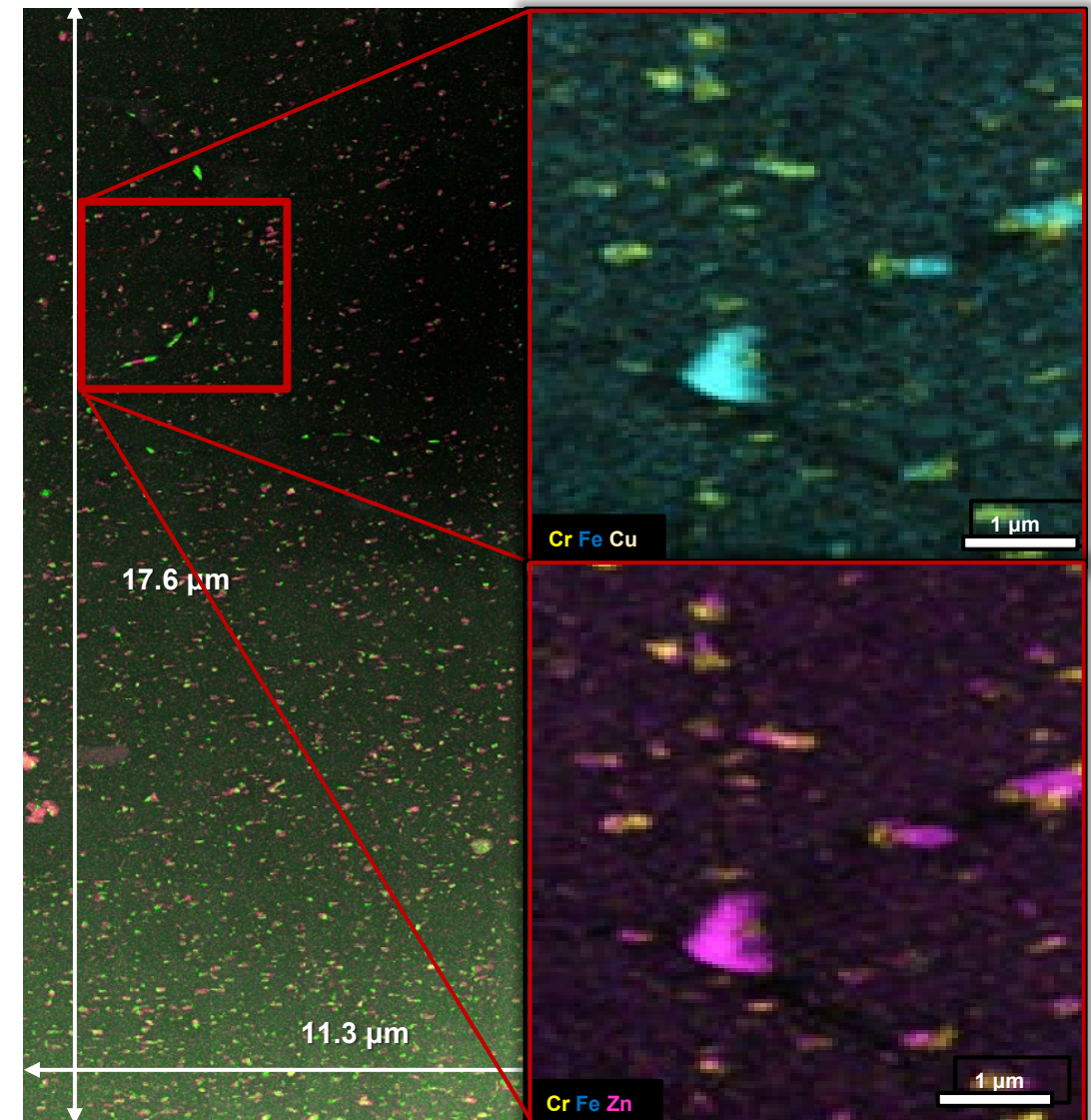


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 - Automated large area EDS at high resolution
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Sample courtesy: University of Manchester / University of Trento

