Developments in detector technology for XRD

Roger Durst

Bruker AXS Inc.

Detector technology for enhanced speed and accuracy

- ***Energy discriminating strip detectors***

- ***2D detectors***

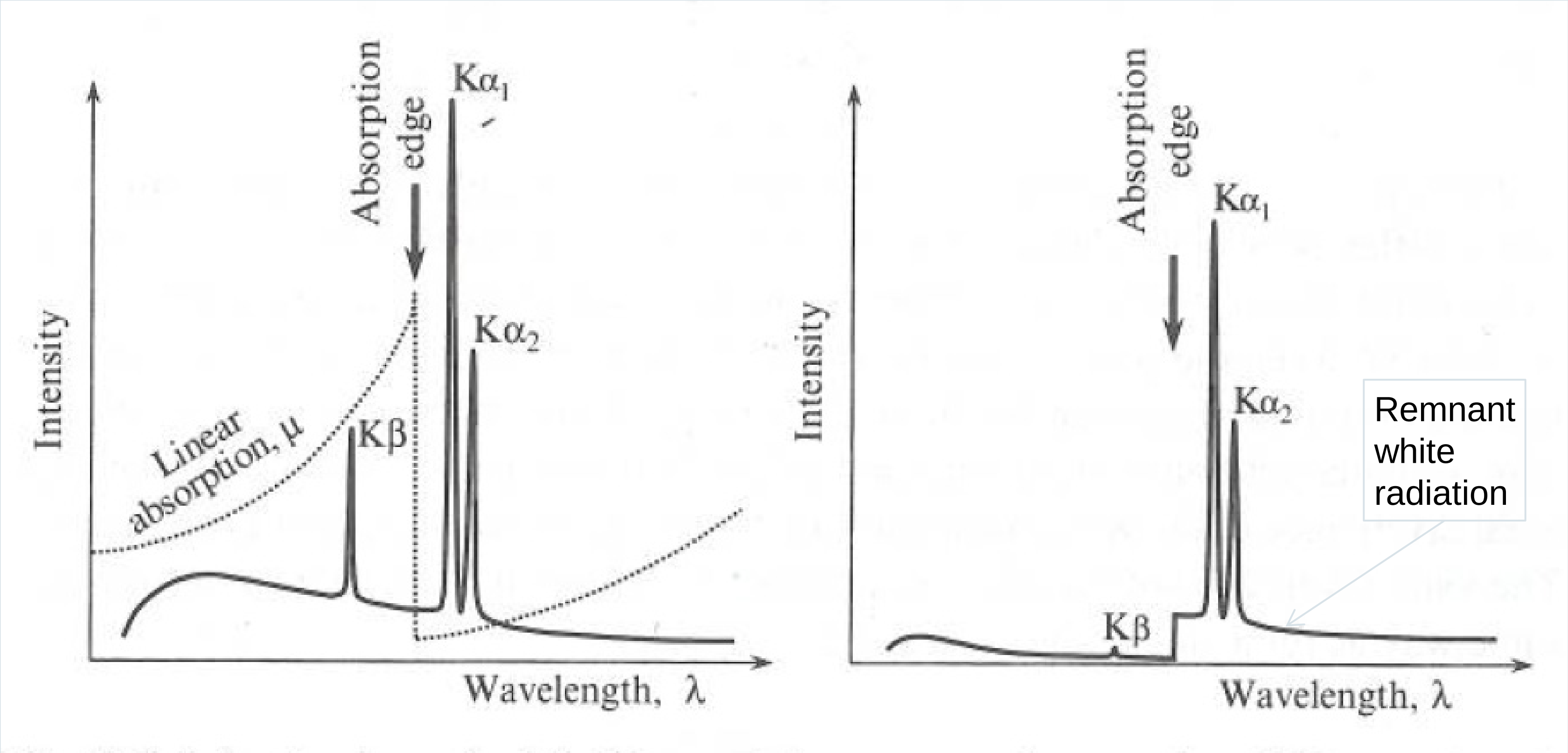
Monochromatization and energy dispersive detectors

Monochromatization

- Before 2000, >90% of all instruments have been equipped with point detectors, > 50% with (secondary) monochromators
  - Intensity loss ~80-90% with respect to unfiltered rad.
- Today, >90% of all instruments are equipped with PSDs, the majority with Kß-Filters
  - Intensity loss ~40-60% with respect to unfiltered radiation
  - Absorption edges
  - Poor filtering of fluorescense

- ***An energy dispesive detector could in principle improve effective sensitivity by 2-10 times and reduce errors due to absorption edges and fluorescence***

Monochromatization
Artifacts from Kß-Filter

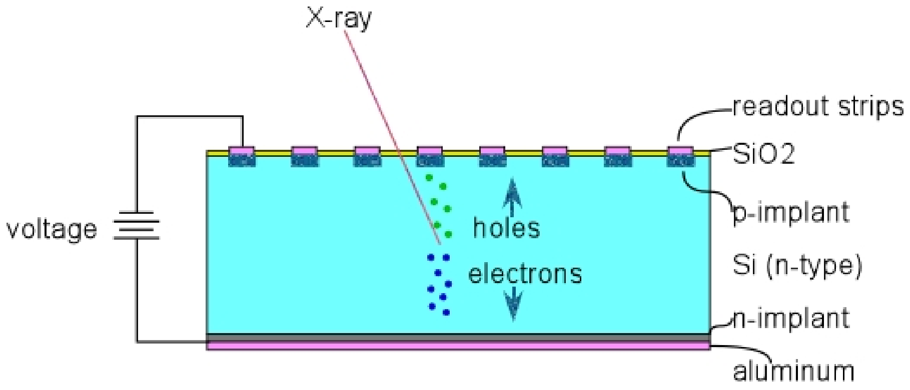


Remnant white radiation

Pecharsky & Zavalij (2009)

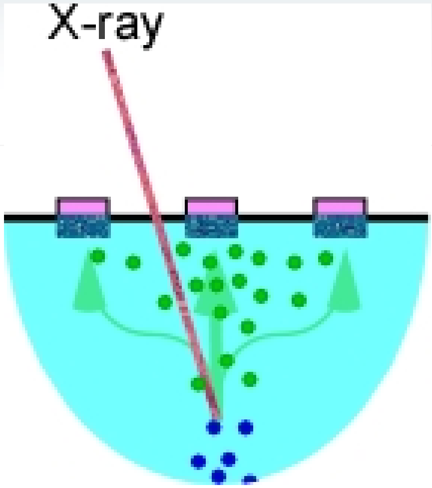
Silicon strip detector principle

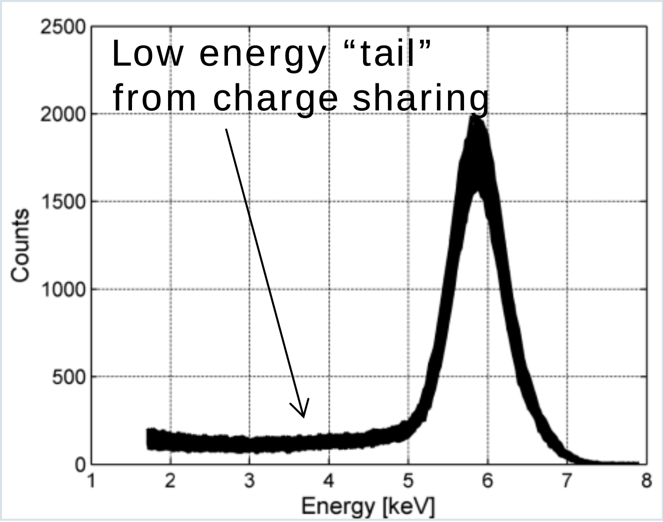
- Electron-hole pairs created in depleted silicon by X-ray photoionization
  - Charge carriers drift to readout strips
- Key advantages
  - High counting rate
    - Typically of order 10^6 counts/strip-sec
  - Good spatial resolution
  - Good energy resolution than other detectors
    - Requires optimized readout



