X-Ray Diffraction From Easy to Hard

AVS TFUG Talk
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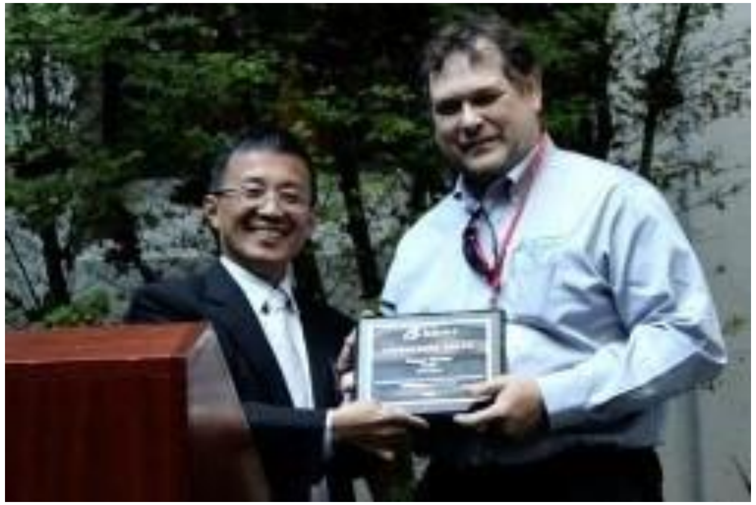

- PhD Solid State Physics , University of Waterloo, 1983
- Extensive experience in metrology technology and systems
  - Lead teams that designed
    - Optical tool UV to mid IR
    - High speed mapping x-ray
    - Photoluminescence
    - Non-contact electrical
    - Contact electrical tools

  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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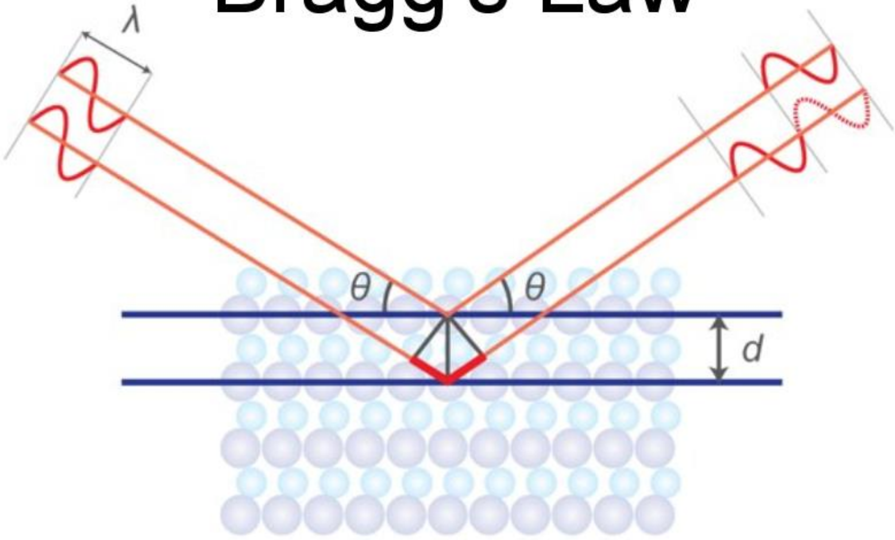
Slides

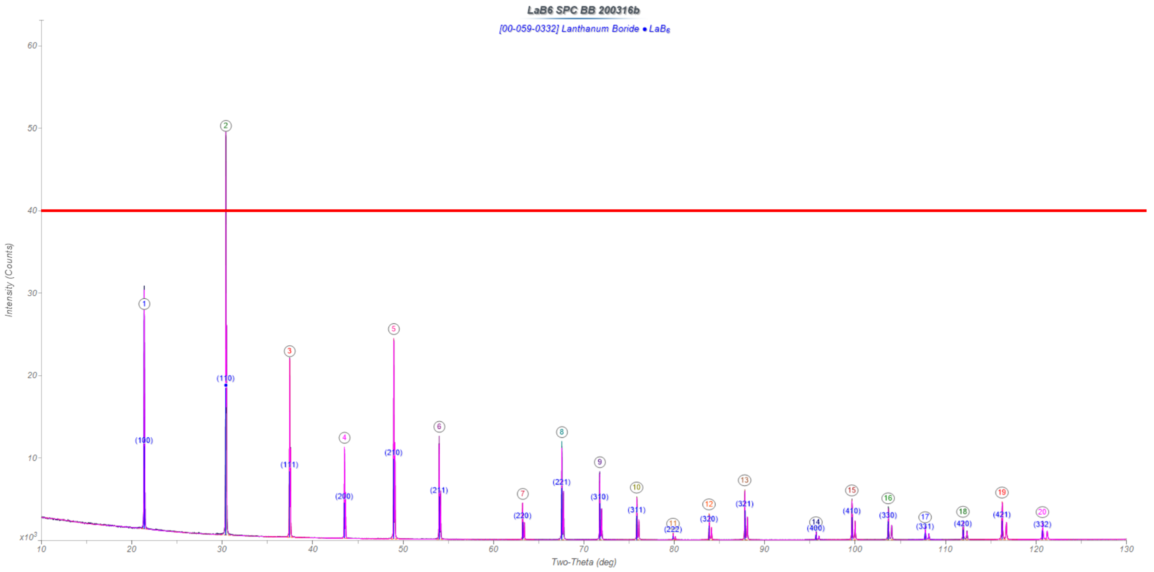
X-Ray Diffraction (the Basics)

