Good Diffraction Practice Webinar Series

In-Plane Grazing Incidence Diffraction – March 23, 2013

www.bruker-webinars.com

Welcome

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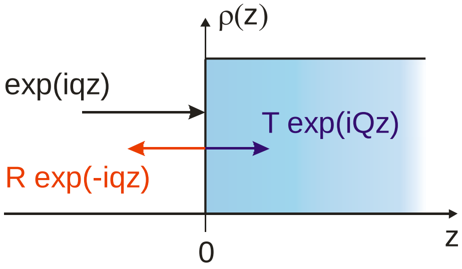
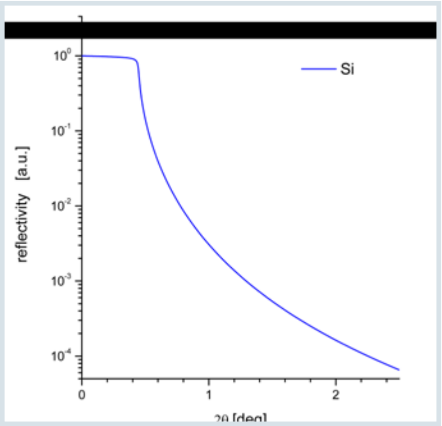
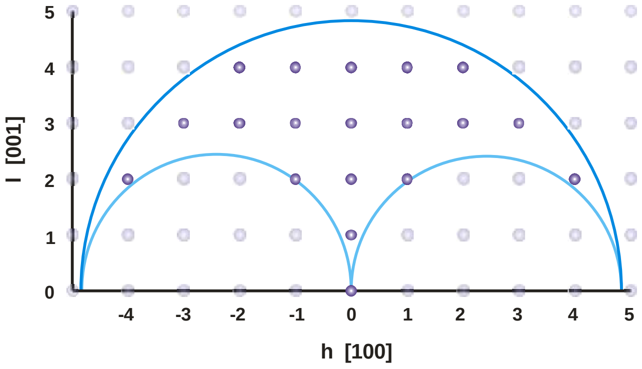
Good Diffraction Practice Webinar Series History

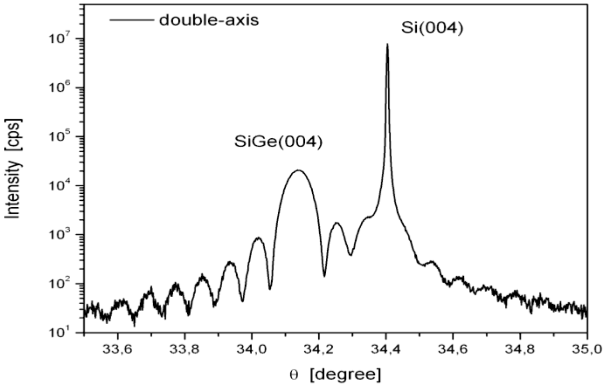
July 2010
X-ray Reflectometry



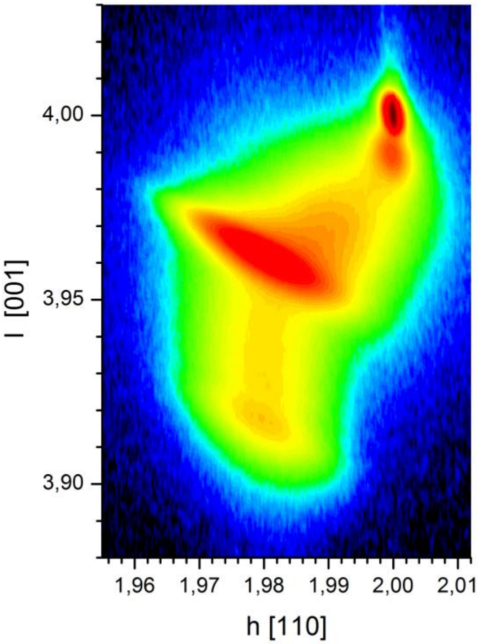


May 2011
High-Resolution X-ray Diffraction (HRXRD)





Jan 2012
HRXRD – Reciprocal Space Mapping



Outline

- Introduction
- Experimental configurations
- Experimental tips
- Examples

- Introduction
- Experimental configurations
- Experimental tips
- Examples

What is In-Plane Grazing Incidence X-Ray Diffraction (IPGID)?

An X-ray scattering technique

- Non-destructive method
- X-rays probe on the nanometer scale

Diffraction technique

- Requires a crystal lattice
- Works for epitaxial and polycrystalline samples

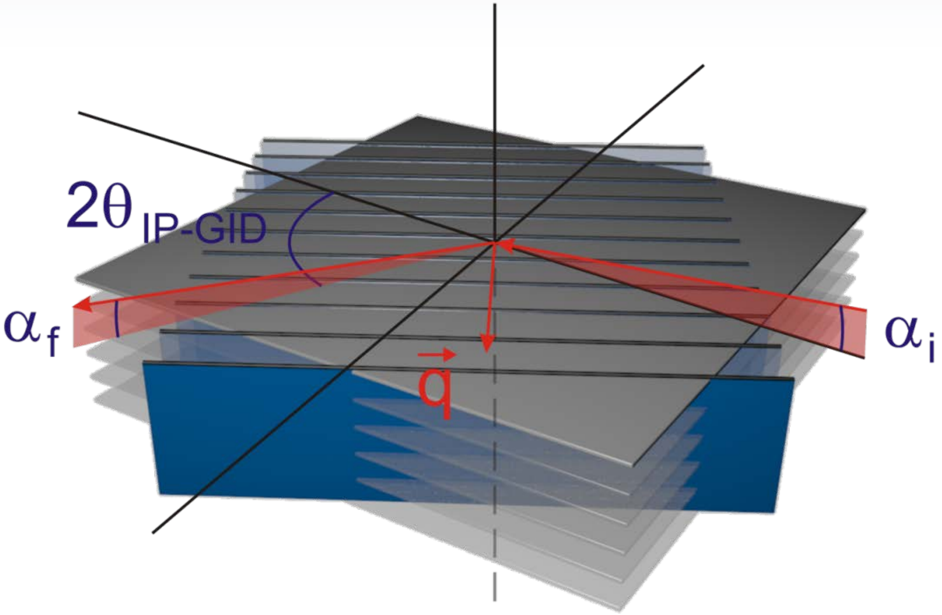
In-plane grazing incidence geometry

- Probes the near-surface part of the sample
- Probes the crystal properties parallel to the surface

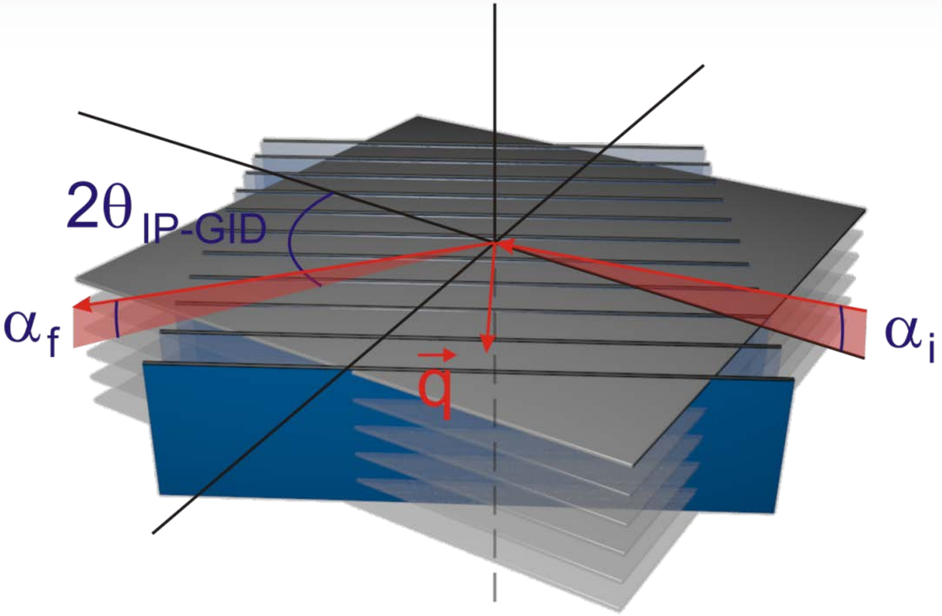
What kind of information does IPGID provide about my sample?

