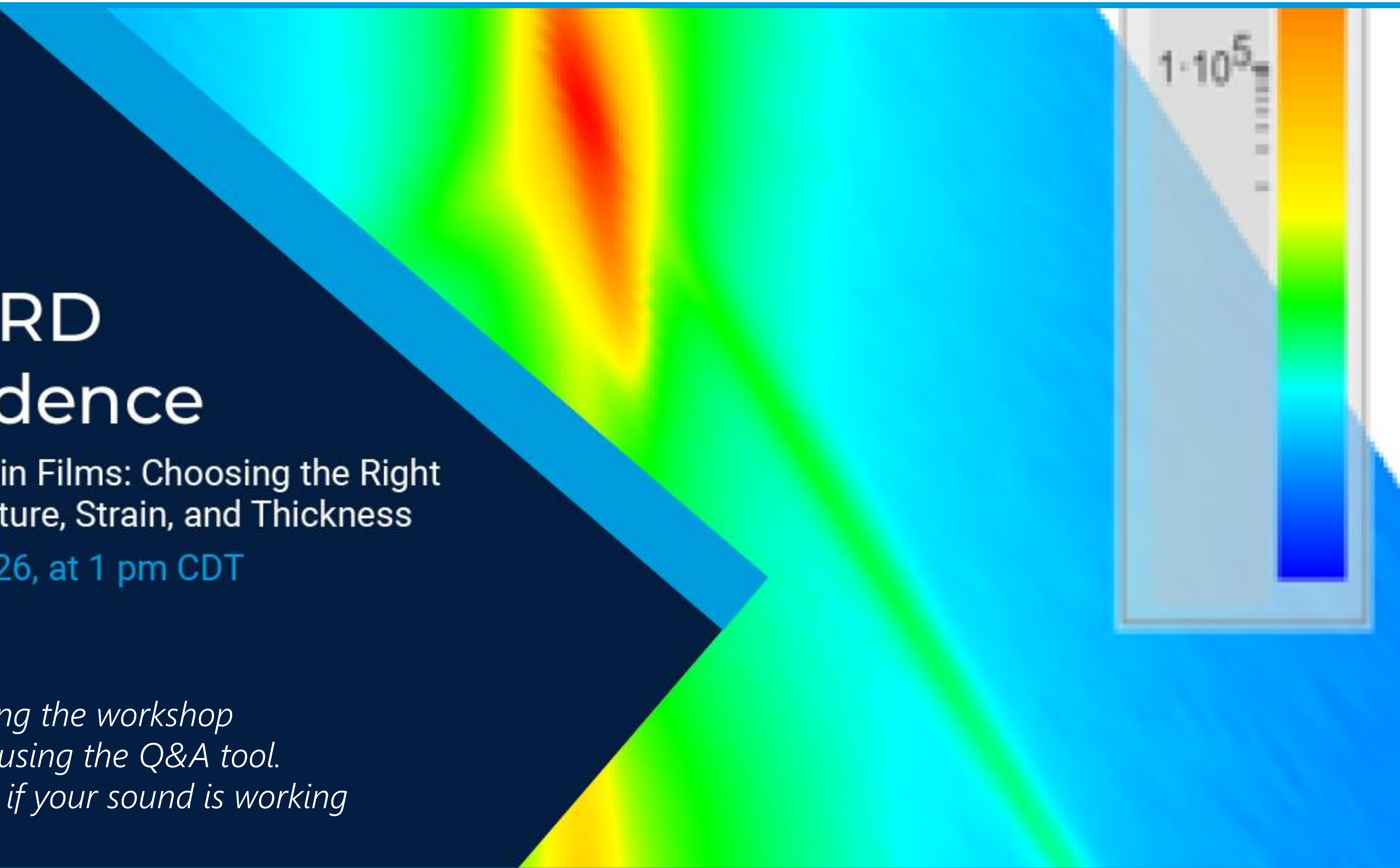
Practical XRD with Confidence

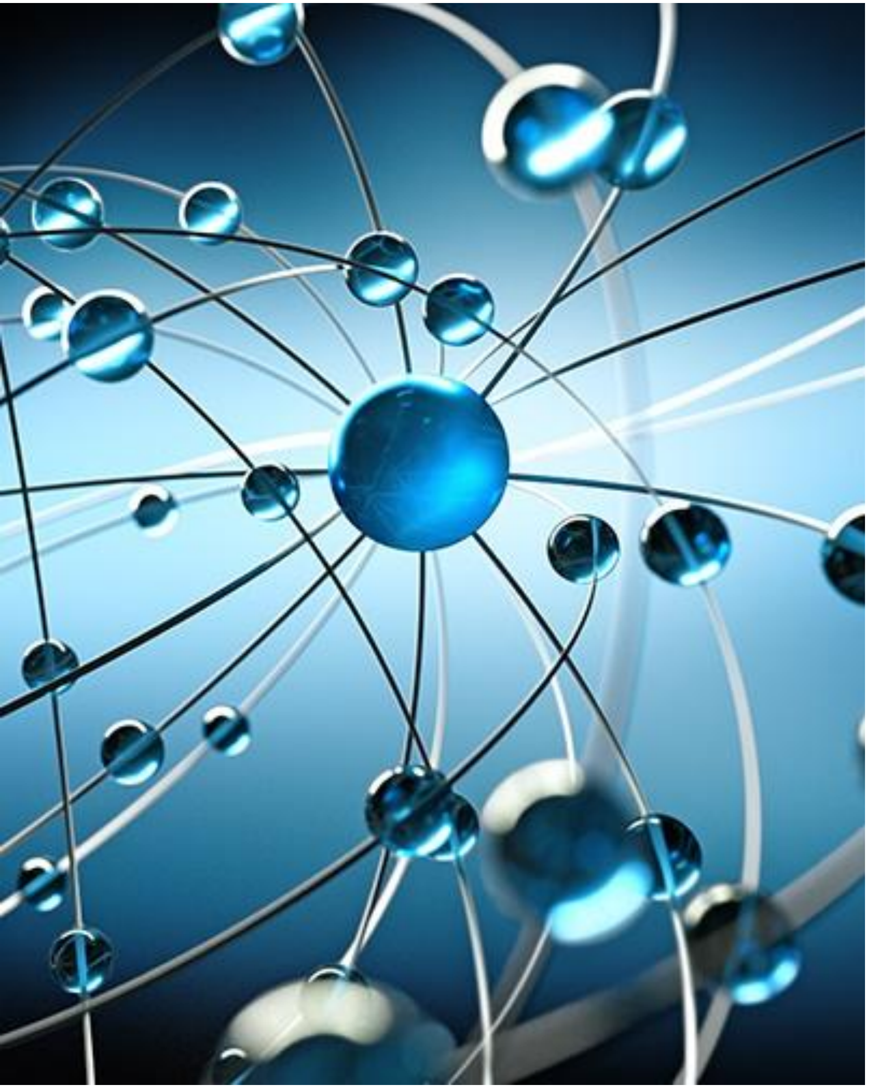
Episode 3 – XRD for Thin Films: Choosing the Right Measurement for Structure, Strain, and Thickness

Wednesday, June 3, 2026, at 1 pm CDT

Presenter: Keisuke Saito, PhD



- *You will be muted during the workshop*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working*

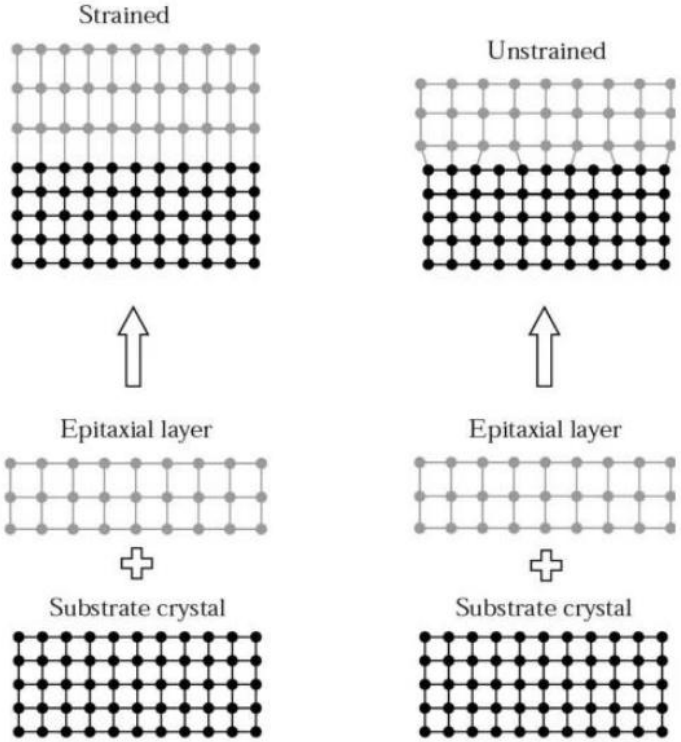


What I will cover today:

1. Preferred orientation of crystals
2. XRD scans for polycrystalline thin films
3. Phase identification of a 5 nm thick Fe–O thin film
4. HRXRD measurement types and instrument configurations
5. HRXRD scans in reciprocal space
6. Epitaxial perovskite thin films: structure and thickness

1. Preferred orientation of crystals

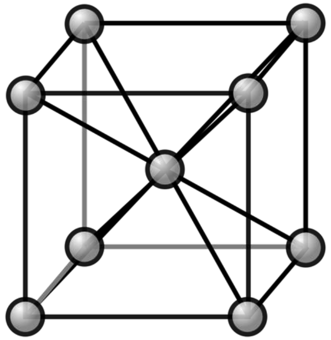
Thin film growth



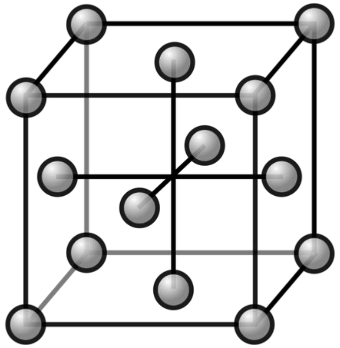
- Epitaxial growth: crystal-aligned thin film growth on a single-crystal substrate

Thin film growth

Body centered cubic



Face centered cubic



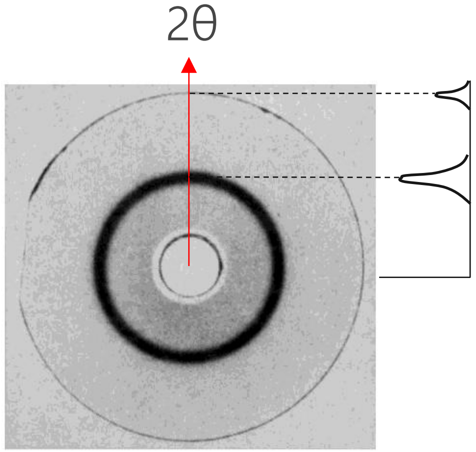
- Amorphous or mismatched substrates: lattice plane with lowest surface energy will orient ∥ to surface
  - Surface energy E_{hkl}

    B.C.C crystals: $E_{110} < E_{100} < E_{111}$

    F.C.C crystals: $E_{111} < E_{100} < E_{110}$

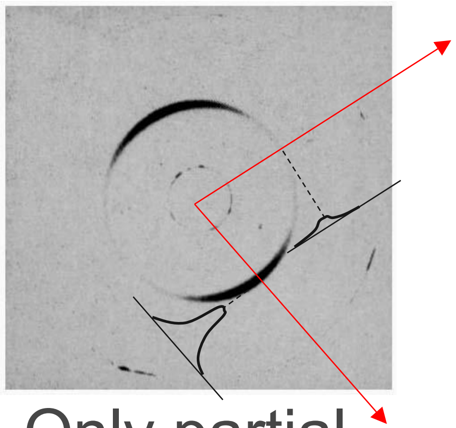
X-ray diffraction pattern & crystal orientation

Powder



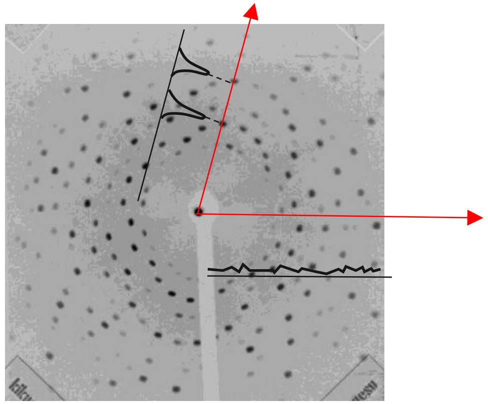
Full Debye ring

Crystals with orientation



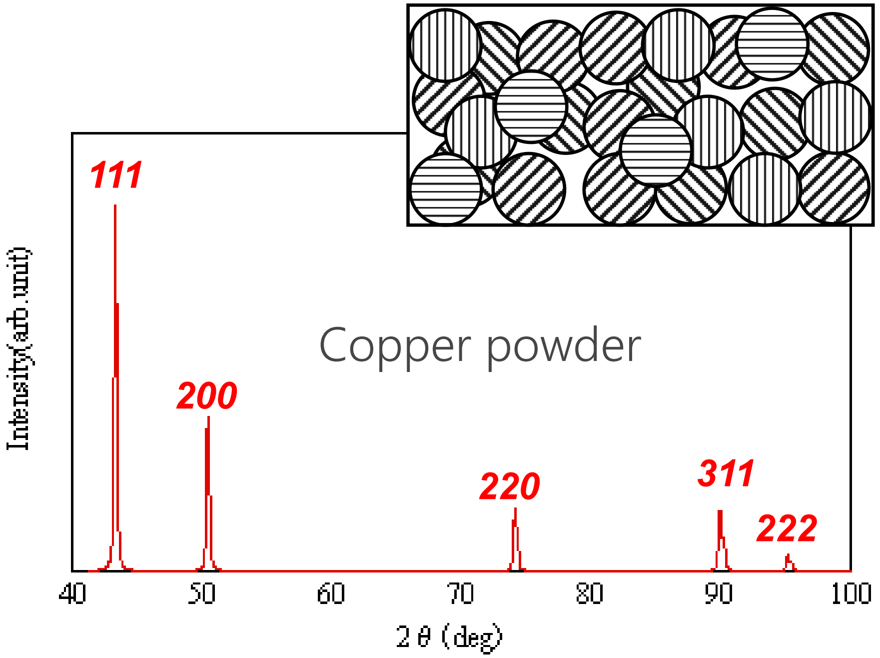
Only partial rings visible

Single crystal or epitaxial film

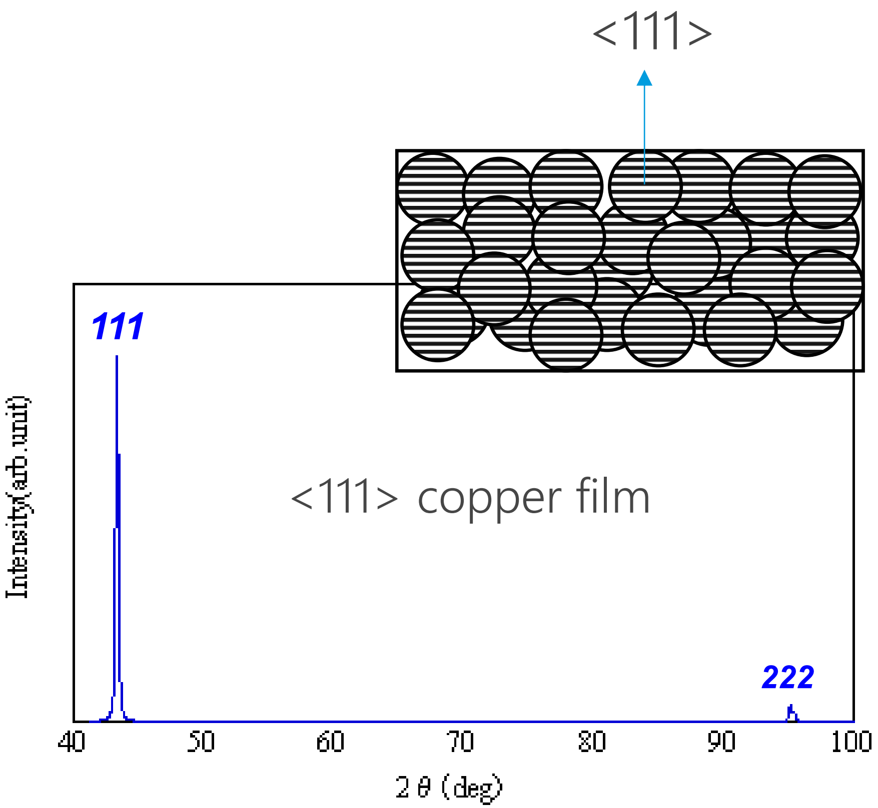


Sharp spots

Preferred orientation



No preferred orientation

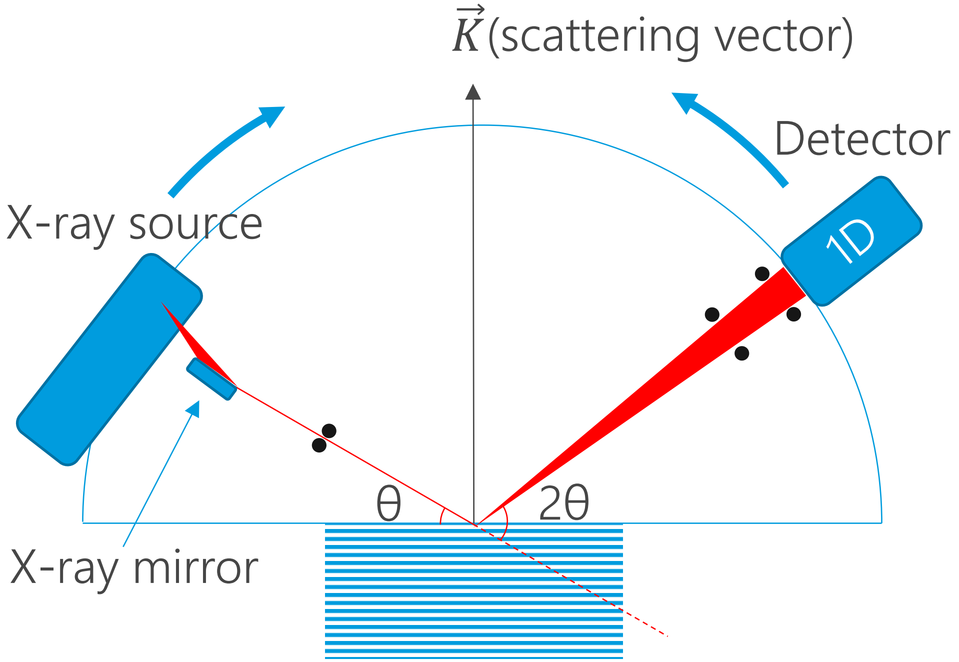


With preferred orientation

▨ Grain

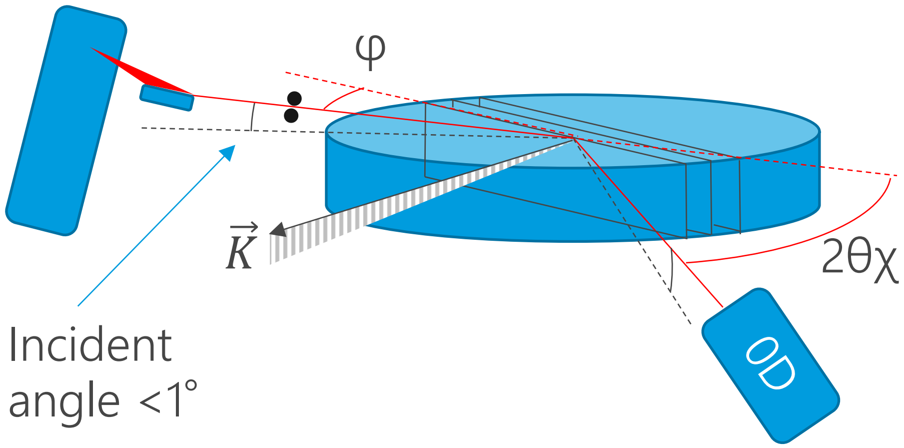
2. XRD scans for polycrystalline thin films

