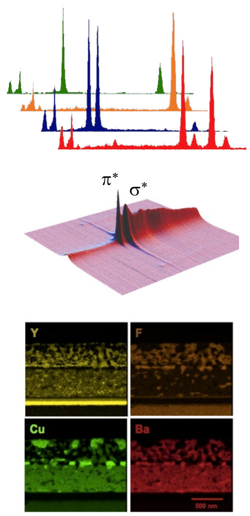
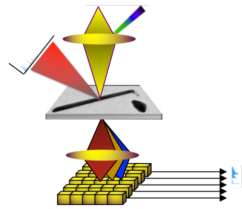
If you can't Detect it – Then you can't Measure it.

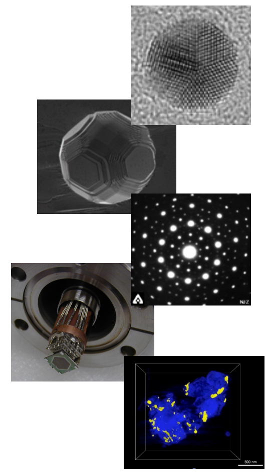




Nestor J. Zaluzec

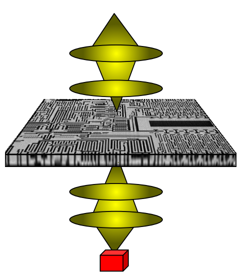
Argonne National Laboratory
Photon Sciences Division

zaluzec@aaem.amc.anl.gov
zaluzec@microscopy.com



1

Types of Microscopes

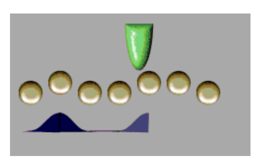


Microscopes with Lenses

Optical Microscopes
Electron Microscopes
Focused Ion Beam Microscopes
X-ray Microscopes

Microscopes without Lenses

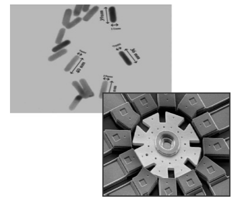
Field Emission Microscopes/Atom Probes
Point Projection Microscopes
Scanning Probe Microscopes



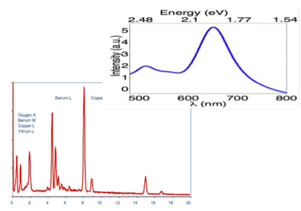
2

2

Charged Particles (e^-, i^+) vs Photons (λ , x)



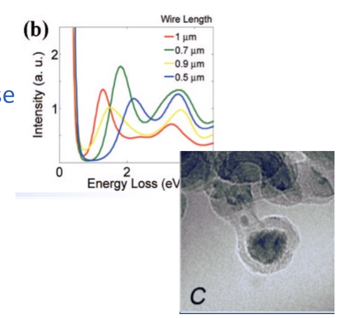
- Charged Particles are good for:
  - High spatial resolution (< 1 nm routine)
  - Probing /visualizing structure
  - Various forms of Microanalysis (in-elastic scattering)
  - Coupling to plasmons (loss function Im{1/ε})
- Photons are good for:
  - In-situ & Atmospheric+ Operation / In-Vivo Studies
  - Various spectroscopies at high spectral (energy) resolution
    - Depending on technique 1 eV -> 1 μeV
    - Microanalysis (X-ray/Optical Spectroscopy)
  - Thick/Bulk specimens (XRD/Synchrotrons)
  - Coupling to electronic excitations/excitons
  - Ultrafast (ps-fs) time resolution
  - $$



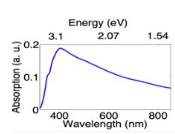
3

Charged Particles (e^-, i^+) vs Photons (λ , x)

- Charged Particles are less desireable because
  - The sample is ubiquitously damaged
  - Time resolution is challenging
  - Usually in Vacuum
    - In-situ possible but more difficult
  - $$



- Photons are less capable at:
  - Very high spatial resolution
    ~λ/2 far-field, 30-200 nm in near field
  - Non-transparent/thick specimens (visible light)
  - Coupling to dark modes
    forbidden excitons, plasmon polaritons



4

X-Ray Microscopy – Capabilities

- Large penetration: 10 μm – mm (W, Si, 20 keV)
  - ⇒ Study of buried layers and complex devices w/o thinning.
- Use of external fields (magnetic, electric).
- No charging effects.
- Good spatial resolution – (but far from fundamental limits)
  - Soft X-ray Microscopy: $\delta \sim 15$ nm
  - Future x-ray optics: $\delta < 10$ nm
- Contrast Mechanisms:
  - X-ray fluorescence: atto-g sensitivity (30 nm), quantitative
  - Diffraction: sensitivity to crystallographic phase, strain
  - Transmission imaging in phase/absorption contrast
- Good working distance (>> mm), depth of focus (> um) (E = 10 keV, dr_N = 10 nm)

