Developments in detector technology for XRD

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Detector technology for enhanced speed and accuracy

- *Energy discriminating strip detectors*
- *2D detectors*
Monochromatization and energy dispersive detectors
• 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 Kβ-Filters
  • Intensity loss ~40-60% with respect to unfiltered radiation
  • Absorption edges
  • Poor filtering of fluorescence

• An energy dispersive detector could in principle improve effective sensitivity by 2-10 times and reduce errors due to absorption edges and fluorescence
Monochromatization
Artifacts from Kβ-Filter

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
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

<table>
<thead>
<tr>
<th>Detector type</th>
<th>Compound silicon strip</th>
</tr>
</thead>
<tbody>
<tr>
<td># strips</td>
<td>192 strips, 75 mm pitch</td>
</tr>
<tr>
<td>Active area</td>
<td>14.4 x 16mm</td>
</tr>
<tr>
<td>Modes</td>
<td>1D and 0D</td>
</tr>
<tr>
<td>Wavelengths</td>
<td>Cr, Co, Cu, Mo, and Ag</td>
</tr>
<tr>
<td>Energy resolution (Cu)</td>
<td>&lt; 680 eV @ 8 KeV</td>
</tr>
</tbody>
</table>
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 fluorescence radiation (Cu)
LYNXEYE XE
Filtering Fe fluorescence: Iron Ore

- Fluorescence filtered completely
- <5% loss of peak intensity
Filtering of Fluorescence Radiation (Cu)

- **Manganese:**
  - Fluorescence filtered completely at <5% loss of peak intensity

- **Iron:**
  - Fluorescence filtered completely at <5% loss of peak intensity

- **Cobalt:**
  - > 90% Fluorescence filtered at <5% loss of peak intensity
  - > 98% Fluorescence filtered at <25% loss of peak intensity

- **Nickel:**
  - > 50% Fluorescence filtered at <5% loss of peak intensity
  - > 90% Fluorescence filtered at about 60% loss of peak intensity using an additional primary Ni filter
LYNXEYE XE Kβ filtering
NIST SRM 1976a (Corundum)

Square root scale to highlight details

LYNXEYE XE, unfiltered radiation
LYNXEYE XE, Kβ filtering
LYNXEYE XE, 0.0125 mm Ni Filter
LYNXEYE XE, 0.02 mm Ni Filter
LYNXEYE XE Kβ filtering
NIST SRM 1976a (Corundum)

<table>
<thead>
<tr>
<th>Device</th>
<th>Remnant Kβ</th>
<th>Intensity loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>A LYNXEYE XE, Kβ filtering</td>
<td>0.8%</td>
<td>~20%</td>
</tr>
<tr>
<td>B LYNXEYE XE, 0.0125 mm Ni Filter</td>
<td>1.2%</td>
<td>~40%</td>
</tr>
<tr>
<td>C LYNXEYE XE, 0.02 mm Ni Filter</td>
<td>0.3%</td>
<td>~60%</td>
</tr>
</tbody>
</table>
2D detectors for XRD$^2$
**XRD^2**: Diffraction pattern with both $\gamma$ and $2\theta$ information

- Integrating over larger $\gamma$ 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 XRD$^2$?

Photon-counting detector

- Photon-counting detectors are preferred
  - In XRD$^2$ 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)
What is important for XRD²
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

**CMS Detector**

- **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 3$^{rd}$ 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.
- $2\theta$ 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 XRD²

Embedded Tooth with its Polished Surface

Measurements Taken Vertically, Lengthwise along the Tooth’s Diameter
Microdiffraction: Forensic analysis of car paint

integration of data: diffractogram for phase identification

video image (for documentation)
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 very 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
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