Limitations of energy resolution due to charge sharing effects

- Energy resolution is accomplished by “counting” the electrons in a strip or pixel
  - $\Delta E \geq 2.35\sqrt{\frac{F}{N}}$
  - F=Fano factor, N=# of electron-hole pairs
- Problem, ***not all electrons are collected in a given strip (or pixel)***, in general some electrons will diffuse to neighboring strips or pixels: “charge sharing”
  - Fall below discriminator level and are thus “lost”
  - Lost electrons cause poor energy resolution

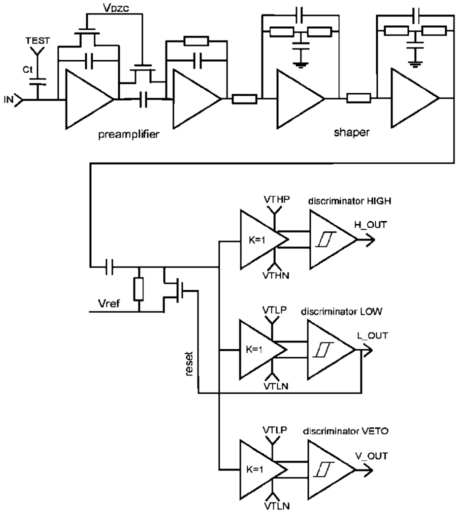



Low energy “tail” from charge sharing

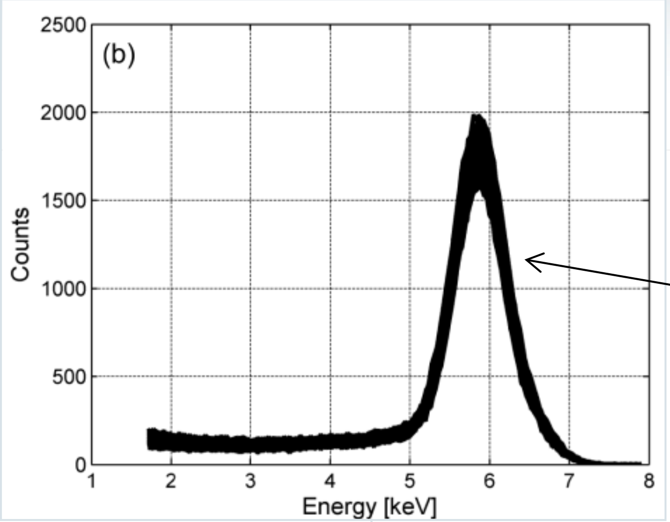
LYNXEYE XE

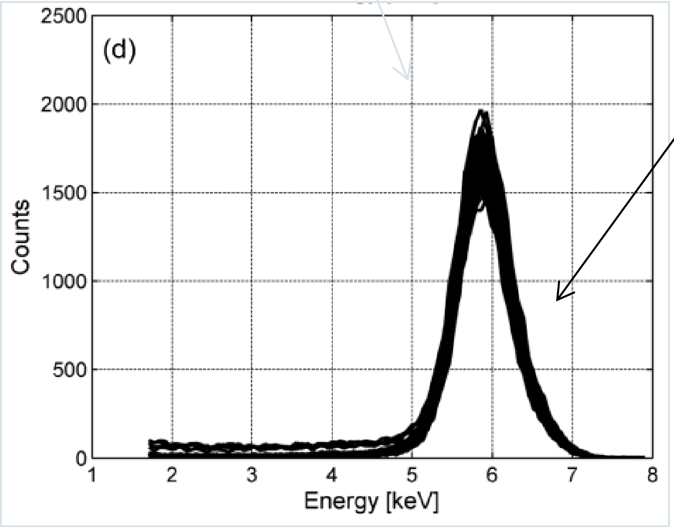
- The LYNXEYE XE is the first **energy dispersive Si strip detector** for home-laboratory X-ray diffraction
  - Inter-strip logic to correct for charge sharing

Detector type	Compound silicon strip
# strips	192 strips, 75 mm pitch
Active area	14.4 x 16mm
Modes	1D and 0D
Wavelengths	Cr, Co, Cu, Mo, and Ag
Energy resolution (Cu)	< 680 eV @ 8 KeV



LYNXEYE XE
Energy Resolution with inter-strip logic



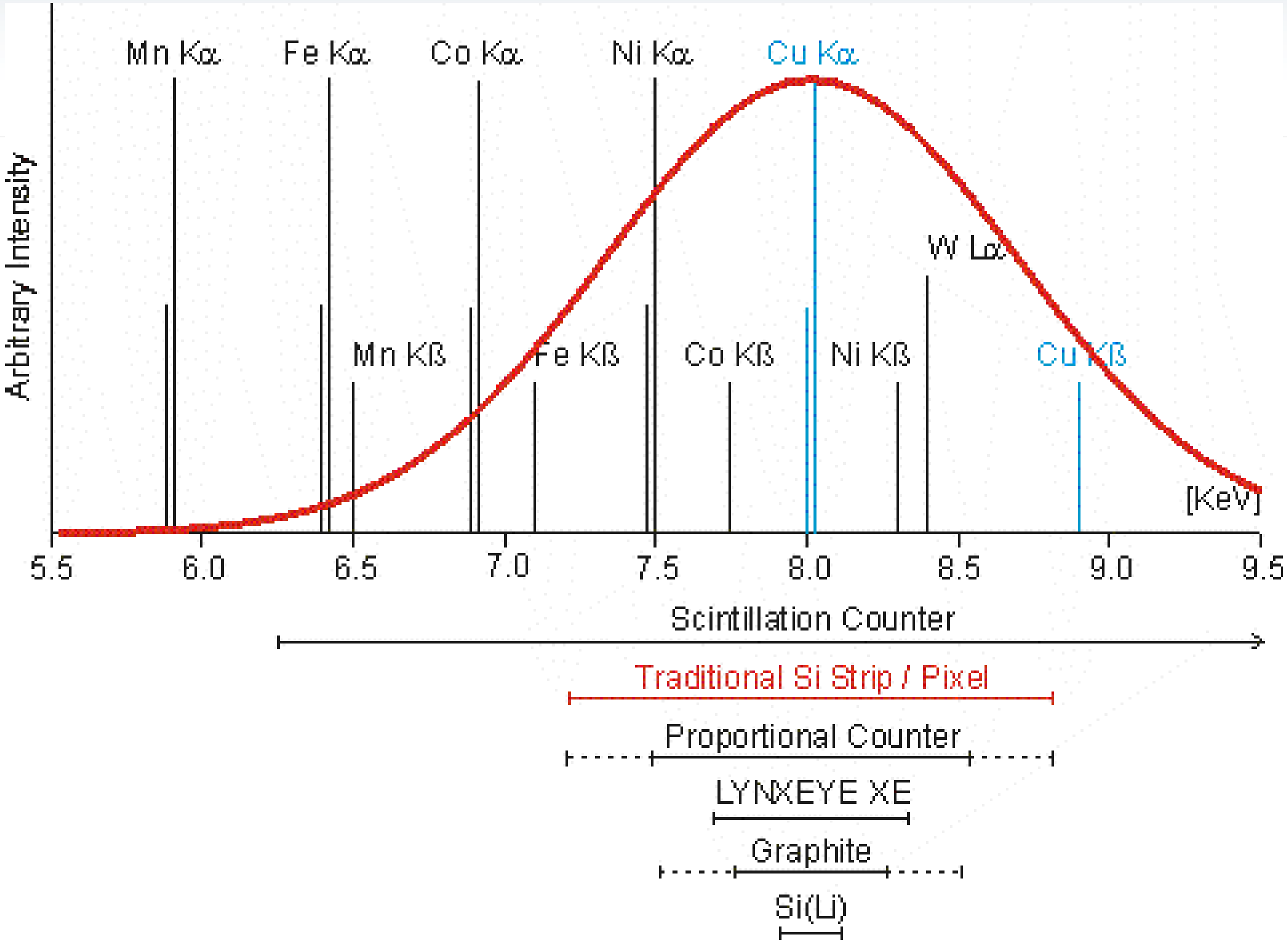


Device	**Resolution (KeV)**
Scintillation counter	~3.5
Traditional silicon strip / pixel	~1.6 - 2.0
Proportional counter	~1.1 - 1.6
LYNXEYE XE	<0.68
Graphite monochromator	~0.5 - 1.0
Si(Li) detector (Peltier cooled)	<0.2