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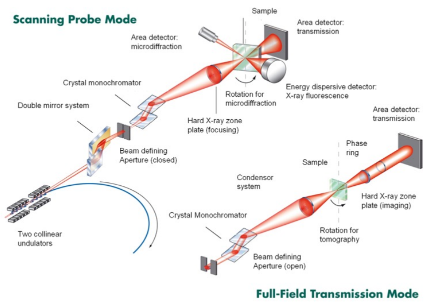
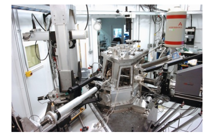
5

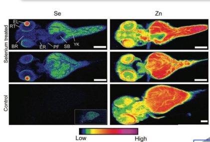
Photon Detectors are Ubiquitous in (IR-VIS-NIR) Light Microscopy

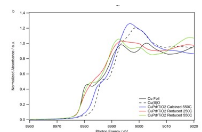
6

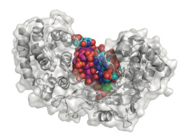
X-ray (Photon) Microscopy & Microanalysis

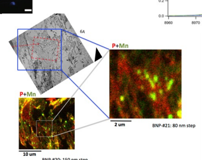


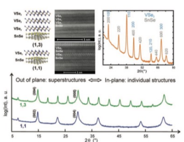


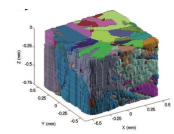








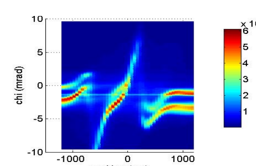




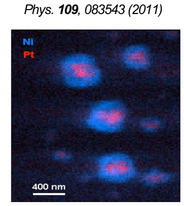
7

Hard X-ray Microscopy Capabilities at ANL/APS

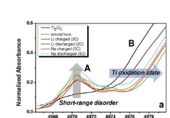
- **Current high-resolution capabilities at APS:**
  - CNM/APS Hard X-ray Nanoprobe, sector 26
    - 40 nm resolution in x-ray fluorescence, 30 nm resolution in transmission
    - Diffraction, fluorescence, tomography
  - APS Bio Nanoprobe, sector 21
    - 30 nm resolution, x-ray fluorescence
    - Cryogenic operation, cryogenic specimen transfer
  - Transmission X-ray microscope, sector 32
    - 30 nm resolution, transmission tomography
    - In-situ capabilities
- **In-Situ Nanoprobe Beamline (APS Upgrade)**
  - Science Thrust: Photovoltaics, Energy Storage, Nanoscale Electronics, Catalysis
  - 20 nm – 50 nm resolution at 10 – 1000x increased flux
  - *In-situ* fluorescence spectroscopy, tomography
  - Sensitivity to << 100 atoms in buried structures/interfaces
  - Coherent scattering to map defects at < 10 nm.



C. Murray et al. Journ. Appl. Phys. ***109****, 083543 (2011)*



Courtesy H. C. Kang

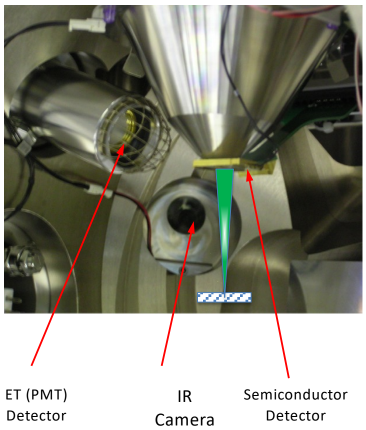


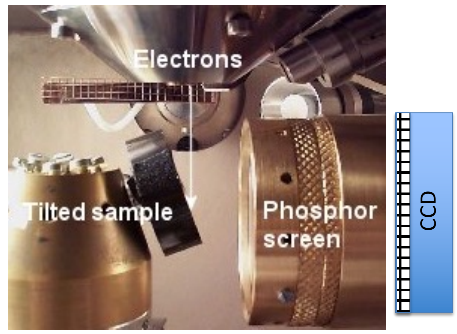
H Xiong et al., J. Phys. Chem. Lett., 2, 2560-2565 (2011)