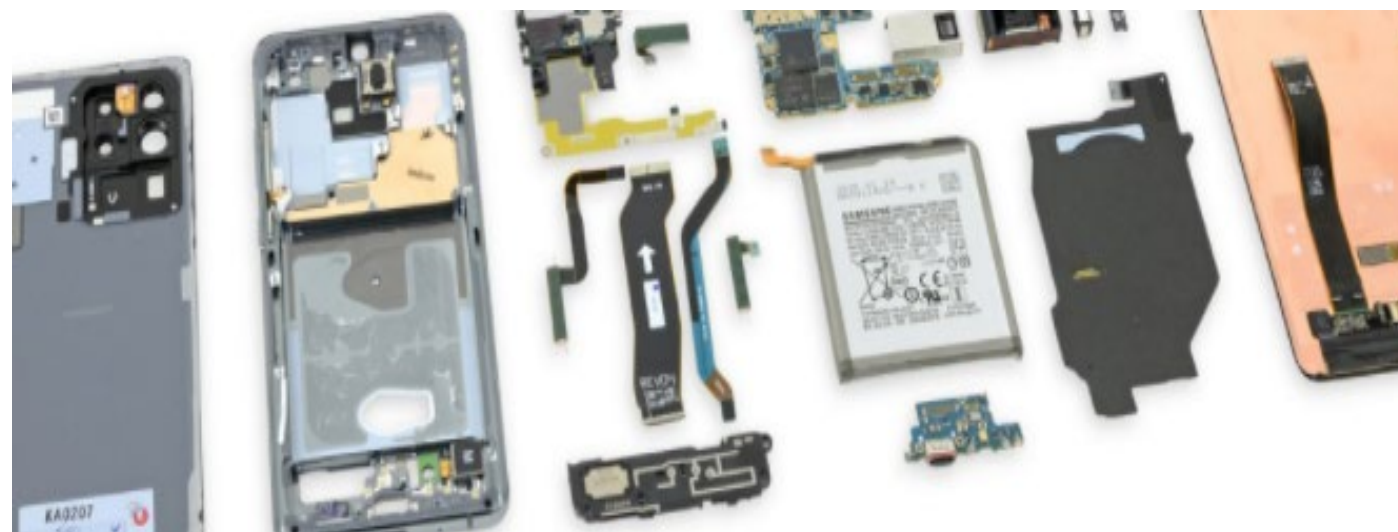
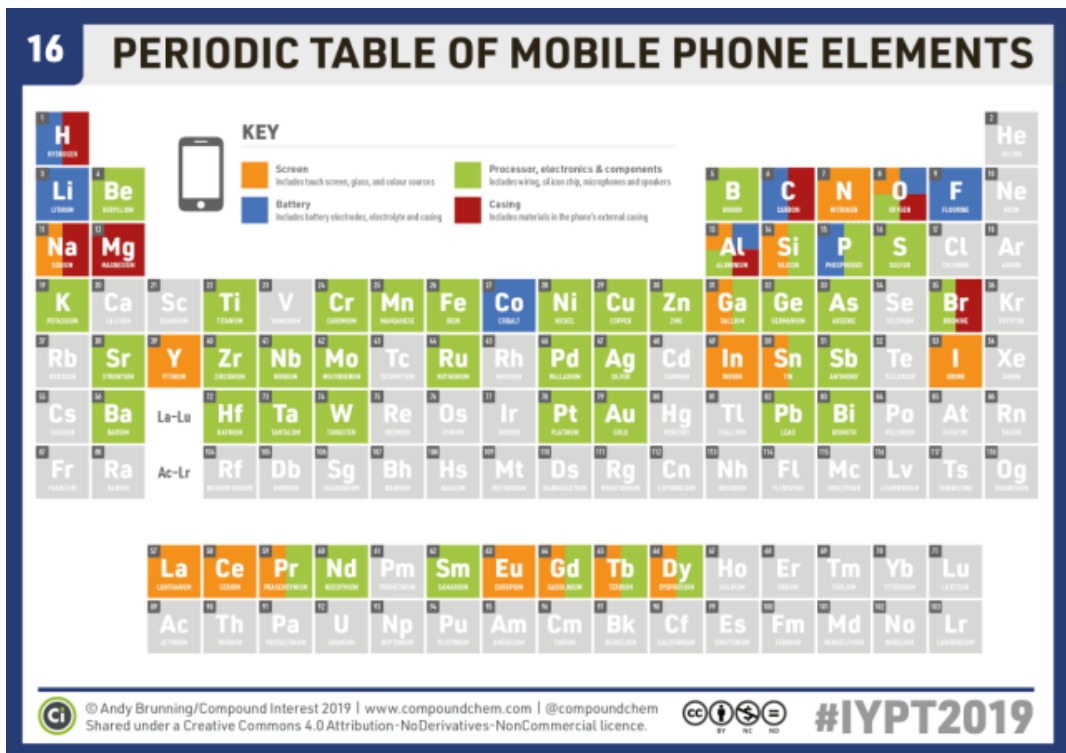
Digital zoom