- In-plane lattice parameter
- Epitaxial relation
- Domain formation and twist
- In-plane texture
- Crystallite size
- Micro strain

In-plane Grazing Incidence Diffraction:
The scattering geometry

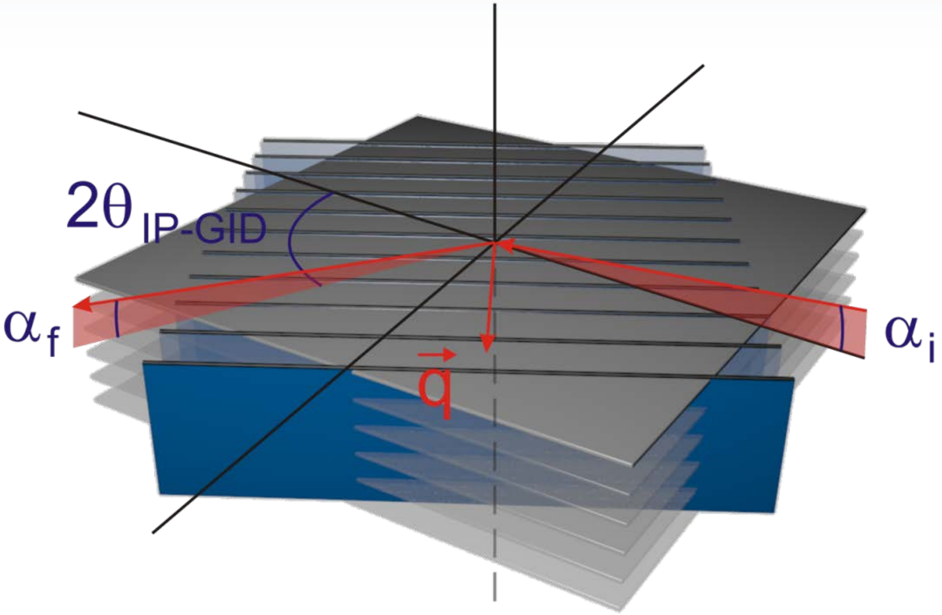


In-plane Grazing Incidence Diffraction:
The scattering geometry



Intensity : $I(\vec{q},\alpha_i,\alpha_f) \propto |T(\alpha_i)|^2\,|F(\vec{q})|^2\,\left|T(\alpha_f)\right|^2$

In-plane Grazing Incidence Diffraction:
The scattering geometry



Probed quantity :

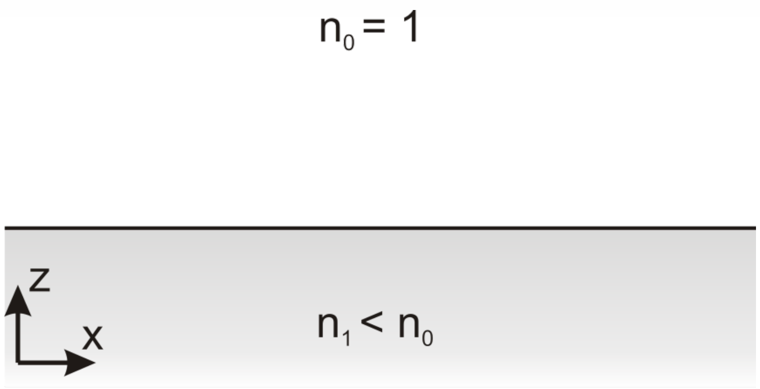
$$|F(\vec{q})|^2 \propto \left| \int_V \rho(\vec{r}) \exp\left(i\overrightarrow{q\vec{r}}\right) d\vec{r} \right|^2$$

Transmission of
incident and exit beam

Intensity :

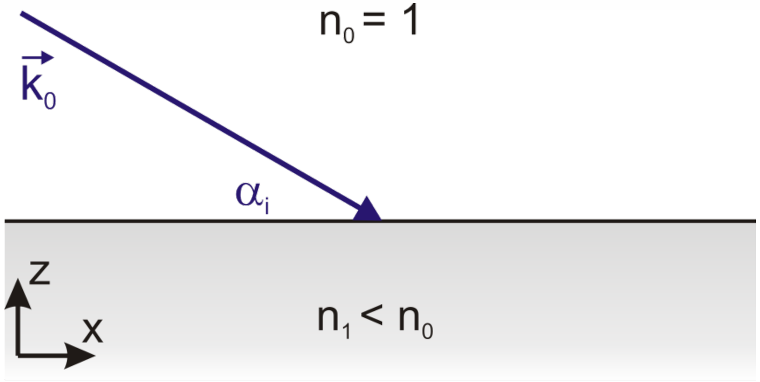
$$I(\vec{q}, \alpha_i, \alpha_f) \propto |T(\alpha_i)|^2 \, |F(\vec{q})|^2 \left|T(\alpha_f)\right|^2$$

Reflectivity and Transmission of a substrate



- Index of refraction: $n = 1 - \delta + i\beta$
- For X-rays, dispersion: $\delta \approx 10^{-5} - 10^{-6}$
- Absorption: $\beta \approx (0.1, \ldots, 0.01)\delta$

Reflectivity and Transmission of a substrate



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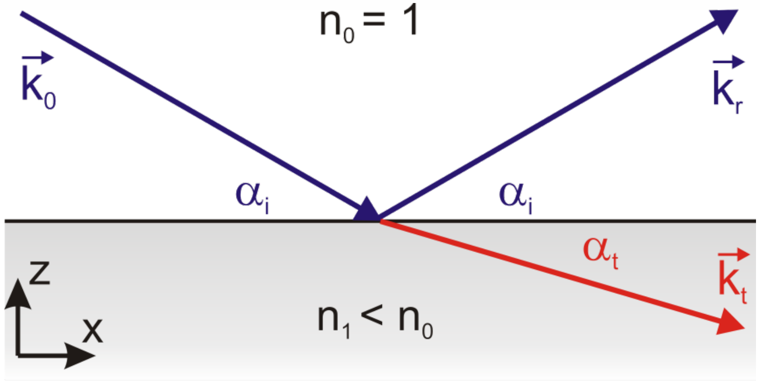
Reflectivity and Transmission of a substrate



Reflection coefficient:

$$r = \frac{k_{0,z} - k_{t,z}}{k_{0,z} + k_{t,z}}$$

Transmission coefficient:

$$t = \frac{2k_{0,z}}{k_{0,z} + k_{t,z}}$$

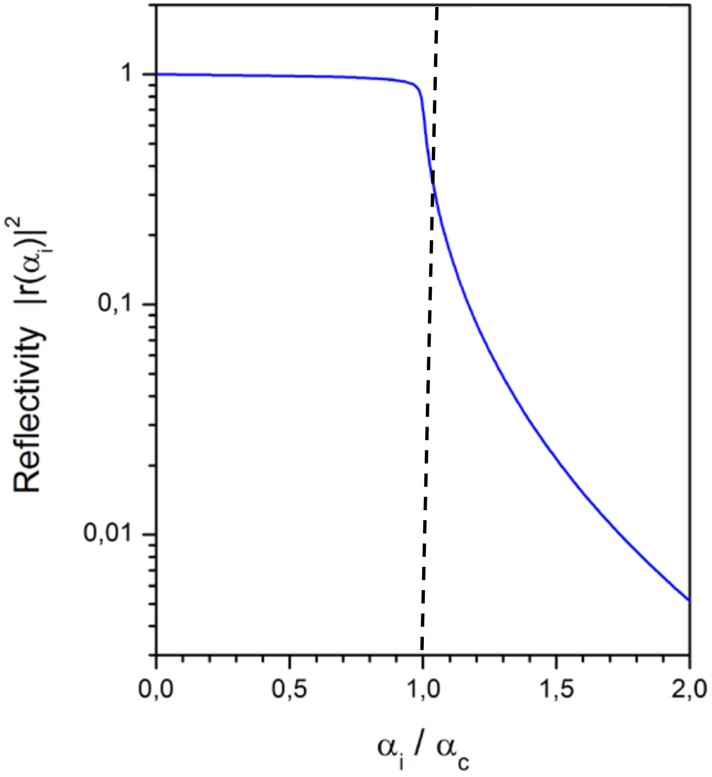
- Index of refraction: $n = 1 - \delta + i\beta$
- For X-rays, dispersion: $\delta \approx 10^{-5} - 10^{-6}$
- Absorption: $\beta \approx (0.1, \dots, 0.01)\delta$