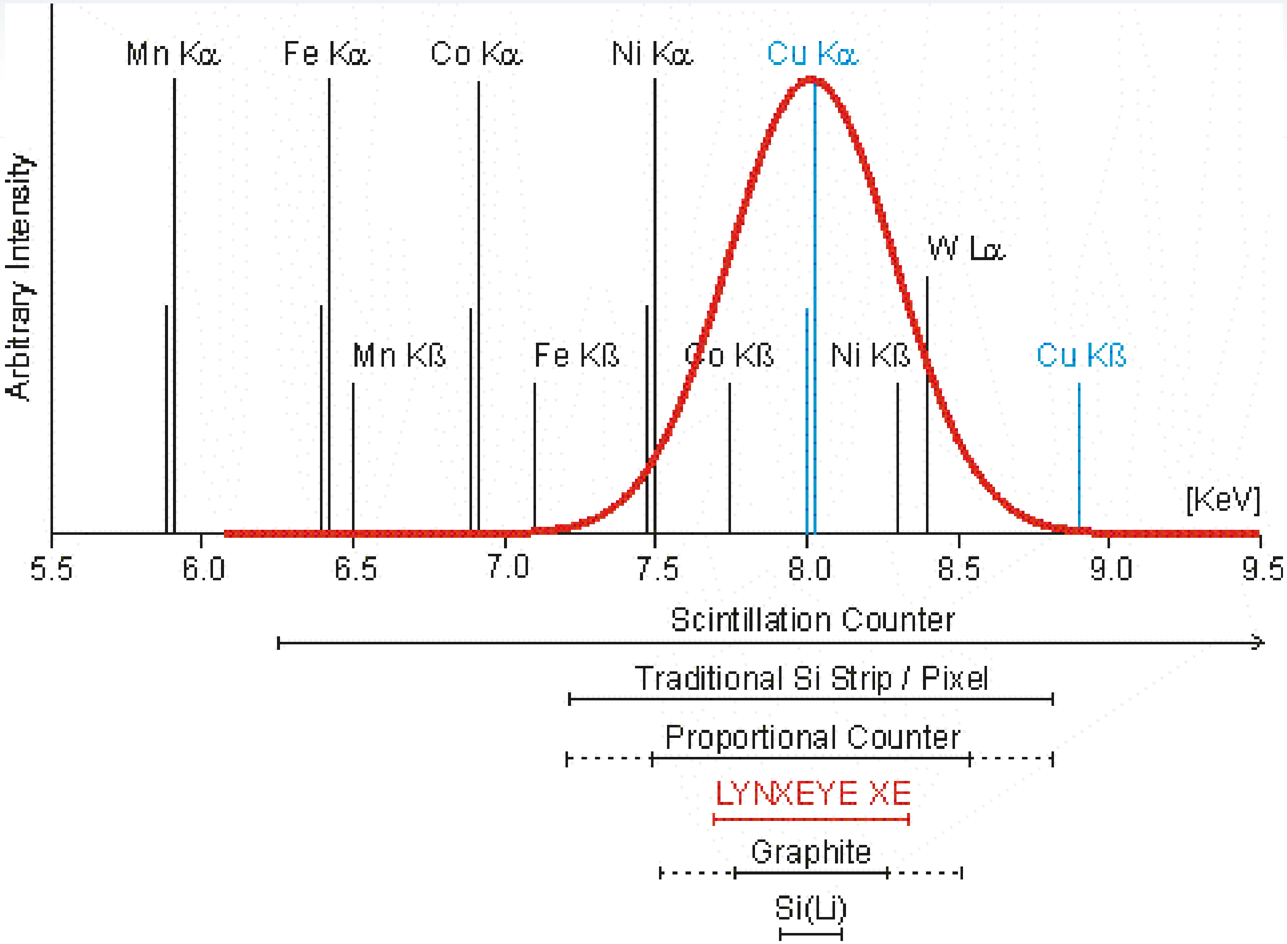
Incorporation in inter-strip logic improves energy resolution 2-3 times compared to conventional Si detector

Comparable to Graphite monochromator

Monochromatization
Traditional Si Strip

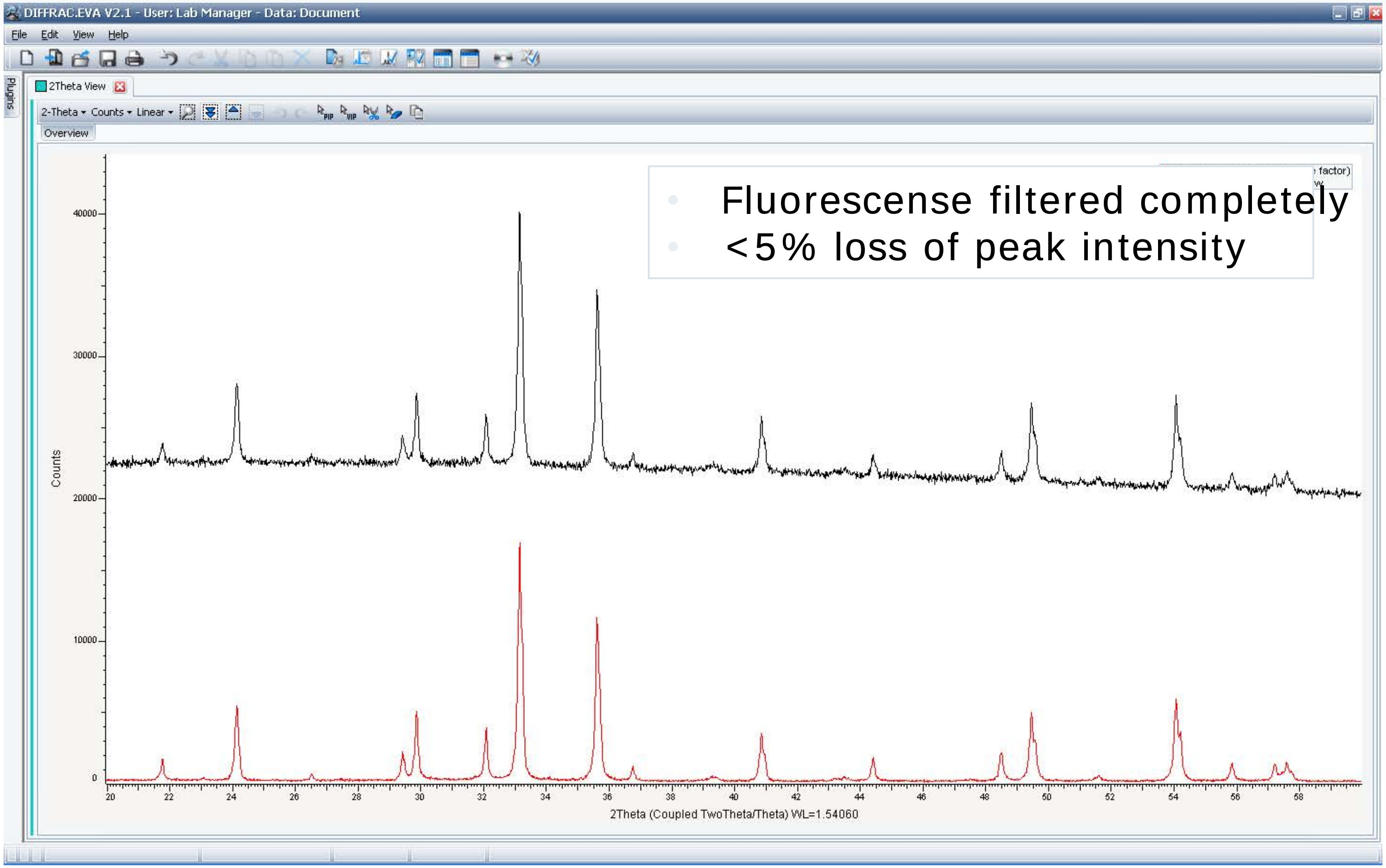


Monochromatization
LYNXEYE XE



Filtering of fluorescense radiation (Cu)