Bragg's Law

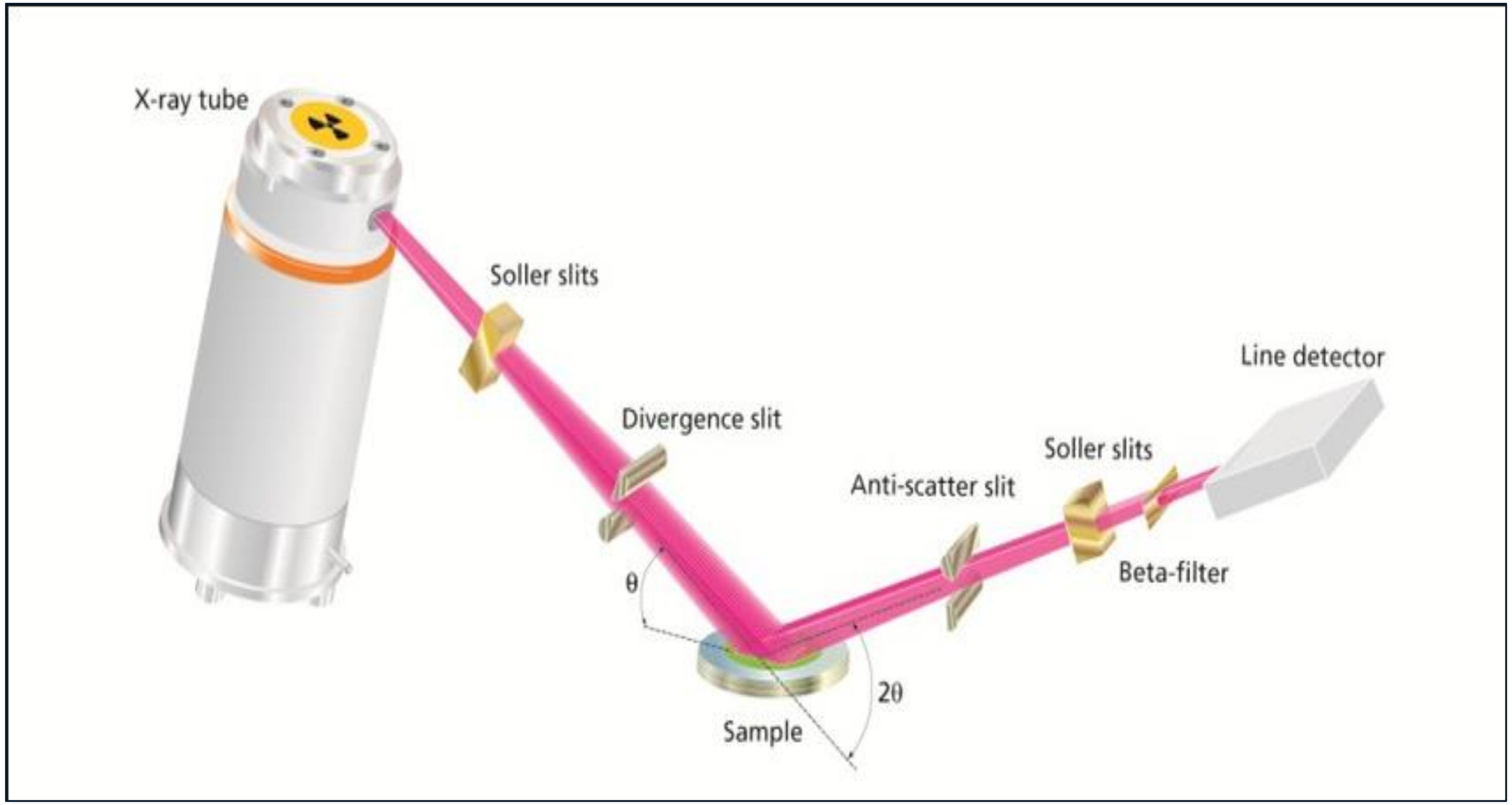


$$n\lambda = 2d \cdot \sin\theta$$

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

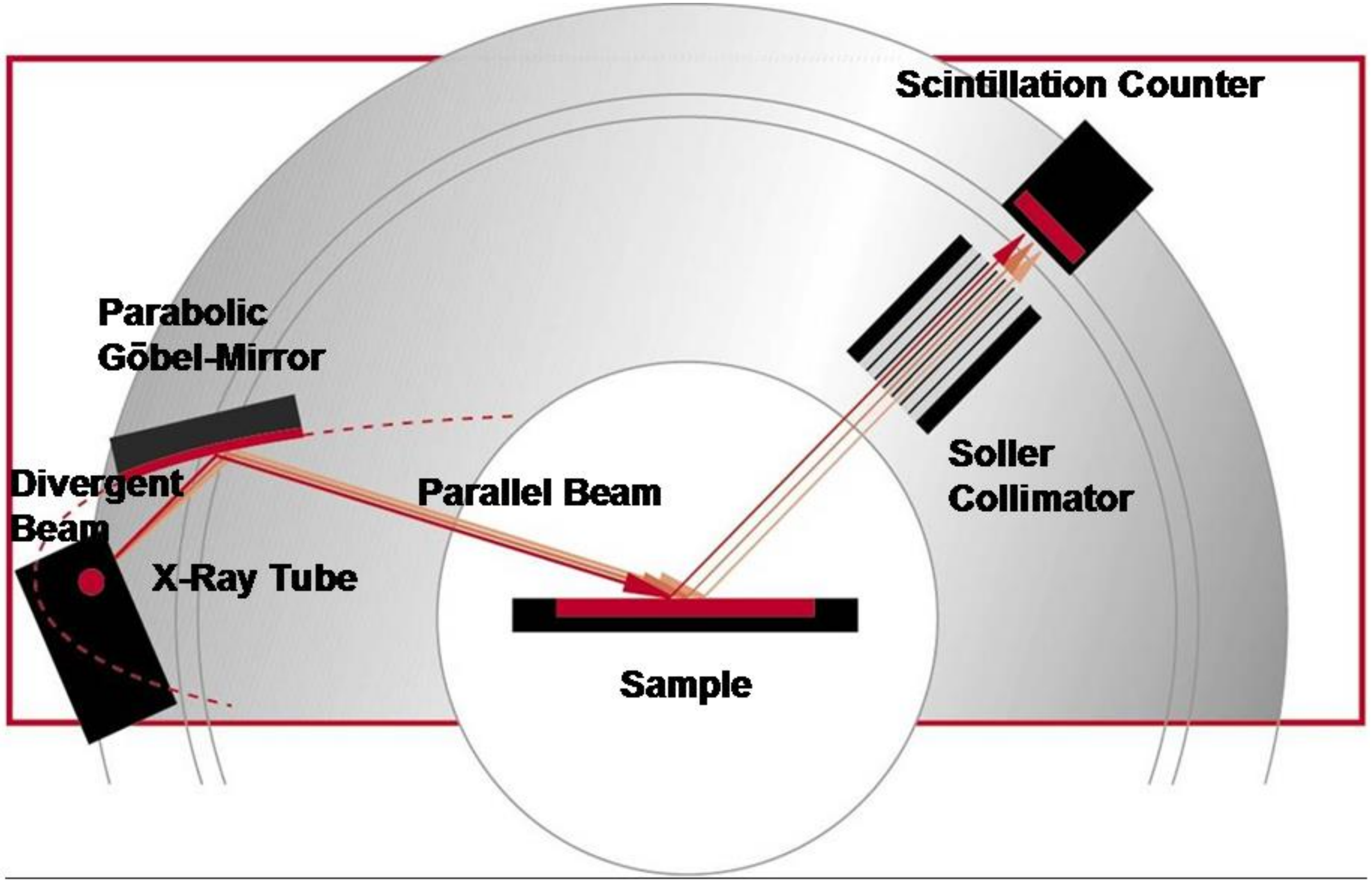


