Basics and Recent Advances in Two-dimensional X-ray Diffraction

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Basic Concept – from XRD to XRD$^2$
Conventional X-ray Diffractometer

- Bragg-Brentano Geometry.
- Point (0D) detector.
- Scanning over $2\theta$ range to collect XRD pattern.
Diffraction Patterns vs. Atomic Arrangement

- Crystal
  - Graph: peaks
  - Description: ordered arrangement of atoms

- Liquid or amorphous solid
  - Graph: broad peak
  - Description: disordered arrangement

- Monatomic gas
  - Graph: continuous curve
  - Description: random arrangement of atoms
XRD²: Two-dimensional X-ray Diffraction
XRD$^2$: From 2D pattern to $\gamma$-2$\theta$ image

Fig. A. 2D diffraction pattern.

Fig. B. 2D pattern in rectangular $\gamma$-2$\theta$ image.
XRD$^2$: 2D pattern in I on $\gamma$-2$\theta$ coordinates

Powder

Texture
XRD$^2$: 2D pattern in I on $\gamma$-2$\theta$ coordinates

Stress

Large grains
Geometry Convention and Diffraction Vector Approach
Laue equation:

\[ \mathbf{a} \cdot (\mathbf{s} - \mathbf{s}_0) = h\lambda \]
\[ \mathbf{b} \cdot (\mathbf{s} - \mathbf{s}_0) = k\lambda \]
\[ \mathbf{c} \cdot (\mathbf{s} - \mathbf{s}_0) = l\lambda \]
XRD$^2$: Debye cones from powders

Bragg law

\[ n\lambda = 2d \sin \theta \]
**XRD²**: Diffraction pattern with both $\gamma$ and $2\theta$ information

- **Debye Cone**

- **Sample**

- **Incident Beam**

**Diffraction vector with $\gamma$**

$$H = \frac{s - s_0}{\lambda} = \frac{1}{\lambda} \begin{bmatrix} \cos 2\theta - 1 \\ -\sin 2\theta \sin \gamma \\ -\sin 2\theta \cos \gamma \end{bmatrix}$$
**XRD²**: Geometry Convention - Diffraction Space

Diffraction rings (blue) in the laboratory axes (red).
The diffraction vector is given in laboratory coordinates by

\[ H = \frac{s - s_0}{\lambda} = \frac{1}{\lambda} \begin{bmatrix} s_x - s_{0x} \\ s_y - s_{0y} \\ s_z - s_{0z} \end{bmatrix} = \frac{1}{\lambda} \begin{bmatrix} \cos 2\theta - 1 \\ -\sin 2\theta \sin \gamma \\ -\sin 2\theta \cos \gamma \end{bmatrix} \]

The direction of each diffraction vector can be represented by its unit vector given by:

\[ h_L = \frac{H}{|H|} = \begin{bmatrix} h_x \\ h_y \\ h_z \end{bmatrix} = \begin{bmatrix} -\sin \theta \\ -\cos \theta \sin \gamma \\ -\cos \theta \cos \gamma \end{bmatrix} \]
The angular relationships between $X_L Y_L Z_L$ and $S_1 S_2 S_3$ are:

$$
\begin{bmatrix}
X_L & Y_L & Z_L \\
S_1 & a_{11} & a_{12} & a_{13} \\
S_2 & a_{21} & a_{22} & a_{23} \\
S_3 & a_{31} & a_{32} & a_{33}
\end{bmatrix}
$$

The transformation matrix from the diffraction space to the sample space is:

$$
\begin{bmatrix}
\frac{-\sin\omega\sin\psi\sin\phi}{-\sin\omega\sin\psi\sin\phi} & \frac{\cos\omega\sin\psi\sin\phi}{\cos\omega\sin\psi\sin\phi} & \frac{-\cos\psi\sin\phi}{-\cos\psi\sin\phi} \\
\frac{-\cos\omega\cos\phi}{-\cos\omega\cos\phi} & \frac{-\sin\omega\cos\phi}{-\sin\omega\cos\phi} & \frac{\cos\psi\cos\phi}{\cos\psi\cos\phi} \\
\frac{\sin\omega\sin\psi\cos\phi}{\sin\omega\sin\psi\cos\phi} & \frac{-\cos\omega\sin\psi\cos\phi}{-\cos\omega\sin\psi\cos\phi} & \frac{\cos\psi\cos\phi}{\cos\psi\cos\phi} \\
\frac{-\cos\omega\sin\phi}{-\cos\omega\sin\phi} & \frac{-\sin\omega\sin\phi}{-\sin\omega\sin\phi} & \frac{\sin\psi}{\sin\psi}
\end{bmatrix}
$$
The components of the unit vector $h_s$ in the sample coordinates $S_1S_2S_3$ is then given by