LYNXEYE XE
Filtering Fe fluoresecence: Iron Ore



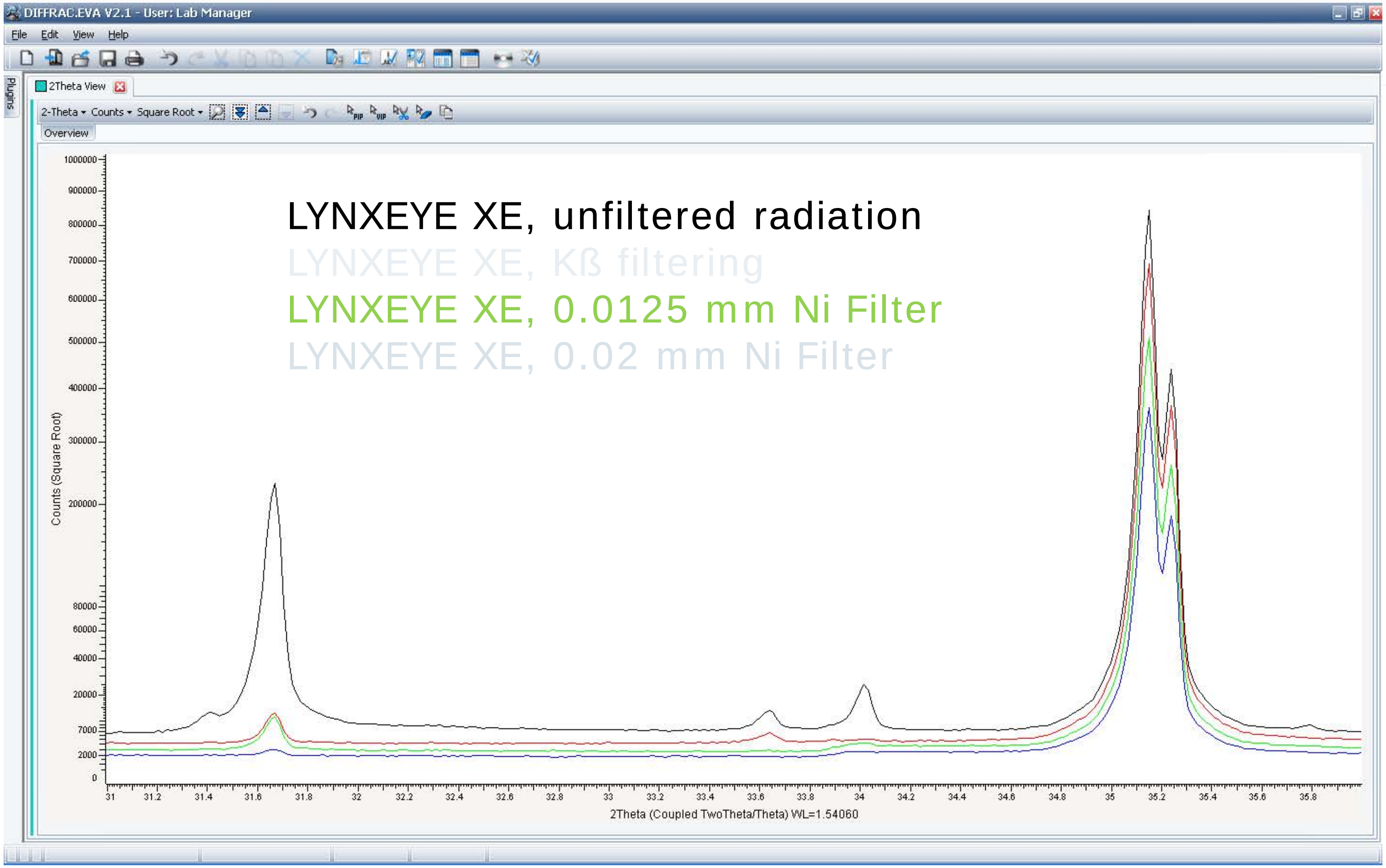
- Fluorescense filtered completely
- <5% loss of peak intensity

Filtering of Fluorescense Radiation (Cu)

- **Manganese:**
  - Fluorescense filtered completely at <5% loss of peak intensity
- **Iron:**
  - Fluorescense filtered completely at <5% loss of peak intensity
- **Cobalt:**
  - > 90% Fluorescense filtered at <5% loss of peak intensity
  - > 98% Fluorescense filtered at <25% loss of peak intensity
- **Nickel:**
  - > 50% Fluorescense filtered at <5% loss of peak intensity
  - > 90% Fluorescense filtered at about 60% loss of peak intensity using an additional <u>primary</u> Ni filter

LYNXEYE XE Kβ filtering
NIST SRM 1976a (Corundum)