Bragg Brentano Geometry



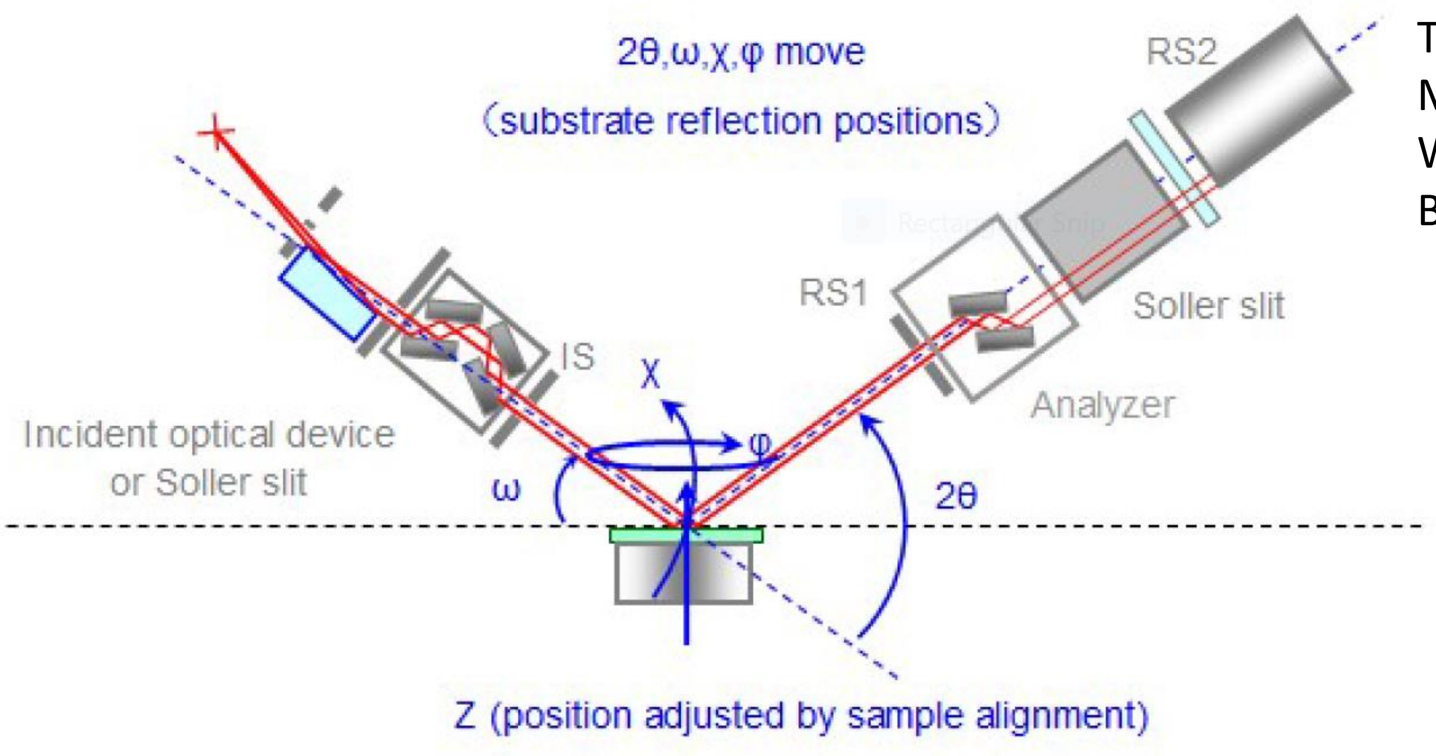
Typically source and detector move
High speed measurement
Need flat sample (or powder)

