Dr Chris Moore, VP Technology

- PhD Solid State Physics, University of Waterloo, 1983
- Extensive experience in metrology technology and systems
  - Lead teams that designed
    - Optical tool UV to mid IR
    - High speed mapping x-ray
    - Photoluminescence
    - Non-contact electrical
    - Contact electrical tools
  - Worked with many other types of tools and imaging systems
- Experience in measuring or characterizing:
  - Semiconductor materials and devices
  - Compound materials and devices
  - Flat panel display
  - Solar cells and batteries
  - MEMS and micro-fluidics
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Bragg's Law

Unlike light reflection (see red below) in x-rays you only get a signal when diffraction condition is satisfied (see mauve below)

\[ n\lambda = 2d \cdot \sin\theta \]
Bragg Brentano Geometry

Typically source and detector move
High speed measurement
Need flat sample (or powder)
Parallel Beam Geometry

- Typically source and detector move
- Medium speed measurement
- Works on rough samples
Typically source and detector move
Measurement speed highly sample dependent
Works best on smooth samples
Best resolution of all geometries
Grazing Incidence Geometry

Typically detector moves
Measurement speed highly sample dependent
Works best on smooth samples
Great to separate substrate/film effects
Using Standards (NIST LaB6) Using Bragg-Brentano

The positions, heights, areas, and FWHM of the peaks can be found through profile fitting.
• The peak position shifts to lower angles with elevated temperature were used to calculate the change in interplanar spacings.
• Whole-pattern fitting was used to calculate lattice parameters for \( \text{Al}_2\text{O}_3 \) powder at 29°C and 1000°C.
• The calculated thermal expansion of the \( \text{Al}_2\text{O}_3 \) powder between 29°C and 1000°C was within 1% of the theoretical value for the “a” lattice parameter and within 5% of the “c” lattice parameter.
Many crystalline materials can have different crystalline structures (phases). Typically these phases have different mechanical, optical and electrical properties. Find out what crystal structures are actually in your crystalline material!

XRD Example Phase Identification

XRD phase identification:
• Is highly repeatable
• Is non-destructive and fast
• Can detect phases in small amounts
• Needs an expert to model and analyze

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<th>Phases Detected in XRD</th>
<th>Possible Effects of Phases</th>
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<td>Austenite phase in Martensite phase steel</td>
<td>increased fatigue strength, unwanted change in dimensions</td>
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<tr>
<td>Silicon Carbide polytypes</td>
<td>unpredictable change in band gap, increase in electron mobility</td>
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<td>CIGS phases for solar cells</td>
<td>increase or decrease of conversion efficiency</td>
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Qualitative
Sample contains the rhombohedral crystal structure of Al₂O₃

Quantitative
Sample is 26.8% ± 5% cubic CaF₂
26.9% ± 5% hexagonal ZnO
46.3% ± 5% rhombohedral Al₂O₃

XRD scans courtesy of MDI...
• ZnO and Al₂O₃ were the phases identified for the mixed powder sample
• Whole Pattern Fitting in JADE software was used to calculate the weight percentages of the phases, shown above
• Overlapping peaks, preferred orientation (texture) and inaccurate RIR (reference intensity ratio) must be addressed to avoid errors
XRD Example  GIXRD on 50nm ZrO$_2$ on Glass

- Bragg-Brentano

Parallel Beam GIXRD
• In the case of a Si substrate, a single peak is measured in 2D to find the lattice parameter with temperature
• The linear coefficient of thermal expansion from 30°C to 1000°C is 1.9526x10^{-6}/C
• The literature value of the linear coefficient is ~2.6x10^{-6}/C but it does not take into consideration anisotropy
Wrap up

• Important to choose best techniques
  – Not all XRD is the same

• Difficult to compare systems and results on a systematic basis
  – Different optics, detectors….

• Real samples are complicated
  – Often need other techniques information to get to the “right” answer

• Acknowledgements
  – Dr. Colleen Frazer
    Director of X-Ray Characterization
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