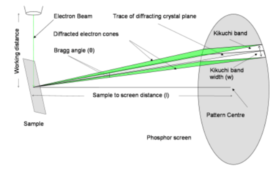
Maser, ICXOM 2013

8

Imaging Detectors in Charged Particle Optical Systems



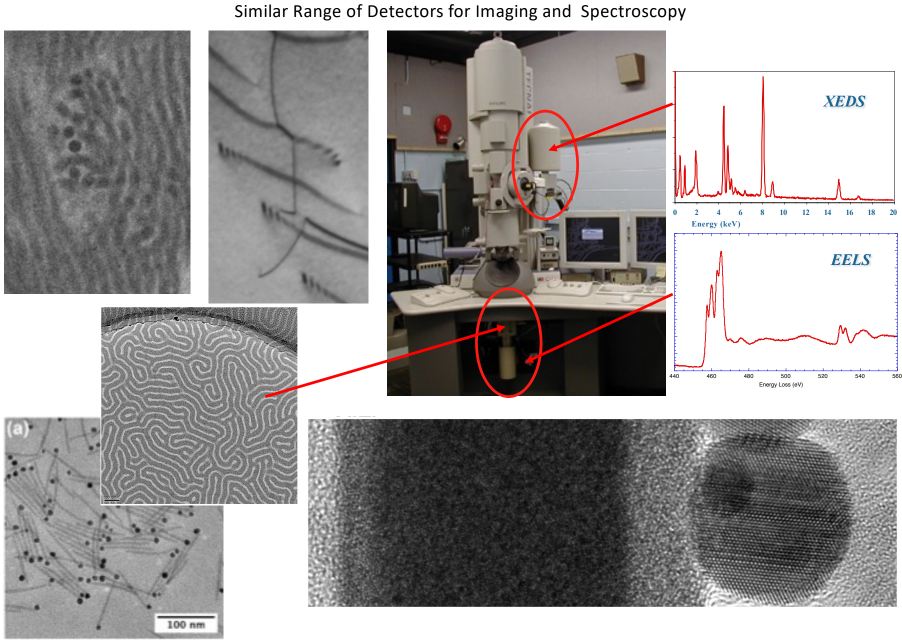




9

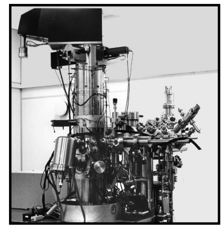
Transmission Electron Microscopy

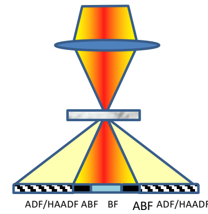
Similar Range of Detectors for Imaging and Spectroscopy

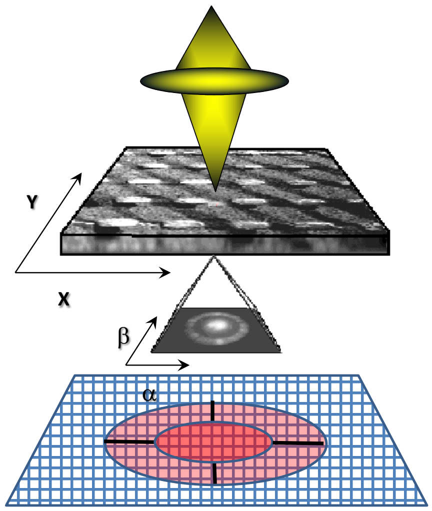


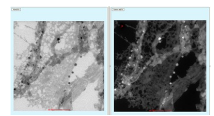
10

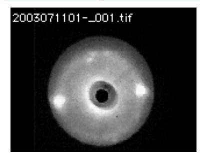
Scanning Transmission Microscopy & Diffraction







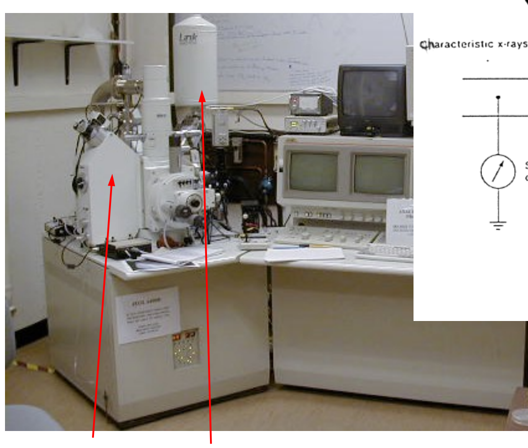




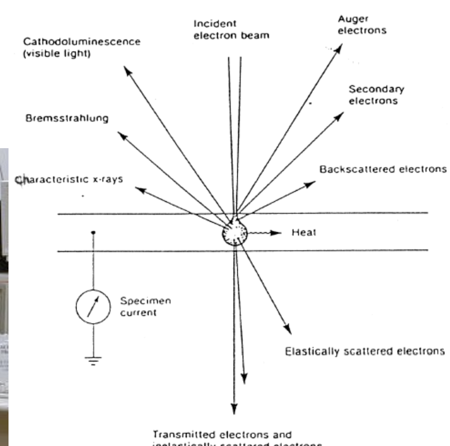
Array Detectors
vs
Single or
Segmented Detector