Parallel Beam Geometry



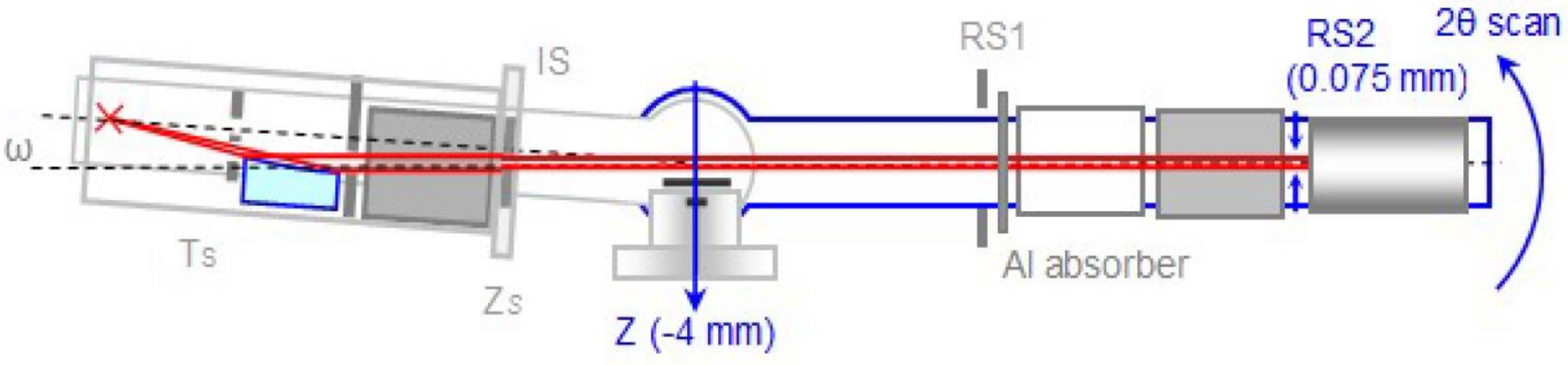
Typically source and detector move
Medium speed measurement
Works on rough samples

High Resolution Geometry



Typically source and detector move
Measurement speed highly sample dependent
Works best on smooth samples
Best resolution of all geometries