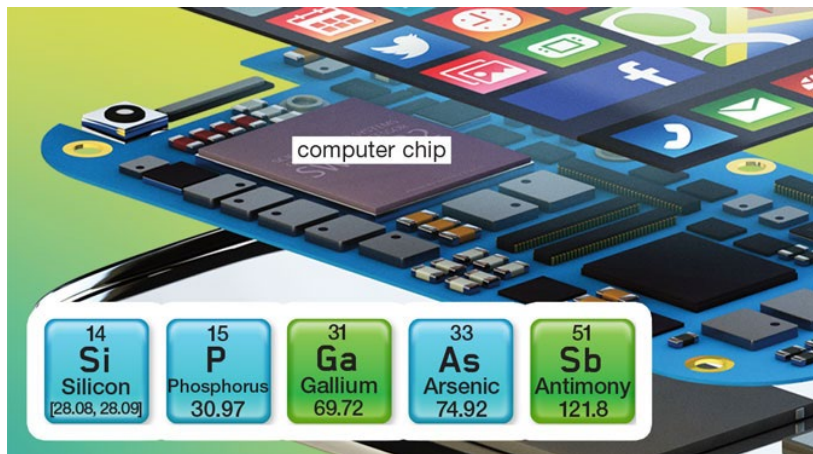


Applications Examples

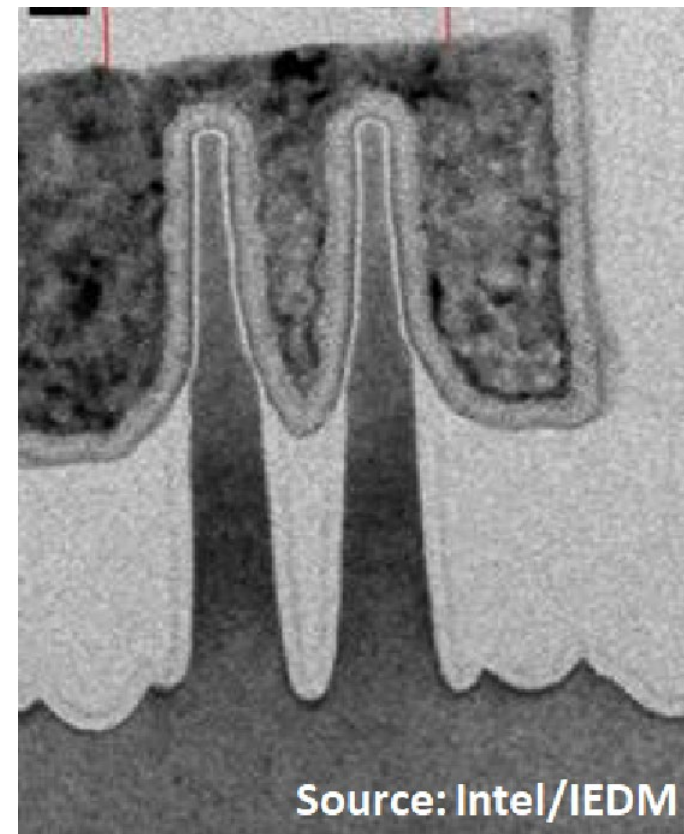
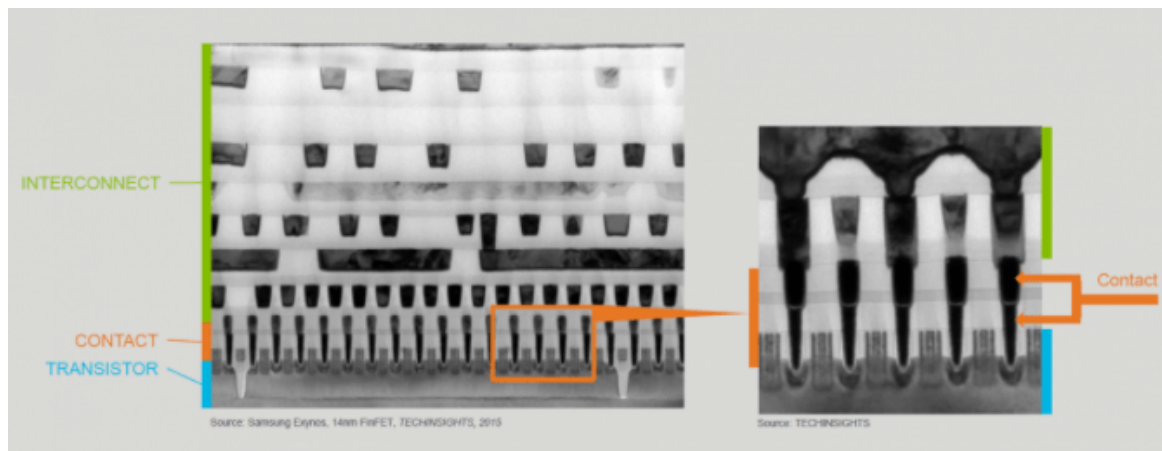
Talos Images

- Over 50 different elements used in modern cell phone





Visualization of the modern logic chips employed by current generation cell phones