with $k_{0,z} = k \sin \alpha_i$

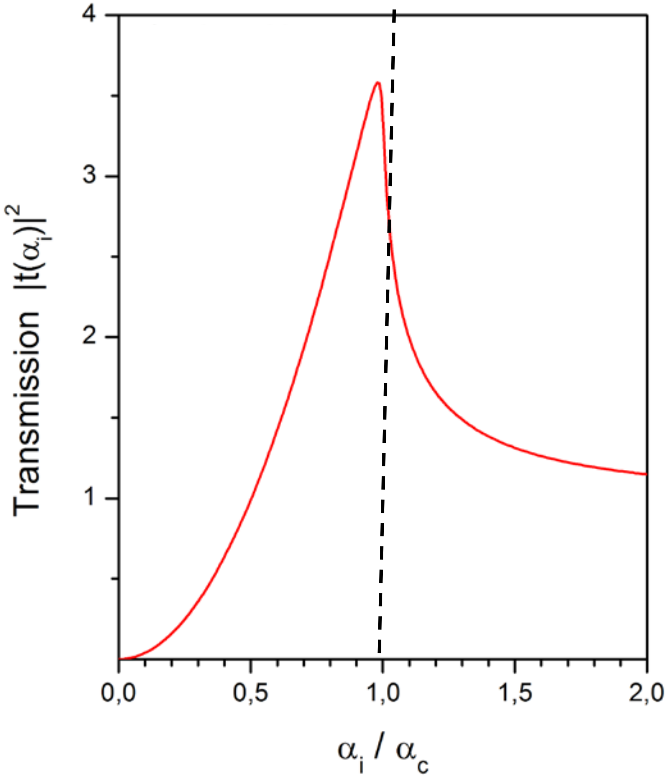
$$k_{t,z} = k \sqrt{n^2 - \cos^2 \alpha_i}$$

Reflectivity and Transmission of a silicon substrate

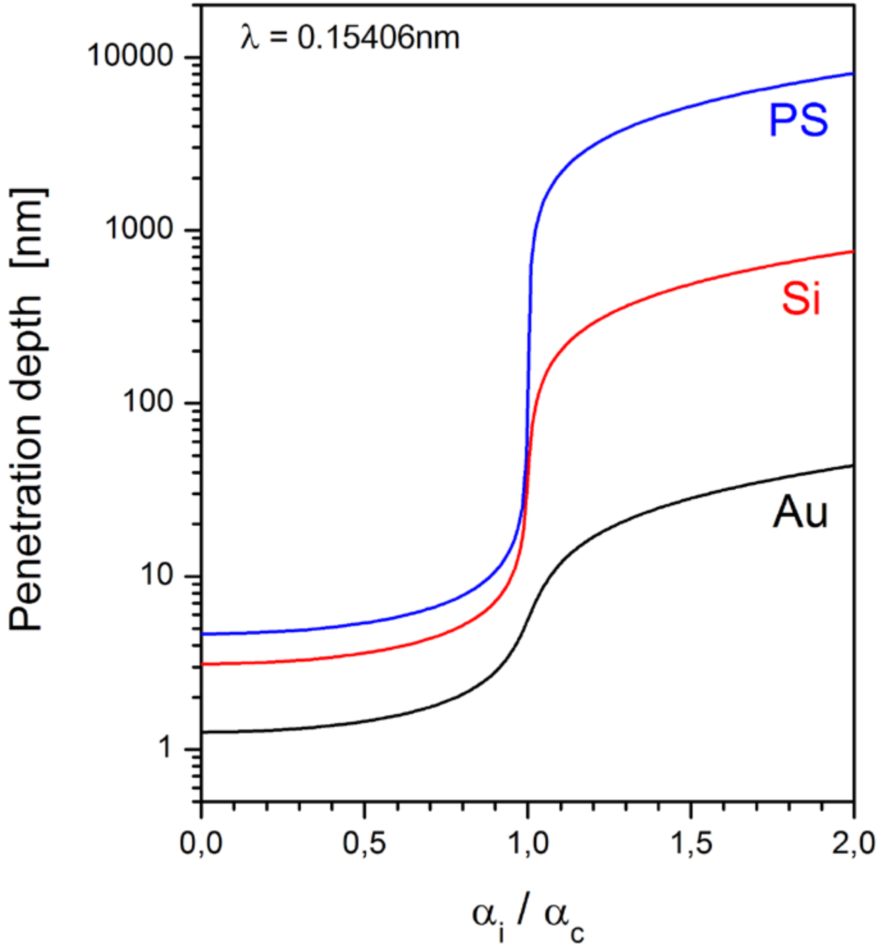
- Reflectivity



- Transmission



Penetration depth for different materials



- Minimum penetration depths

$$\Lambda_0 = \frac{1}{\sqrt{4\pi r_e \rho}}$$

r_e classical electron radius
ρ electron density

- Maximum penetration at high angles

$$\Lambda_{max} = \frac{\lambda}{2\pi\sqrt{\beta}}$$

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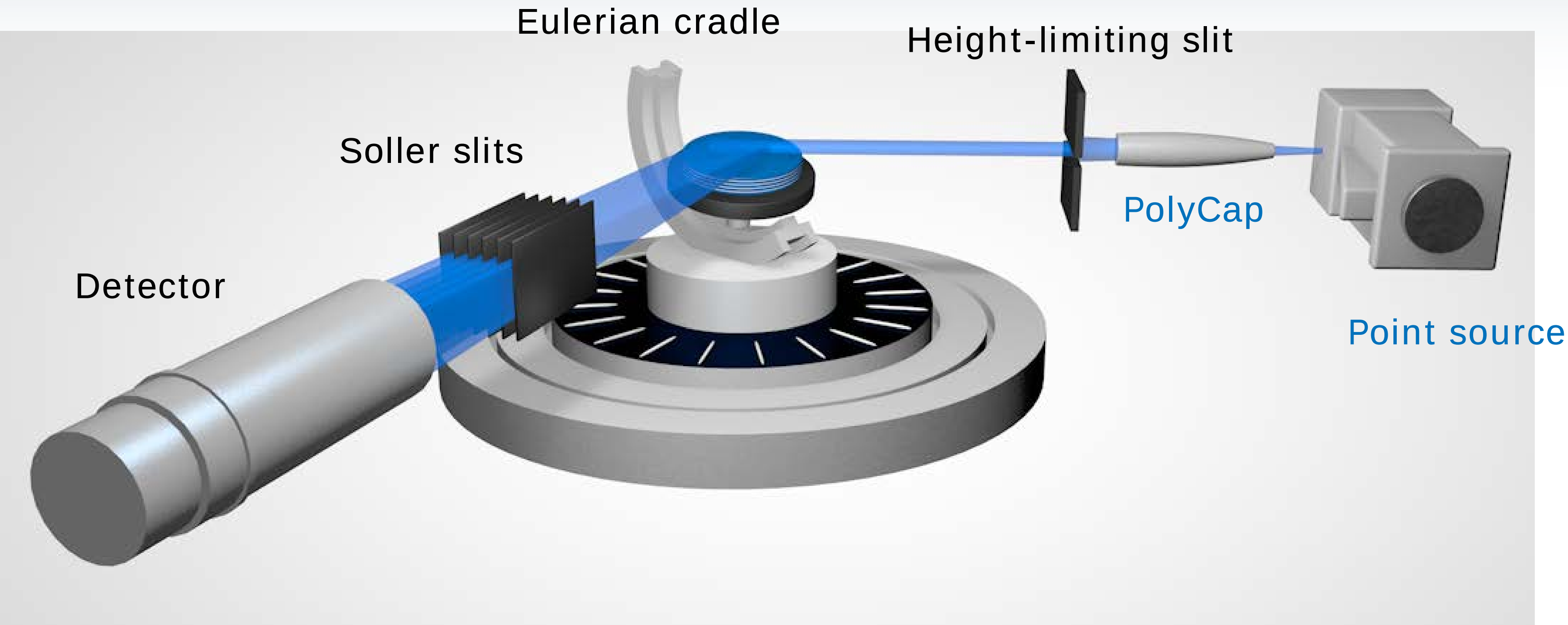
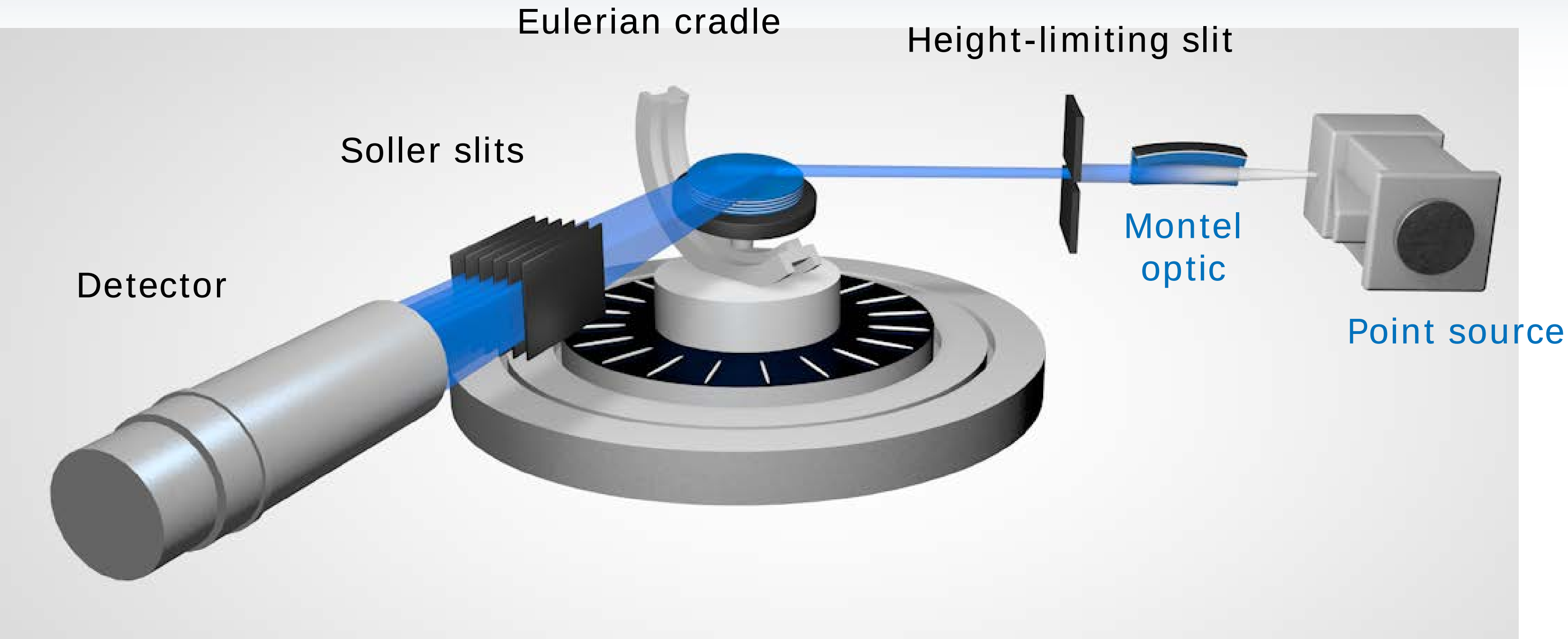
Experimental setup for IPGID with PolyCapillary