$$
\begin{bmatrix}
  h_1 \\
  h_2 \\
  h_3
\end{bmatrix}
= \begin{bmatrix}
  a_{11} a_{12} a_{13} \\
  a_{21} a_{22} a_{23} \\
  a_{31} a_{32} a_{33}
\end{bmatrix}
\begin{bmatrix}
  h_x \\
  h_y \\
  h_z
\end{bmatrix}
$$

Or in expanded form:

$$
\begin{align*}
  h_1 &= \sin \theta (\sin \phi \sin \psi \sin \omega + \cos \phi \cos \omega) + \cos \theta \cos \gamma \sin \phi \cos \psi \\
       &\quad - \cos \theta \sin \gamma (\sin \phi \sin \psi \cos \omega - \cos \phi \sin \omega) \\
  h_2 &= -\sin \theta (\cos \phi \sin \psi \sin \omega - \sin \phi \cos \omega) - \cos \theta \cos \gamma \cos \phi \cos \psi \\
       &\quad + \cos \theta \sin \gamma (\cos \phi \sin \psi \cos \omega + \sin \phi \sin \omega) \\
  h_3 &= \sin \theta \cos \psi \sin \omega - \cos \theta \sin \gamma \cos \psi \cos \omega - \cos \theta \cos \gamma \sin \psi
\end{align*}
$$
Sources
Characteristic X-ray generation

- All present monochromatic home laboratory sources are based on characteristic radiation from a material anode
- The efficiency of this process is very low
  - Approximately 99% of the incident electron power is converted to heat, not X-rays
- Dissipation of this waste heat fundamentally limits the brightness of the source
How to make brighter source I: Microfocus sources

- Brightness ($B$) is proportional to power loading ($p$)
- Power loading is higher for smaller spot focus

- Large spot
- Quasi-one dimensional heat flow limits power loading

- Small spot
- Two dimensional heat flow (more efficient cooling)
- Relative performance improved by 10 times

\[ p_{\text{max}} = \frac{2\kappa(T_m - T_0)}{r \sqrt{\pi} \ln(2)} \]
IμS microfocus source

• Intensity $3 \times 10^{10}$ X-rays/mm$^2$-sec (Cu K$_\alpha$)
  • 8 times higher than conventional 5.4 kW rotating anode
• Typical lifetime >5 years
  • High reliability
  • 3 year warranty
  • >300 installed
• Air-cooled
• Available in Cr, Cu, Mo, Ag
**IμS & VÅNTÉC-2000 vs. Classic Set-up**

**Corundum Comparison**

**Sealed Tube with Göbel Mirror**
45kV, 40mA, (1800 W)
0.3mm collimator

**total counts:** 78K

**Microsource (IμS)™**
45kV, 0.650mA, (30 W)
0.3mm collimator

**total counts:** 1235K

**Intensity:** 15.8x; **Efficiency:** 948x!
How to make a brighter source II: Rotating anode sources

- Power loading can be increased by over an order of magnitude by rotating the anode surface to spread out the heat load.
  - Power load is also (modestly) increased by smaller spot.
- In latest generation rotating anodes angular velocity is 10,000 rpm.
- This improves performance by 50 times.

\[
p_{\text{max}} \propto \kappa (T_m - T_0) \sqrt{\frac{v}{w}}
\]

- \(w\) = beam width
- \(v\) = anode velocity
TXS HB High brilliance rotating anode

- Highest intensity rotating anode
  - $2 \times 10^{11}$ X-rays/mm$^2$-sec Cu K$\alpha$
  - 50 times the intensity of a 5.4 kW classic RAG with multilayer optics
- Cu, Mo, Ag anodes
- Low maintenance
- Easy to align, highly stable mount
  - No alignment base
- Precrystallized, prealigned filaments
  - *No realignment required after filament changes (2X per year)*
- Precision aligned anode
  - *No realignment required after anode exchange (1X per year)*
What are the limits of rotating anode performance?