Symmetric 2θ-θ scan



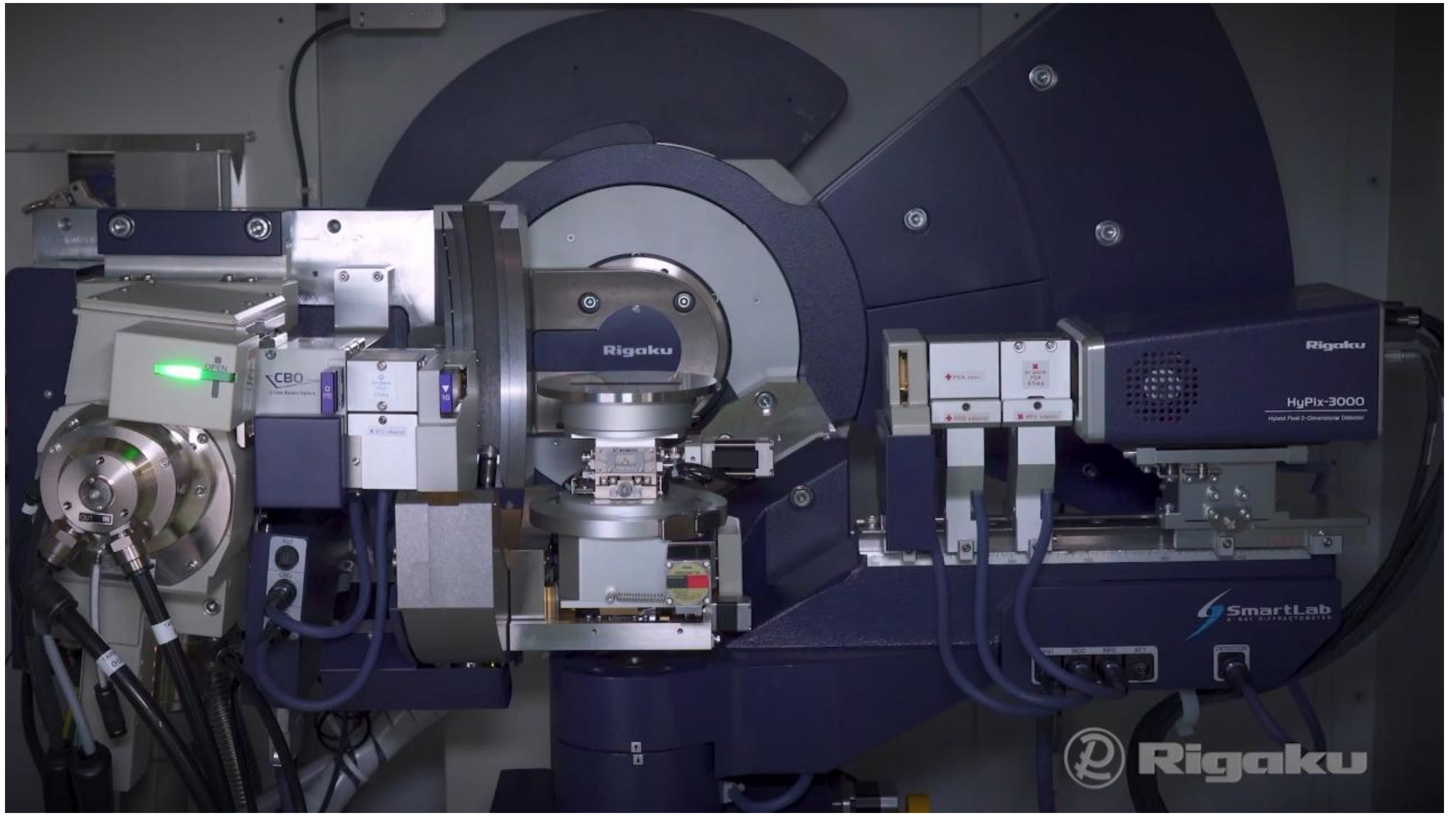
- d-spacings ⊥ from surface
- Preferred orientation
- Phase identification
- Large penetration depth of X-ray

In-plane scan (2θχ-φ scan)



- d-spacings ‖ to surface
- Preferred orientation ‖ to surface
- Phase identification ‖ to surface
- Depth profile

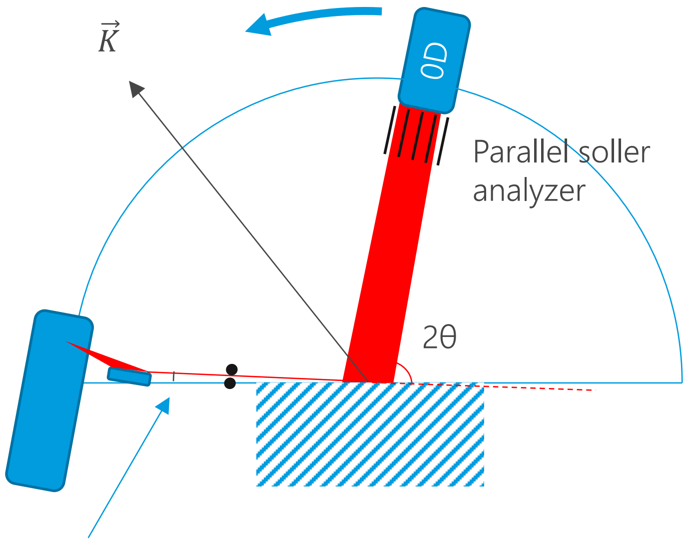
In-plane scan



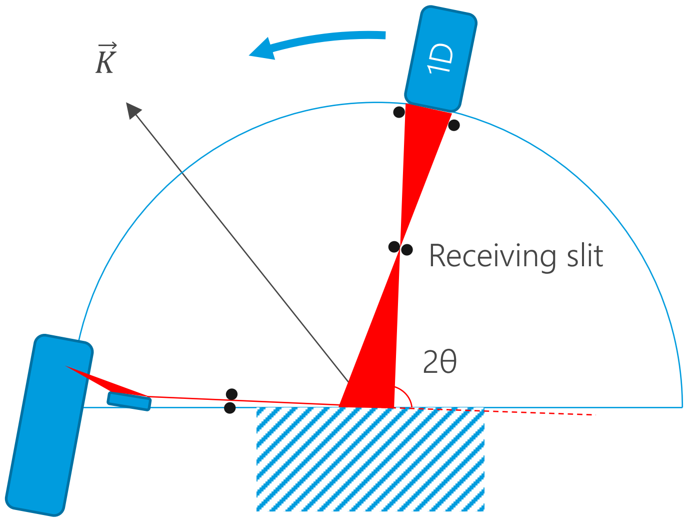
https://www.youtube.com/watch?v=rzC5amxVaf0&t=1s

Grazing incidence XRD (2θ scan)

Traditional PSA scan



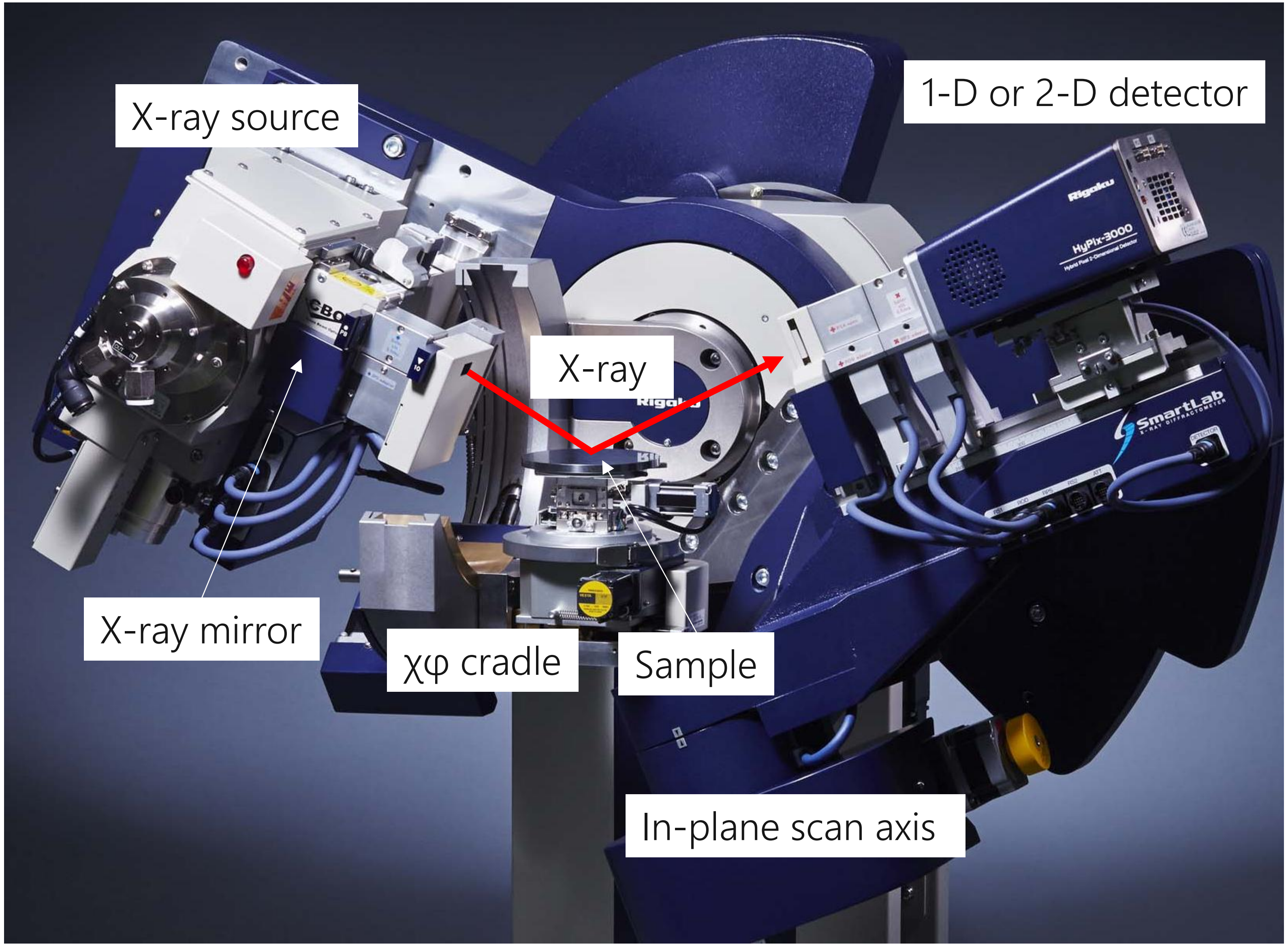
1D fast GID scan



Next-Gen GI-XRD Technology Cuts Scan Times to Minutes

- d-spacings from tilted direction
- Tilt angle depends on 2θ
- Phase identification for non-textured thin film

Configurations

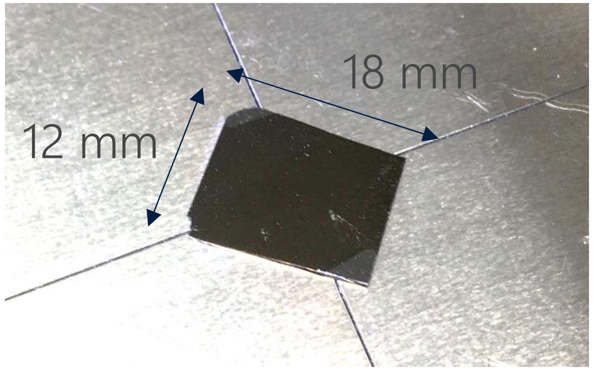


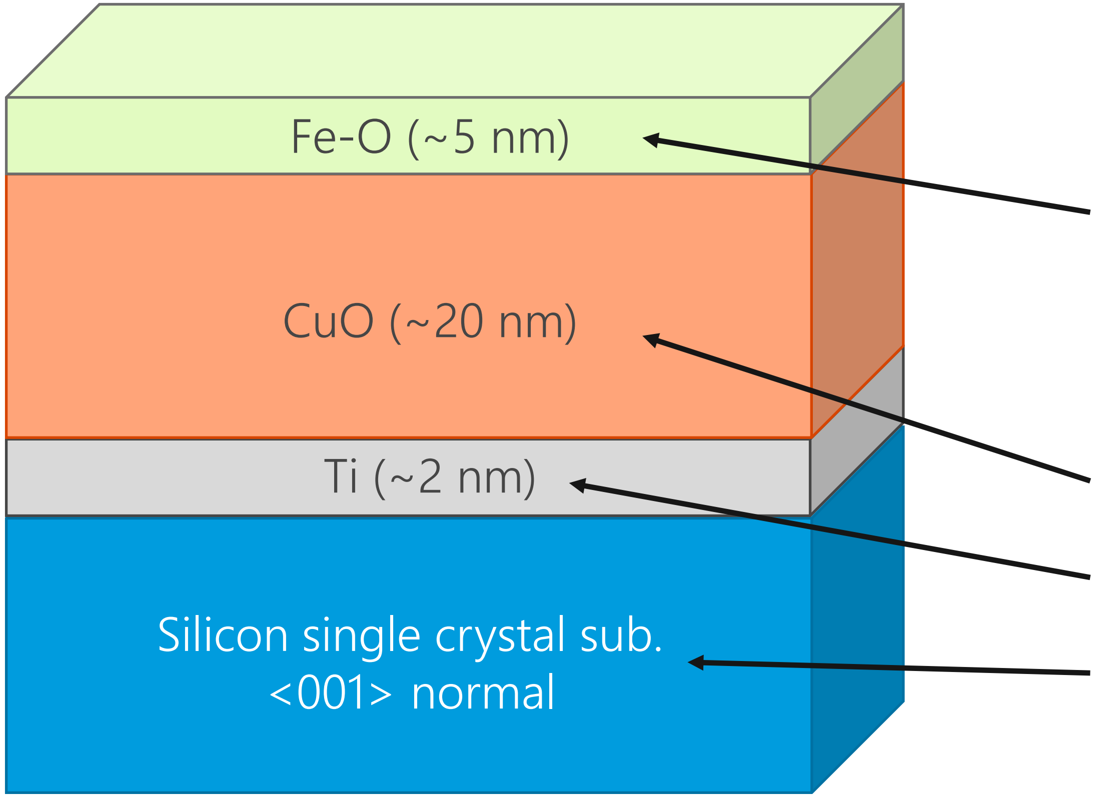
2D detector

- Hybrid pixel array detector
- Attenuator free
- Pixel resolution ~ 0.02°
- >29° (2θ) coverage

3. Phase identification of a 5 nm thick Fe–O thin film

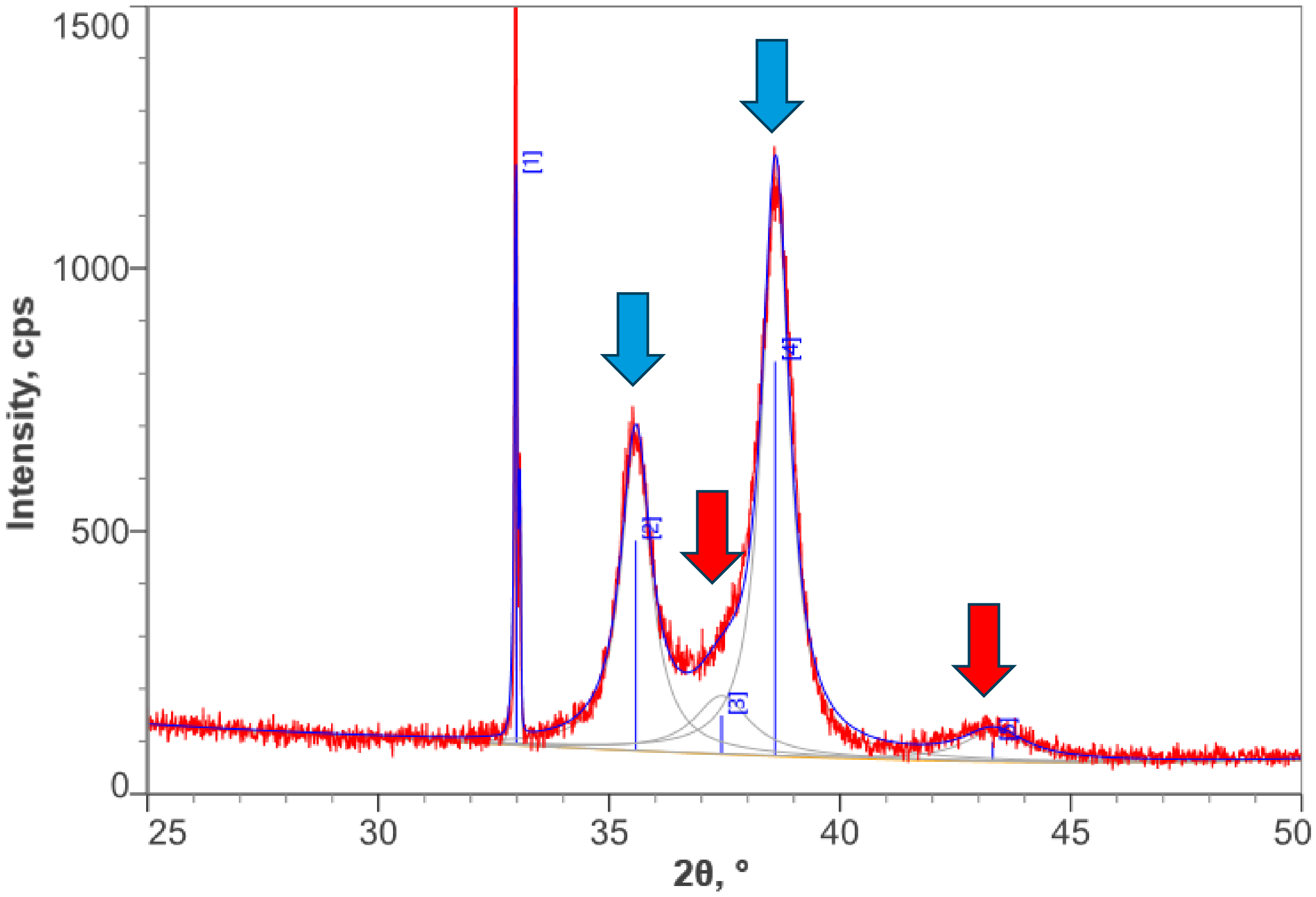
Sample structure





- Fe-O
  - FeO wüstite
  - Fe_2O_3 hematite
  - Fe_2O_3 maghemite
  - Fe_3O_4 magnetite
- CuO: tenorite
- Ti: adhesive layer, amorphous
- Si: substrate

Symmetric 2θ-θ scan



- Peak FWHM ➔ Crystallite size
  - Scherrer equation
- Peaks 2&4
  - Peak FWHMs ~ 0.9°
  - Crystallite sizes ~ 11 nm
- Peaks 3 & 5
  - Peak FWHMs ~ 1.9°
  - Crystallite sizes ~ 5 nm

Scherrer equation

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos\theta}$$

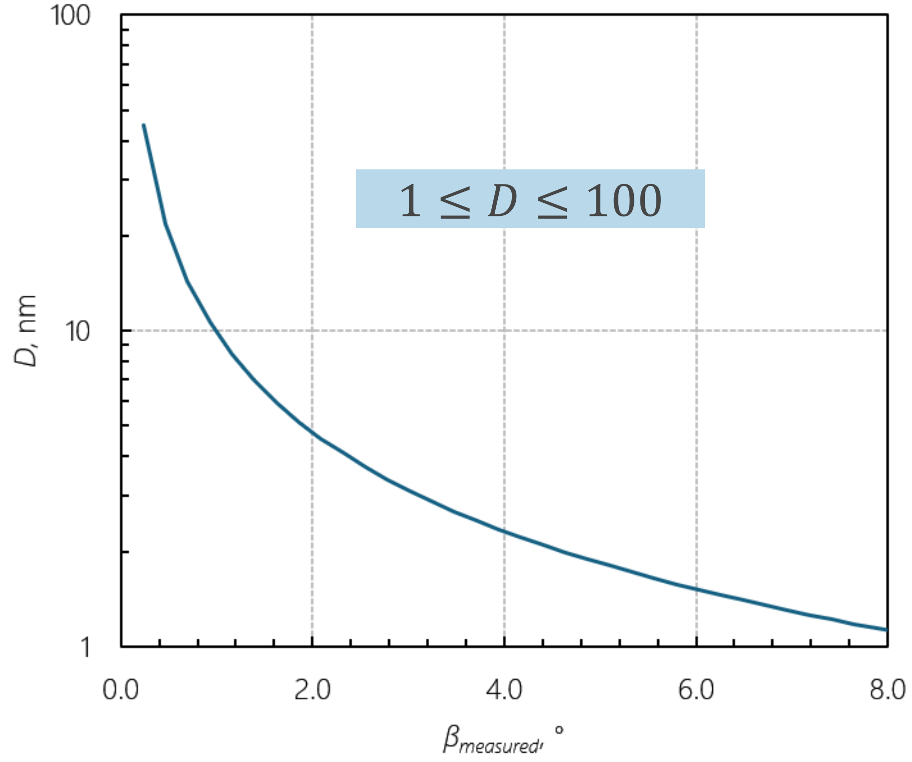
D: average crystallite size

K: shape factor (1.0 oriented thin film, 0.89 sphere)