Square root scale to highlight details



LYNXEYE XE Kß filtering
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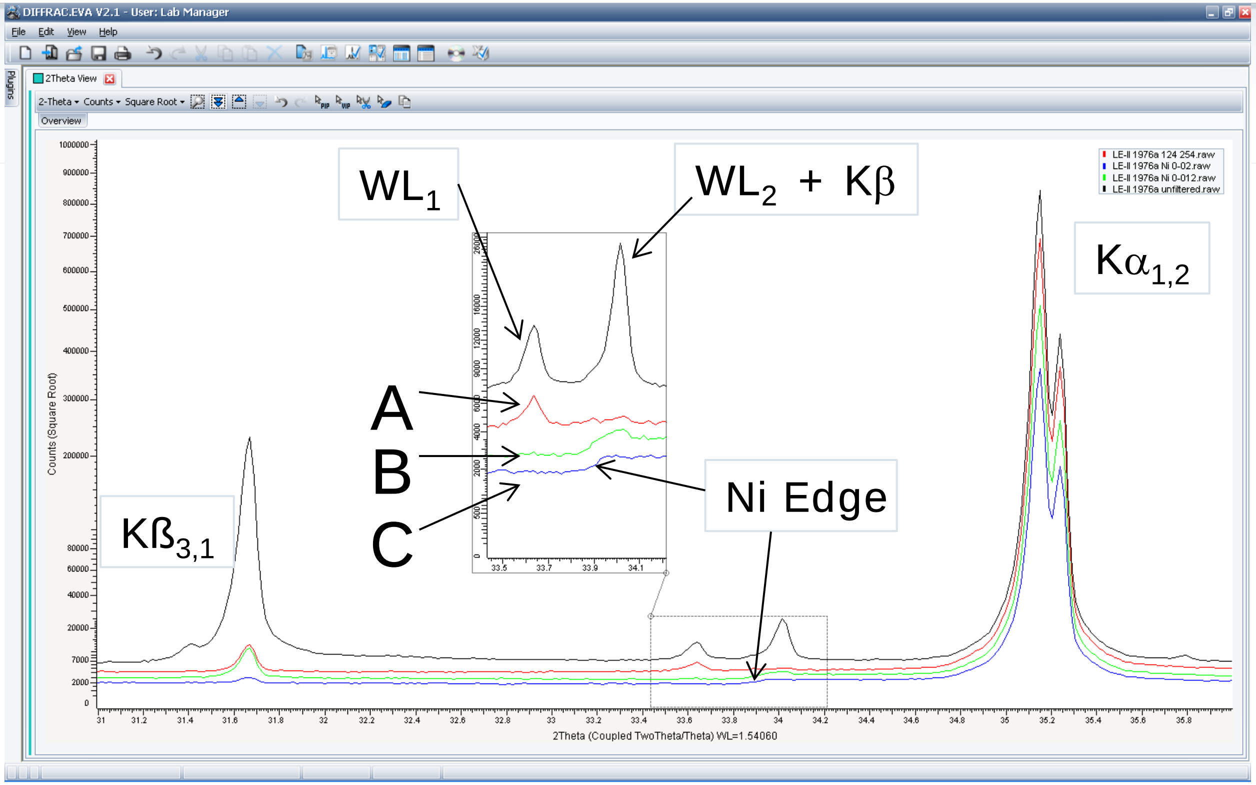
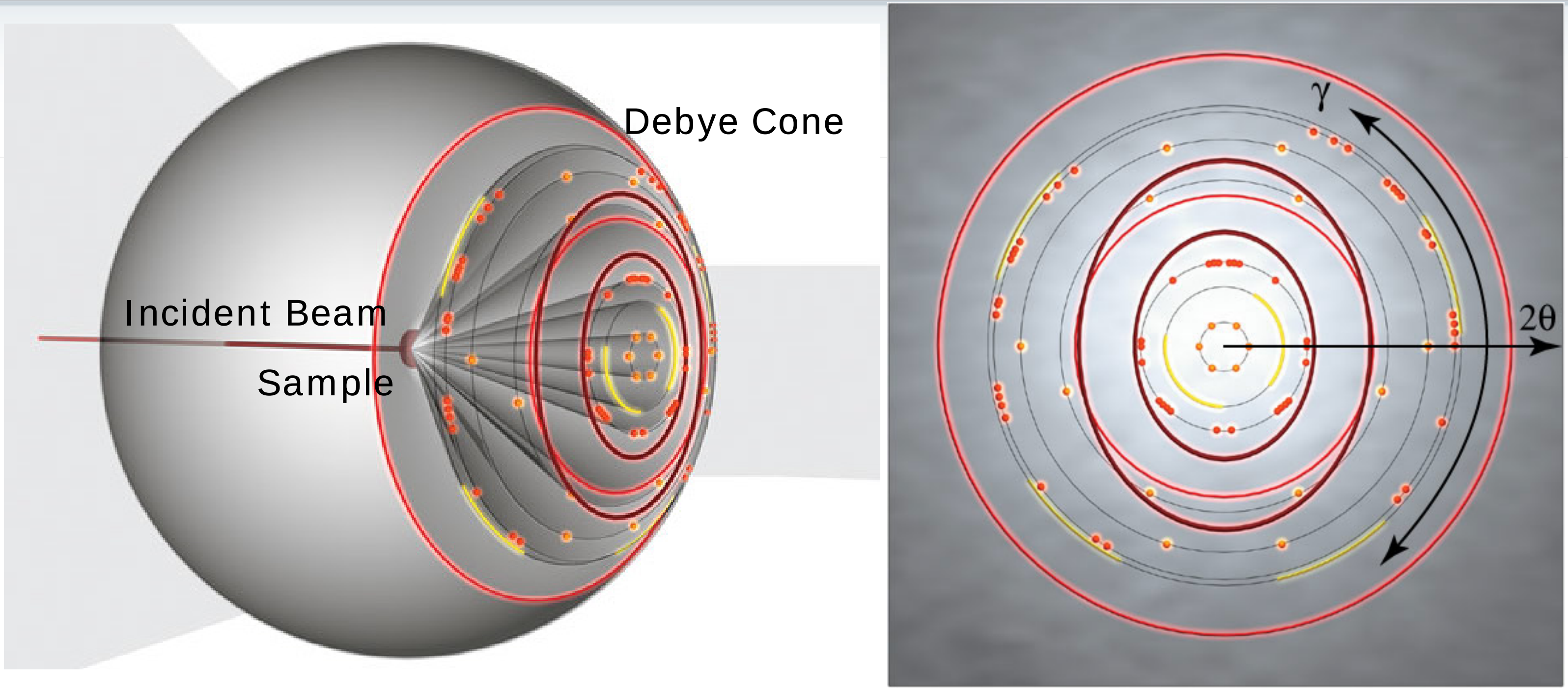


Device	Remnant Kß	Intensity loss
A LYNXEYE XE, Kß filtering	0.8%	~20%
B LYNXEYE XE, 0.0125 mm Ni Filter	1.2%	~40%
C LYNXEYE XE, 0.02 mm Ni Filter	0.3%	~60%

2D detectors for XRD^2

XRD^2: Diffraction pattern with both γ and 2θ information

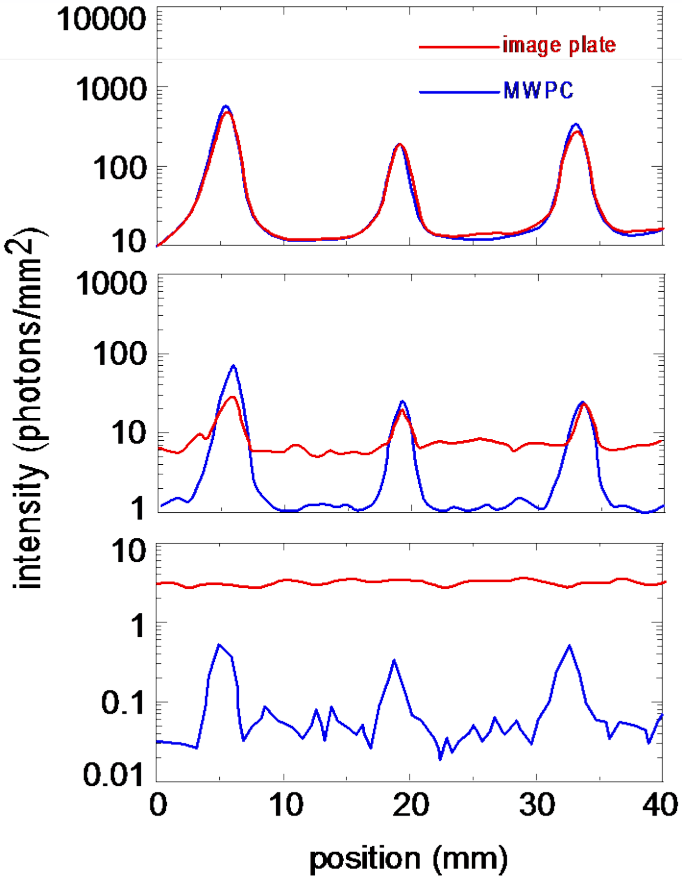


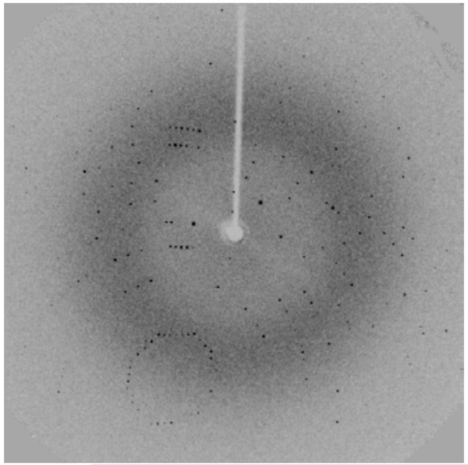
- Integrating over larger γ range improves statistics
  - Especially for microdiffraction, mapping or time resolved measurements
- 2D structure of Debye rings gives additional information
  - Stress, texture, particle size

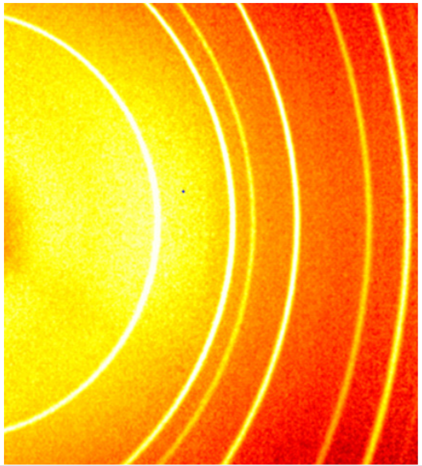
What is important for XRD²? Photon-counting detector