11

X-ray Detectors for Electron Beam Microanalysis

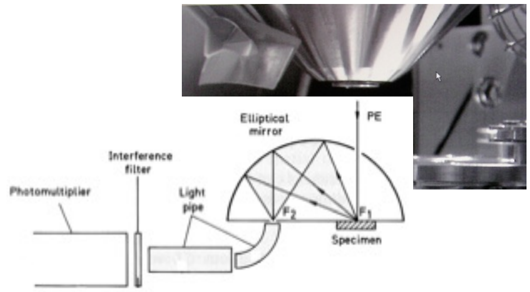


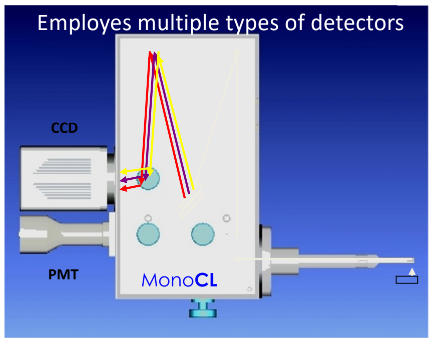
WDS EDS

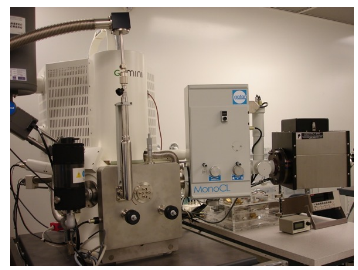


12

Cathodoluminescence Instrumentation In Electron Microscopy

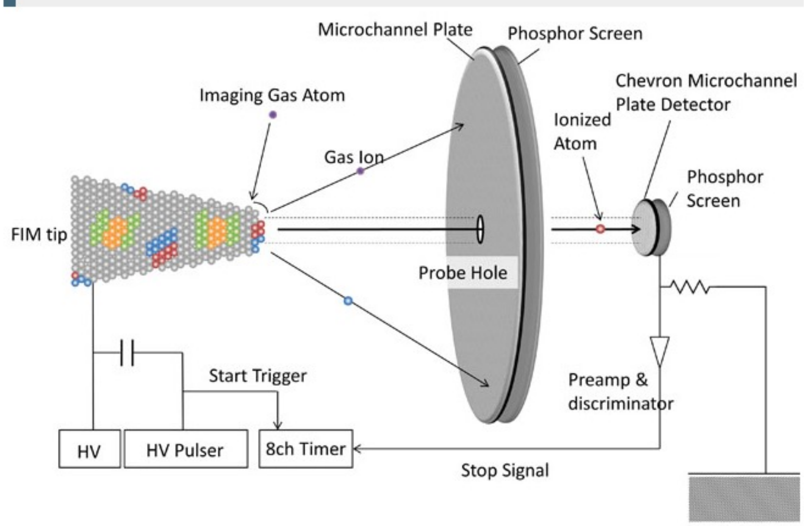






13

Atom Probe Field Ion Microscope



14

Detectors

- Vacuum/Ionization devices: - Traditional Technology
  - o Photomultiplier Tubes (PMT)
  - o MicroChannel Plate (MCP)
  - o Hybrid Detectors (HD)
  - o Ion Detectors (ID)
  - o Ionization Chambers (IC)

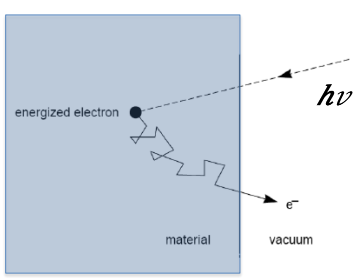
- Solid State devices: Rapidly Evolving Technology
  - o PhotoDiodes (PD)
  - o Silicon Photomultipliers (SiPM)
  - o Avalanche Photo Diode : APD
  - o Geiger Mode APD : Arrays of SPAD: SiPM / MPPC …
  - o Imaging devices: CCD and sCMOS, EMCCD
  - o X-ray Detectors: Si(Li), SDD….