λ: wavelength of X-ray

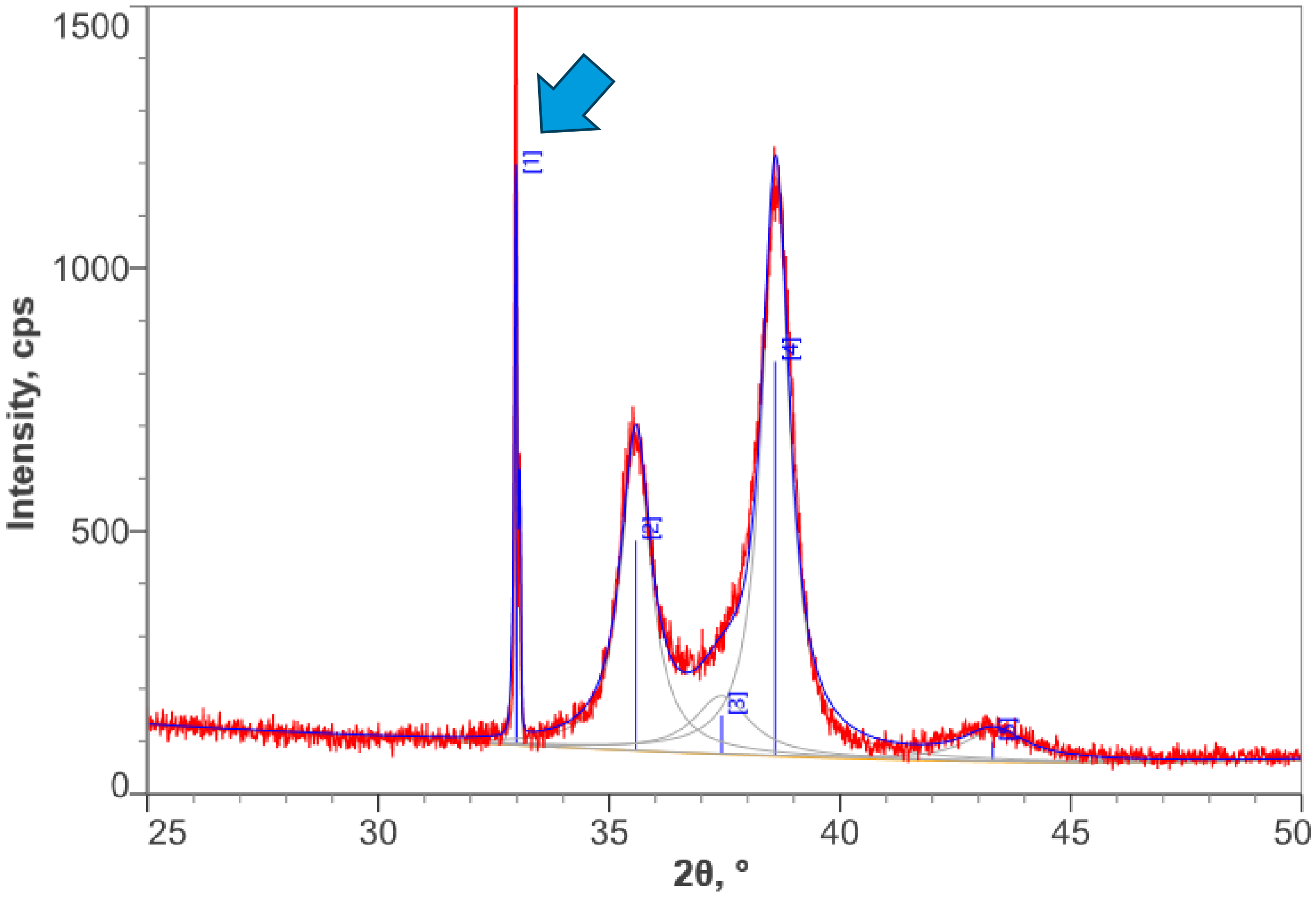
$\beta = \sqrt{\beta_{measured}^2 - \beta_{instrument}^2}$: FWHM of Gaussian

θ: Bragg angle

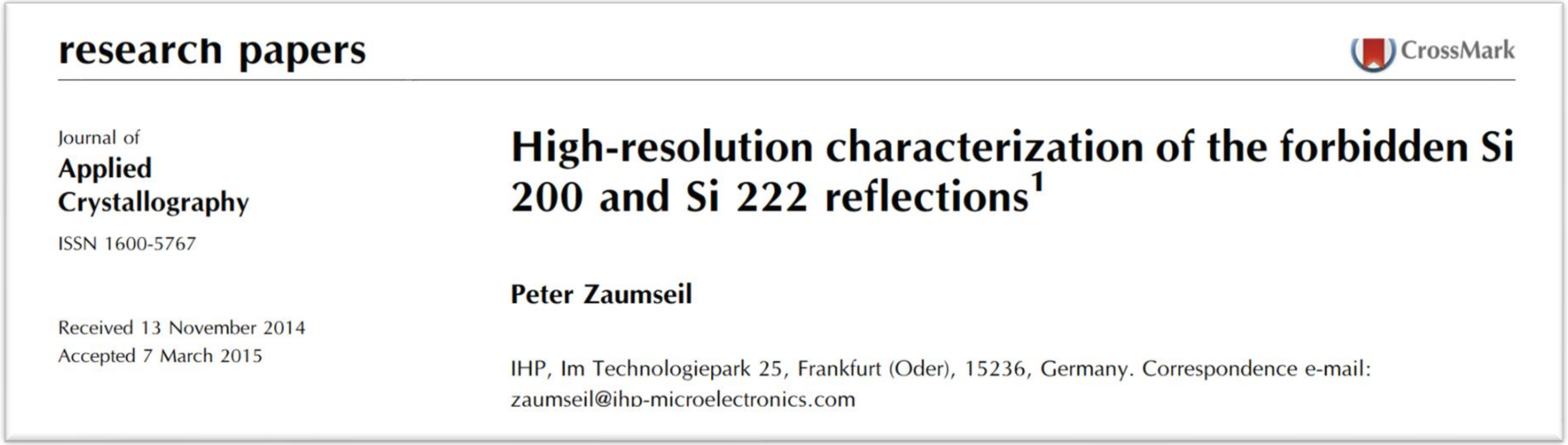


Peak FWHM β_m dependence of crystallite size D
β_I =0.2°, 2θ =40°, CuKα

Symmetric 2θ-θ scan



- Is peak 1 silicon *002, which is a forbidden diffraction?*



research papers

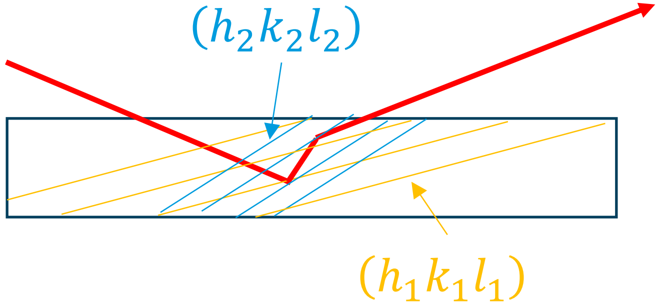
Journal of
**Applied
Crystallography**
ISSN 1600-5767

Received 13 November 2014
Accepted 7 March 2015

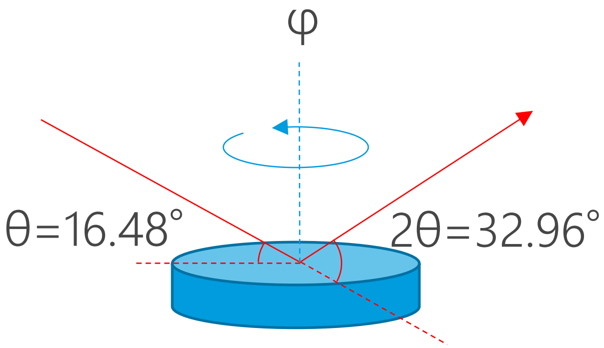
High-resolution characterization of the forbidden Si 200 and Si 222 reflections[1]

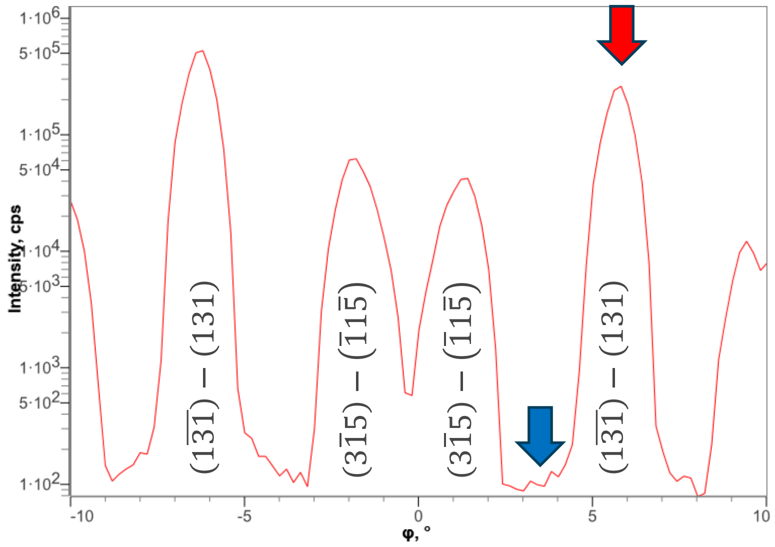
Peter Zaumseil

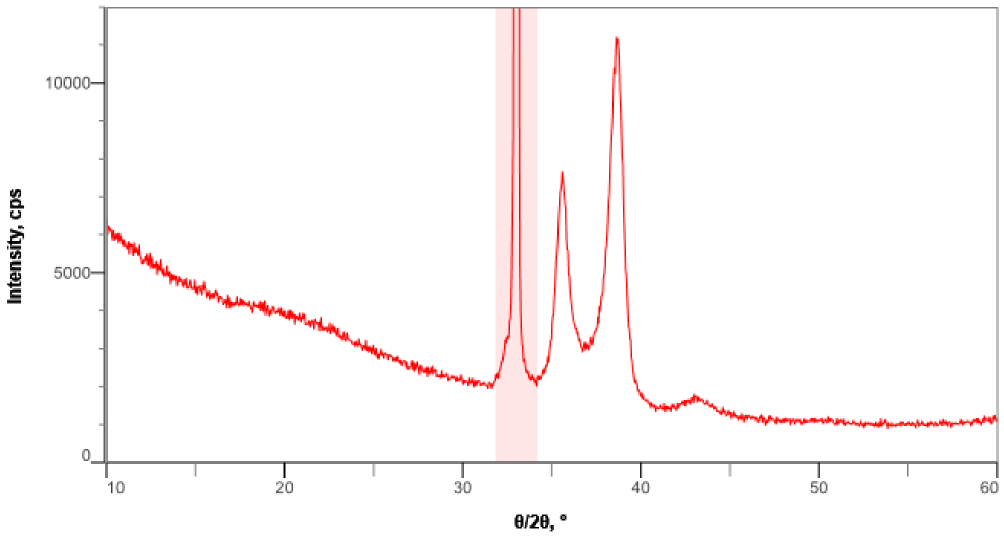
IHP, Im Technologiepark 25, Frankfurt (Oder), 15236, Germany. Correspondence e-mail: zaumseil@ihp-microelectronics.com

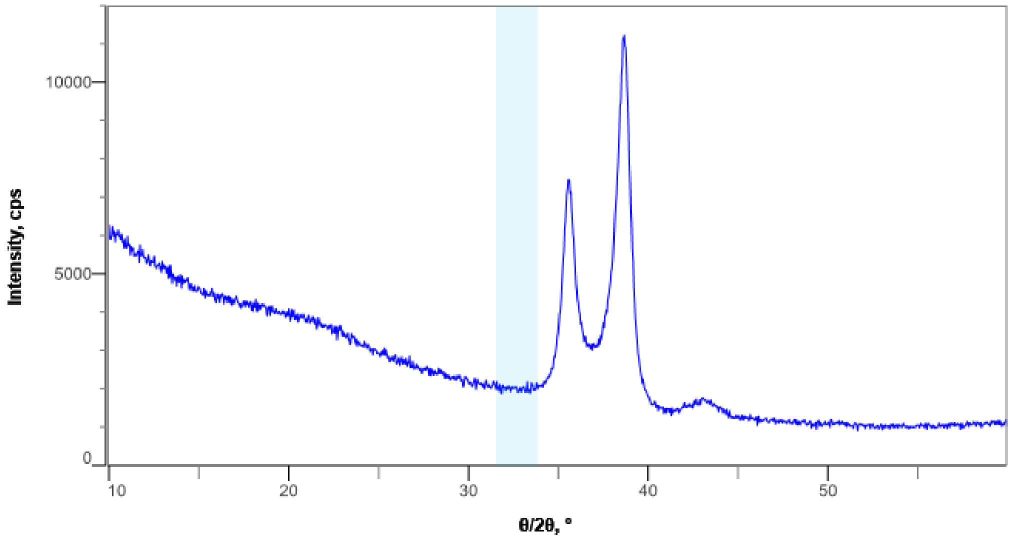


Multiple diffraction



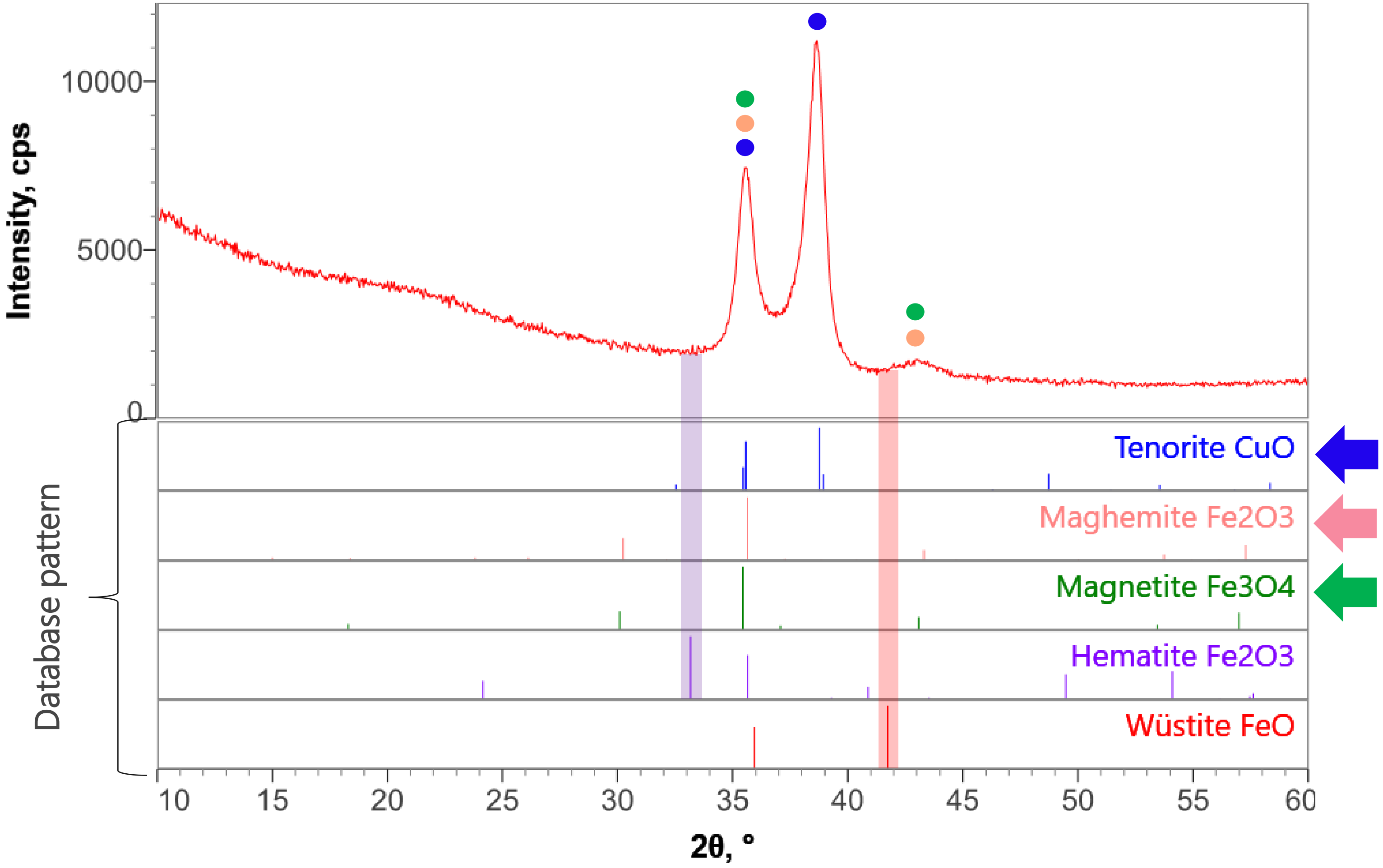






2θ-θ scan - phase identification

- Cu-O layer is Tenorite.
- Fe-O layer could be Maghemite or/and Magnetite.
- Can we probe surface layer only?



Probing surface layers with X-rays

- Penetration depth (D) of X-ray around θ_C

$$D = \frac{\lambda}{4\pi B}$$

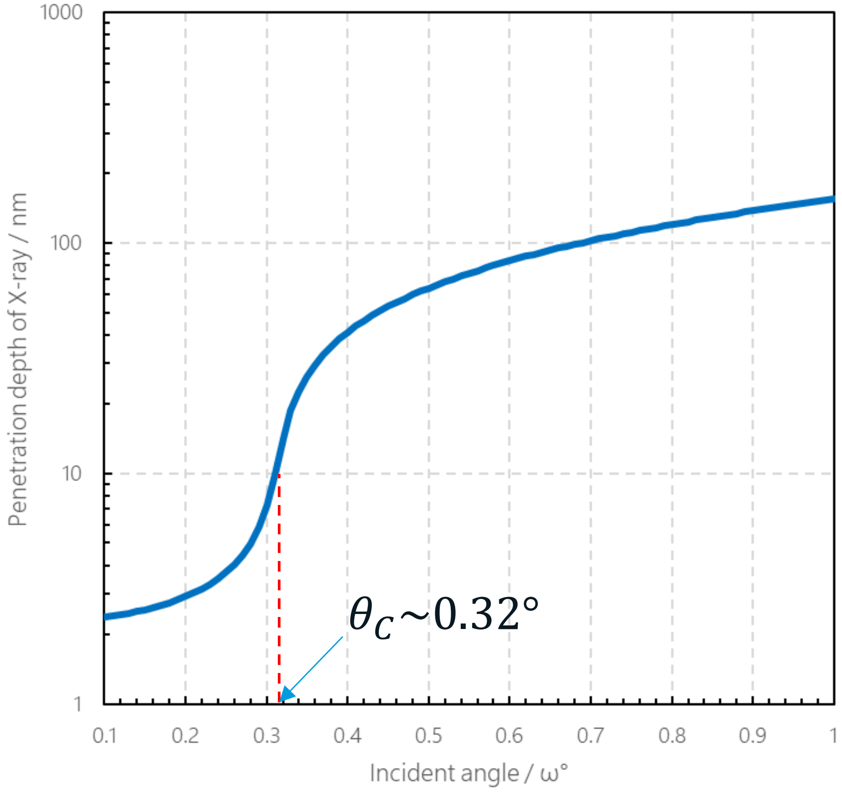
$$2B^2 = (-\omega^2 + 2\delta) + [(\omega^2 - 2\delta)^2 + 4\beta^2]^{1/2}$$