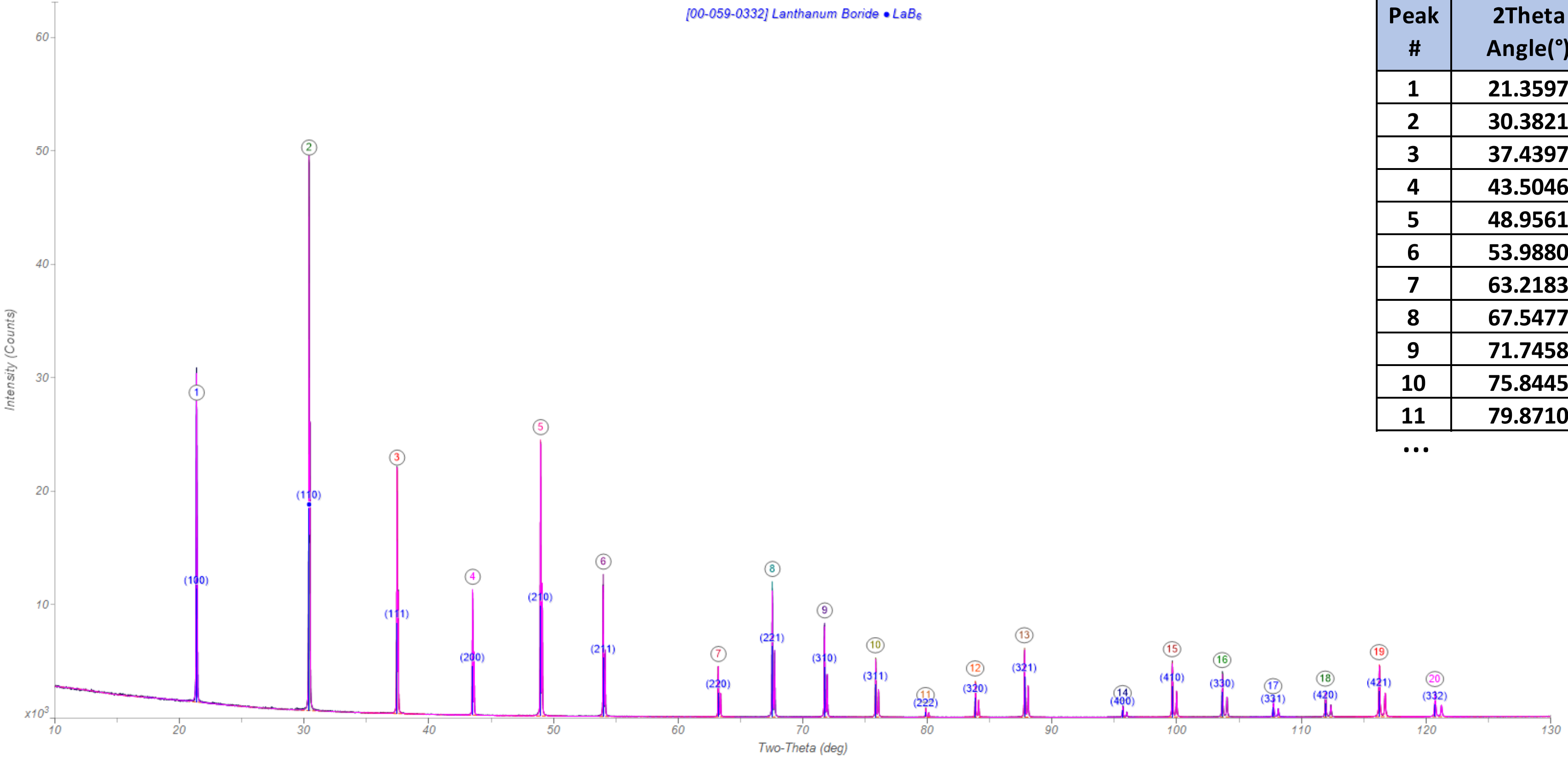
Grazing Incidence Geometry



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

NIST LaB_6 660c

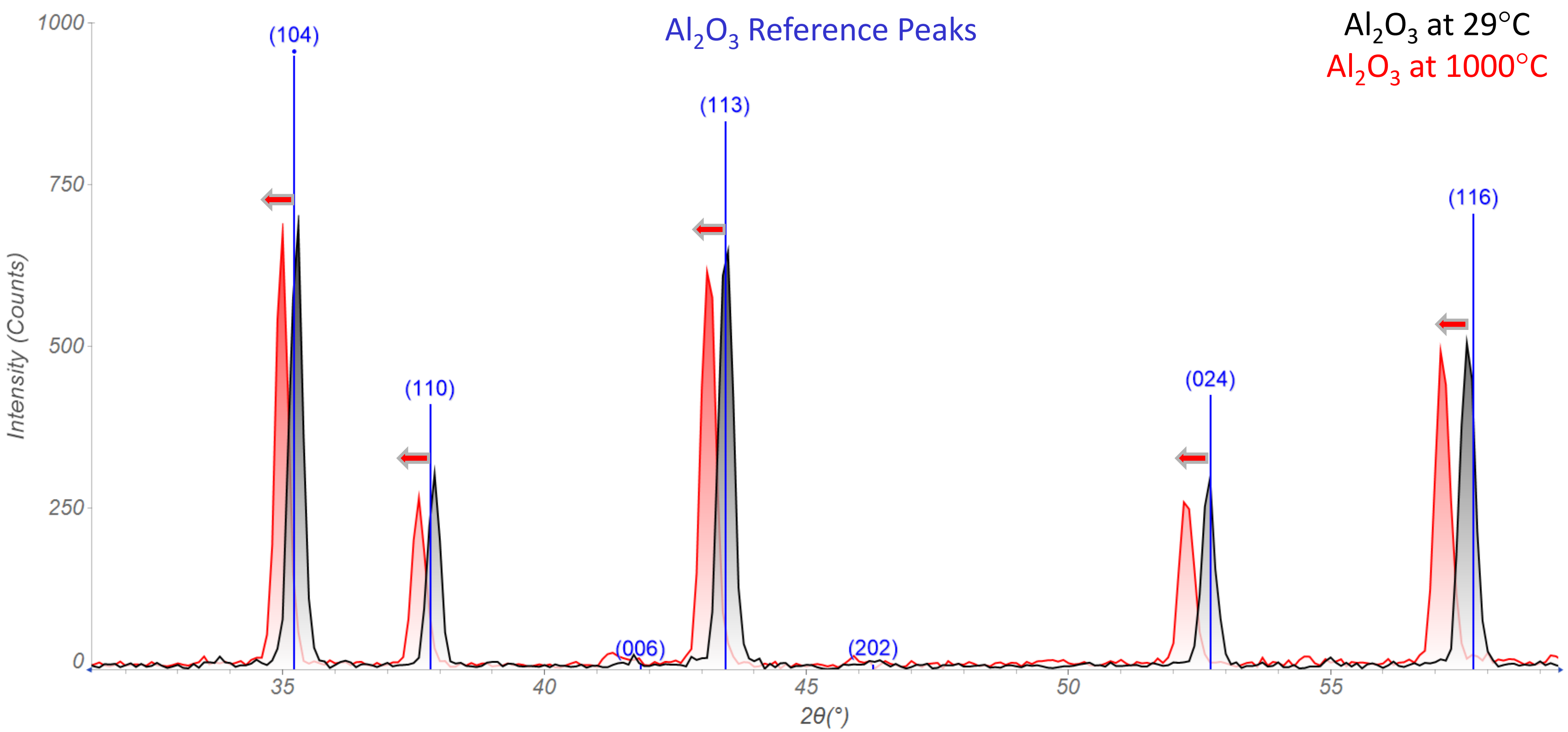


Peak #	2Theta Angle(°)	d(Å)	Height (cts)	Area (α1)	FWHM (°)	LaB6 {HKL}
1	21.3597	4.1566	26149.7	1761.9	0.0560	100
2	30.3821	2.9396	48502.6	2914.8	0.0482	110
3	37.4397	2.4001	21449.9	1264.6	0.0467	111
4	43.5046	2.0785	11083.7	655.2	0.0466	200
5	48.9561	1.8591	24338.9	1473.2	0.0465	210
6	53.9880	1.6971	12528.7	789.6	0.0478	211
7	63.2183	1.4697	4427.0	301.2	0.0507	220
8	67.5477	1.3856	11928.5	828.3	0.0514	221
9	71.7458	1.3145	8285.4	591.6	0.0517	310
10	75.8445	1.2533	5218.1	393.2	0.0541	311
11	79.8710	1.2000	838.6	70.6	0.0601	222

…

- The positions, heights, areas, and FWHM of the peaks can be found through profile fitting

XRD Example High Temperature Lattice Parameters

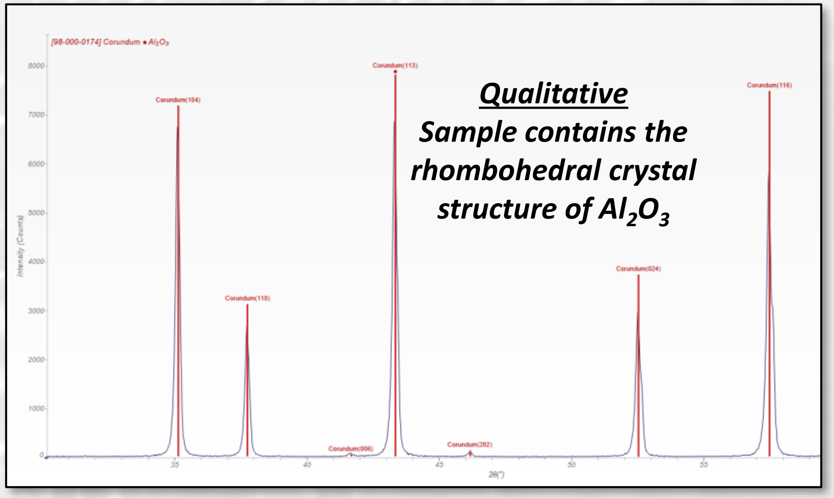


- The peak position shifts to lower angles with elevated temperature were used to calculate the change in interplanar spacings
- Whole-pattern fitting was used to calculate lattice parameters for Al_2O_3 powder at 29°C and 1000°C
- The calculated thermal expansion of the Al_2O_3 powder between 29°C and 1000°C was within 1% of the theoretical value for the “a” lattice parameter and within 5% of the “c” lattice parameter