- Faster rotation (ω) allows higher power loading
  \[ p_{\text{max}} \propto \sqrt{\frac{\omega R}{w}} \]

- However, faster rotation also increases mechanical stress
  \[ \sigma_h = \frac{PR}{t} = \frac{\rho \omega^2 R^3}{2t} + \rho \omega^2 R^2 \]

- State-of-the-art rotating anodes are operated close to mechanical failure limits

- **Little room for further improvement**
NEW: Liquid metal sources

- High-speed liquid-metal-jet anode
- Anode is regenerative
- No longer limited by melting
- >500 kW/mm² e-beam power density
  - Rotating anode limited to maximum 50 kW/mm²
Spot Size

<table>
<thead>
<tr>
<th>Spot size [µm, FWHM]</th>
<th>Voltage [kV]</th>
<th>Power [W]</th>
<th>Ga Kα Brightness [Photons/(s × mm² × mrad² × line)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>60</td>
<td>50</td>
<td>$1.5 \times 10^{11}$</td>
</tr>
<tr>
<td>10</td>
<td>60</td>
<td>100</td>
<td>$7.6 \times 10^{10}$</td>
</tr>
<tr>
<td>20</td>
<td>60</td>
<td>200</td>
<td>$3.8 \times 10^{10}$</td>
</tr>
</tbody>
</table>

- X-ray photon energy: Ga Kα 9.25 keV ($\lambda=1.34$ Å)
- Suitable replacement for Cu Kα 8.06 keV ($\lambda=1.54$ Å)
So, is it possible to put a synchrotron beamline on a table top?

- Yes, at least the equivalent of a typical present generation bending magnet beamline.
Detector
Characteristics of area detectors:
Sensitivity vs. Counting Rate

- Detective Quantum Efficiency (DQE):
  - The DQE is a parameter defined as the square of the ratio of the output and input signal-to-noise ratios (SNR).
  \[
  DQE = \left( \frac{(S/N)_{out}}{(S/N)_{in}} \right)^2
  \]
  - The DQE of a real detector is less than 100% because not every incident x-ray photon is detected, and because there is always some detector noise.
  - MiKroGap™ has the best overall performance.

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**Diagram Details**

- **MiKroGap**
- **MWPC**
- **CCD**
- **Image Plate**

**Graph**

- X-axis: Fluence (x-rays/reflection)
- Y-axis: Effective DQE
- Int = 30 sec

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**Graph Labels**

- MiKroGap
- MWPC
- CCD
- Image Plate
XRD²: Choice of Detectors: Active Area and Pixel Size

The angular coverage of 2D detectors with various active areas (D=150mm)
At $D=150$ mm, the angular resolution per pixel is $0.026^\circ$ for the large detector, but $0.21^\circ$ and $0.11^\circ$ for two smaller 2D detectors.
VÅNTEC-500 – Outperforms all previous gaseous detectors.

- Large active area: 140 mm in dia.
- Frame size:
  - 2048 x 2048 pixels
  - 1024 x 1024 pixels
  - 512 x 512 pixels
- Pixel size:
  - 68 µm x 68 µm
  - 136 µm x 136 µm
  - 272 µm x 272 µm
- High sensitivity: 80% DQE for Cu
- High max linear count rate:
  - 0.9 Mcps – global;
  - 160 kcps/reflection -local
- Low background noise:
  - <10^{-5} cps/pix
- Maintenance-free:
  - no re-gassing
Phase ID
XRD²: Phase ID Measurement Geometry
XRD\textsuperscript{2}: Single Frame Covering All

- Multilayer battery anode.
- $2\theta$ coverage: 70° at 8 cm detector distance
- A single frame showing information on phase, stress, texture and grain size
- 2D detector is essential for In-situ measurement
XRD$^2$: Frame Merge and Integration

- 4 frames at 20 cm
- Merged frames 2θ coverage: 100°
- Integrated profile for phase ID search/mat ch
Stress
XRD: Sample Space & Unit Diffraction Vector