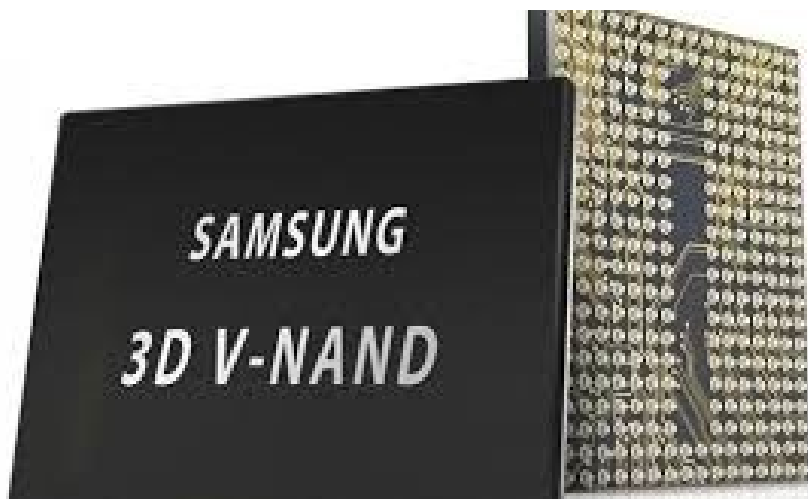


<https://semiengineering.com/finfet-metrology-challenges-grow/>

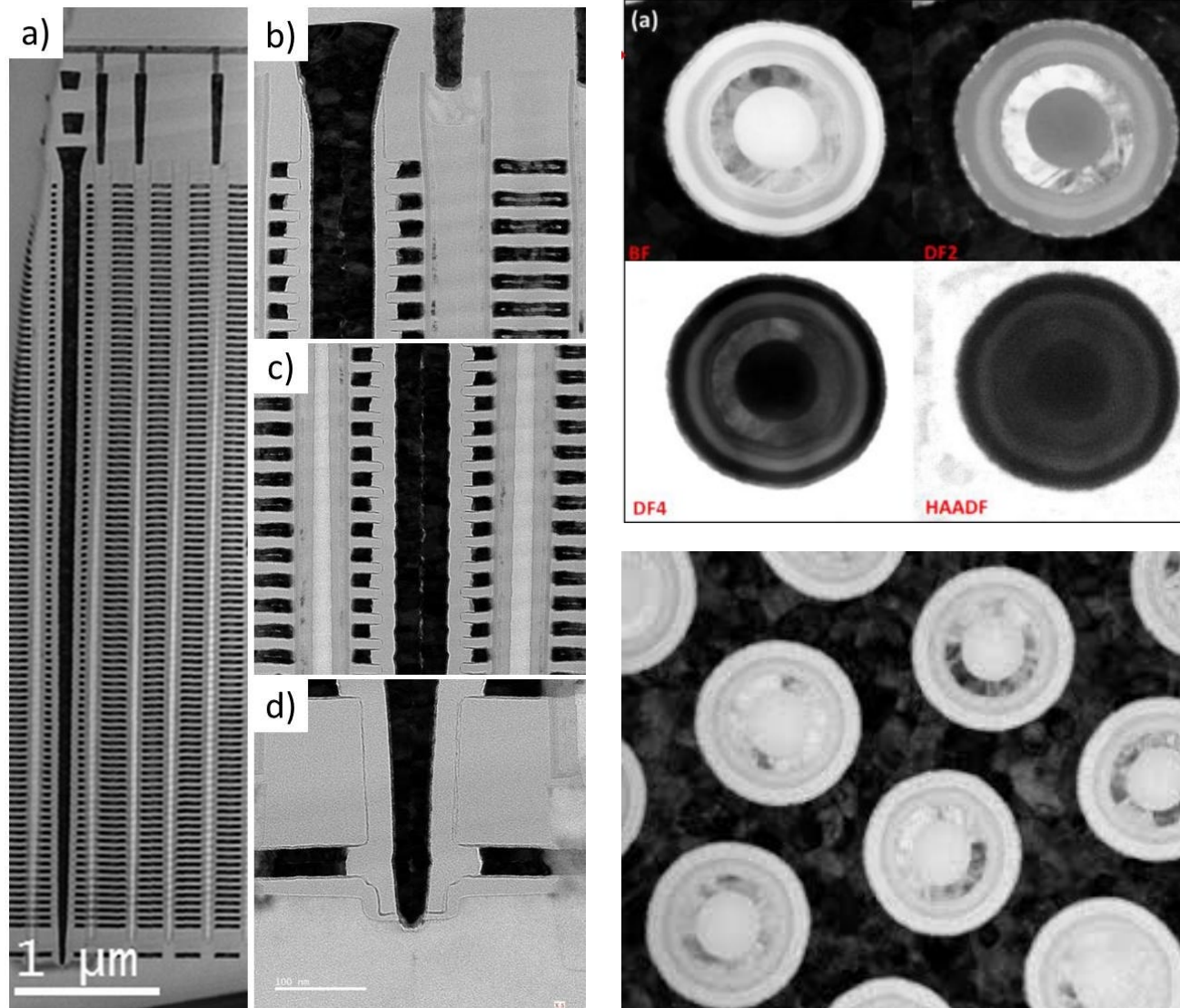
Memory Chip

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- 3D NAND allows for high capacity storage on our mobile devices.
 - Advanced NAND is vertical and hosts a variety of challenges in its design and manufacturing