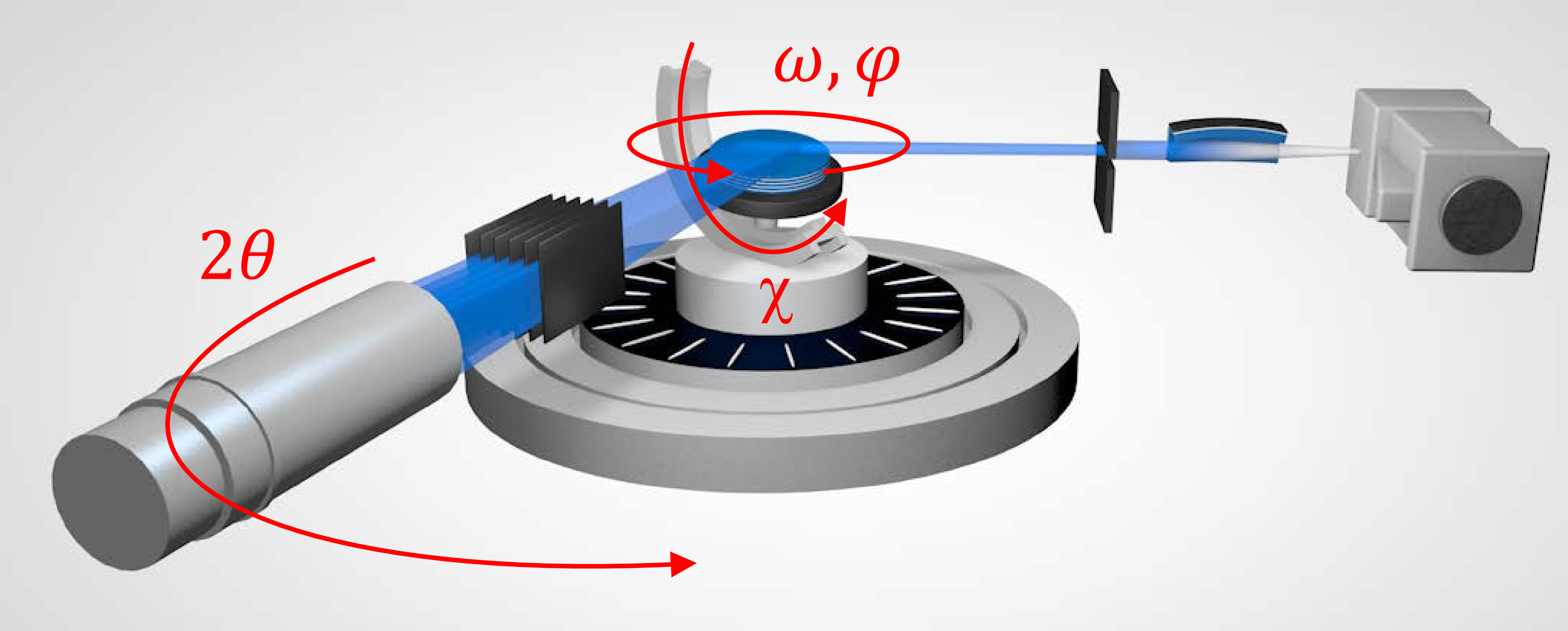
- PolyCap beam has poor resolution in surface-normal direction, no real depth control.
- Control of the incident angle via sample inclination.

Experimental setup for IPGID with Montel optic



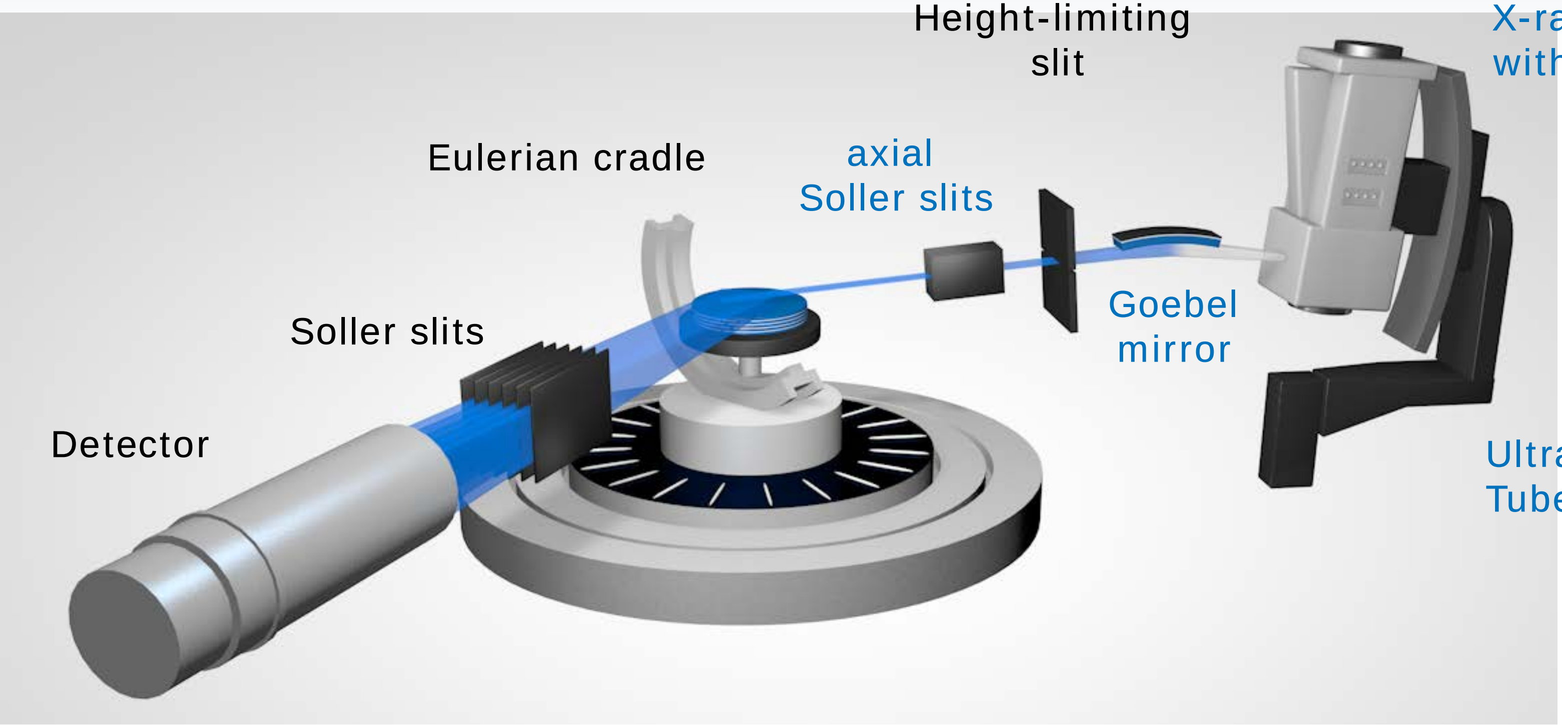
- Small incident beam (1 mm x 1 mm) with good in-plane resolution.
- Control of the incident angle via sample inclination.