- Photon-counting detectors are preferred
  - In XRD² typically integrate over hundreds or thousands of pixels
  - Detectors with a finite noise background (e.g., CCD, IP) result in lower data quality
    - CCDs and IPs better for crystallography (where reflections span only a few pixels)

Source: *C. Hall et al., Nucl. Instr. and Meth. in Phys. Res. A 348 (1994) 627*

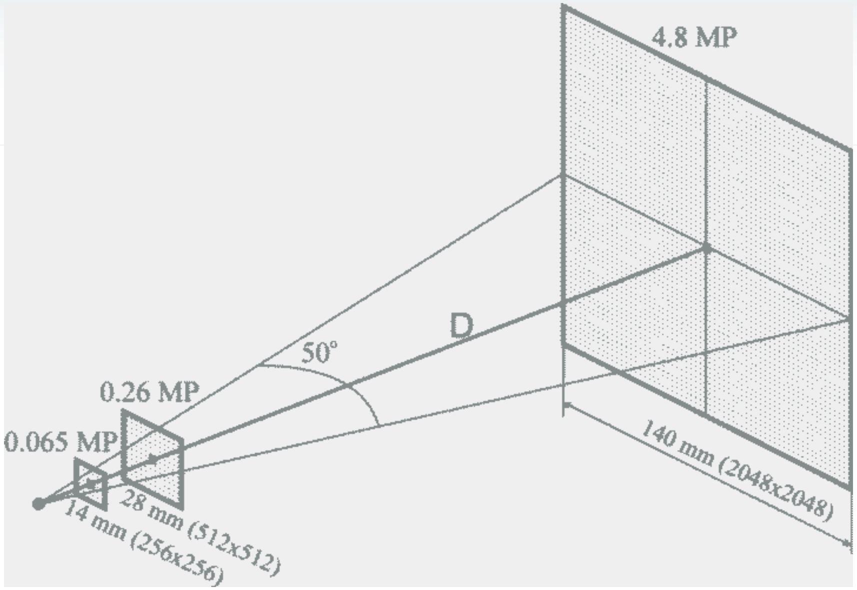


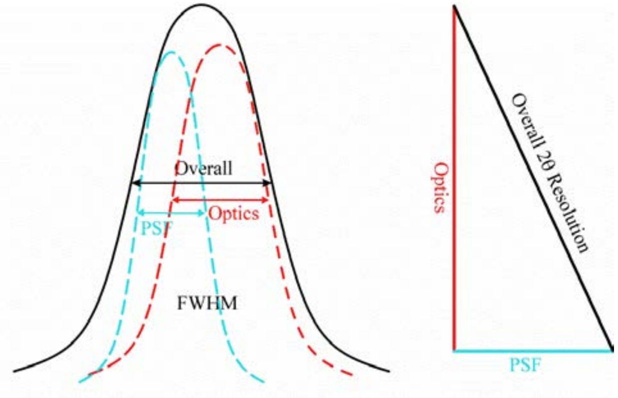




What is important for XRD^2
BIGGER is BETTER

- To cover a given solid angle we may use a big detector **farther away** or a small detector **closer** to the sample
  - E.g., a 140 mm detector at D=150 mm and a 28 mm detector at D=30 mm cover the same solid angle
- For a given angular resolution, a smaller detector requires a smaller beam, this means LESS INTENSITY
  - However, if the 140 mm detector employs a **500 micron beam**, to achieve the same resolution the 28 mm needs to employ a **50 micron beam**
  - ***This results in a 100 times loss in intensity for the small detector!***

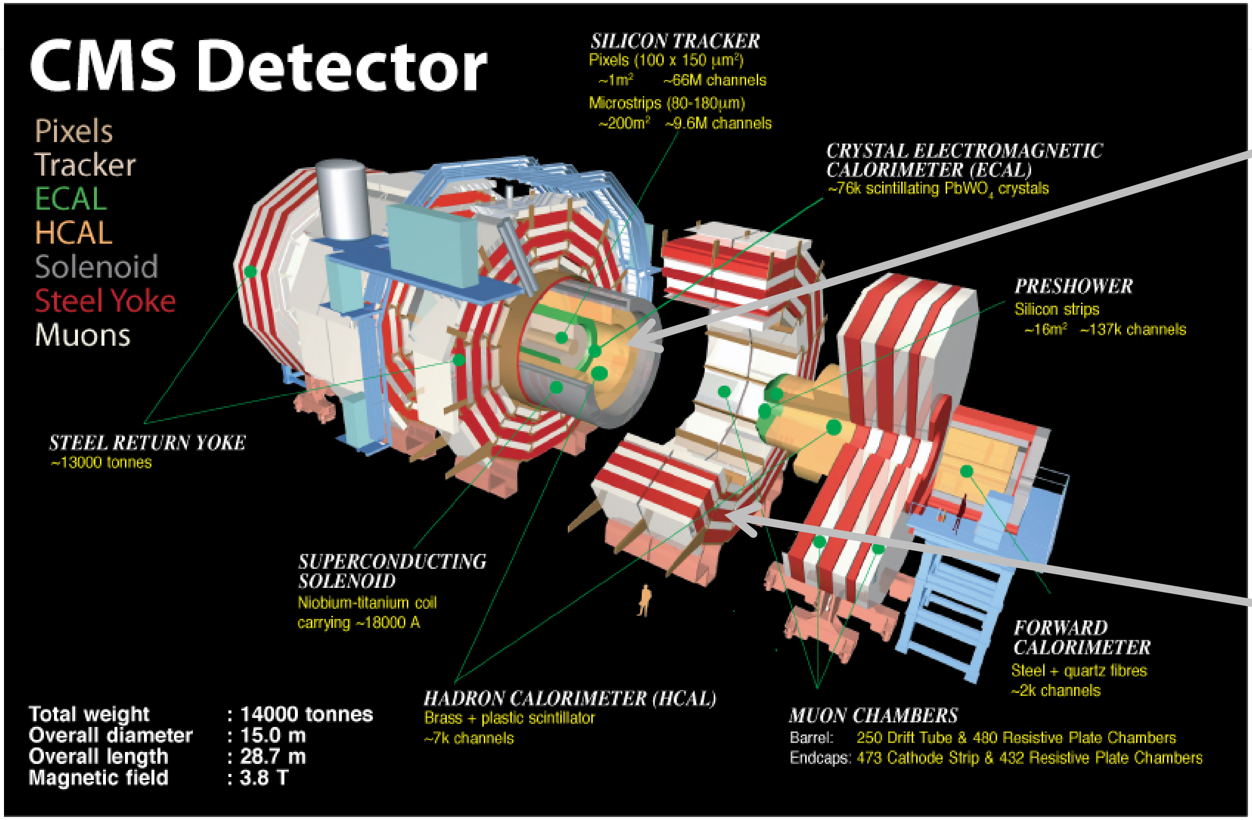




Types of photon-counting area detectors: Gas and Silicon Pixel Array (Si-PADs)

- Photon counting area detectors employ either conversion in gas (usually Xenon) or conversion in a semiconductor (usually Silicon)
  - **Gas detectors:** VANTEC (Bruker), Triton (Rigaku)
    - Advantages
      - Large active areas, no gaps
      - ***Lower cost per unit area*** ⬅ *Key advantage for lab*
    - Disadvantages
      - Lower count rate capability
  - **Silicon pixel arrays:** Pilatus (Dectris), others in development (e.g., Rigaku HPAD)
    - Advantages
      - ***Higher count rate*** ⬅ *Key advantage for synchrotron*
    - ***Disadvantages***
      - ***Higher cost per unit area***