The components of the unit vector $h_S$ in the sample coordinates $S_1S_2S_3$ is then given by

$$h_{\phi\psi} = \begin{bmatrix} h_{1}^{\phi\psi} \\ h_{2}^{\phi\psi} \\ h_{3}^{\phi\psi} \end{bmatrix} = \begin{bmatrix} \cos \phi \sin \psi \\ \sin \phi \sin \psi \\ \cos \psi \end{bmatrix}$$

which is a single value at each sample orientation.
The $\sin^2\psi$ - Method for Stress Measurements

Linear $\varepsilon_{\psi\phi} - \sin^2\psi$ - Function

- $\phi = \text{constant}$

- The strain-free state of the sample does not need to be known exactly ($\sigma_{33} = 0$), since from the slope of the $\varepsilon_{\psi\phi} - \sin^2\psi$ - function the normal stress component $\sigma_{11}$ or $\sigma_{22}$ respectively (for $\phi = 0^\circ$ or $\phi = 90^\circ$) can be evaluated.
The $\sin^2\psi$ - Method for Stress Measurements

Elliptical $\varepsilon_{\psi\phi} - \sin^2\psi$ - Function ($\psi$ - Splitting)

- From the slope of the averaged $\varepsilon_{\psi\phi} - \sin^2\psi$ - function the normal stress component $\sigma_{11}$ or $\sigma_{22}$ respectively (for $\phi = 0^\circ$ or $\phi = 90^\circ$) can be evaluated.

- From the splitting of the $\varepsilon_{\psi\phi} - \sin^2\psi$ - function the shear stress component $\sigma_{13}$ or $\sigma_{23}$ respectively (for $\phi = 0^\circ$ or $\phi = 90^\circ$) can be evaluated.
Effects of Texture and Large Grain on $\varepsilon_{\psi\phi} - \sin^2\psi$ – Functions

- **Linear Function**
  - $\varepsilon_{\psi\phi}$ vs $\sin^2\psi$
  - Data points

- **Oscillating Function (Wavy Line)**
  - Textured material (elastic constants depend on $\psi$- and $\phi$-direction)

- **Scattered Data Points**
  - Large grains
  - Large error in due to poor sampling statistics
As a second order tensor, the relationship between the measured strains and the strain tensor is given by:

\[
\varepsilon_{\phi\psi} = \varepsilon_{ij} \cdot h_i^{\phi\psi} \cdot h_j^{\phi\psi} \quad \text{0D/1D:} \\
\varepsilon_{(\gamma,\omega,\psi,\phi)} = \varepsilon_{ij} \cdot h_i \cdot h_j \quad \text{2D:}
\]

Both equations are in the same form except the difference in \( h_s \)

\[
\varepsilon_{\phi\psi} = \varepsilon_{11} \cos^2 \phi \sin^2 \psi + \varepsilon_{12} \sin 2\phi \sin^2 \psi + \varepsilon_{22} \sin^2 \phi \sin^2 \psi + \varepsilon_{13} \cos \phi \sin 2\psi + \varepsilon_{23} \sin \phi \sin 2\psi + \varepsilon_{33} \cos^2 \psi
\]

Introducing \( \gamma = 90^\circ \) or \( 270^\circ \) (diffractometer plane),

the above equation can be derived from the bottom equation

\[
\varepsilon_{(hkl)}^{(\gamma,\omega,\psi,\phi)} = h_1^2 \varepsilon_{11} + 2h_1 h_2 \varepsilon_{12} + h_2^2 \varepsilon_{22} + 2h_1 h_3 \varepsilon_{13} + 2h_2 h_3 \varepsilon_{23} + h_3^2 \varepsilon_{33}
\]
Introducing the elasticity constants: \( E \) & \( \nu \) or
the macroscopic elastic constants:

\[
\frac{1}{2} S_2 = \frac{(1 + \nu)}{E}
\]