Experimental setup for IPGID with fixed point source



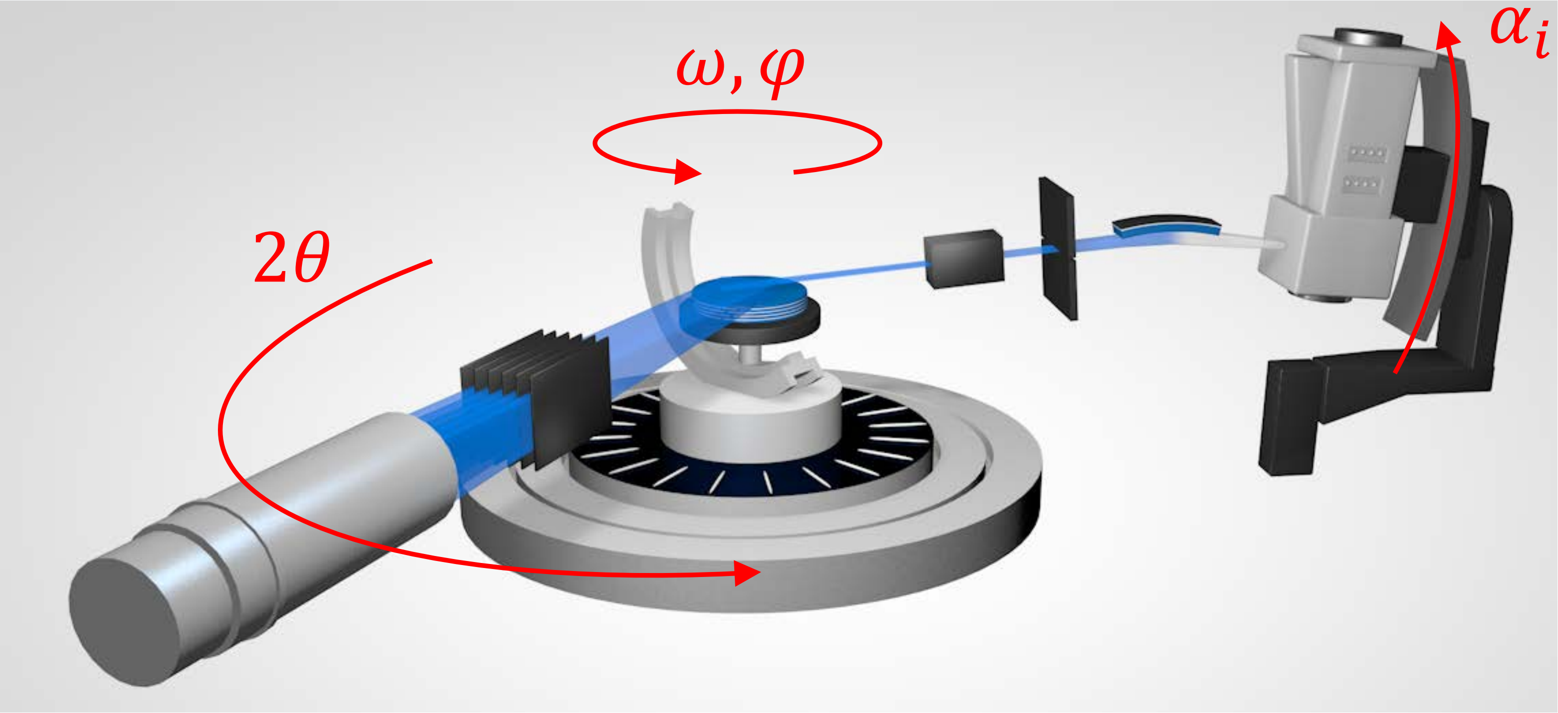
- Control of the incident angle via inclination of the sample using χ
- Not independent from ω

Ultra-GID configuration : Optimized setup for surface diffraction



- Line focus is parallel to the sample surface: Good depth control.
- Angle of incidence is controlled by a separate drive.

Ultra-GID configuration : Optimized setup for surface diffraction

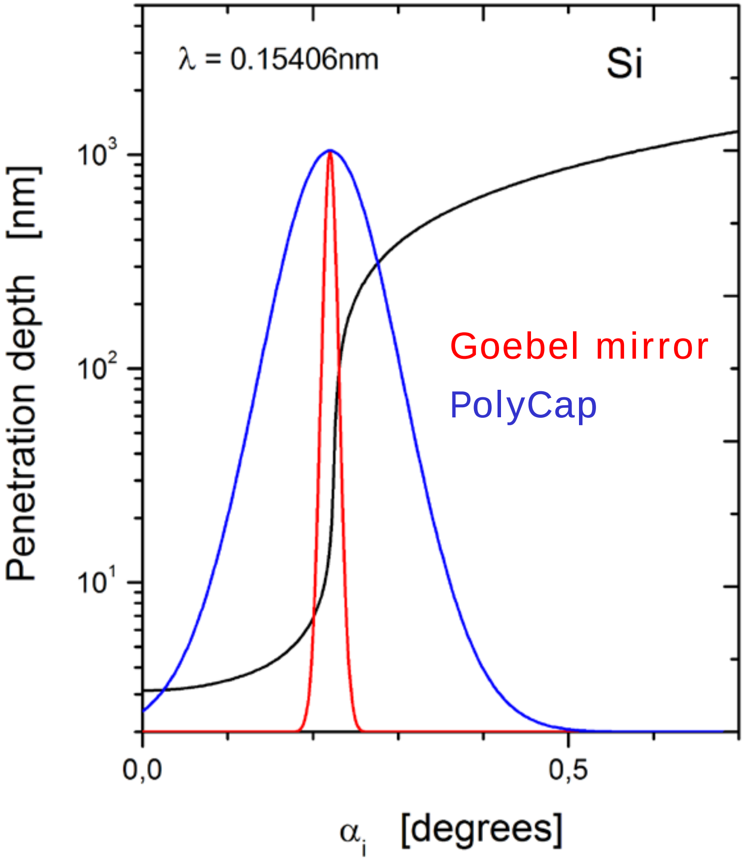


- Line focus is parallel to the sample surface: Good depth control.
- Angle of incidence is controlled by a separate drive.

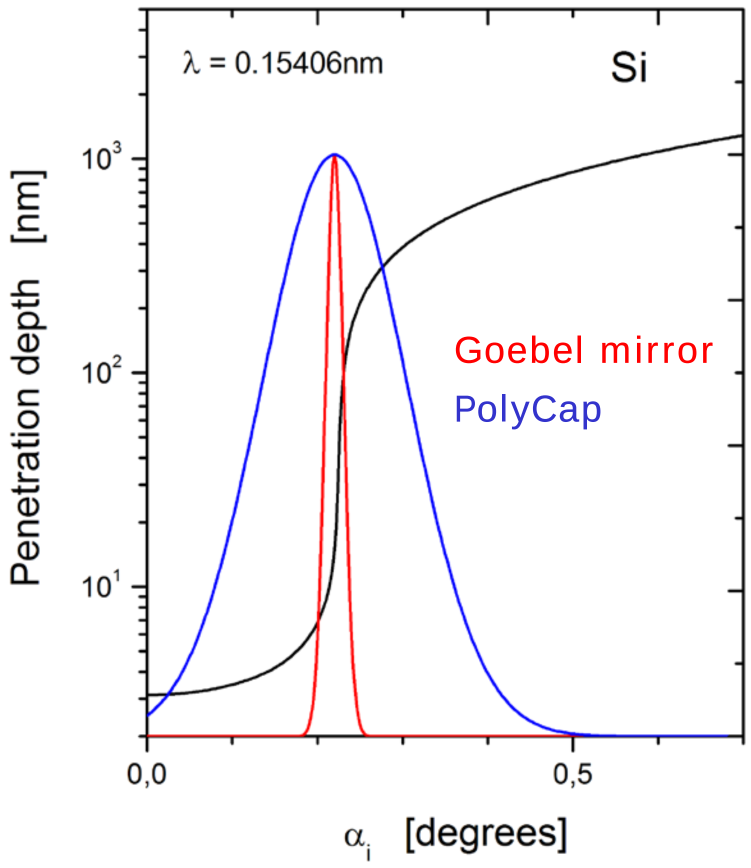
Preparing a beam with 200-µm height: Comparison of the different setups