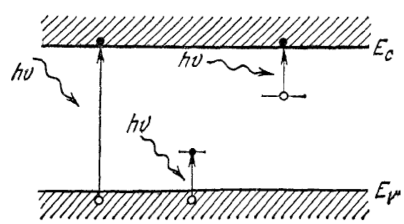
15

The photoelectric effect is generally responsible for the Signal (*hv*) detection by creating a measurable signal

Two types:

1. External: the Photo-electron is emitted into the vacuum from a photocathode material.
2. Internal: the Photo-electron is excited and occupies the conduction band of the semiconductor material, the photoconductive effect





External
PhotoElectron
escapes the material

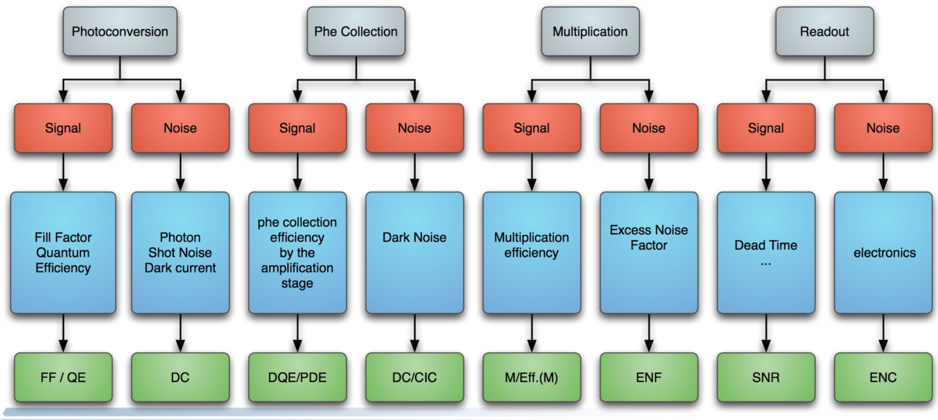
Internal
PhotoElectron
in the Conduction band

16

The 4 generic steps in the Detection process:

1. The initial charge carrier (pe, e/h) is produced by the Signal ($h\nu$)
2. The primary charge carrier is collected
3. The primary charge carrier is multiplied/amplified or not (CMOS/CCD/PD/SDD/Si-Li)
4. The secondary (or primary) charges are collected and read out

The measurement process is modified by noise sources and by signal collection inefficiency at each step:



Adapted from R. Barbie – Univ. Of Lyon

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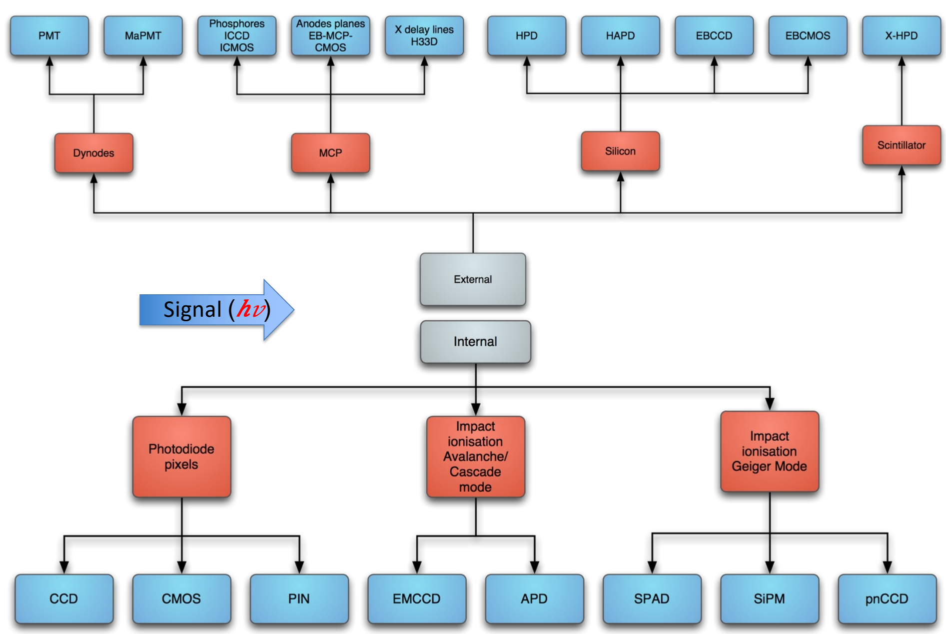