Current generation XRD detectors derived from HEP technology

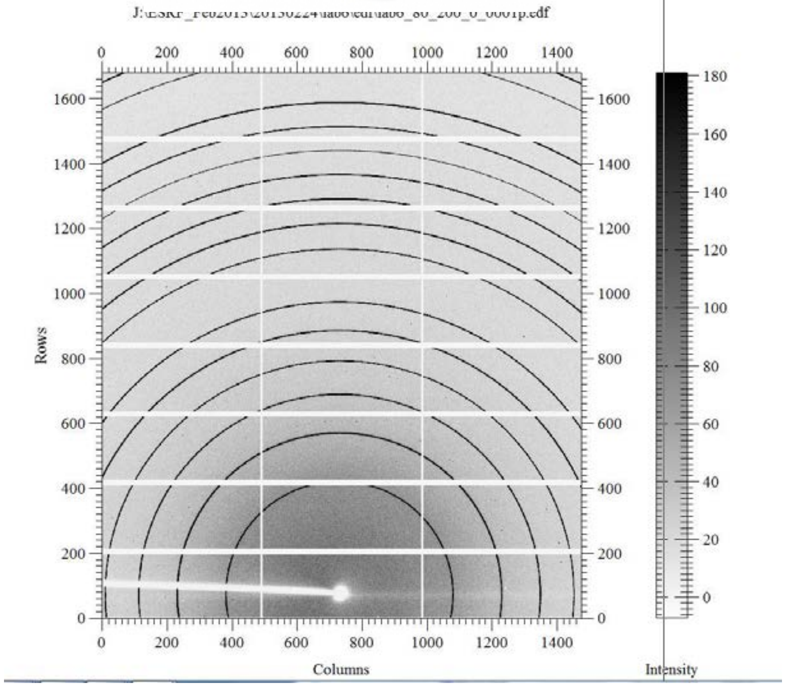


Inner tracker:
Silicon pixel detectors

Outer muon chambers:
Gas (RPC) detectors

Silicon Pixel Arrays Detectors for XRD^2

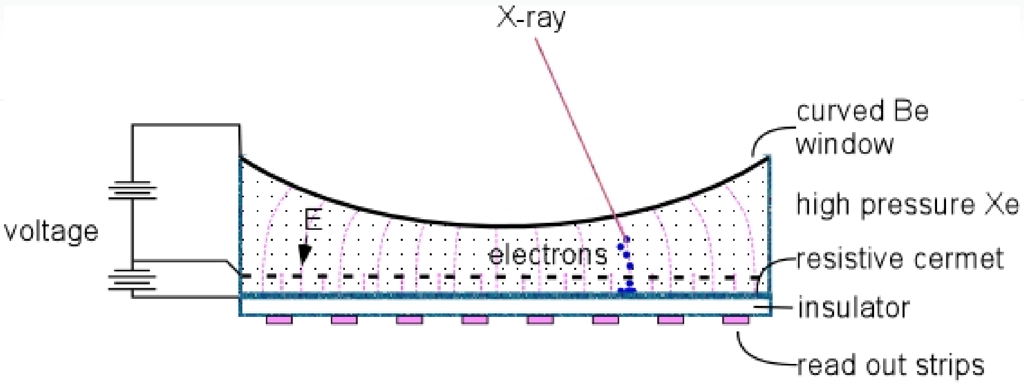
- Silicon Pixel Array detector (Dectris Pilatus 2M) proven to collect very high quality XRD^2
  - Uniquely capable of handling the very high flux at 3rd generation beamlines
- However, large Si-PAD arrays are so far less common for home laboratory use because of the relatively high cost



Courtesy P. Pattinson, ESRF

Gas detector principle: Xe microgap detector

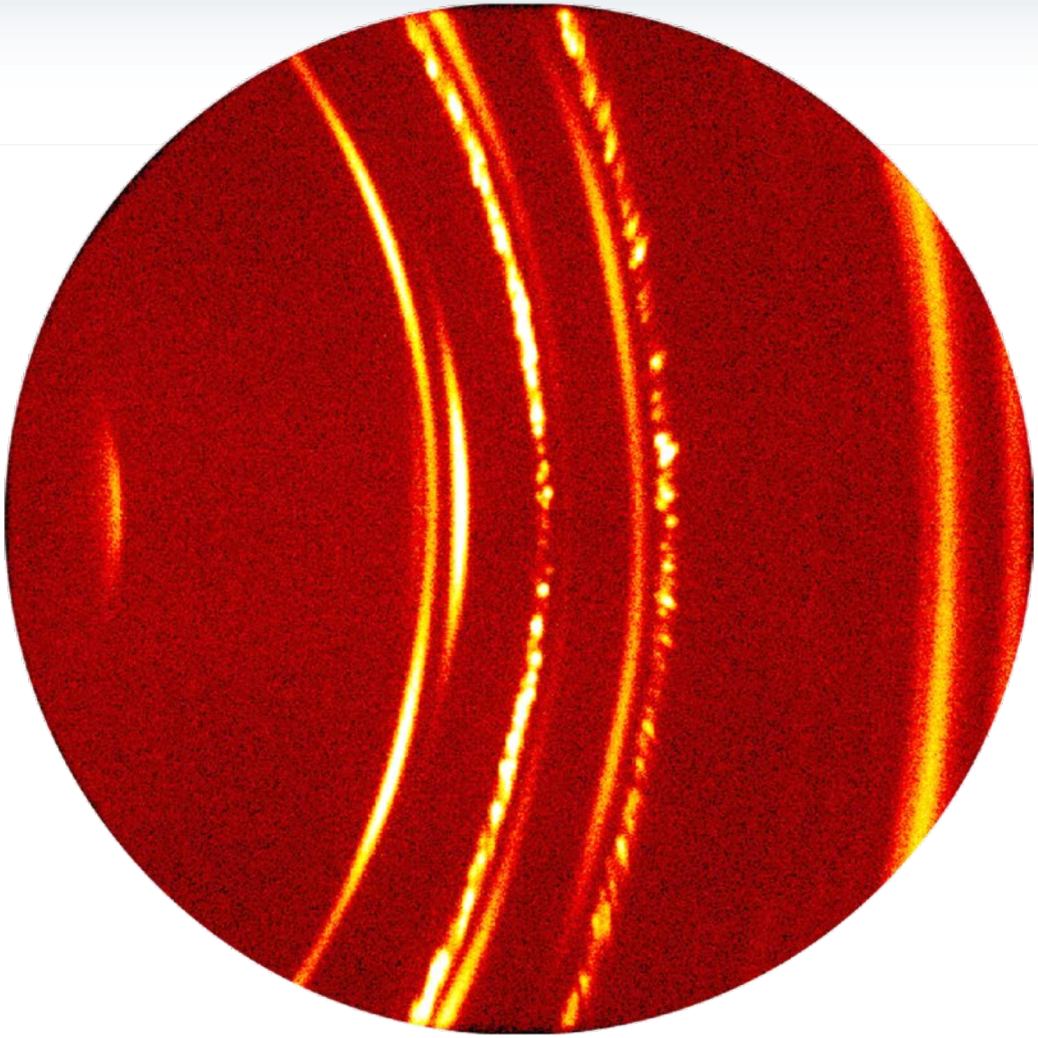
- X-rays absorbed in high pressure Xenon
  - Entrance window spherical to minimize parallax
- Electrons drift to amplification grid
  - Electrons undergo Townshend avalanche multiplication
  - Gain $>10^6$
  - Results in noiseless readout
- Resistive anode protects against discharges (US patent 6340819)
- Readout strips capacitively coupled



VÅNTEC-500 – Xe microgap detector

- Large active area: 140 mm in dia.
- Frame size: 2048 x 2048 pixels
- Pixel size: 68 µm x 68 µm
- High sensitivity: 80% DQE for Cu
- No gaps
- Highly uniform response (<1%)
- High max linear count rate: 0.9 Mcps
- Low background noise: $<10^{-5}$ cps/pix
- Maintenance-free: no re-gassing
- Very radiation hard
- Proven reliability: >500 installed worldwide