Optic	**PolyCap**	**Montel**	**Ultra-GID with Goebel Mirror**	
Focus orientation	Point focus	Point focus	Line focus	
Resolution $q_{surface}$	0.2°	0.05°	0.023°	
Resolution $q_{in\text{-}plane}$	0.2°	0.05°	0.2° soller	0.5° soller
Beam width	5 cm	1 mm	16 mm	16 mm
Spectral purity	Tube spectrum	Cu K$\alpha_{1,2}$, few Kβ	Cu K$\alpha_{1,2}$	

Choice of the incident beam optics



Choice of the incident beam optics



- The optimum choice of the incident beam configuration depends on the sample.
- Depth control requires good resolution perpendicular to the surface -> Goebel mirror
- Epitaxial samples with low mosaicity will except only small angular range of the incident beam -> Goebel mirror
- Polycrystalline samples and thick layers can be measured with a higher beam spread -> PolyCap

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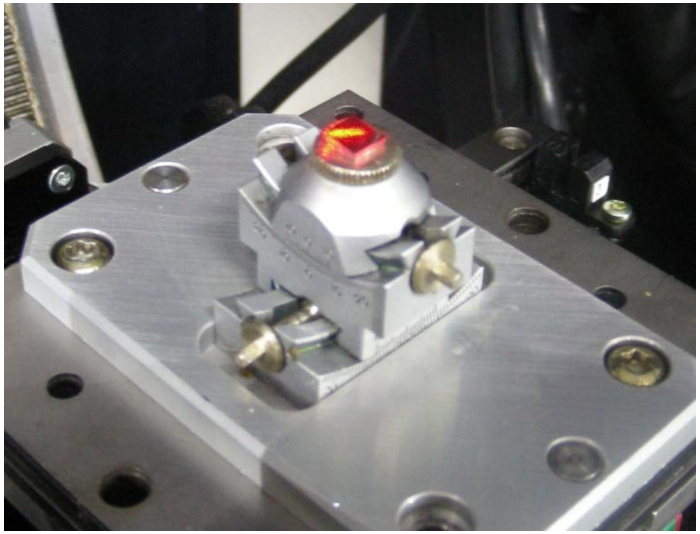
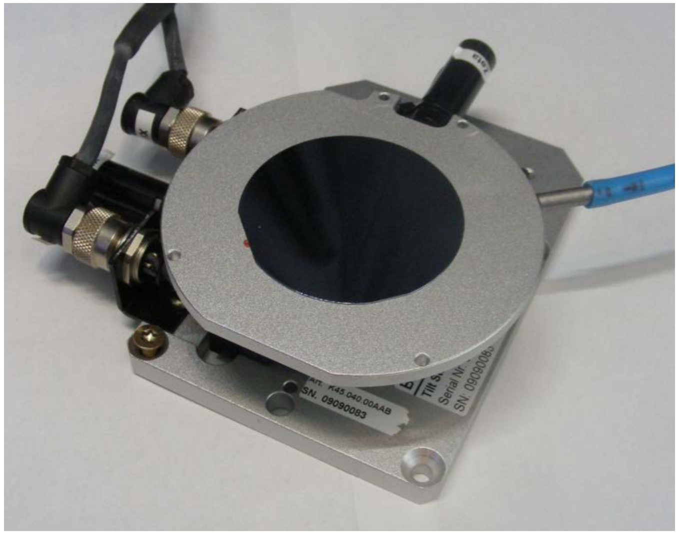
Aligning the sample surface parallel to the φ-axis : tilt stages

- Manual goniometer head



- For small samples
- Optical alignment using a laser beam

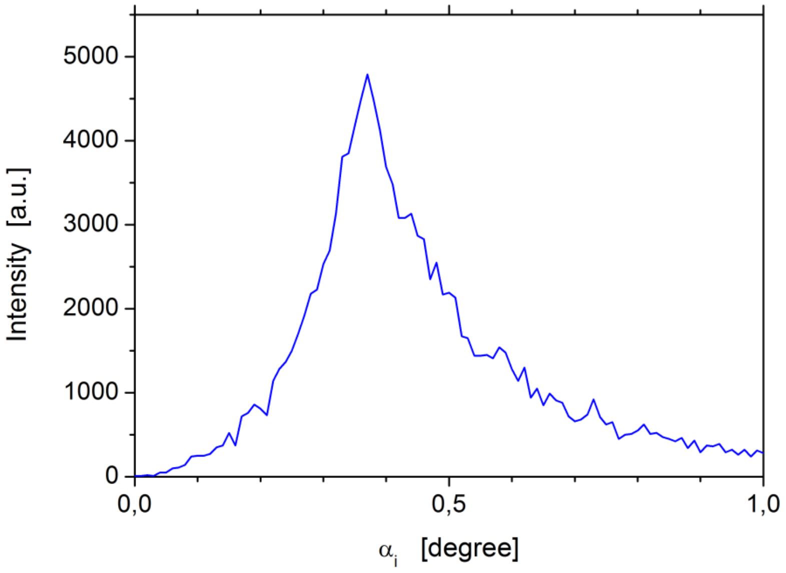
- Motorized tilt stage



- For larger samples fixed by vacuum
- Can use the X-ray beam for surface alignment

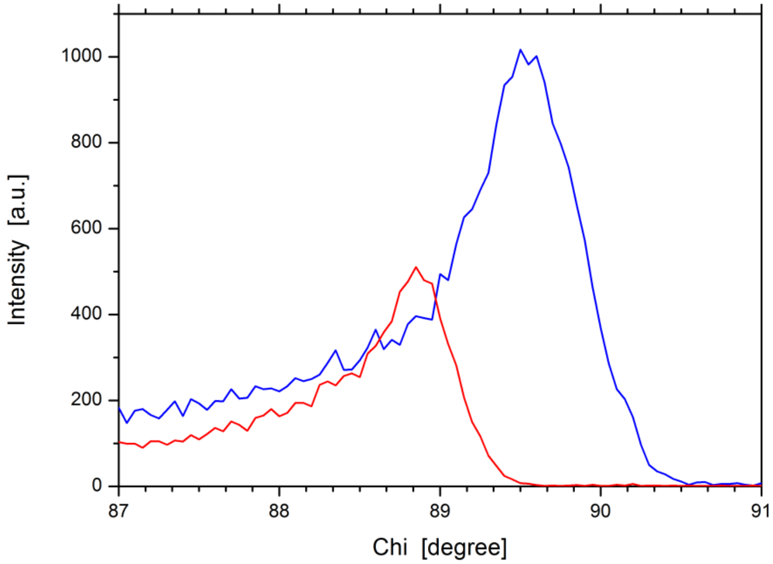
Optimizing the angle of incidence

- With Ultra-GID tube stand



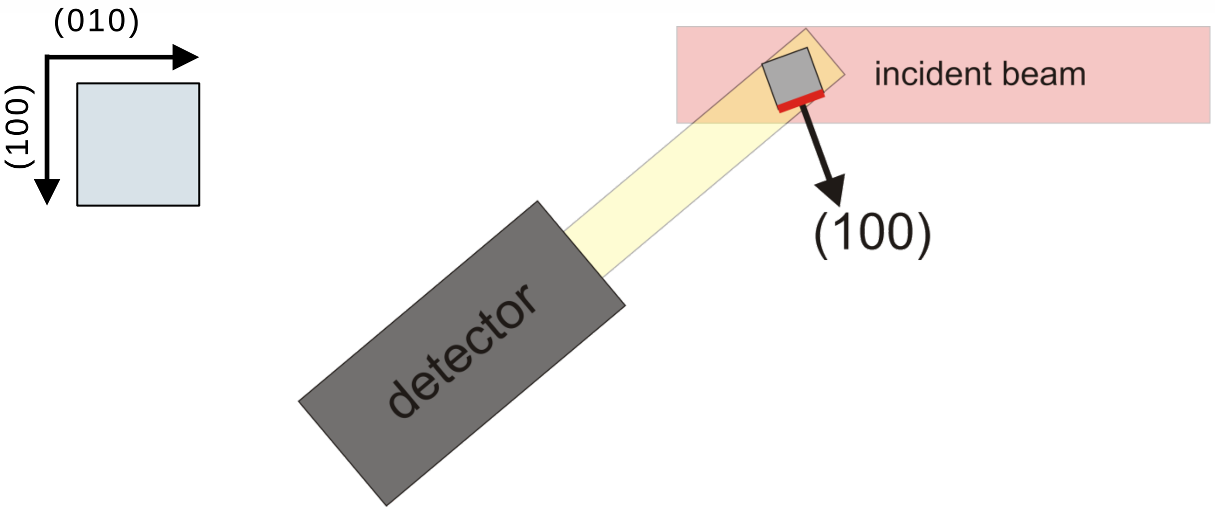
- Alignment of the optimal angle of incidence by α_i drive.
- Independent of ω.