Adapted from R. Barbie – Univ. Of Lyon

18

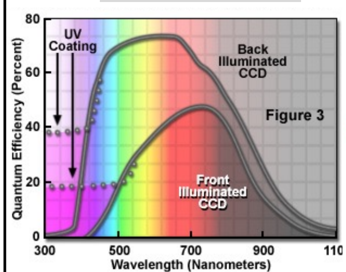
Detector Quantum Efficiency

Detector Quantum Efficiency (DQE): probability that a single event triggers a detectable output signal

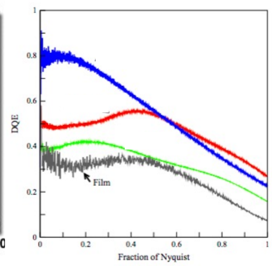
$$DQE = \frac{Output\ Signal}{Input\ Signal} \leq 1$$

Different Detectors have Different DQE's

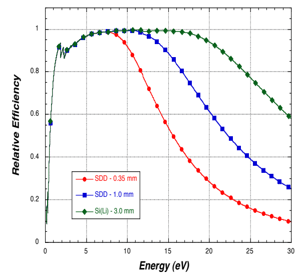
Visible Photons



High Energy Electrons



X-rays



DQE of a "Detector" ≠ DQE of the "Signal Processing Chain"

19

The choice of Detector should be made in light of the question:

What do you want to measure?

Choose your hierarchy of priorities:

- Counting ? Linearity ? Single Photon sensitivity ?
- Localize ? Imaging ? Large Field of View ? Detection surface ?
- dynamic range ?
- temporal resolution? dead time ?
- …

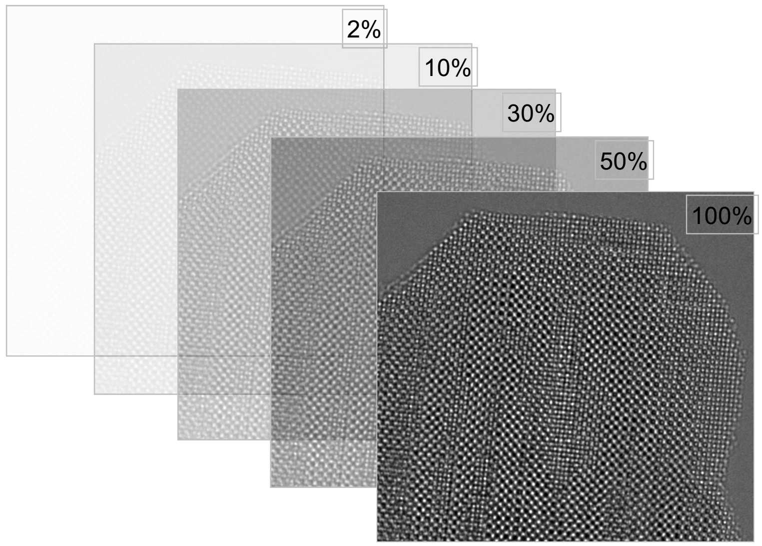
Different detectors exist and the "best" for the application is not always obvious.

The detection technologies are evolving rapidly.

Our speakers will be addressing the range typical to Microscopy/Microanalysis

20

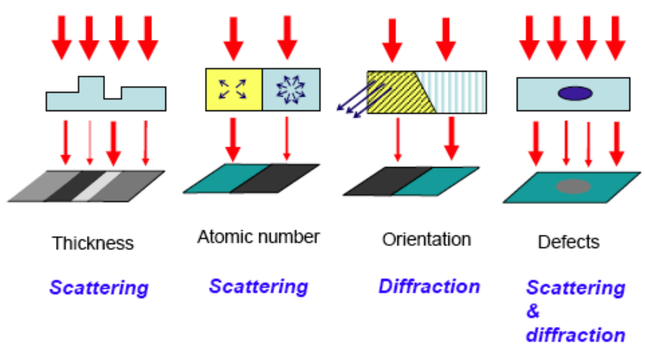
What is the limit ? Signal vs Contrast



$$Intensity = \Gamma_{scatteringprobability} \bullet N_{atoms} \bullet \eta_{electrons} \bullet \tau_{time} \bullet \Omega_{efficiency}$$

21

Factors Affecting Image Contrast (Physics vs Technology)



22

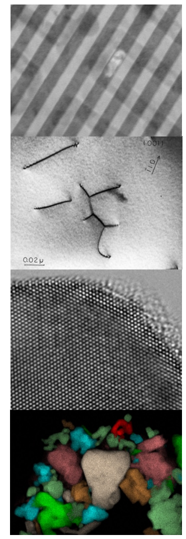
Contrast Mechanisms

Elastic

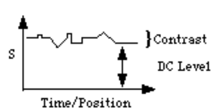
- Amplitude (Mass/Thickness) Contrast
  - Varying mass of the specimen attenuates the beam
- Diffraction Contrast
  - Scattered beams are removed or signal from scattered events is used
- Phase (Interference) Contrast
  - Scattered beams constructively or destructively intereact with each other