&

\[
S_1 = -\frac{\nu}{E}
\]

the fundamental equation for stress measurement in XRD\(^2\):

\[
S_1(\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1}{2} S_2 \left( \sigma_{11} h_1^2 + \sigma_{22} h_2^2 + \sigma_{33} h_3^2 \right) + 2\sigma_{12} h_1 h_2 + 2\sigma_{13} h_1 h_3 + 2\sigma_{23} h_2 h_3 = \ln \left( \frac{\sin \theta_0}{\sin \theta} \right)
\]
The simulated diffraction ring distortion in radar chart:
(a) equibiaxial stress with $\psi$ scan;
(b) uniaxial stress with $\omega$ scan for Fe (211) with Cr radiation.
The D8 DISCOVER with DAVINCI VÅNTEC-500 for stress measurement

- Almen strip (spring steel)
- (211) & (200) rings
- Ring distortion when sample rotates with $\psi$ and $\phi$
XRD$^2$: Stress Measurement – Example

Parameter setting

Evaluate the data quality

Results reporting:
\[\sigma_{11} = -1068 \pm 18 \text{ MPa}\]
\[\sigma_{22} = -1085 \pm 18 \text{ MPa}\]

Principal stress and stress ellipse – equibiaxial
Comparison of the $\sin^2\psi$ and 2D method for stress measurement

With increasing data points, the new 2D method can measure stress with higher accuracy than the conventional $\sin^2\psi$ method for the same amount of data collection.
XRD²: Stress Measurement with 2D method: Effect of Texture

- low intensity and poor profile due to texture tends to give larger $2\theta$ shift error.
- Elastic anisotropy results in error in stress calculation
**XRD²**: Stress Measurement with 2D method: Effect of Large Grains

- Poor profile due to large grain size gives larger $2\theta$ shift error.
- Diffraction profile of extreme large grain or substrate at a rocking angle brings error in $2\theta$. 
**XRD²: Innovations to 2D method:**
Intensity Weighted Least Squares Regression

Low intensity and poor profile due to texture and large grain tends to give larger 2θ shift error.


\[
S = \sum_{i=1}^{n} w_i r_i^2 = \sum_{i=1}^{n} w_i (y_i - \hat{y}_i)^2
\]

\[
w_i = \frac{I_i}{\sigma_i^2}
\]

- maximum (or integrated) intensity
- the standard error of profile fitting of the \(i\)th data point
**XRD²**: Innovations to 2D method: Stress Measurement from Multiple \((hkl)\) Rings

Regression

Improve the accuracy and reduce the effect of anisotropy and texture.
Texture, Orientation and Fiber
The pole figure angles (α, β) can be calculated from the unit vector components by the pole mapping equations:

$$\alpha = \sin^{-1} |h_3| = \cos^{-1} \sqrt{h_1^2 + h_2^2}$$

$$\beta = \pm \cos^{-1} \frac{h_1}{\sqrt{h_1^2 + h_2^2}} \begin{cases} \beta \geq 0^\circ & \text{if} \; h_2 \geq 0 \\ \beta < 0^\circ & \text{if} \; h_2 < 0 \end{cases}$$
The D8 DISCOVER with DAVINCI VÅNTEC-500 for texture measurement

- Steel can
- (200) & (110) rings
- Intensity variation during $\phi$ scan
XRD$^2$: Data Collection Speed vs. 2D or 0D

2D: 108 exposures (frames); 0D: 973 exposures (points)

multiple pole-figures; single pole-figures

2D detector is 1~2 orders of magnitude faster than 0D detector.
Texture Measurement Using VANTEC-500: Magnetron sputter-deposited Cu films

- Four diffraction rings are observed at 10 cm detector distance.
- Four pole figures can be measured simultaneously.
- Diffraction spots from Si wafer appear on some frames.
Arts from XRD$^2$: Pole-figure from Cu film and Si
XRD$^2$: Miscut Angle

- Two spots are produced by sample rotates during data collection
- 2theta integration shows the gamma angle between two spots

$\gamma$ in degrees
XRD$^2$: Miscut Angle

\[ \Delta \psi = 2 \arcsin[\cos \theta \sin(\Delta \gamma / 2)] \]

\[ \alpha = 2 \arcsin[\cos \theta \sin(\Delta \gamma / 4)] \]

- The angle between two diffraction vector is given by the virtual oscillation angle $\Delta \psi$.
- The miscut angle $\alpha$ is from half of the $\Delta \gamma$. 