- With fixed incident beam

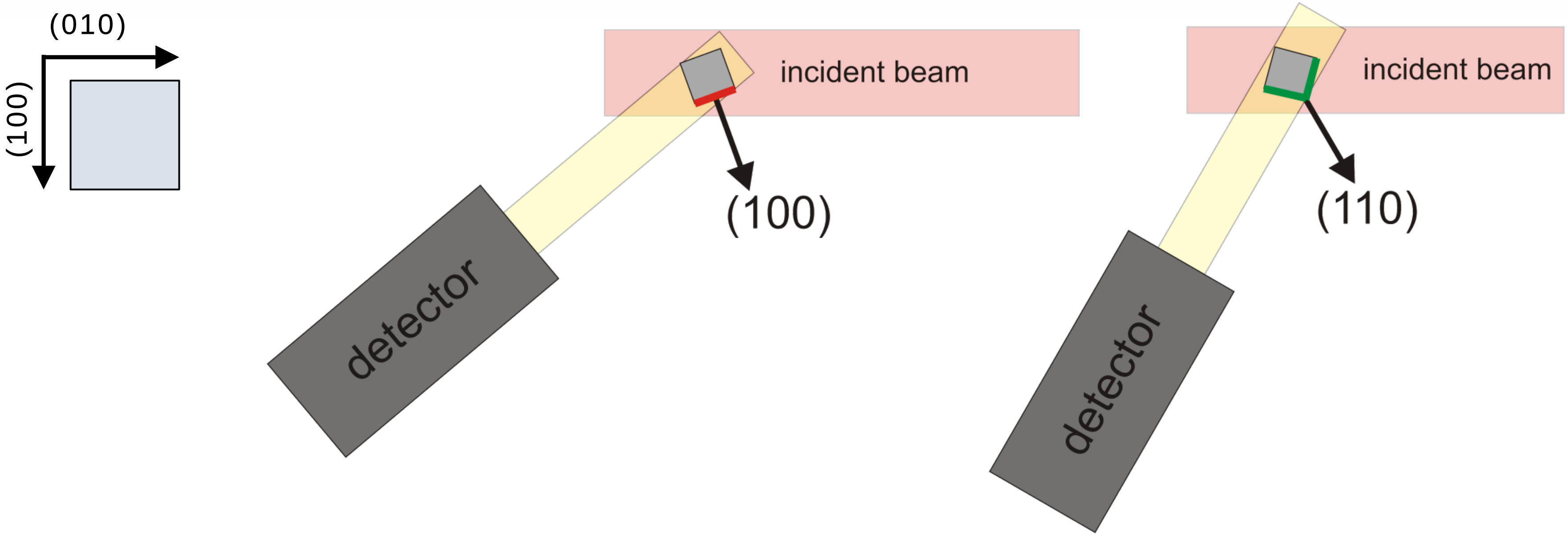


- Using χ for inclining the sample surface.
- $\alpha_i = \cos(\chi)\,\omega$. Depends on ω.