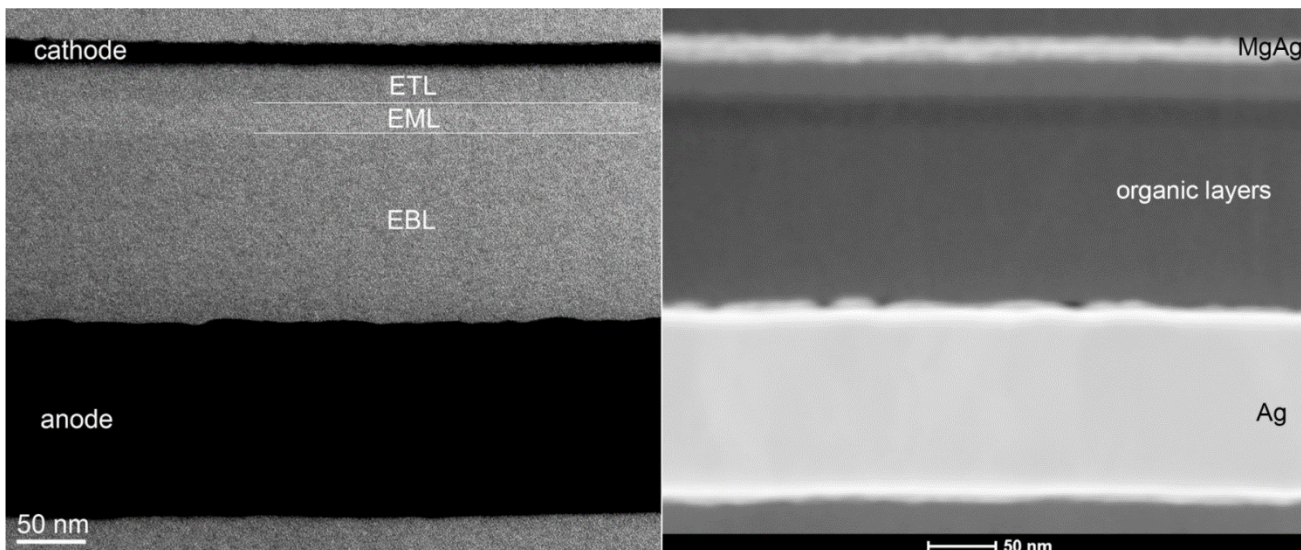
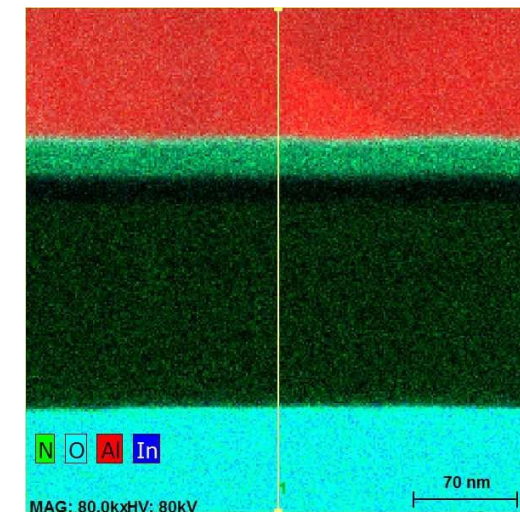
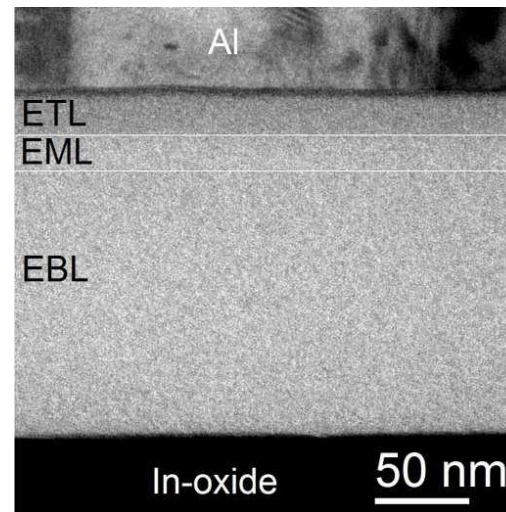