\[
\Delta \psi = \frac{2}{\sin(\arcsin[\cos^2 \gamma \Delta \theta])}
\]

\[
\alpha = \frac{2}{\sin(\arcsin[\cos^2 \gamma \Delta \theta])}
\]
D8 Discover with VÅNTEC-500
PE Fiber: Orientation and Crystallinity
XRD$^2$: Fiber with VÅNTEC-500
Crystal Size
**XRD^2**: Crystal Size by $\gamma$ profile analysis:
Organic glass for food & drugs

*Courtesy of Prof. Lian Yu, U. of Wisconsin - Madison*
XRD\(^2\): Data Collection: Acetaminophen powder

- The spotty diffraction ring is due to the large crystallites compared to the sampling volume (beam size).
- The number of spots on the ring is determined by crystallite size, instrumental window (\(\gamma\)-range), multiplicity of the crystal plane, and effective diffraction volume.
- The size of jelly beans and candy bin determines how many you can fill.
For XRD$^2$, the instrumental window $\Omega$ is given by

$$\Omega = \beta_1 \beta_2 = 2 \beta \arcsin[\cos \theta \sin(\Delta \gamma / 2)]$$
XRD²: Particle Size Analysis

2θ profile analysis

γ profile analysis

1 nm  10 nm  100 nm  1 μm  10 μm  100 μm  1 mm

Scherrer equation:

\[ t = \frac{C\lambda}{B \cos \theta} \]

Gold nano-particle
Thin film applications

contributed by Jon Giencke
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Innovative XRD² Techniques
Rapid X-ray Reflectometry with XRD²

Instrument Setup
- IₜS microfocus Cu Tube
- Montel Optic
- 0.5 mm Collimator
- Knife Edge
- VÅNTEC 500

Ewald Sphere

Specular Reflection

θ

α

γ

XRD² coverage
Innovative XRD² Techniques
Rapid X-ray Reflectometry with XRD²

Layers of Various Thicknesses

Superlattice with 4nm period

Scan Mode

One Frame

Step Mode

9.748 nm
18.095 nm
42.348 nm
116.199 nm
Innovative XRD² Techniques
In Plane Grazing Incidence Diffraction with XRD²

- **Standard GID (Polycrystalline Film)**
- **In Plane GID (Polycrystalline Film)**
- **In Plane GID (Epitaxial Film)**
Innovative XRD\textsuperscript{2} Techniques

In Plane Grazing Incidence Diffraction with XRD\textsuperscript{2}
10 nm Polycrystalline Film on Si – Conventional GID

In Plane GID
Innovative XRD\(^2\) Techniques

In Plane Grazing Incidence Diffraction with XRD\(^2\)

Comparison of Conventional and XRD\(^2\)
Innovative XRD² Techniques
In Plane Grazing Incidence Diffraction with XRD²

Ewald Sphere Construction

Ray Tracing Diagram

IµS with Montel
VÅNTEC 500
Sample
Innovative XRD² Techniques

In Plane Grazing Incidence Diffraction with XRD²
Crystal Truncation Rod Measurement

**Raw Data**

![Raw Data Image]

**What is a Crystal Truncation Rod?**

Fig. 3: Examples of (a) miscut cubic lattice and (b) ordered surface roughness, and (c,d) the corresponding CTR profiles, respectively.
Innovative XRD\textsuperscript{2} Techniques

In Plane Grazing Incidence Diffraction with XRD\textsuperscript{2}
Crystal Truncation Rod Measurement

\begin{itemize}
\item 25 nm Si / 50 nm SiGe / Si
\item 20 nm STO on Si
\end{itemize}
Innovative XRD² Techniques

In Plane Grazing Transmission Diffraction with XRD²

Z is set slightly low, so only the edge is tilted into the beam

STO 200  Si 220
Innovative XRD² Techniques

Reciprocal Space Mapping with XRD²
Comparison of RSM with XRD² and 1D LynxEye

Total Time for XRD² RSM (27 peaks): 15 minutes
Total Time for 1D RSMs (4 peaks): 1.5 hours
Innovative XRD\textsuperscript{2} Techniques
Lattice Parameter Refinement with DIFFRAC.TOPAS
3D Display
Visualization of 3D Reciprocal Space with MAX3D