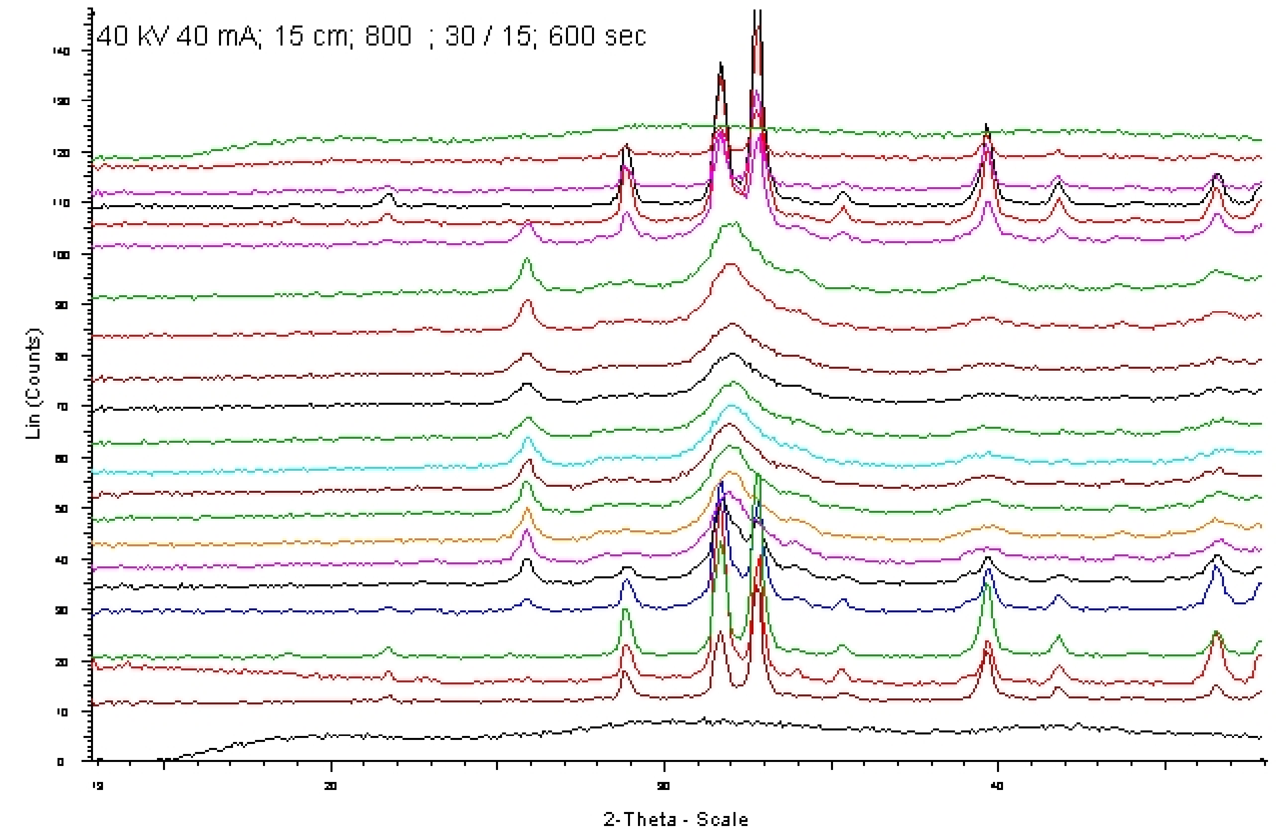
XRD^2 data



- Multilayer battery anode.
- 2θ coverage: 70° at 8 cm detector distance
- A single frame showing information on phase, stress, texture and grain size

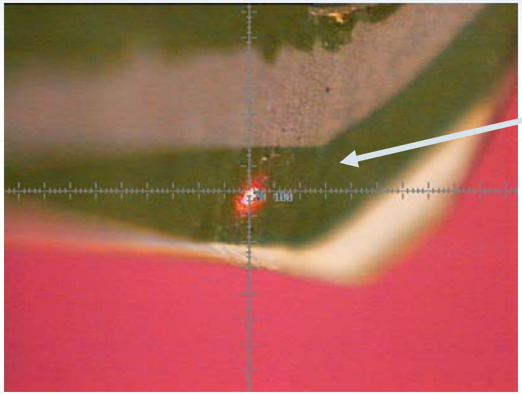
Fast mapping:
Scanning Over a Tooth by XRD^2

Embedded Tooth with its Polished Surface



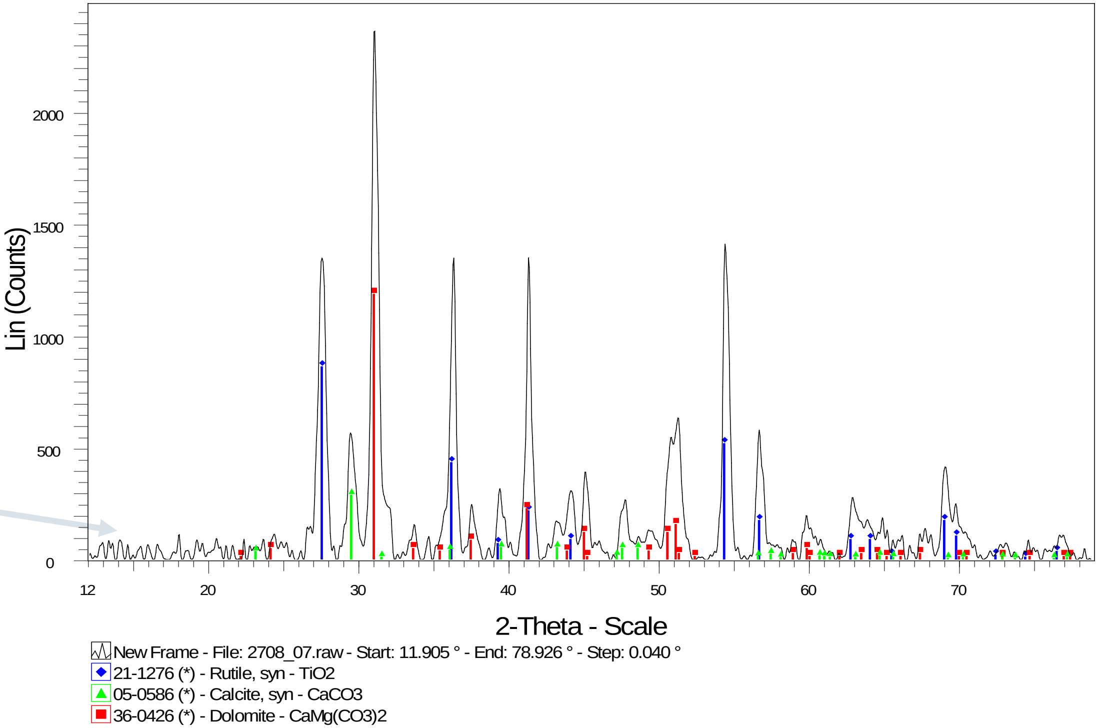
Measurements Taken Vertically, Lengthwise along the Tooth´s Diameter

Microdiffraction:
Forensic analysis of car paint



video image (for documentation)

integration of data: diffractogram for phase identification



Conclusions

- Energy dispersive detector arrays represents a paradigm change in laboratory X-ray diffraction
  - Highly effective filtering of fluorescence, white radiation and K-beta radiation at greatly reduced intensity losses compared to conventional detectors
  - No absorption edges associated with metal filters
  - Significantly improved intensity, peak-to-background-ratio, lower limits of detection
- XRD^2 is a powerful technique for a variety of applications including mapping, microdiffraction, stress, texture
- Gas detectors and silicon pixel array detectors both deliver vey high quality XRD^2
  - For applications with higher count rates silicon detectors are preferred
  - For applications with lower count rates gas detectors deliver comparable data

Acknowledgments

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