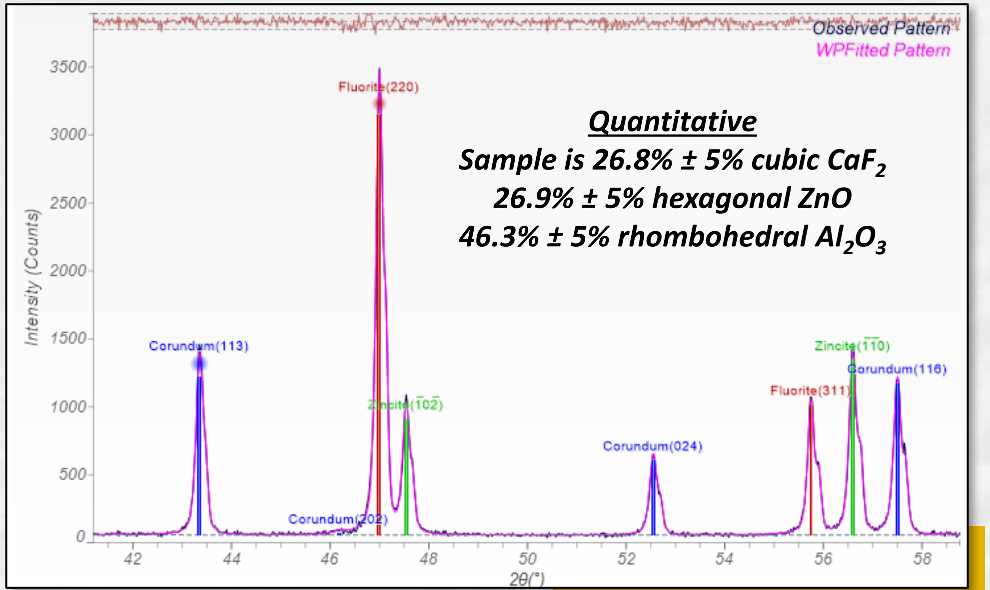
XRD Example Phase Identification

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!

Phases Detected in XRD	Possible Effects of Phases
Austenite phase in Martensite phase steel	increased fatigue strength, unwanted change in dimensions
Silicon Carbide polytypes	unpredictable change in band gap, increase in electron mobility
CIGS phases for solar cells	increase or decrease of conversion efficiency



Qualitative
Sample contains the rhombohedral crystal structure of Al_2O_3



Quantitative
Sample is 26.8% ± 5% cubic CaF_2
26.9% ± 5% hexagonal ZnO
46.3% ± 5% rhombohedral Al_2O_3

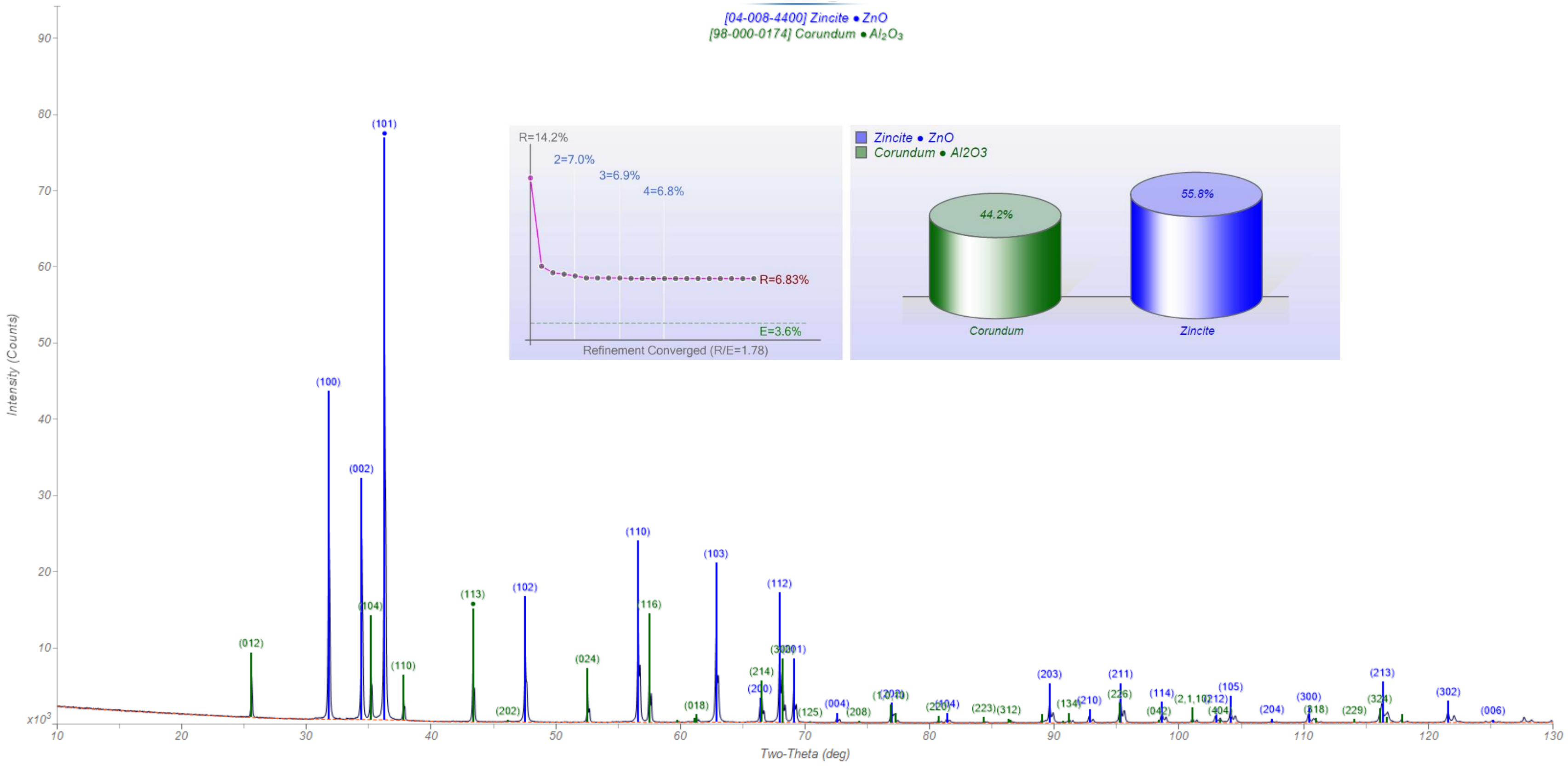
XRD phase identification:

- *Is highly repeatable*
- *Is non-destructive and fast*
- *Can detect phases in small amounts*
- *Needs an expert to model and analyze*

XRD scans courtesy of MDI…

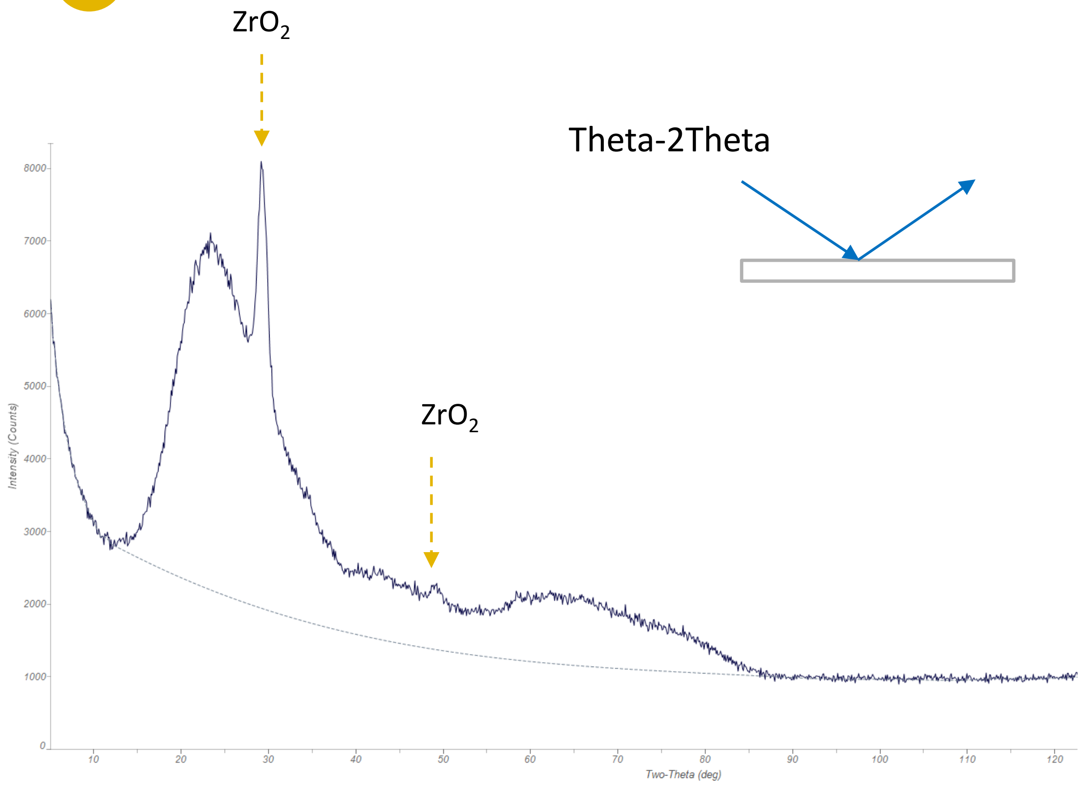
XRD Example Quantitative Phase Identification