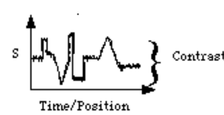
Inelastic

- Signal derived from probe changing energy/momentum in the specimen

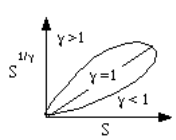


23



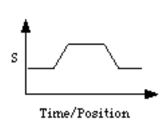


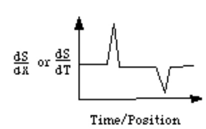
Black Level Suppression



γ=1 Linear amplification of all Signals
γ < 1 Preferentially Enhances Low Signal regions
γ > 1 Preferentially Enhances Dark Signal regions

Non-Linear Amplification





Differentiation

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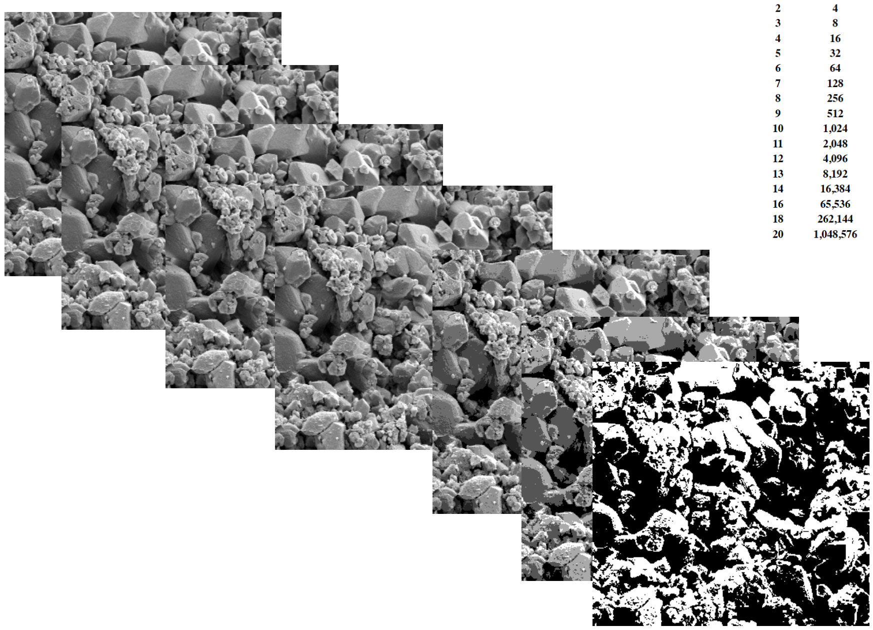
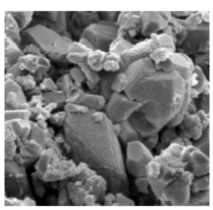
Bit Depth

Bit Depth	Grayscale Levels
1	2
2	4
3	8
4	16
5	32
6	64
7	128
8	256
9	512
10	1,024
11	2,048
12	4,096
13	8,192
14	16,384
16	65,536
18	262,144
20	1,048,576

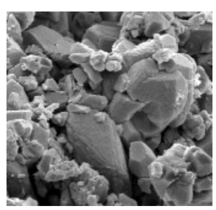


25

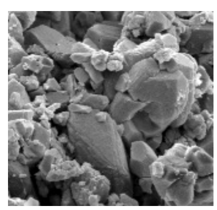
8 Bits (0-255)



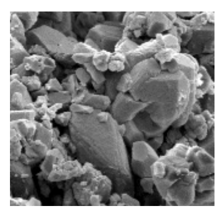
7 Bits (0-127)



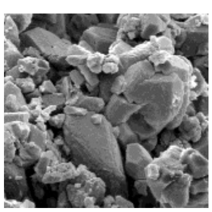
6 Bits (0-63)



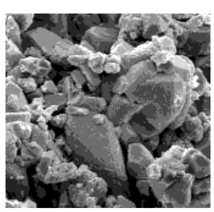
5 Bits (0-31)



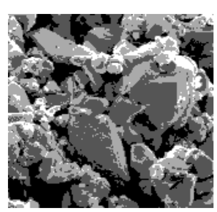
4 Bits (0-15)



