2D Detectors for X-ray Diffraction

Joe Ferrara
Deputy Director, XRL Rigaku Corporation
CSO, Rigaku Americas Corporation
VP, American Crystallographic Association

Denver X-ray Conference,
August 6, 2018
Introduction

• Basic concepts
• Detector performance
• Detector technology
• Applications
Introduction

• Basic concepts
• Detector performance
• Detector technology
• Applications
Key Components of an X-ray Diffraction System

**Generator**
- Wavelength, Intensity

**Sample**
- Crystal structure, Crystal quality, etc.

**Optics**
- Geometry, Spectrum etc.

**Detector**
- Energy resolution, Efficiency, Size, etc.
Single Crystal

Powder
- Texture
- Orientation

Ideal Powder

2D

1D
Sample

- Peak positions
  - d values > Phase ID
  - d shift > Lattice parameters
    - Residual stress
    - Solid solution

- FWHM
  - Crystal quality
  - Crystallite size
  - Lattice distortion

- Intensity vs. Orientation
  - Preferred orientation
  - Fiber structure
  - Pole figure

- Integrated Intensity of amorphous
- Integrated Intensity of crystal > Qualitative analysis

- Crystallinity

Scattering Angle vs. Intensity
Position

- Lattice parameters
  \[ d = f(a, b, c, \alpha, \beta, \gamma) \]
  \[ 2d \sin \theta = n\lambda, (n = 1, 2, 3 \cdots) \]

- Systematic errors
  - Flat sample
  - Sample displacement
  - Absorption
  - Axial divergence
  - Misalignment

- Calibration
  - Internal/external SRM with known lattice parameters
Width

- Crystallite size
  \[ D = \frac{K\lambda}{\beta \cos \theta} \]

- Lattice distortion
  \[ e = \frac{\beta}{4 \tan \theta} \]

- Systematic errors
  - Resolution
  - Flat sample
  - Absorption
  - Axial divergence

- Calibration
  - Internal/external SRM with uniform crystallite size larger than about 5~10 µm.
Diffracted Intensity

- Crystal structure
  \[ I_{hkl} \propto |F_{hkl}|^2 pK_{LPA} e^{-2M}, F_{hkl} = \sum_{j=1}^{N} f_j e^{2\pi i (hu_j + kv_j + lw_j)} \]

- Preferred orientation
  \[ I'_{hkl} \propto I_{hkl} \cdot P_{hkl}, P_{hkl} = \frac{1}{m_{hkl}} \sum_{j=1}^{m_{hkl}} \left( r^2 \cos^2 \alpha_j + r^{-1} \sin^2 \alpha_j \right)^{-3/2} \]

- Particle statistics
- Calibration
  - Internal/external SRM with uniform crystallite size larger than about 1 µm.
What is an Ideal Detector?

- The ideal detector measures every photon
- The ideal detector tells you when and where it landed
- There is nothing else
  - Jim Pflugrath, 1994
- And, it does not add information (noise)
  - Ed Lattman, 2013
Detectors and their Properties

• Film
• Ionization chambers
• Scintillation counters
• Multiwire Proportional Counting (MWPC) Detectors and MicroPattern Gas Detectors (MPGD)
• Imaging Plate (IP) detectors
• Charge Coupled Device (CCD) detectors
• Active Pixel Sensor/Complementary Metal Oxide Semiconductor (CMOS) detectors
• Hybrid Pixel Array Detectors (HPAD)
  • Photon counting (HPC)
  • Integrating
Integrating versus Counting Detection

**Integrating**
- Integrates all charge generated by incoming photons and noise.

**Photon Counting**
- Discriminates X-ray pulse from noise by amplitude of the signal.
Direct vs Indirect Detection

“Towards Photon Counting X-Ray Image Sensors”
Bart Dierickx, Benoit Dupont, A. Defernez, and P. Henckes
Introduction

• Basic concepts
• Detector performance
• Detector technology
• Applications
Detective Quantum Efficiency

\[ DQE = \frac{SNR_{out}^2}{SNR_{in}^2} \]
What does DQE depend upon?

- Noise – arises from ANYTHING that degrades the signal
  - Integrating Detectors
    - Transmission windows, phosphor noise, point spread function, dynamic range, read noise, dark current noise, digitization noise, linearity fluctuations …
  - Counting Detectors
    - Transmission windows, electrodes, electron-hole pair recombination, K edge fluorescence, PSF, charge sharing noise, dead time …

\[ DQE = f(I, E, \text{psf}, N, t, ...) \]
Dynamic Range

• Dynamic Range is the ratio of the largest signal that can be measured and the larger of the smallest signal and the noise floor

\[ DNR = \frac{\max(I)}{\max(\min(I), n)} \]
Aperture

• How do we account for the aperture of the detector in order to account for photons that are not detected?
• We need to incorporate information about the resolving power of the detector as this determines the sample-to-detector distance (which also affects x-ray background).

\[ DCE = DQE \cdot \frac{A}{A_{\text{ideal}}} \]
Detector Models

• Stanton and Phillips model

\[
DQE = \frac{T_W}{1 + R_s + \frac{1}{G} + \frac{A(n_R^2 + N_D t)}{IT_W G^2} + R_E T_W I}
\]