$\mathrm{n} = 1 - \delta + i\beta$: refractive index

$\theta_C \sim \sqrt{2\delta}$: critical angle for total reflection

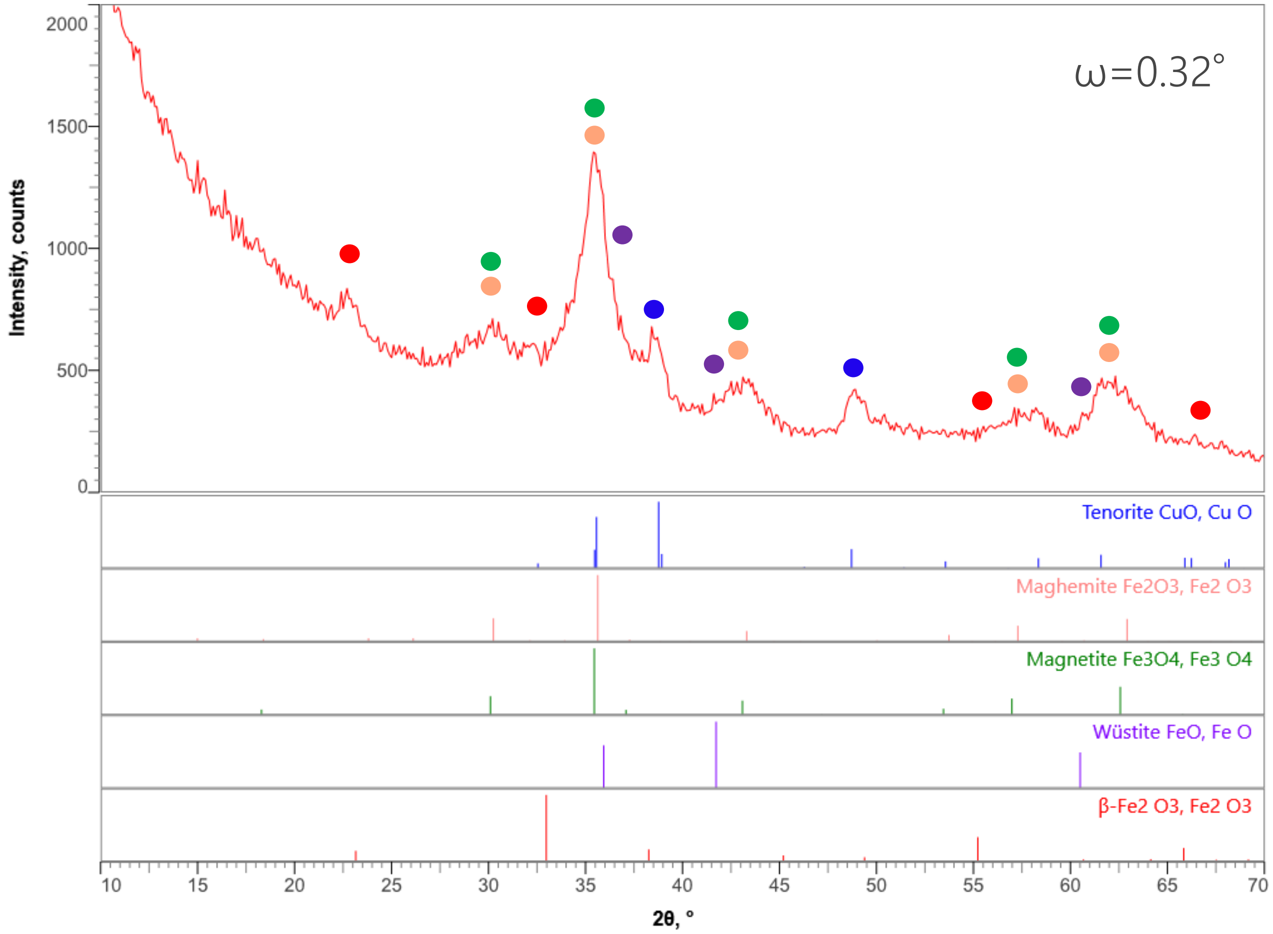
λ: wavelength of X-ray

ω: incident angle of X-ray



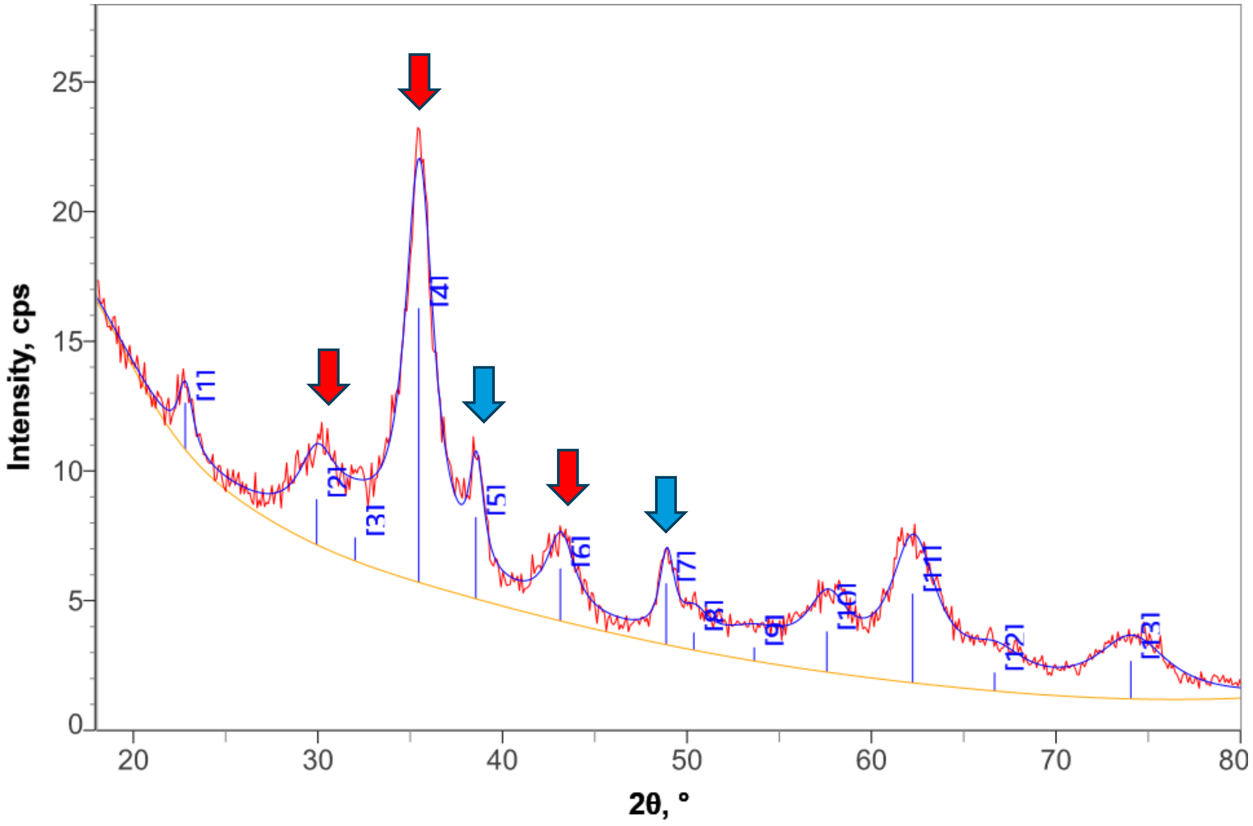
Penetration depth of Cu-$K\alpha$ X-ray for Fe_2O_3 Maghemite

In-plane scan & phase identification



- Small CuO peaks
- Fe-O layer
  - Maghemite
  - Magnetite
  - Wüstite
  - β-Fe_2O_3

In-plane XRD

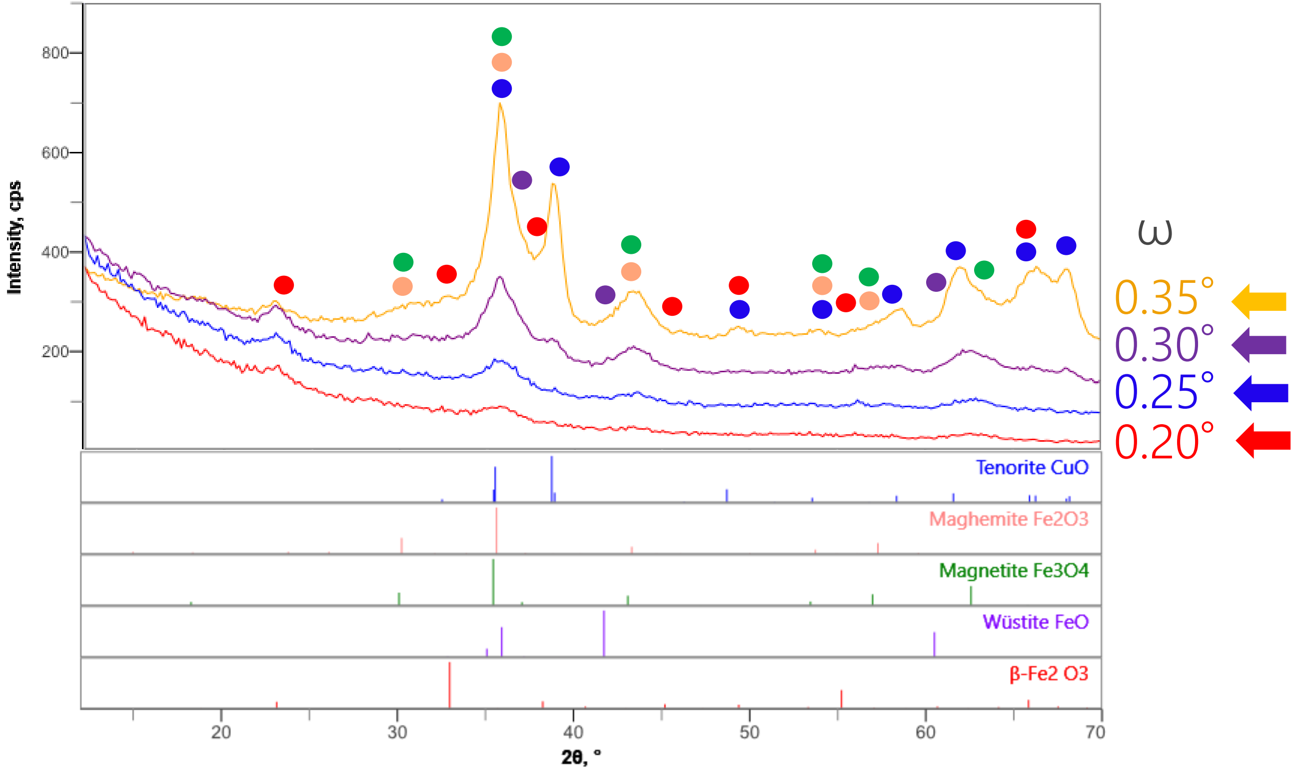


- Peak FWHM ➔ Crystallite size
  - Scherrer equation
- Peaks 5 & 7 (CuO)
  - Peak FWHMs ~ 0.9°
  - Crystallite sizes ~ 11 nm

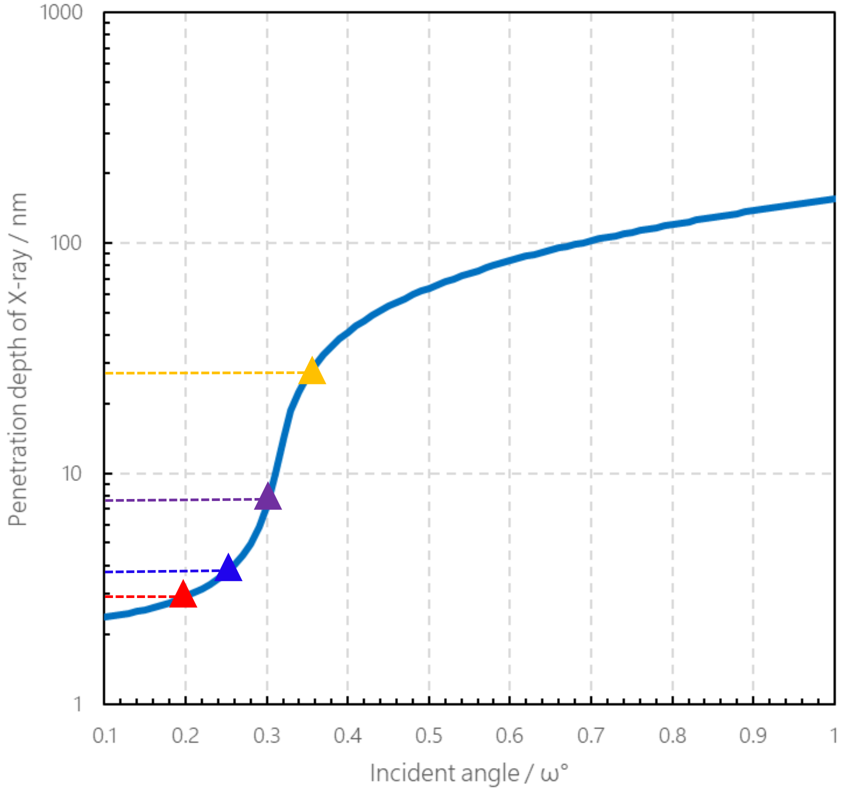
    ~Half thickness
- Peaks 2 & 4 & 6 (Fe-O)
  - Peak FWHMs ~ 2.3°
  - Crystallite sizes ~ 4 nm

    ~ Thickness

GIXRD – incident angle dependence



Penetration depth of Cu-*Kα* X-ray for Fe_2O_3 Maghemite

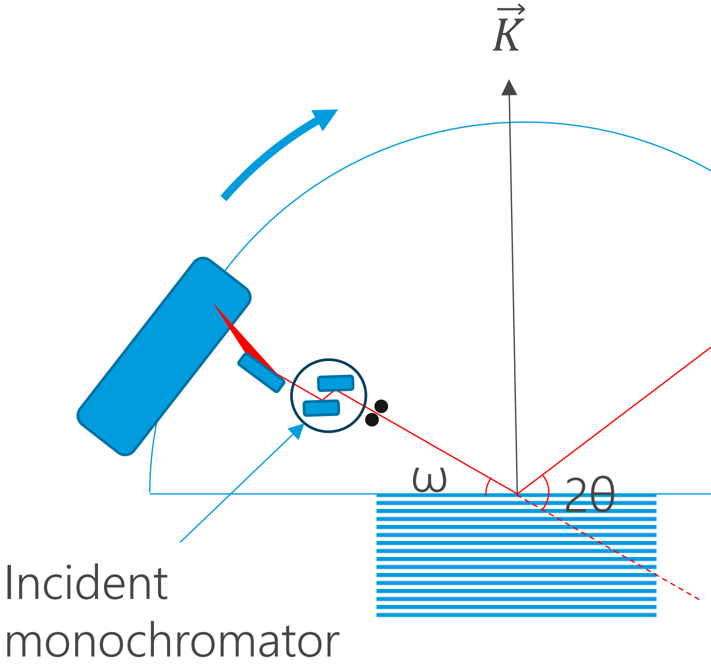


Summary

- 2θ-θ scan
  - Surface normal information
  - Not sensitive to thin layer(s) but should be done first (!)
- In-plane XRD
  - Sensitive to surface layer(s), depth profile
- Grazing incidence XRD
  - Same as in-plane scan but only applicable on non-textured thin films

4. High-resolution X-ray diffraction measurement types & instrument configurations

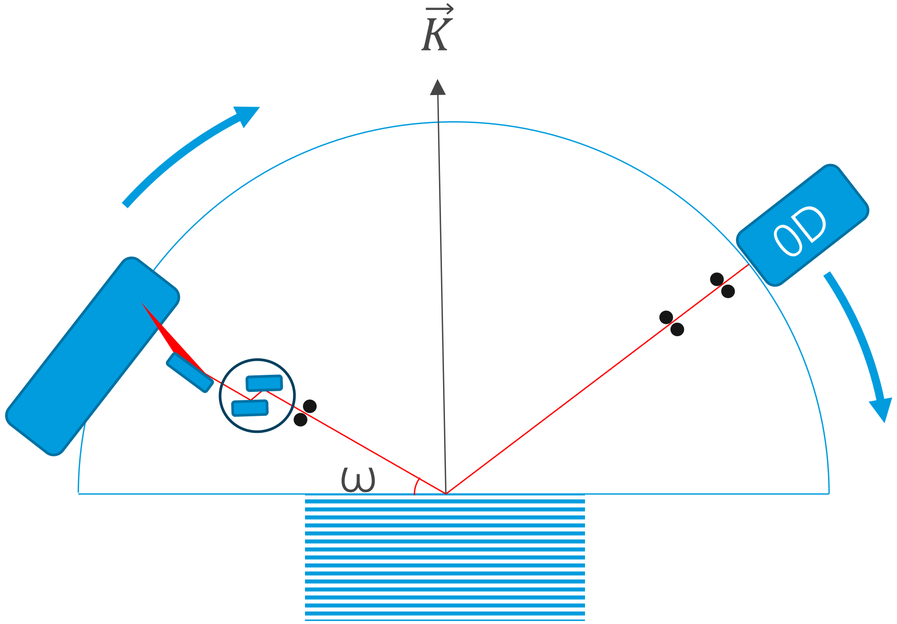
Symmetric 2θ-ω scan, ω=θ



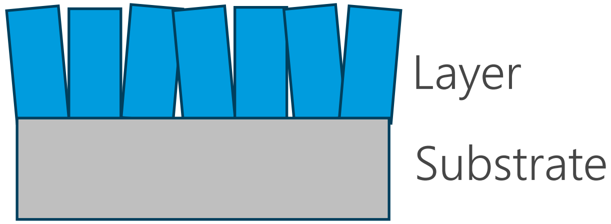
Incident monochromator

- d-spacings ⊥ from surface
- Lattice mismatch
- Thickness from fringes / satellites

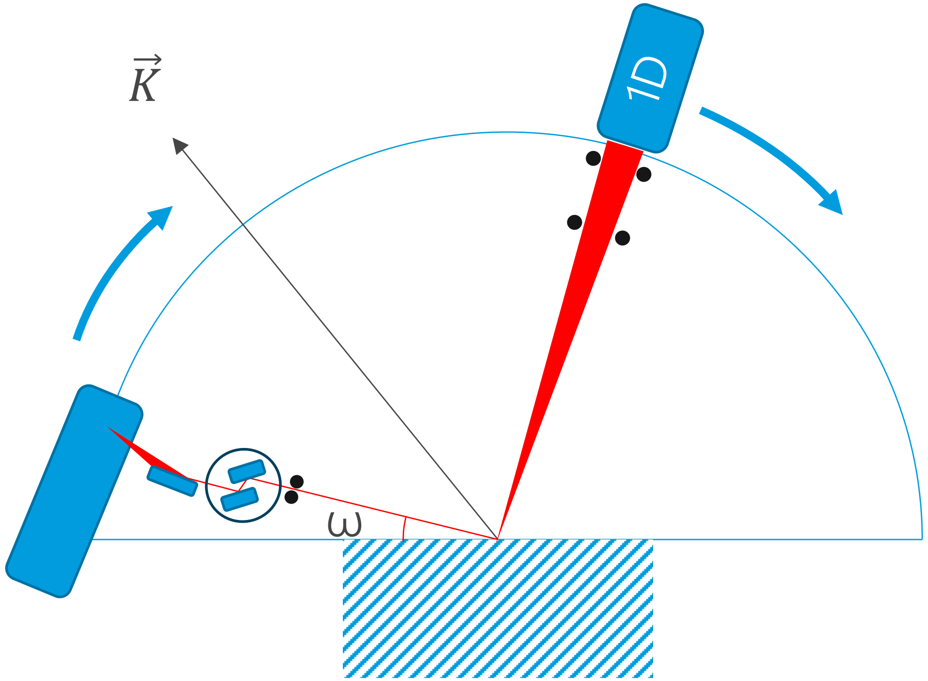
Rocking curve (ω scan)



- Mosaicity
- Dislocation density

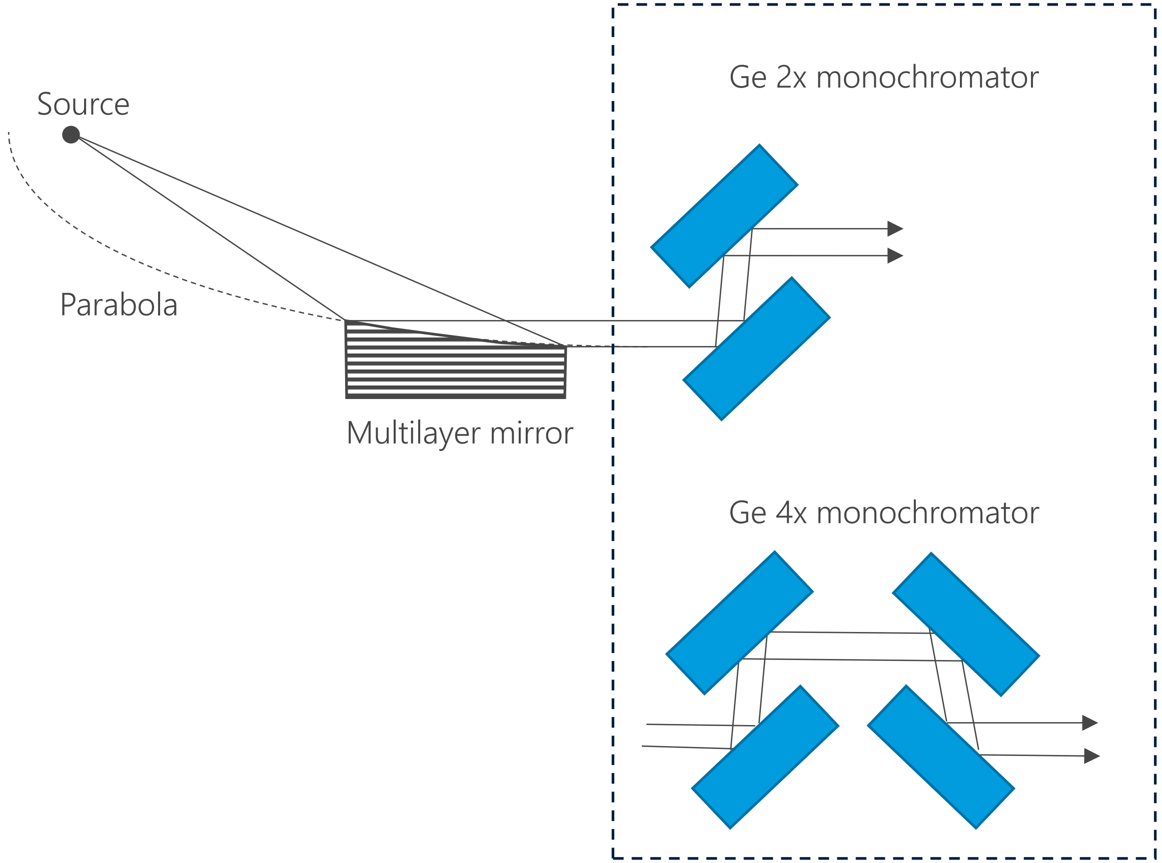


Reciprocal space mapping
(ω scan + 2θ covered by 1D det.)