<https://en.msscorgs.com/products/?mode=data&id=131&top=0>

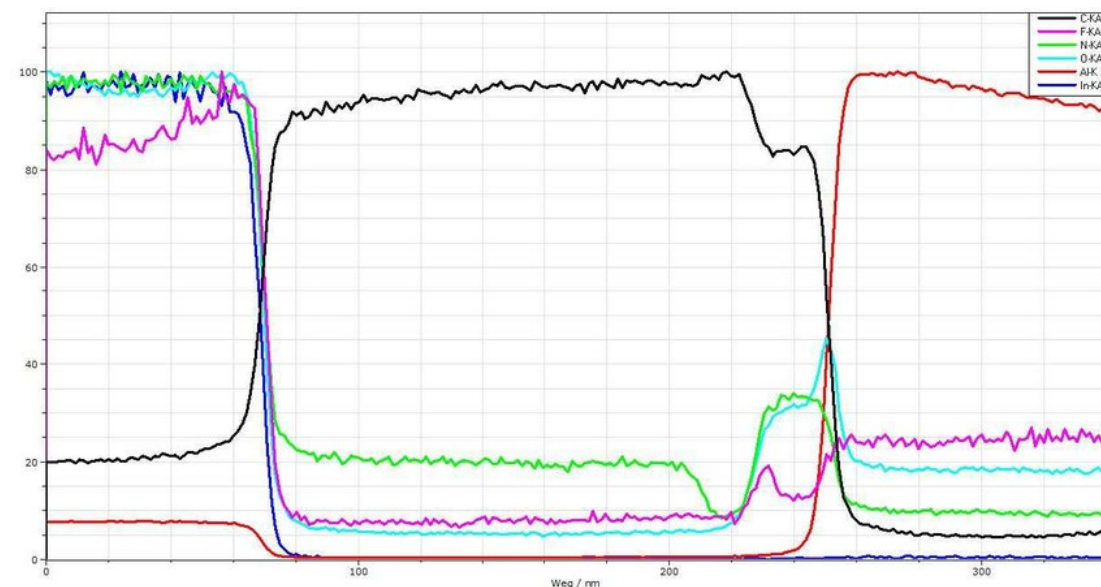




Cell phone Displays provide an ability to review layer composition and understand how EDS can impact measurements