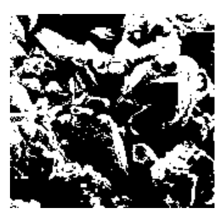
3 Bits (0-7)



2 Bits (0-3)



1 Bit (0-1)



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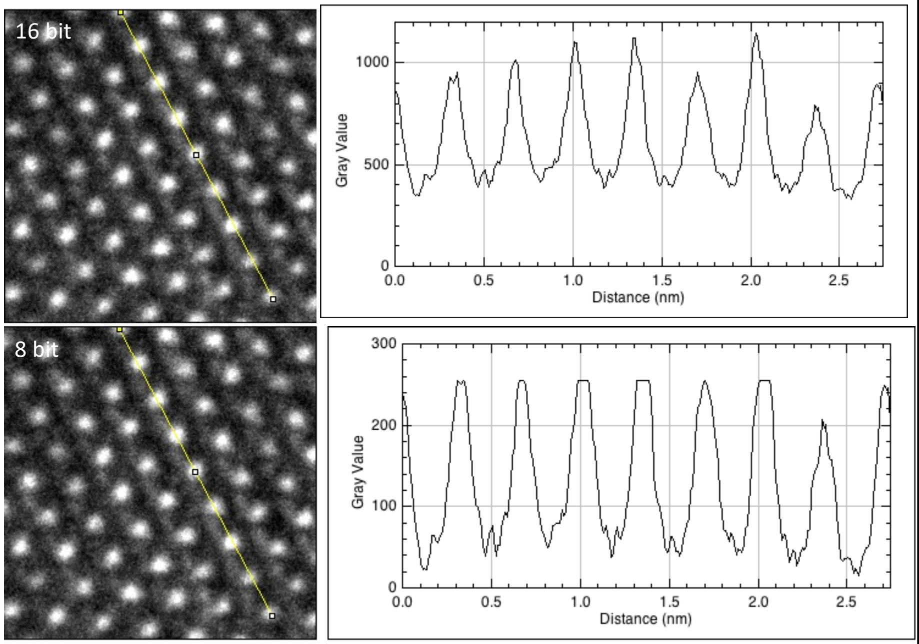
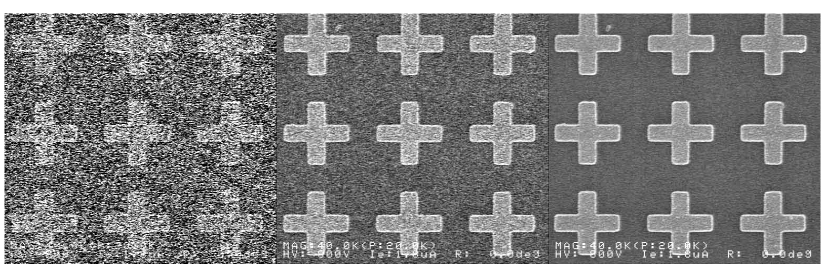


27

Signal-to-noise Ratio

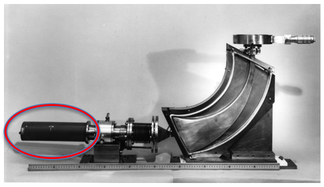


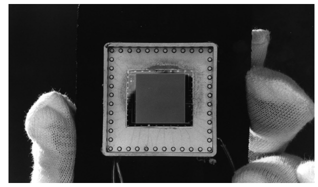
2 frames 8 frames 32 frames

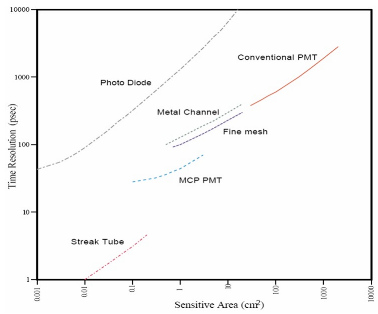
Collect data with adequate signal-to-noise ratio through image frame summation and/or by proper dwell time (pixel or frame time) setting.

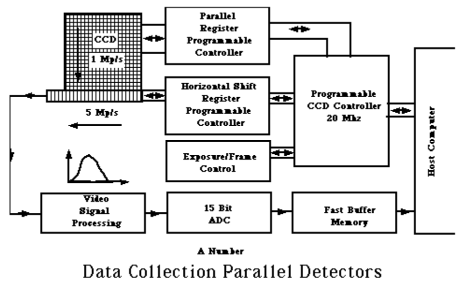
28

Temporal Resolution









Single Event Detectors
counts/picosecond

Array Detectors
frames / sec

29

Part II

30