- d-spacings both ⊥ & ∥ to surface
- Stress & relaxation

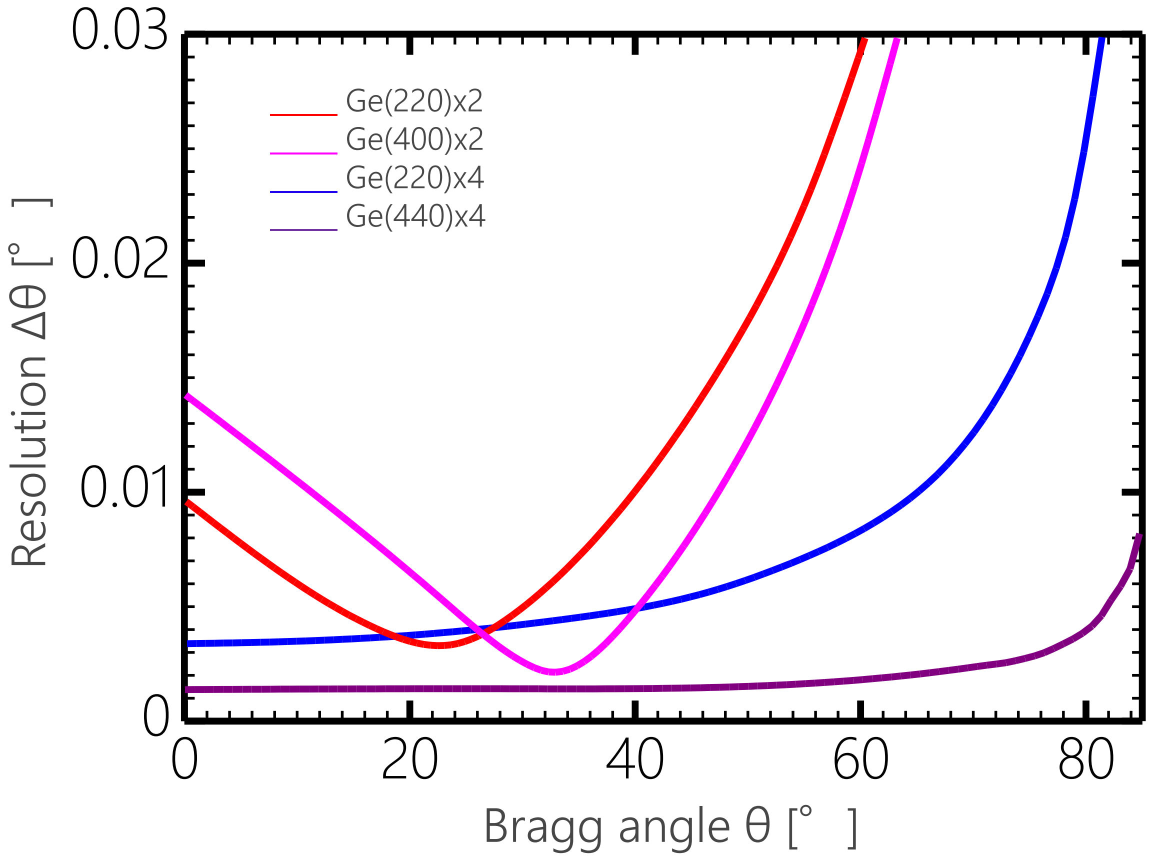
Channel cut monochromator



- 2 bounce monochromator
  - Pros: High intensity
  - Cons: Large dispersion

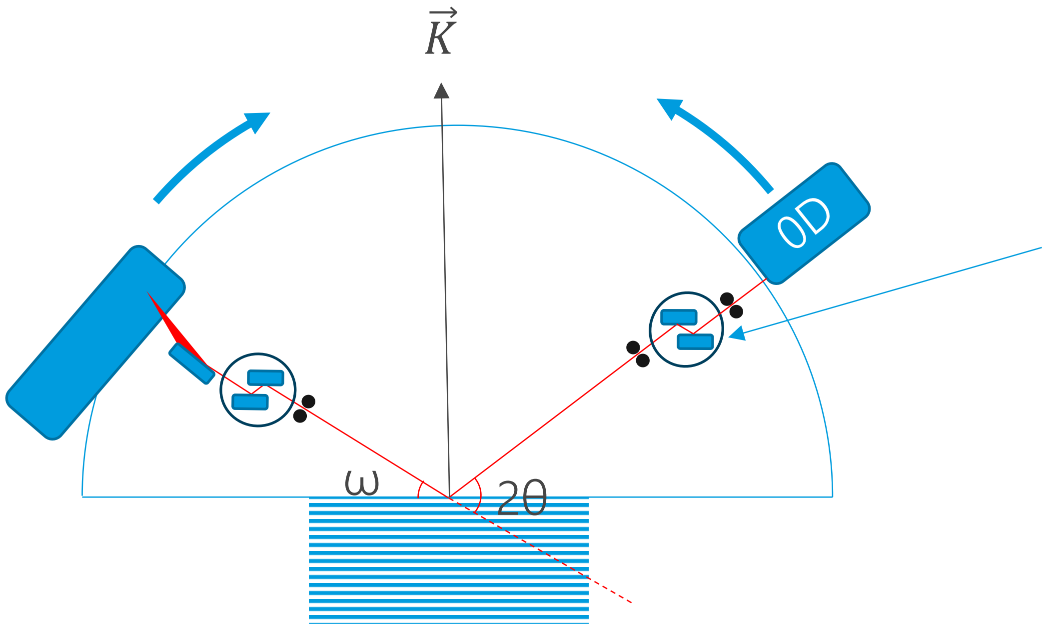
- 4 bounce monochromator
  - Pros: Small dispersion
  - Cons: Low intensity

Monochromator resolution



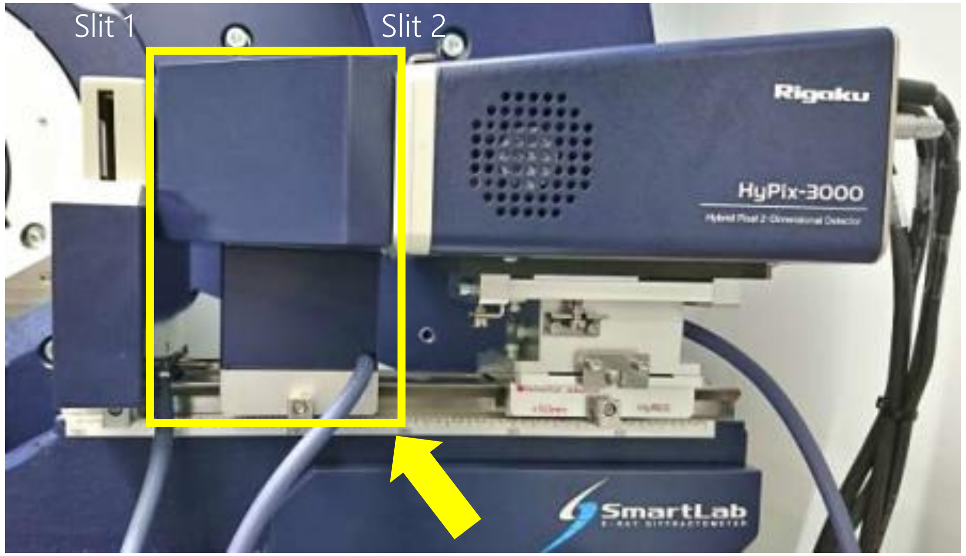
Primary monochromator	Relative intensity	Applications examples
Ge(220) 2-bounce	~ 360	GaN, ZnO, SiC, oxides, metals
Ge(400) 2-bounce	~ 100	Si, GaAs, InP, ZnSe, SiGe
Ge(220) 4-bounce	~ 20	Epitaxial thin films from low to high 2θ angle
Ge(440) 4-bounce	1	Substrate analysis

Symmetric 2θ-ω triple axis scan

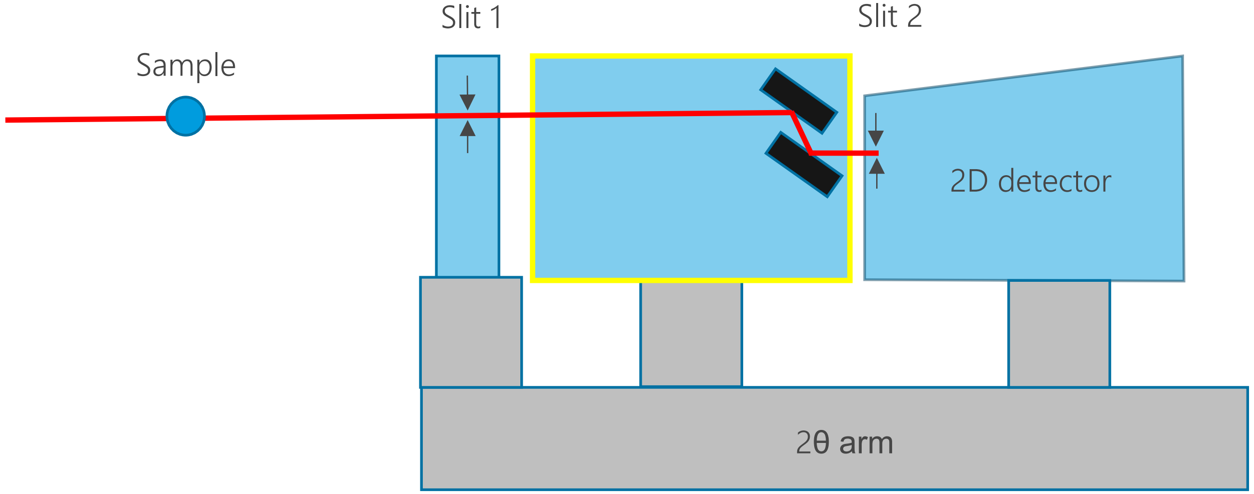


- Ge-channel cut analyzer crystal
- $\Delta 2\theta \sim 0.003°$
- Resolution is too high for sample alignment

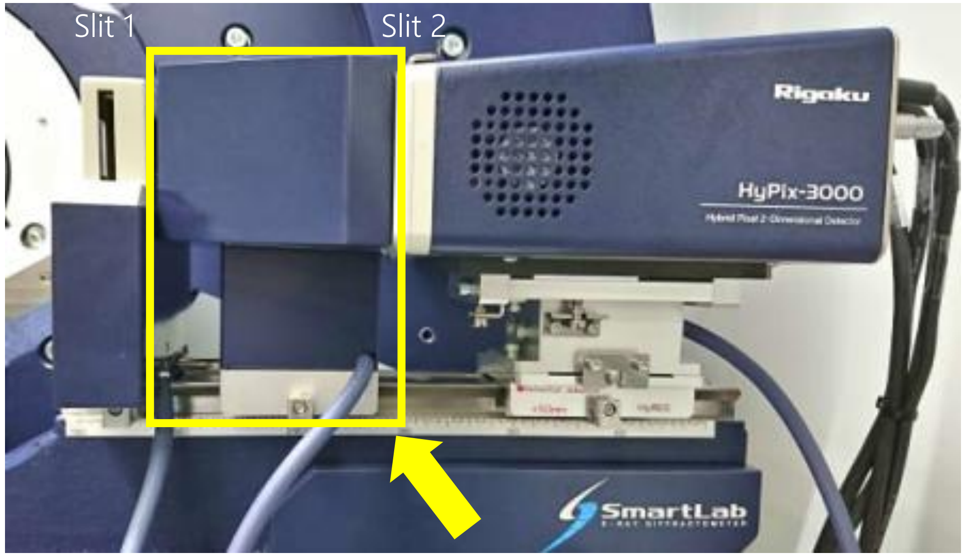
Receiving analyzer crystal



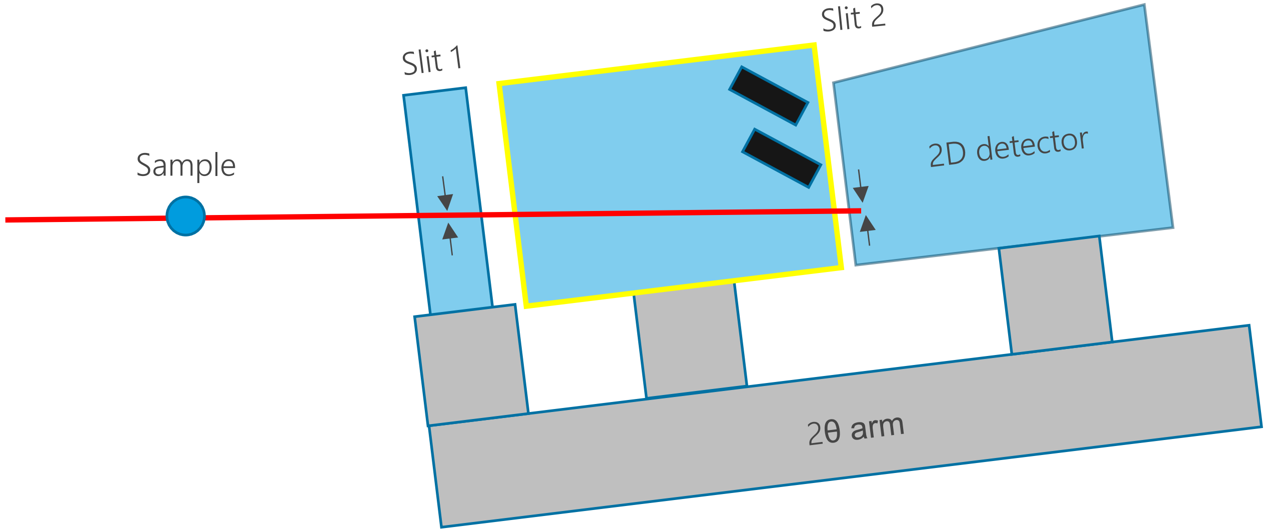
Triple axis



Receiving analyzer crystal

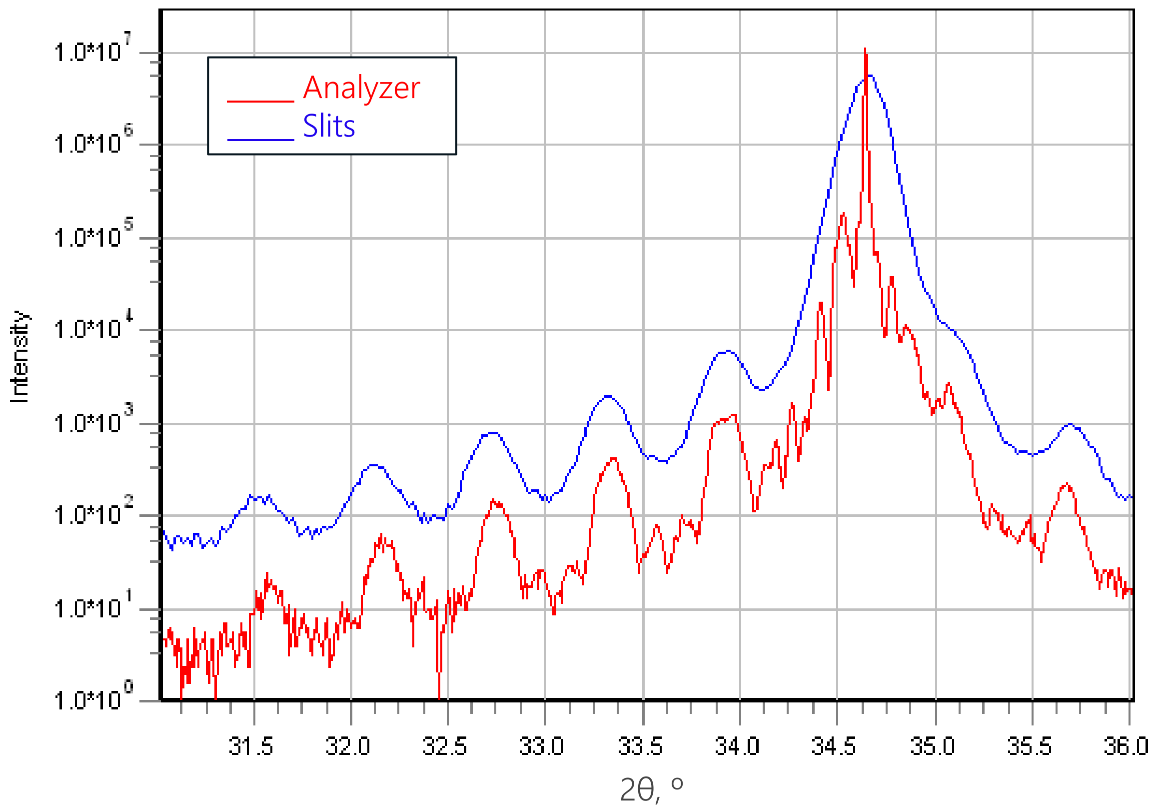


Slits



Slit path vs. Analyzer path

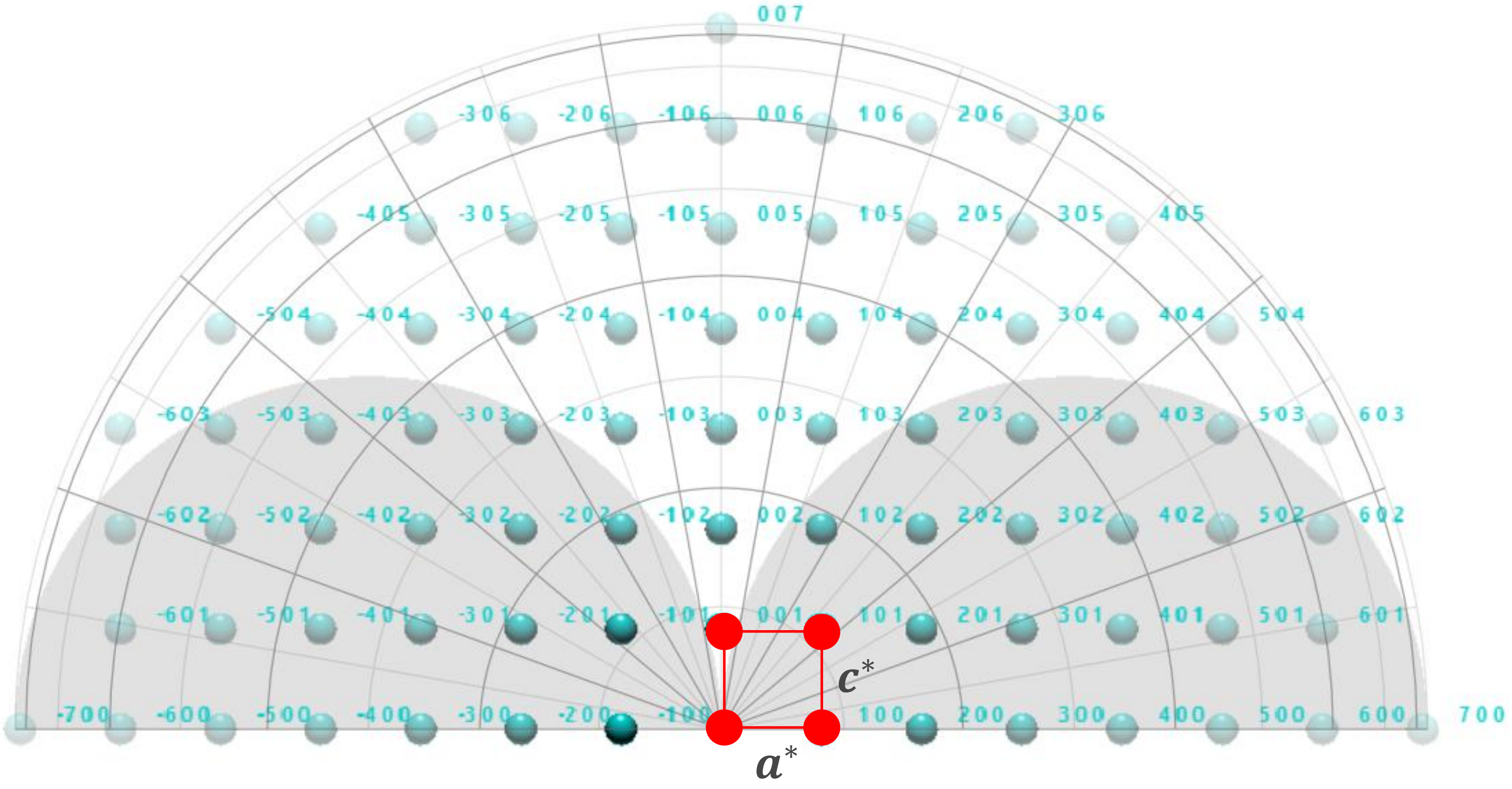
2θ-θ @ *0002* [InGaN/GaN]$_4$ on GaN



- Analyzer
  - Suppresses peak broadening along ω
  - Film with mosaicity, e.g. GaN
- Slits
  - High quality film, e.g. III-V compound semiconductors