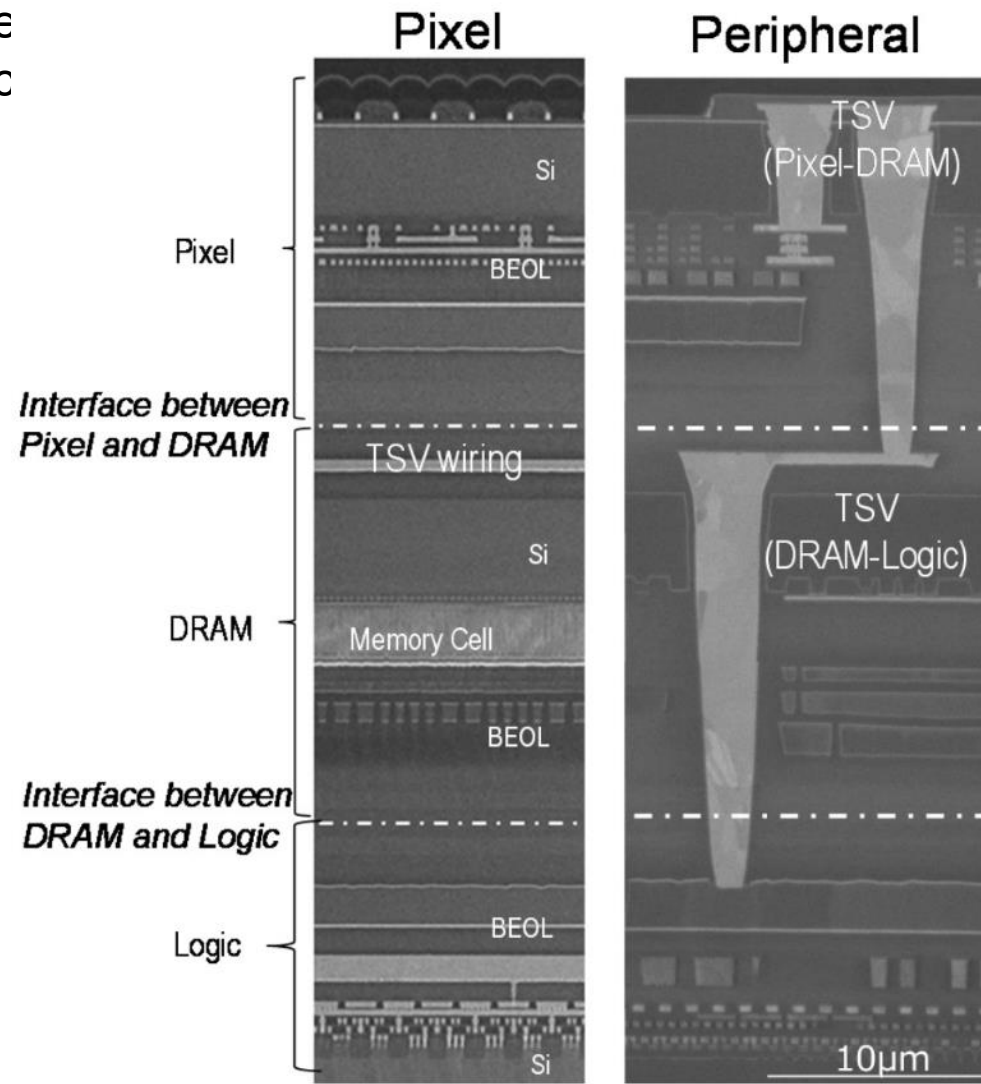
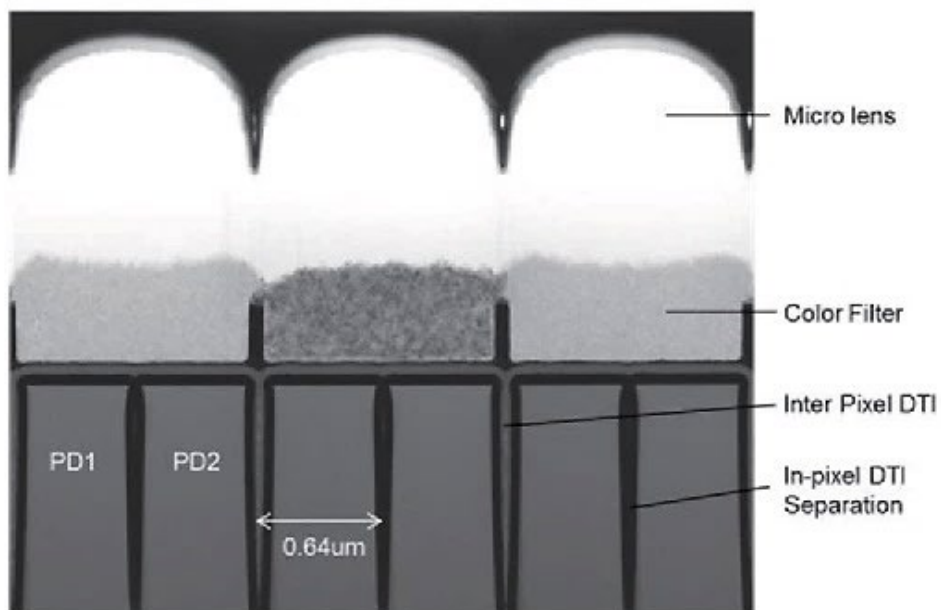
Left: TEM bright field image of the organic layer stack. **Right:** STEM HAADF image of the organic layers. Density variations between the organic layers are visible.



Active Pixel Sensor

47

- The Active Pixel Sensor of the Cell Phone camera allows for ke analysis of a component that bridges from the macro to the mic
- TEM allows for imaging of the
 - Lens
 - CMOS sensor
 - Electronics




- Mainstream of advance techniques
- 4D STEM – EMPAD
 - Collect Diffraction and STEM image at the same time
- Precession
- Tomography
- Cryo for Material science
- Interactive holders (Cryo, heating, probing, ...)
- Low kV
- Integrated Signals
- EF TEM (energy filter)
- DPC

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Q & A Session



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Thank you

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