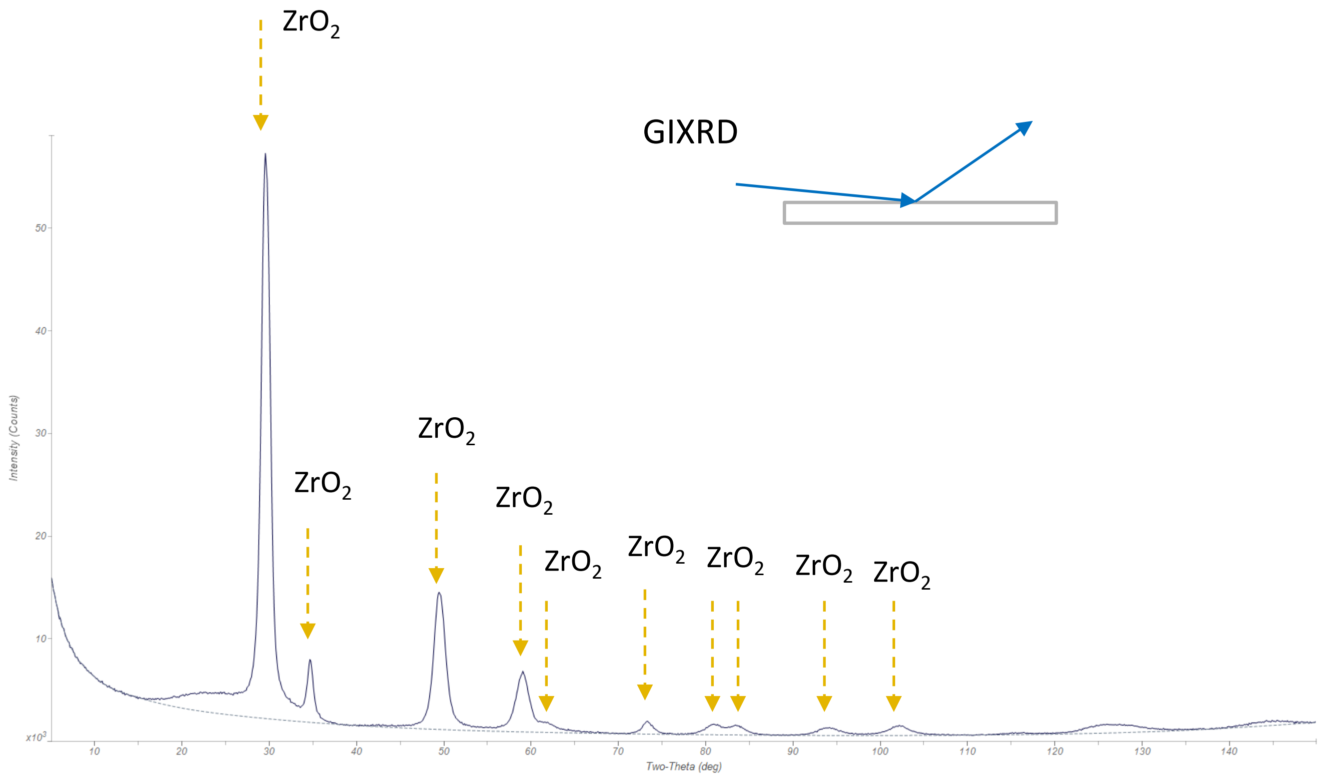
$ZnO + Al_2O_3$



- ZnO and Al_2O_3 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

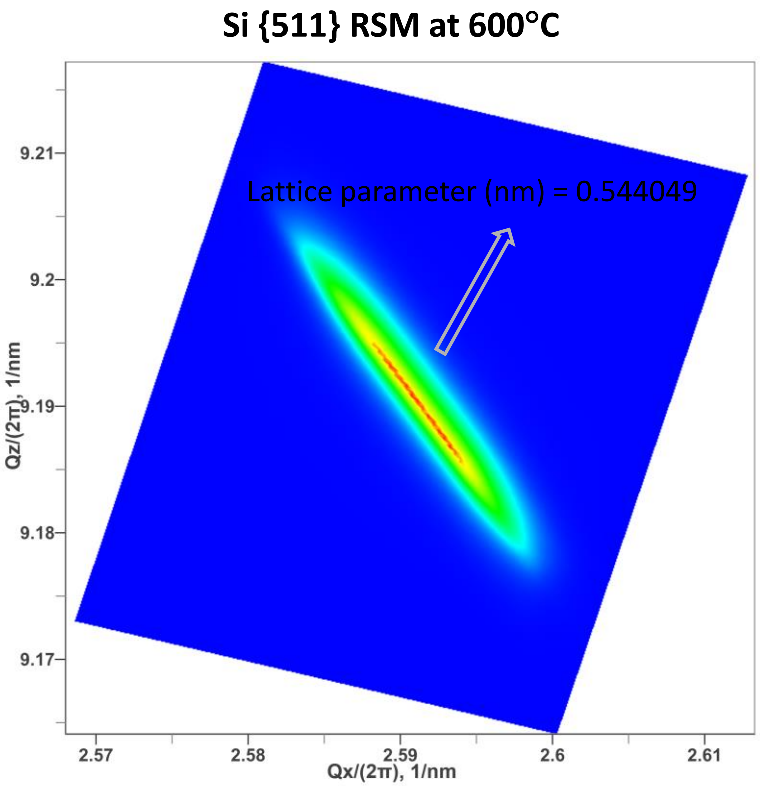


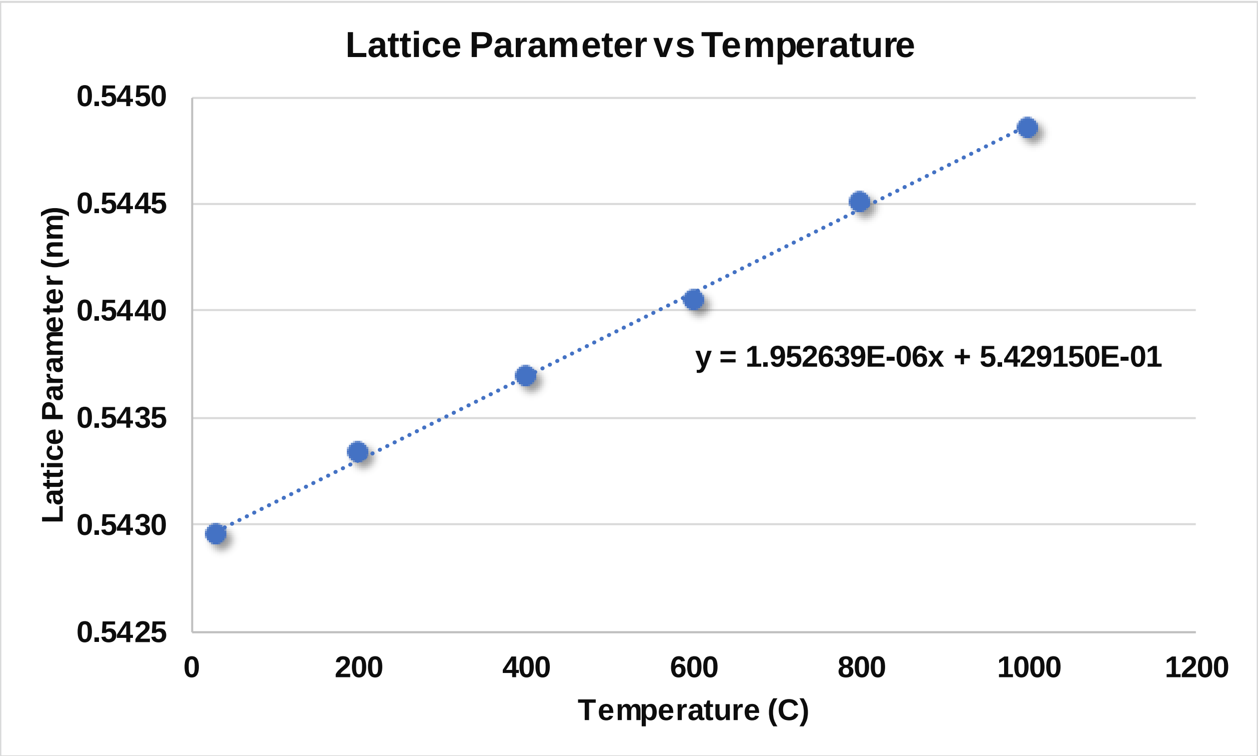


- Bragg-Brentano

Parallel Beam GIXRD

XRD Example High Temperature, High Resolution Mapping





- 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 1.9526×10^{-6}/C
- The literature value of the linear coefficient is ~2.6×10^{-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

    Covalent Metrology

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