5. HRXRD scans in reciprocal space

Reciprocal lattice point



Crystal: a cubic crystal

$$\overrightarrow{g_{hkl}} = h\overrightarrow{a^*} + k\overrightarrow{b^*} + l\overrightarrow{c^*}$$

$$\overrightarrow{a^*} = \frac{\vec{b} \times \vec{c}}{v_c}, \overrightarrow{b^*} = \frac{\vec{a} \times \vec{c}}{v_c}, \overrightarrow{c^*} = \frac{\vec{a} \times \vec{b}}{v_c}$$

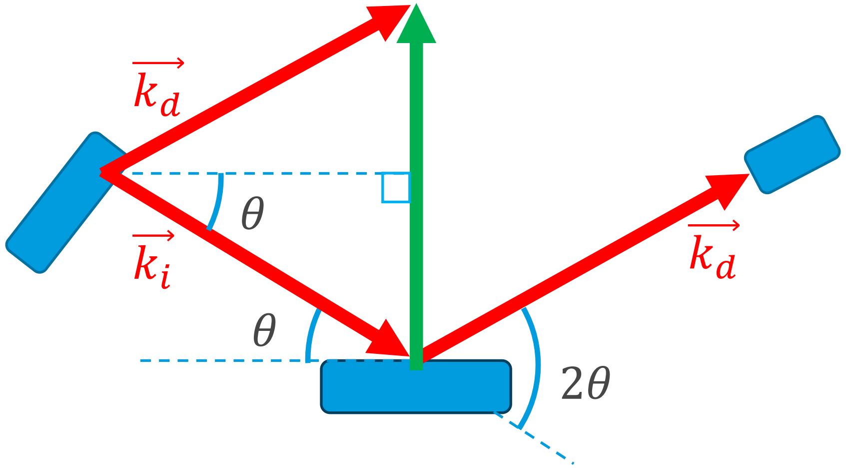
$$|\overrightarrow{g_{hkl}}| = \frac{1}{d_{hkl}}$$

$\overrightarrow{g_{hkl}}$: reciprocal lattice vector

$\overrightarrow{a^*}, \overrightarrow{b^*}, \overrightarrow{c^*}$: unit vector of a reciprocal lattice

v_c: Volume of a unit cell

Bragg equation



Bragg equation

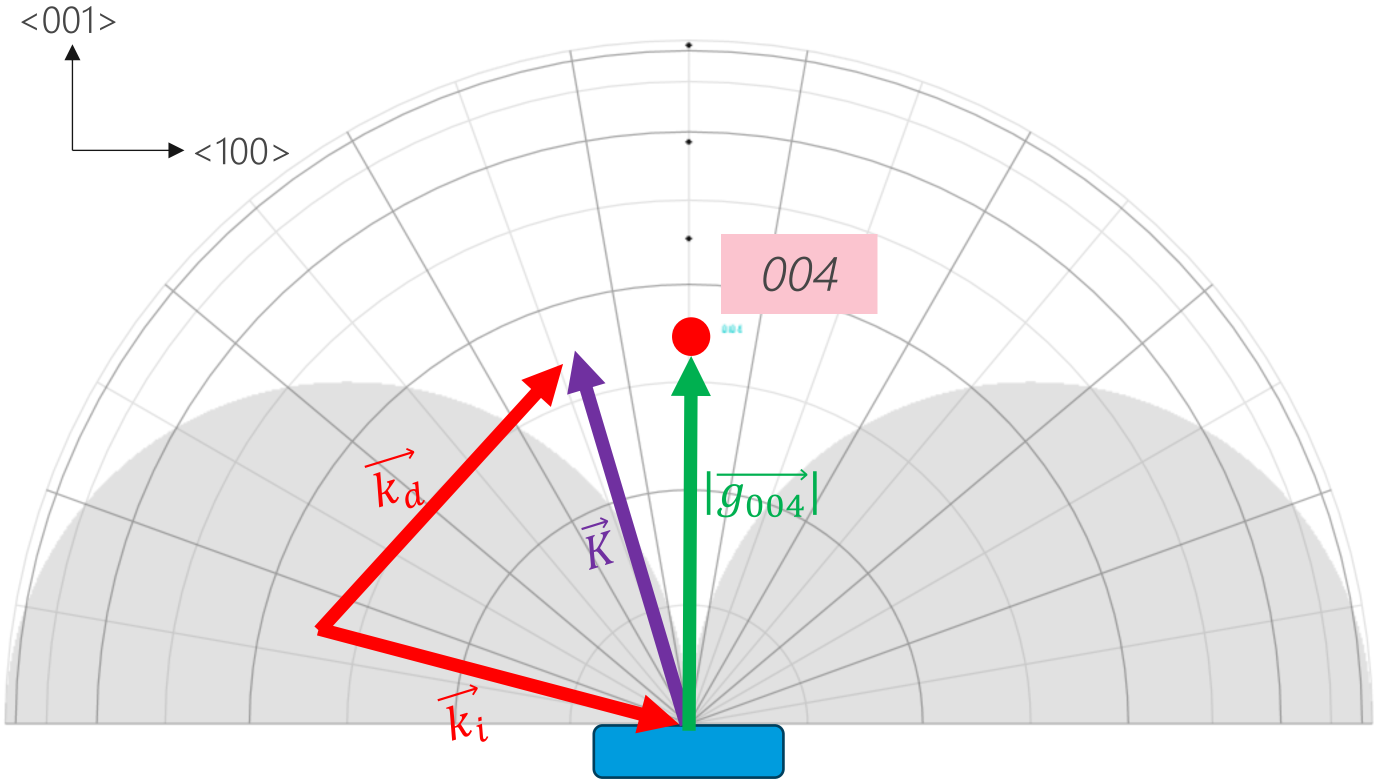
$$2 \cdot d_{hkl} \cdot \sin\theta = \lambda$$

$$\lambda = \frac{1}{\left|\overrightarrow{k_i}\right|} = \frac{1}{\left|\overrightarrow{k_d}\right|}$$

$$\therefore 2 \cdot \left|\vec{k}\right| \cdot \sin\theta = \frac{1}{d_{hkl}} = \left|\overrightarrow{k_d} - \overrightarrow{k_i}\right|$$

d_{hkl}: interplanar spacing of hkl plane
λ: wavelength of X-ray
$\overrightarrow{k_i}$: incident X-ray wave vector
$\overrightarrow{k_d}$: diffracted X-ray wave vector

Diffraction condition in reciprocal space



Scattering vector $\vec{K}$

$$\vec{K} = \overrightarrow{k_d} - \overrightarrow{k_i}$$

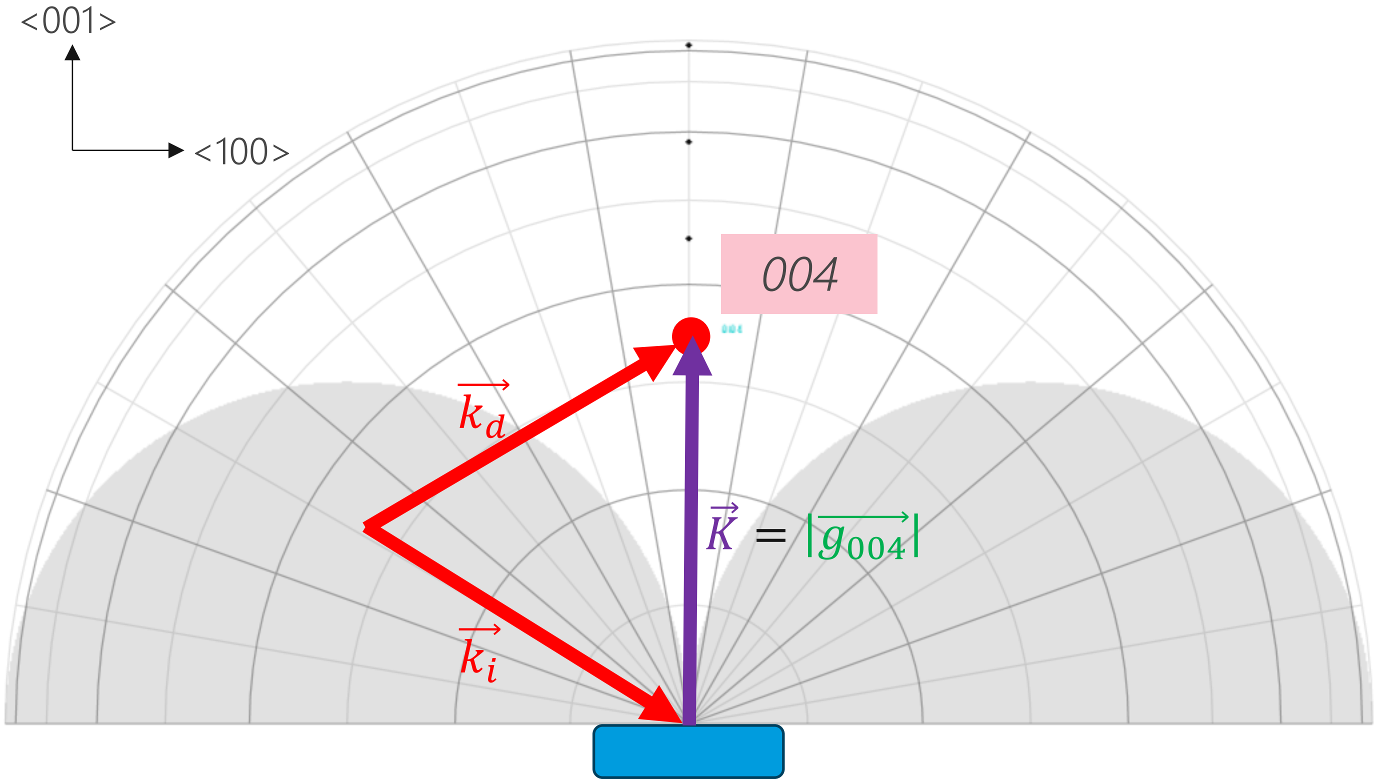
Diffraction is observed when

$$\vec{K} = \overrightarrow{g_{hkl}}$$

$\vec{K}$: scattering vector

$\overrightarrow{g_{hkl}}$: reciprocal lattice vector

Diffraction condition in reciprocal space



Scattering vector $\vec{K}$

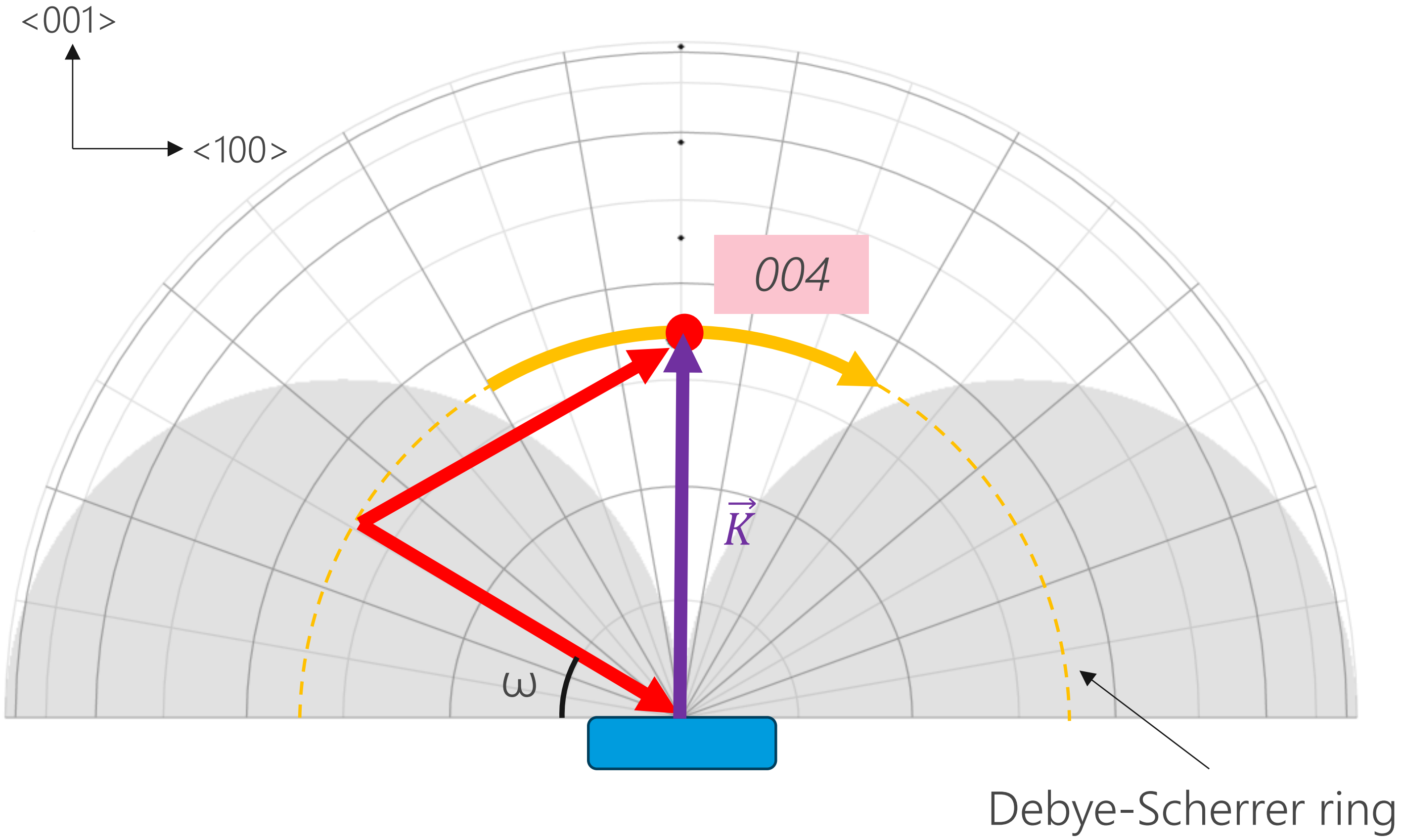
$$\vec{K} = \overrightarrow{k_d} - \overrightarrow{k_i}$$

Diffraction is observed when

$$\vec{K} = \overrightarrow{g_{hkl}}$$

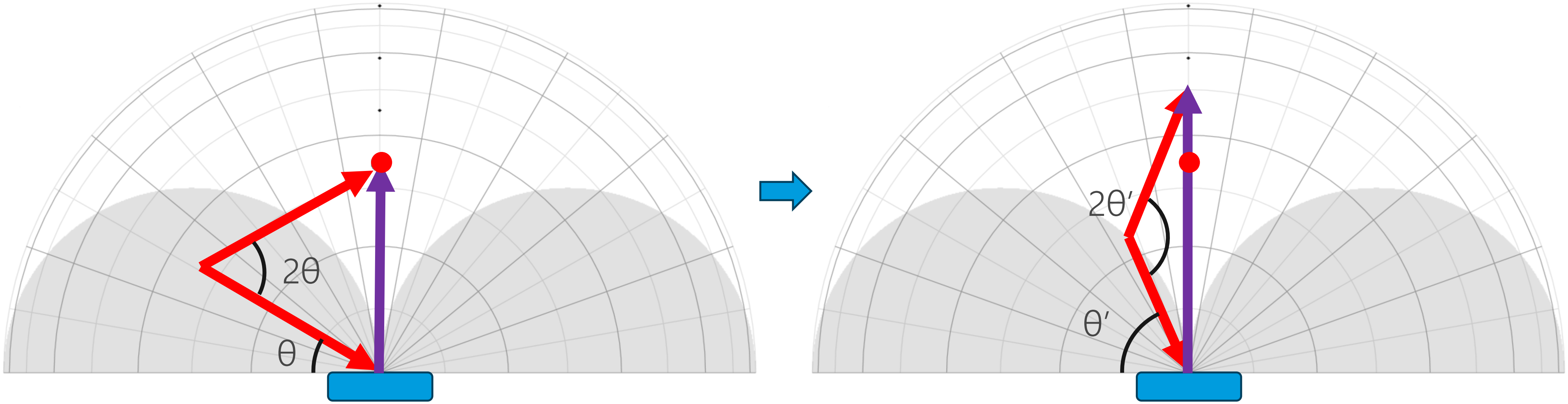
$\vec{K}$: scattering vector
$\overrightarrow{g_{hkl}}$: reciprocal lattice vector

Omega scan



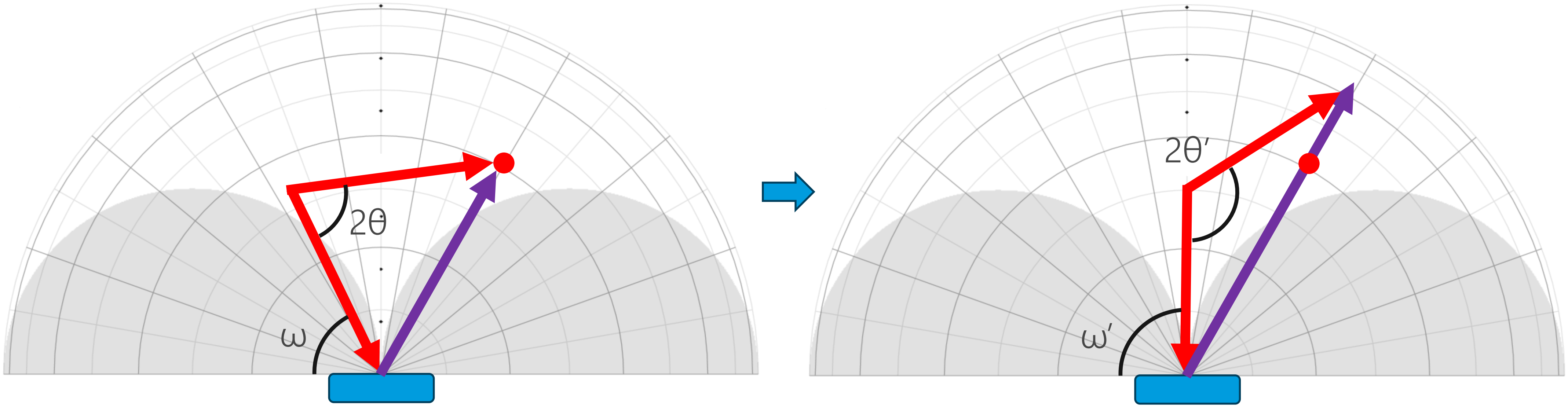
- ω scan ‖ Debye-Scherrer ring
- Also called “rocking curve”

2θ-θ scan



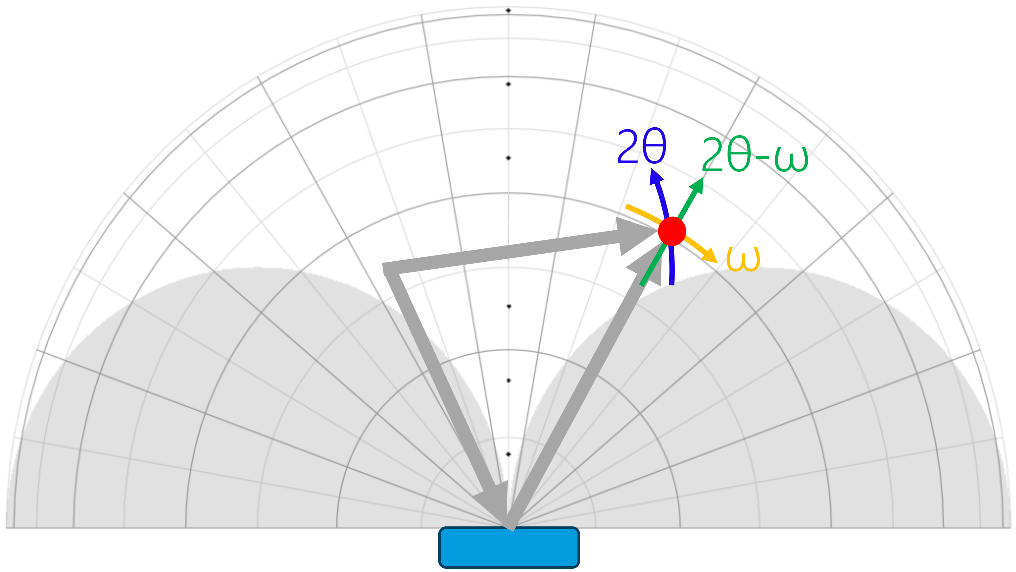
- 2θ-θ scan ⊥ sample surface
- Also called “radial scan”