Contributed by
Jim Britten, Weiguang Guan, Victoria Jarvis
McMaster University, Hamilton, Ontario, Canada
Example 1 – Random Orientation
GaAs NW on Carbon nanotube ‘fabric’

Why bother with XRD^3?
Sometimes there are surprises!
Example 2 – Multiple (8) Orientation GaAs NW’s on Si Substrate

2D scan

Full scan in MAX3D
More About XRD

1. Introduction.
2. Geometry Conventions.
3. X-Ray Source and Optics.
4. X-Ray Detectors.
5. Goniometer and Sample Stages.
6. Data Treatment.
7. Phase Identification.
8. Texture Analysis.
10. Small-Angle X-Ray Scattering.
11. Combinatorial Screening.
12. Quantitative Analysis.
13. Innovation and Future Development.
More About XRD²

Volume H with a chapter on two-dimensional X-ray diffraction will be published in 2014 and available at
XRD² Publications
ICDD Most Downloaded AXA Papers

30 Most Downloaded AXA Publications
(Updated February 2014 with 30 most downloaded AXA files in 2013)

XRD - Most Frequent Downloads

- X-ray Diffraction Analysis in the Forensic Science: The Last Resort in Many Criminal Cases
- Fundamentals of Two-Dimensional X-ray Diffraction (XRD2)
- Powder X-ray Diffraction Detection of Crystalline Phases in Amorphous Pharmaceuticals
- Apatite Structures
- Grazing Incidence in-plane X-ray Diffraction in the Laboratory
- Advances in Quantitative XRD Analysis for Clinker, Cements, and Cementitious Additions
- Applications of X-ray Diffraction in Conservation Science and Archaeometry
- Quantitative Analysis of Calcium Oxide Desiccant Conversion to Calcium Hydroxide Using X-ray Diffraction
- Peak Identification of Conventional X-ray Diffraction Patterns for MBE FePt Thin Films on MgO Single-crystal Substrates
FUNDAMENTALS OF TWO-DIMENSIONAL X-RAY DIFFRACTION (XRD²)

Baoping Bob He, Uwe Preckwinkel and Kingsley L. Smith
Bruker Analytical X-ray Systems
Madison, Wisconsin, USA

Figure 5. The geometric definition of diffraction rings in laboratory axes.

Figure 6. Schematics of an ideal detector covering 4π solid angle.
XRD$^2$ Publications

Most Downloaded AXA Papers #1

Copyright © JCPDS - International Centre for Diffraction Data 2003, Advances in X-ray Analysis, Volume 46.

X-RAY DIFFRACTION ANALYSIS IN THE FORENSIC SCIENCE: THE LAST RESORT IN MANY CRIMINAL CASES

Werner Kugler

Forensic Science Laboratory, Landeskriminalamt Baden-Wuerttemberg, Taubenheimstraße 85, D – 70 372 Stuttgart, Germany

Fig. 2 General Area Detector System (GADDS).

Fig. 3 System configuration, symmetrical and asymmetrical detector position with the primary beam and the scattered beam with the Debye-Scherrer cones.

Fig. 4 Collection of diffraction data in symmetrically or in asymmetrically detector position.

Fig. 30 Pistol mounted on the XYZ stage,
Fatal Bicycle Accident
Collection of Evidence

Dr. W. Kugler
Landeskriminalamt
Baden-Württemberg
Stuttgart, Germany

traces of car paint found on the bicycle
Fatal Bicycle Accident
Mapping of Car Paint with GADDS

video image (for documentation)

2-dimensional diffraction pattern

integration of data: diffractogram for phase identification
Fatal Bicycle Accident
The Car’s Identification

sequence of coatings is characteristic of car type
Police Officer’s Pistol – Excessive Force?
Death of a Bank Robber

Crime Scene Showing the Path of the Bank Robber
Police Officer’s Pistol – Excessive Force?
Contact Trace on the Barrel

XRD Pattern from the Contact Trace and the Material from the Sidewalk
Police Officer’s Pistol – Excessive Force?
Contact Trace on the Projectile

- Projectile Mounted on the Stage
- Measurement Point, Adjusted with the Laser Beam