• Duty cycle correction
  • Needed to account for time spent not collecting data

• Aperture correction
  • Needed to account for diffracted photons not hitting the detector

\[
DCE = DQE \cdot \frac{A}{A_{\text{ideal}}} \cdot T_{\text{duty cycle}}
\]

W. C. Phillips, M. Stanton, A. Stewart, H. Qian, C. Ingersoll and R. M. Sweet
Introduction

• Basic concepts
• Detector performance
• Detector technology
• Applications
Image Plate Technology

- Photostimulated luminescence (PSL)

![Image Plate Technology Diagram]

- Unrecorded Imaging Plate
- \( \text{BaFBr}_{0.85}\text{I}_{0.15}:\text{Eu}^{2+} \)
- Support
- X-ray Photons
- Stored Image
- Exposure
- Laser Beam Scanning
- Excitation light 658 nm
- Luminescence 400 nm
- Visible Light
- Erasing

Plate is ready for use again.
Hybrid Photon Counting Detectors

Direct detection

X-ray photon
HyPix-3000

• Shutterless data collection
• Fluorescence suppression
  • $\Delta E/E < 15\%$ (Cu)
• Wide Dynamic range
  • Attenuator free
• Modes of operation
  • 2D – 1D – 0D
Two 16-bit counters in each pixel

Three different modes:

STANDARD MODE WITH ENERGY WINDOW:
\[ \text{discriminator1} \Rightarrow \text{counter1 (16 bits)}, \text{discriminator2} \Rightarrow \text{counter2 (16 bits)} \]

STANDARD MODE WITH SINGLE THRESHOLD AND LONG COUNTER:
\[ \text{discriminator1} \Rightarrow \text{counter12 (32-bits)} \text{ or discriminator2} \Rightarrow \text{counter12 (32-bits)} \]

CONTINUOUS MODE WITH SINGLE THRESHOLD:
Phase 1: \[ \text{discriminator1} \Rightarrow \text{counter1 (M-bits), counter2(M-bits)} \Rightarrow \text{data readout} \]
Phase 2: \[ \text{discriminator1} \Rightarrow \text{counter2 (M-bits), counter1(M-bits)} \Rightarrow \text{data readout} \]
Number of readout bits M can be controlled 1-16 to increase the frame rate
XRF suppression Fe$_2$O$_3$

- Standard mode
- XRF suppression mode (HyPix-3000)
- XRF suppression mode (newest HyPix-3000)
P/B ratio Fe₂O₃

10

3

1

Standard mode
XRF suppression mode (HyPix-3000)
XRF suppression mode (new HyPix-3000)

Hematite, Fe₂O₃
Zero Dead-Time mode

Zero Dead-Time mode

Normal mode
Precise measurement at high frame rate

The high frame rate allows for better sampling of the intensity data resulting in more accurate measurements.

The real purpose

Measure the peak intensity precisely!

By fine slicing you reduce counting loss errors

Fast image rate is needed

Coincidence correction must be applied

Stability is important
Shutterless high-temp data collection
Introduction

• Basic concepts
• Detector performance
• Detector technology
• Applications
HyPix-3000

- Shutterless data collection
- Fluorescence suppression
  - \( \Delta E/E < 15\% \) (Cu)
- Wide Dynamic range
  - Attenuator free
- Modes of operation
  - 2D – 1D – 0D
2D powder diffraction

- 2D patterns include grain size and preferred orientation effects.
- This information is taken into consideration in phase identification.
In operando measurement

- Electrochemical cell and pouch cell attachments are available for Li-ion related materials.
- EC-cell: reflection either by Cu or Mo
- Pouch: Mo-transmission (CBO-E)
Stress

- Radiation choices: Cr, Co, Cu and Mo
- Supports \( \sin^2 \psi \), multiple HKL and 2D triaxial stress
- Micro area as an option
Pole figure

- Complete pole figure by in-plane or transmission method
- $\alpha$-$\beta$ attachment allows $\gamma$ oscillation
- 2D detector accelerates data acquisition especially it covers more than two HKLs simultaneously
- Quantitative texture analysis by orientation distribution function followed by inverse pole figure, pole figure simulation and texture component calculation
Pair distribution function (PDF)

- PDF analyzes interatomic distances, coordination number and periodicity.
- SmartLab Studio II software can simulate and overly expected peak positions calculated from database.
- Transmission
  - CBO-E (Mo)
  - Capillary spinner
- Reflection
  - CBO (Mo)
In-plane thin film diffraction

True In-plane scan enables one to see:
- Depth profile
- Ultra-thin film (t < 10 nm)
- In-plane d-spacings
- In-plane strain and stress
- Complete pole figure
Advanced thin film RSM

- Ultra-fast RSM by HyPix-3000 (1D)
- Wide area RSM by HyPix-3000 (2D)
2D SAXS, WAXS, GI-SAXS

- Dedicated attachment to achieve real 2D transmission SAXS and WAXS
- Various sample holders are available
- Dedicated attached to achieve real 2D reflection GI-SAXS