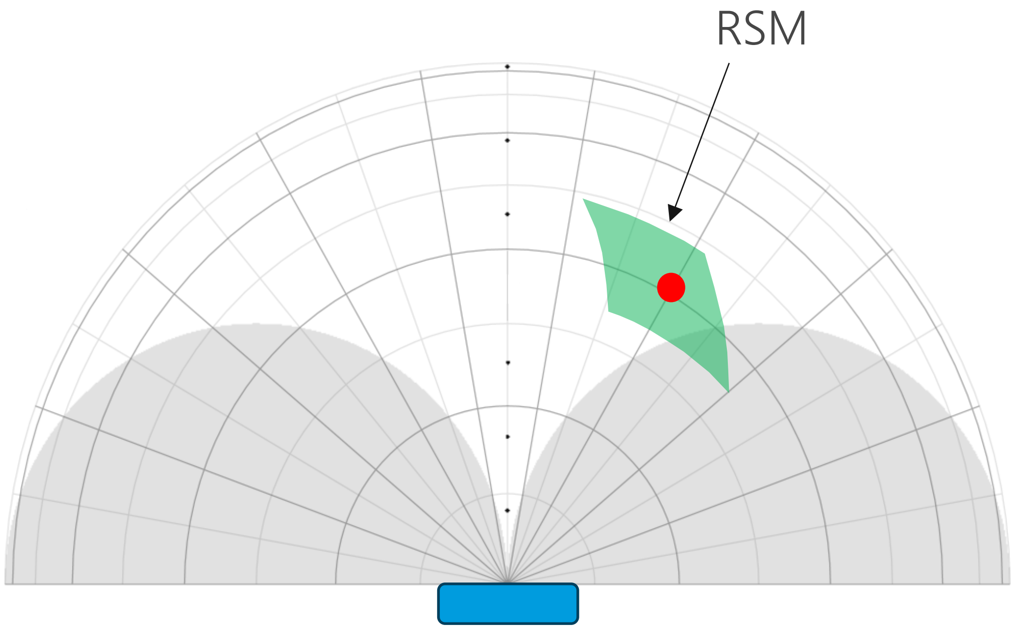
2θ-ω scan



- θ≠ω
- Radial scan along tilted direction

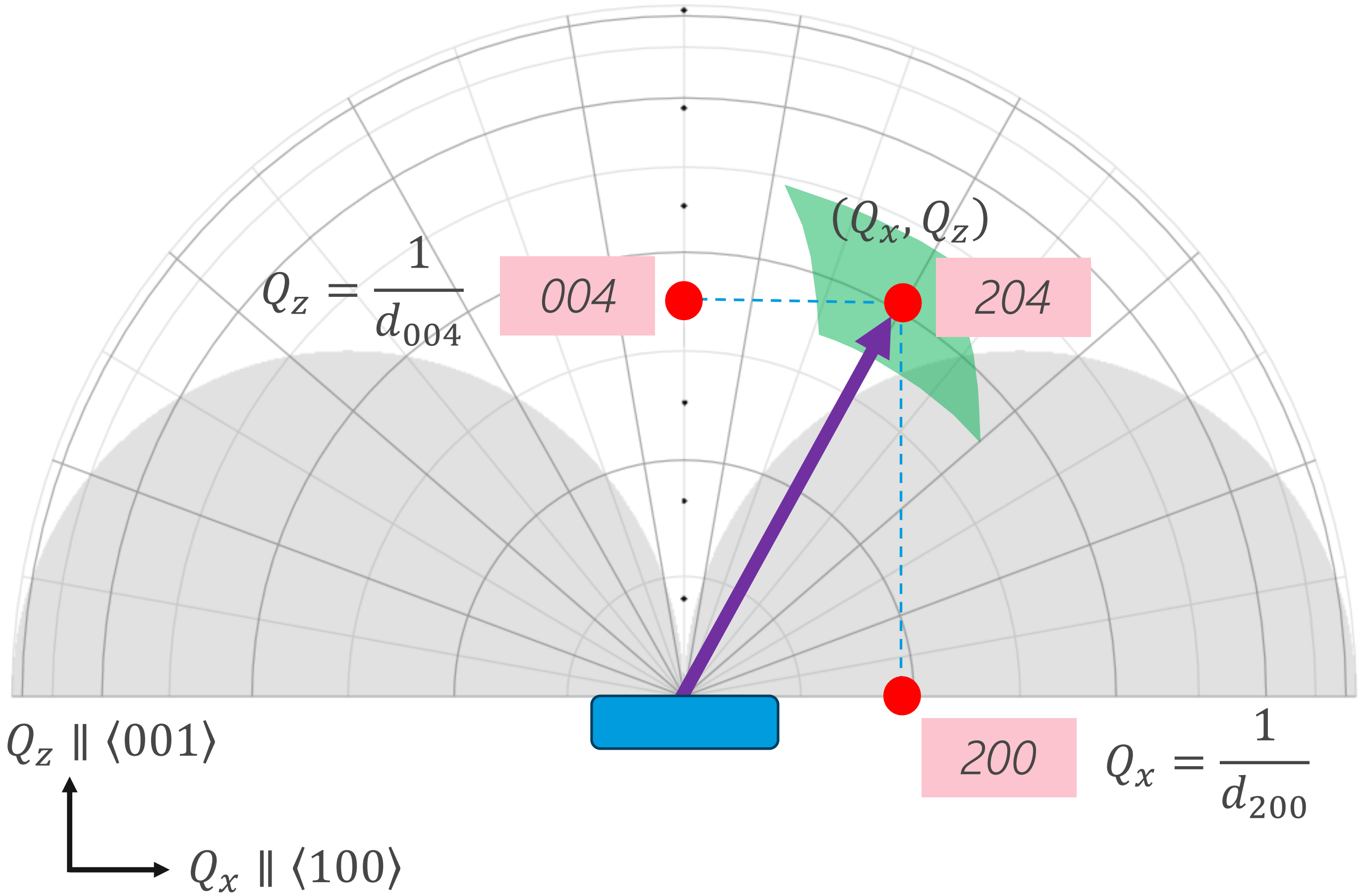
Reciprocal space mapping (RSM)





- 2θ-ω scan + ω offset
- 2θ scan + ω offset
- ω scan + 1D detector (2θ)

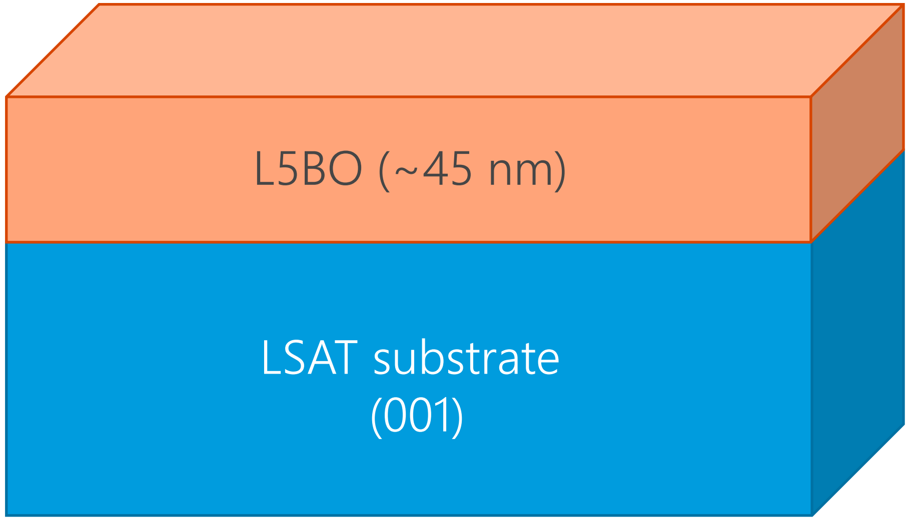
Reciprocal space mapping (RSM)



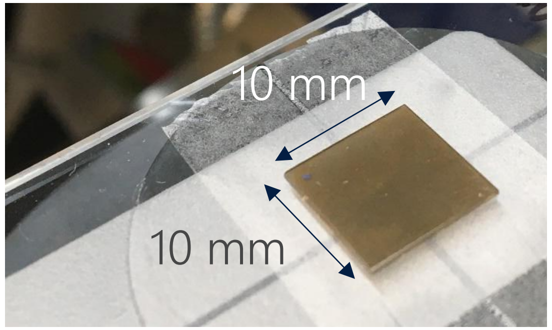
- RSM tells (Q_x, Q_z) of diffraction spot
- $Q_x \propto$ in-plane d-spacing
- $Q_z \propto$ out of plane d-spacing

6. Epitaxial perovskite thin films: structure and thickness

Sample

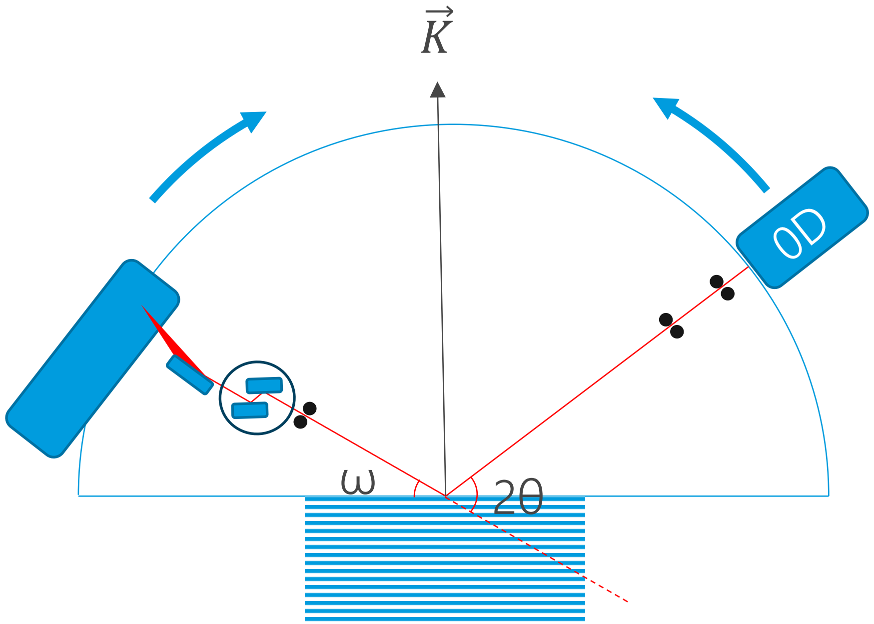


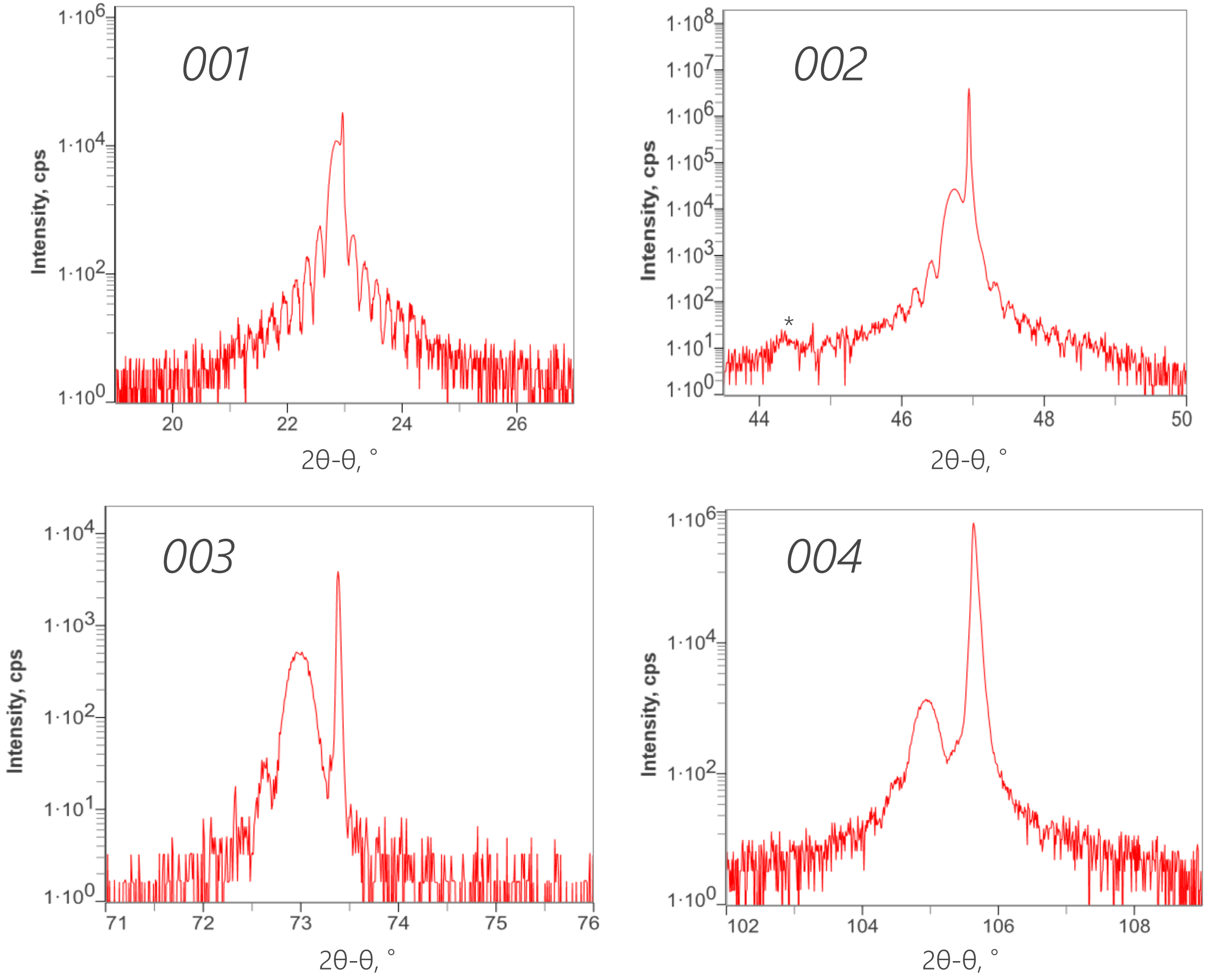
Sample courtesy:
Dr. Alessandro Mazza (Los Alamos Nat. Lab.)
Dr. Jim Browning (Oak Ridge Nat. Lab.)



- L5BO
  - La$(Cr_{0.2}Mn_{0.2}Fe_{0.2}Co_{0.2}Ni_{0.2})O_3$
  - $LaCrO_3$, $LaMnO_3$, $LaFeO_3$ ➔ orthorhombic
  - $LaCoO_3$, $LaNiO_3$ ➔ rhombohedral
- LSAT
  - $(La_{0.18}Sr_{0.82})(Al_{0.59}Ta_{0.41})O_3$
  - $Pm\bar{3}m$, a=0.3868 nm

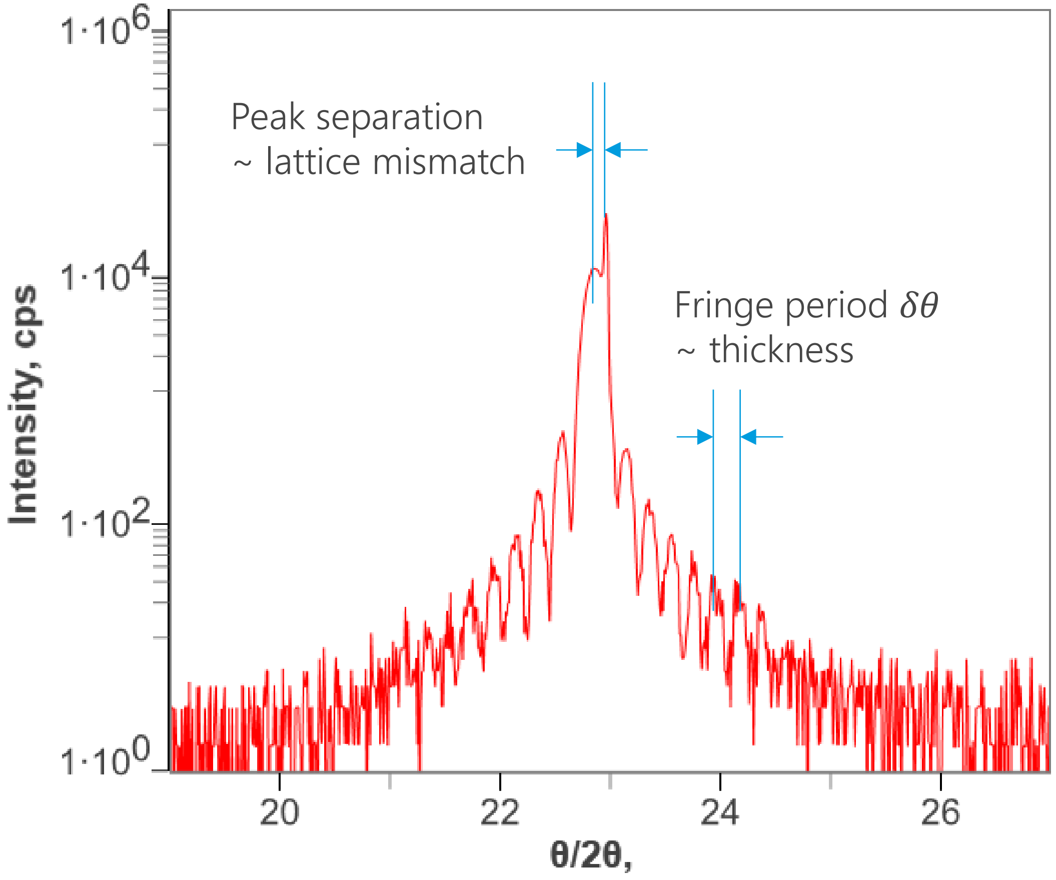
HR 2θ-θ scan





* Unassigned peak

Lattice mismatch & Thickness



Peak separation
~ lattice mismatch

Fringe period $\delta\theta$
~ thickness

- Lattice mismatch: M

$$M = \frac{\Delta c}{c} = \frac{C_L - C_S}{C_S} = \frac{\sin\theta_S}{\sin\theta_L} - 1$$

$$= 0.00471 = 0.471\ \%$$

- Layer's c-lattice parameter: C_L

$$C_L = (1 + M)\cdot C_S = 0.3887\ nm$$

- Layer's thickness: t

$$t = \frac{\lambda\cdot\sin\theta}{\sin 2\theta\cdot\delta\theta}$$

$$= 44.2\ nm$$

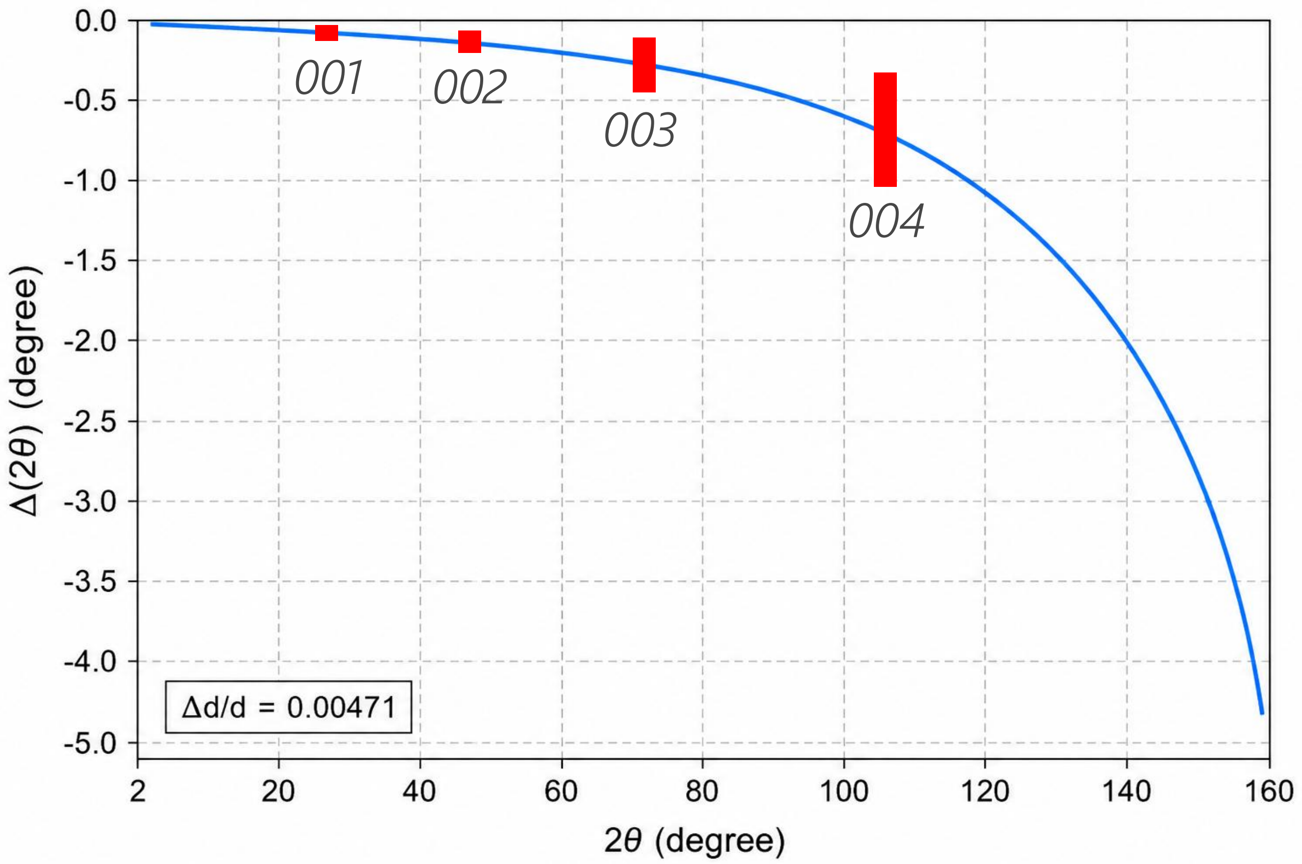
C_L: layer's c-lattice constant

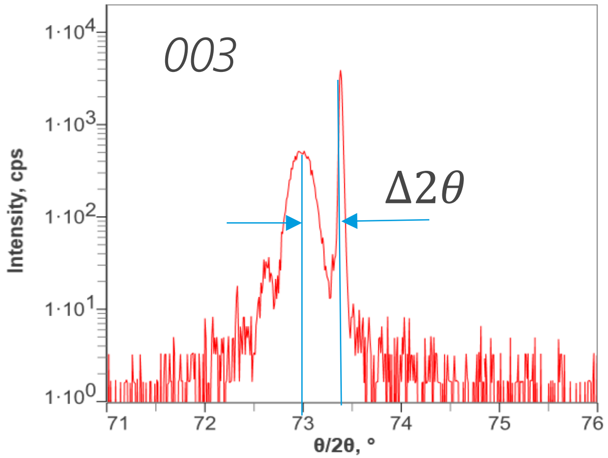
C_S: substrate's c-lattice constant

θ: Bragg angle

λ: X-ray wavelength (0.15406 nm)

Peak separation layer & substrate





- 1^{st} derivative Bragg equation

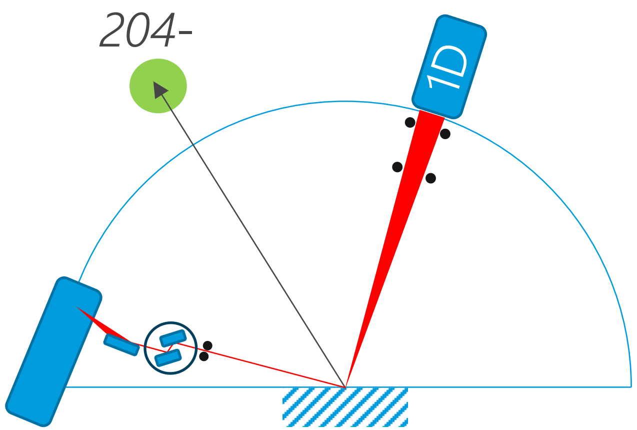
$$\frac{\Delta d}{d} = -\cot\theta \cdot \Delta\theta$$

- Peak separation $\Delta\theta$

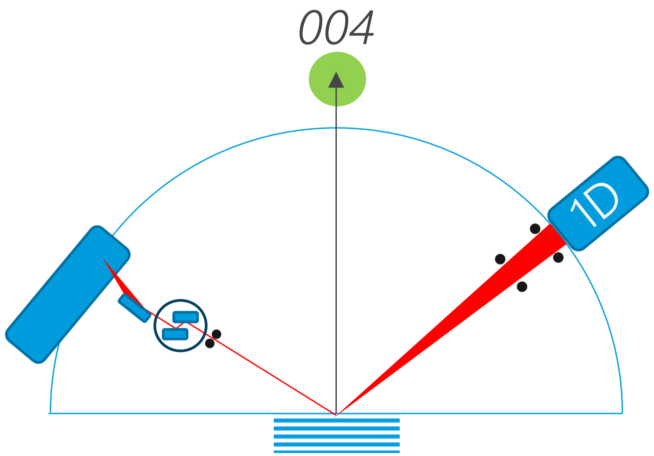
$$\Delta 2\theta = -2 \cdot \tan\theta \cdot \frac{\Delta d}{d}$$

$$\frac{\Delta d}{d} = M = 0.00471$$

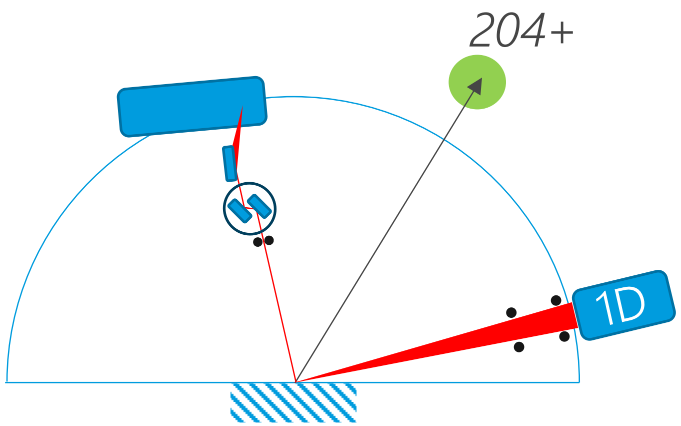
RSMs



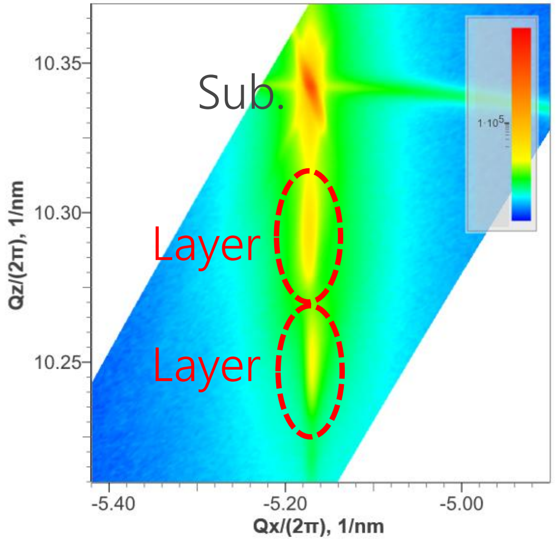
204-
1D



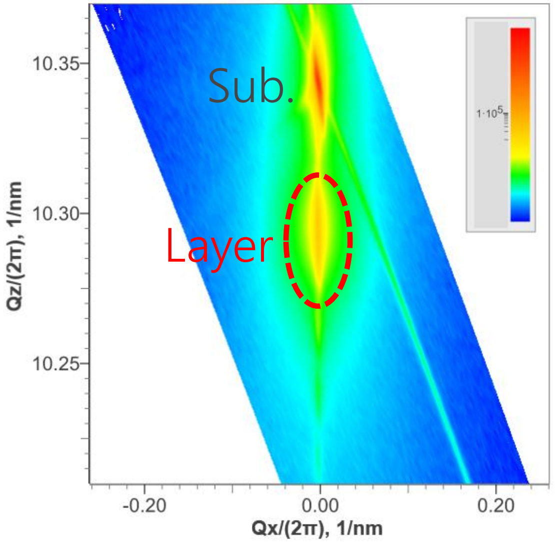
004
1D



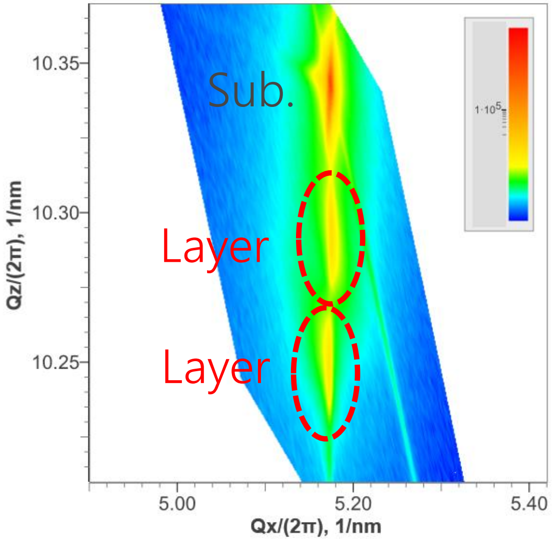
204+
1D



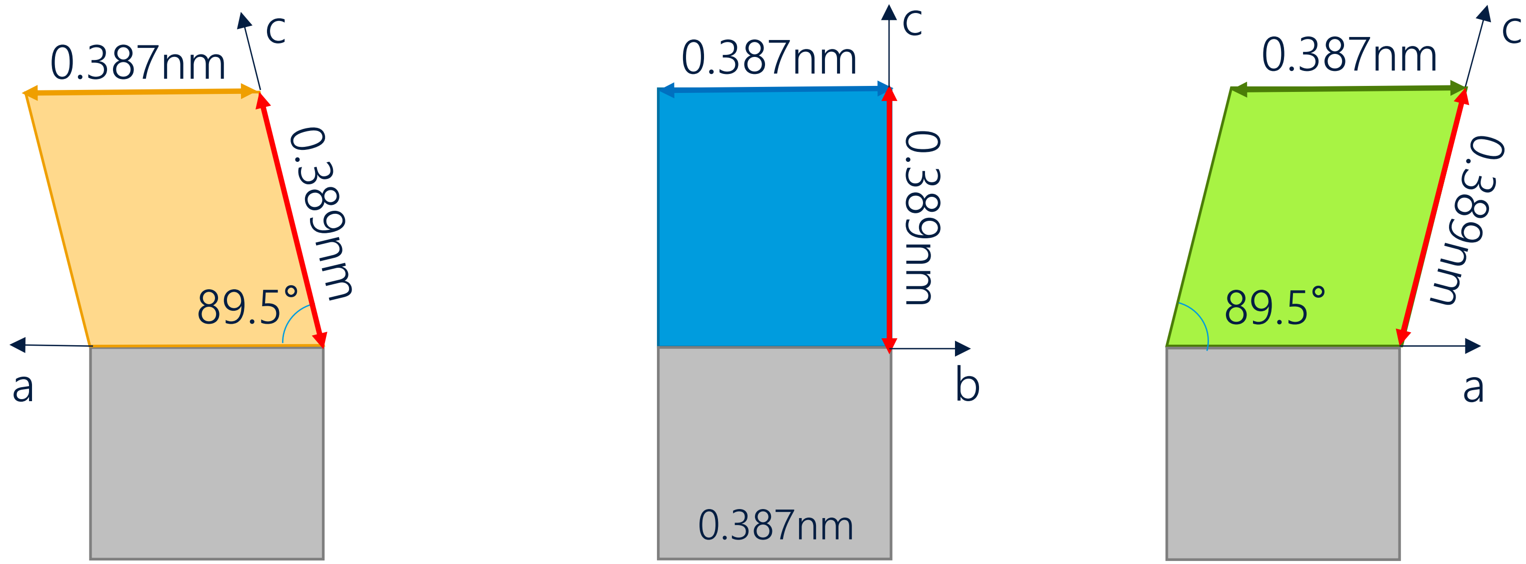
Sub.
Layer
Layer
$1\cdot10^5$
10.35
10.30
10.25
Qz/(2π), 1/nm
-5.40
-5.20
-5.00
Qx/(2π), 1/nm



Sub.
Layer
$1\cdot10^5$
10.35
10.30
10.25
Qz/(2π), 1/nm
-0.20
0.00
0.20
Qx/(2π), 1/nm



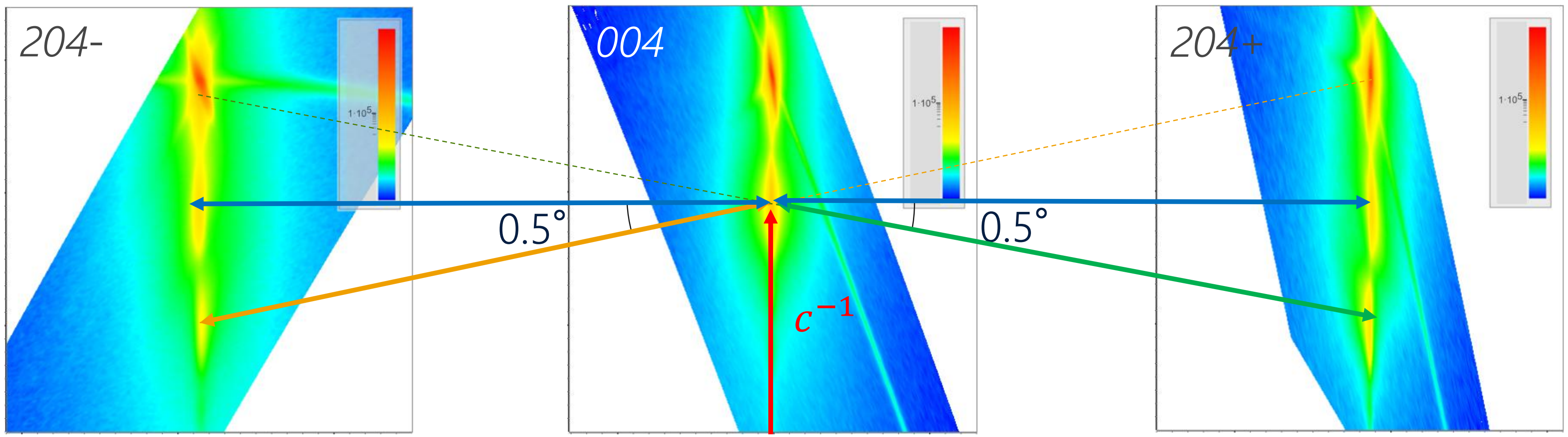
Sub.
Layer
Layer
$1\cdot10^5$
10.35
10.30
10.25
Qz/(2π), 1/nm
5.00
5.20
5.40
Qx/(2π), 1/nm



L5BO layer

LSAT sub.

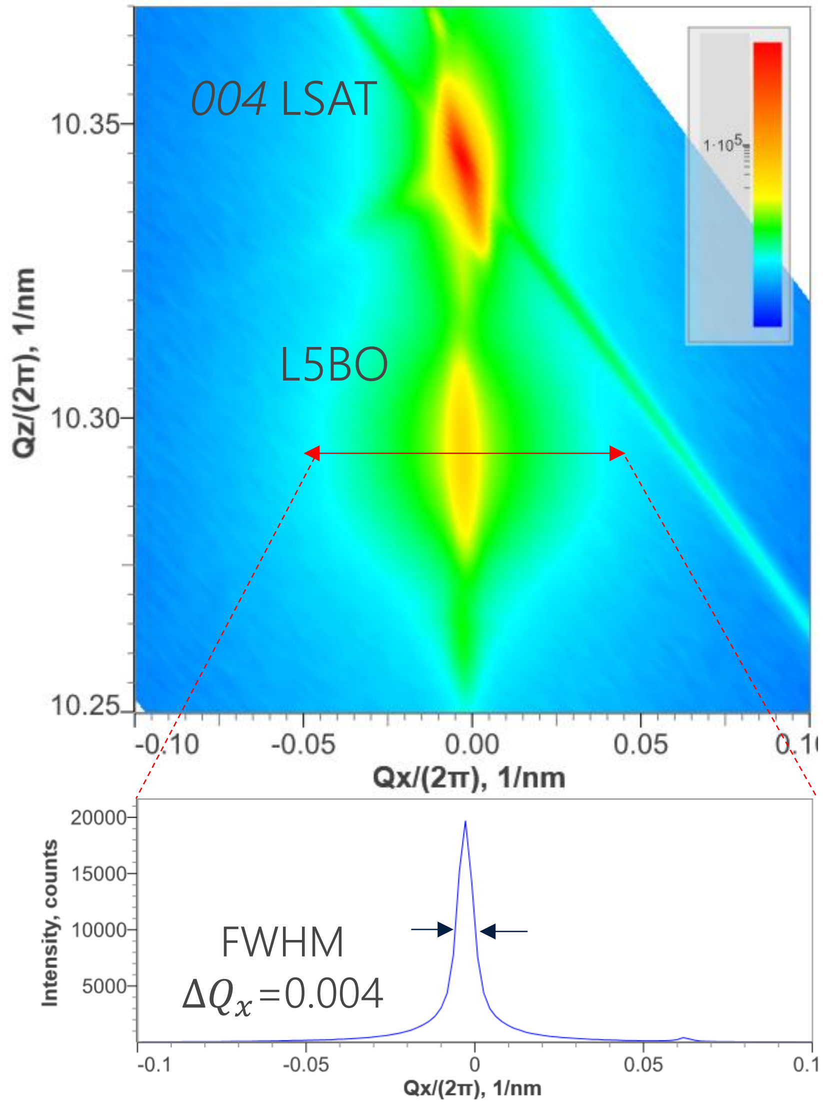
- Monoclinic symmetry
- a=b=0.387 nm
- c=0.389 nm
- β=89.5 °



Rocking curve from RSM

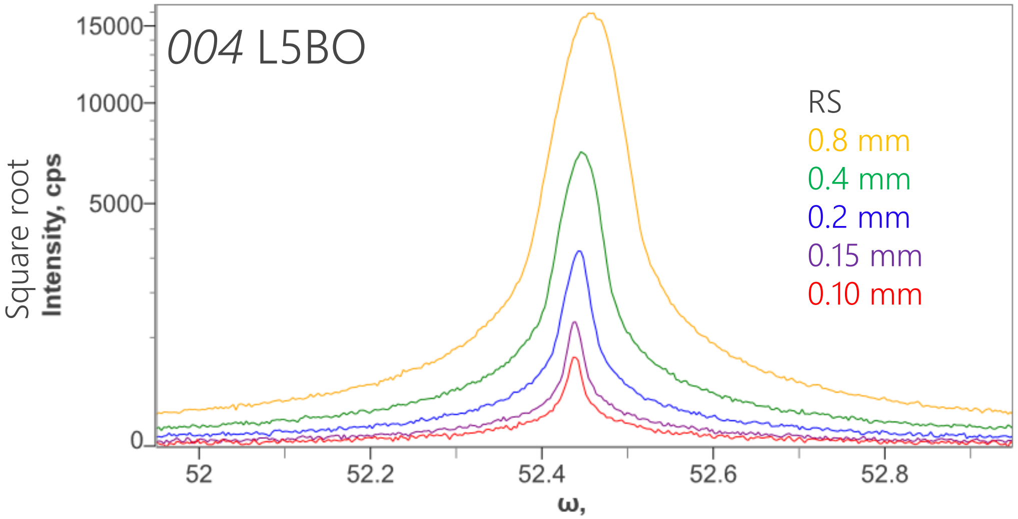
- Conversion of ΔQ_x to rocking curve FWHM ($\Delta\omega$).

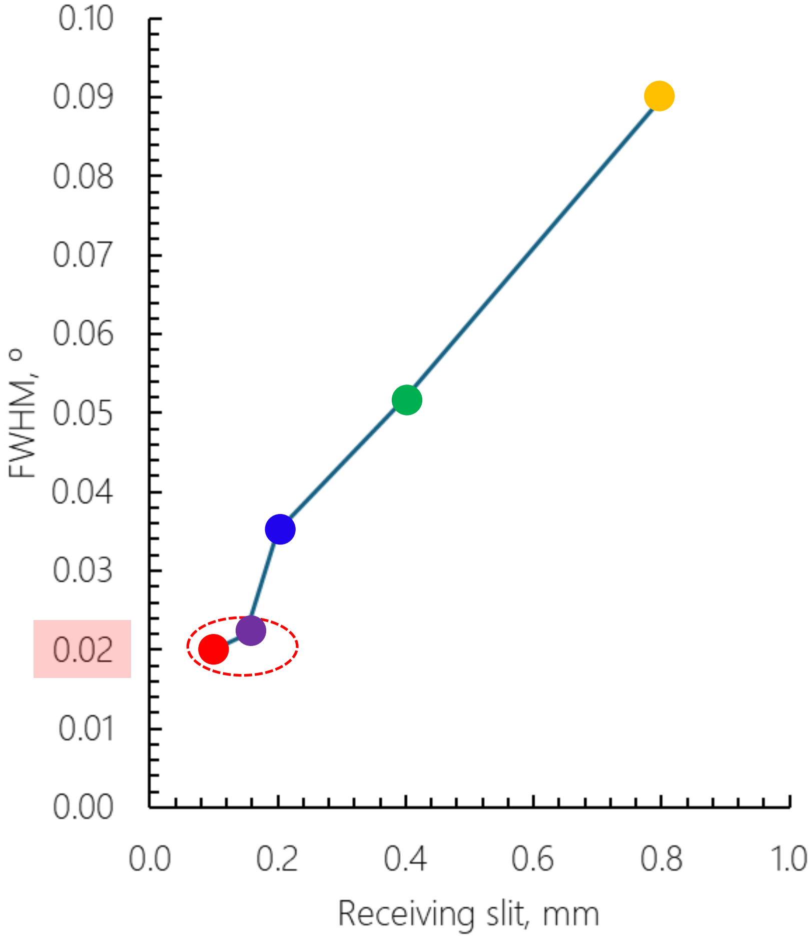
$$\Delta\omega \sim \frac{\Delta Q_x}{Q_z} = \frac{0.004}{10.29} = 0.02°$$



XRC: receiving slit dependence

- Wider receiving slits convolves ΔQ_z with ΔQ_x.
- Narrow receiving slit is required to measure the real XRC peak width ~ 0.02°.





Summary

- 2θ-θ scan
  - Lattice mismatch ⊥ to sample surface
  - Thickness from thickness fringes
- Reciprocal space mapping
  - Lattice mismatch both ⊥ & ‖ to sample surface
  - Crystal symmetry & domain structure detemination
  - Rocking curve extraction