Choosing the appropriate reflection to avoid substrate scattering

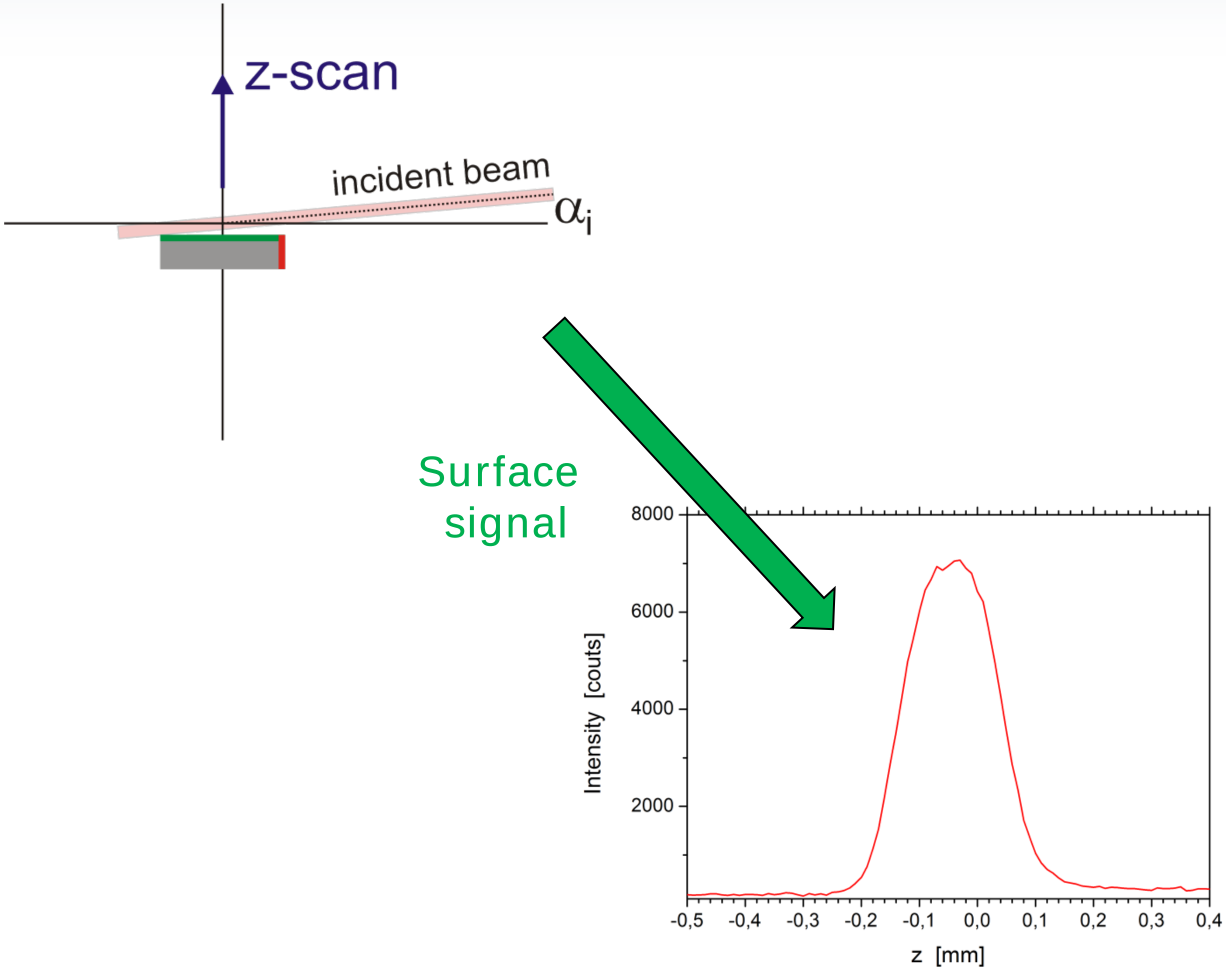


Choosing the appropriate reflection to avoid substrate scattering



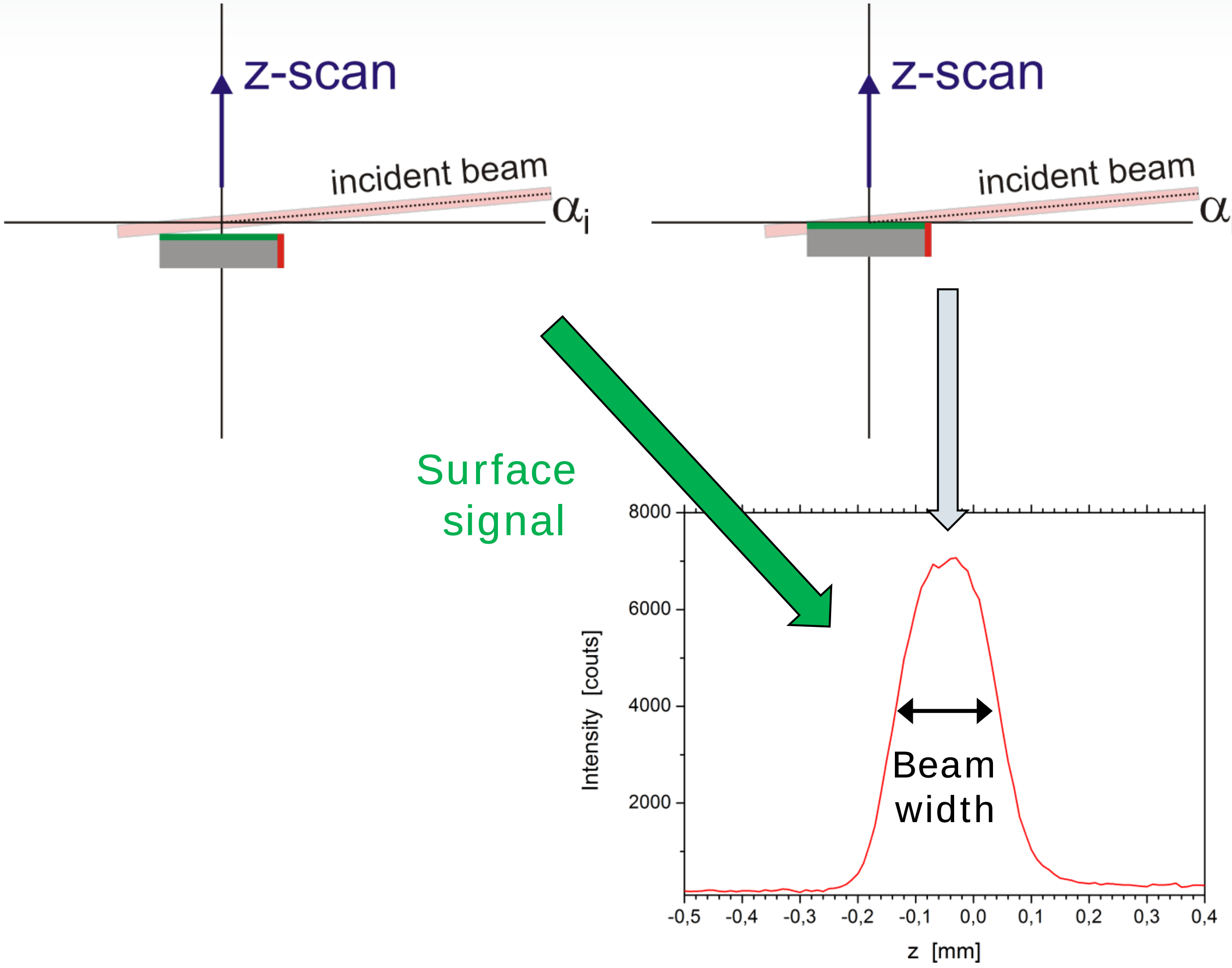
- Not the reflection with the highest intensity is aways the best choice.
- Choosing an appropriate reflection helps to avoid scattering from the substrate edge.

Alignment tips: Change sample height to avoid substrate scattering



Surface signal

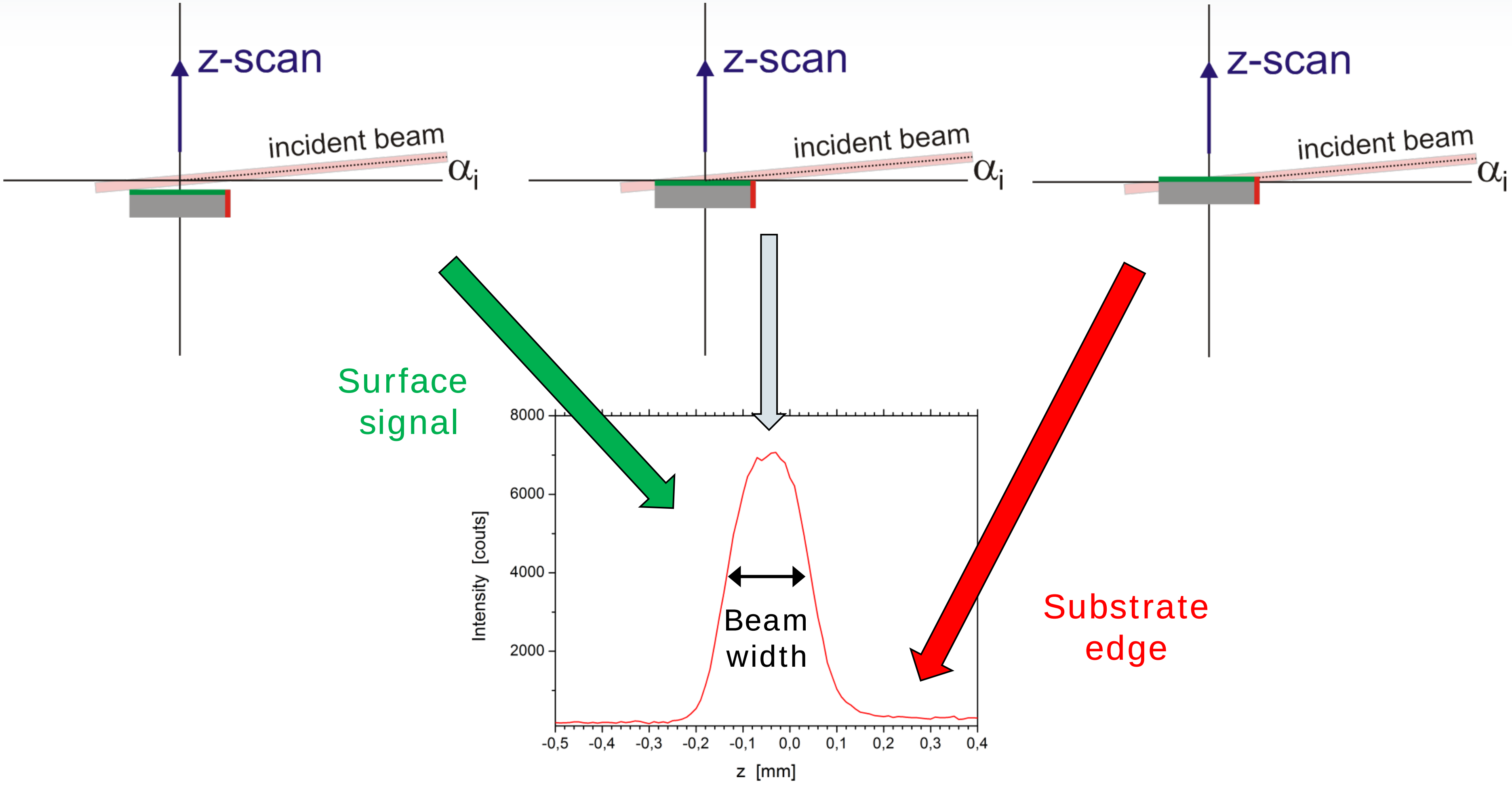
Alignment tips: Change sample height to avoid substrate scattering



Surface signal

Beam width

Alignment tips: Change sample height to avoid substrate scattering

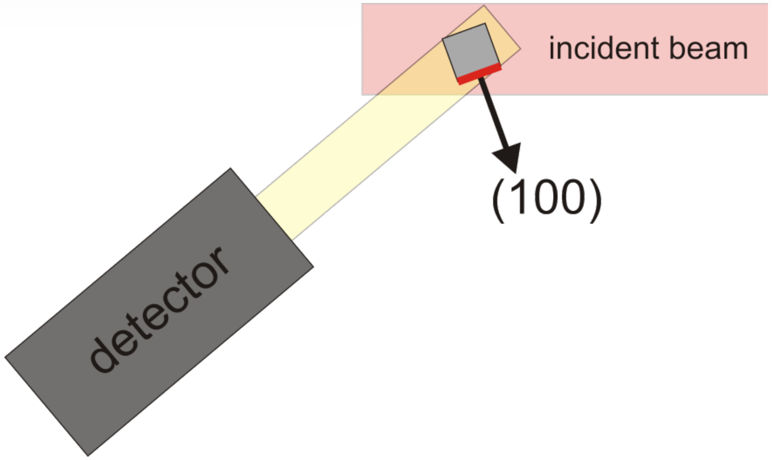


Surface signal

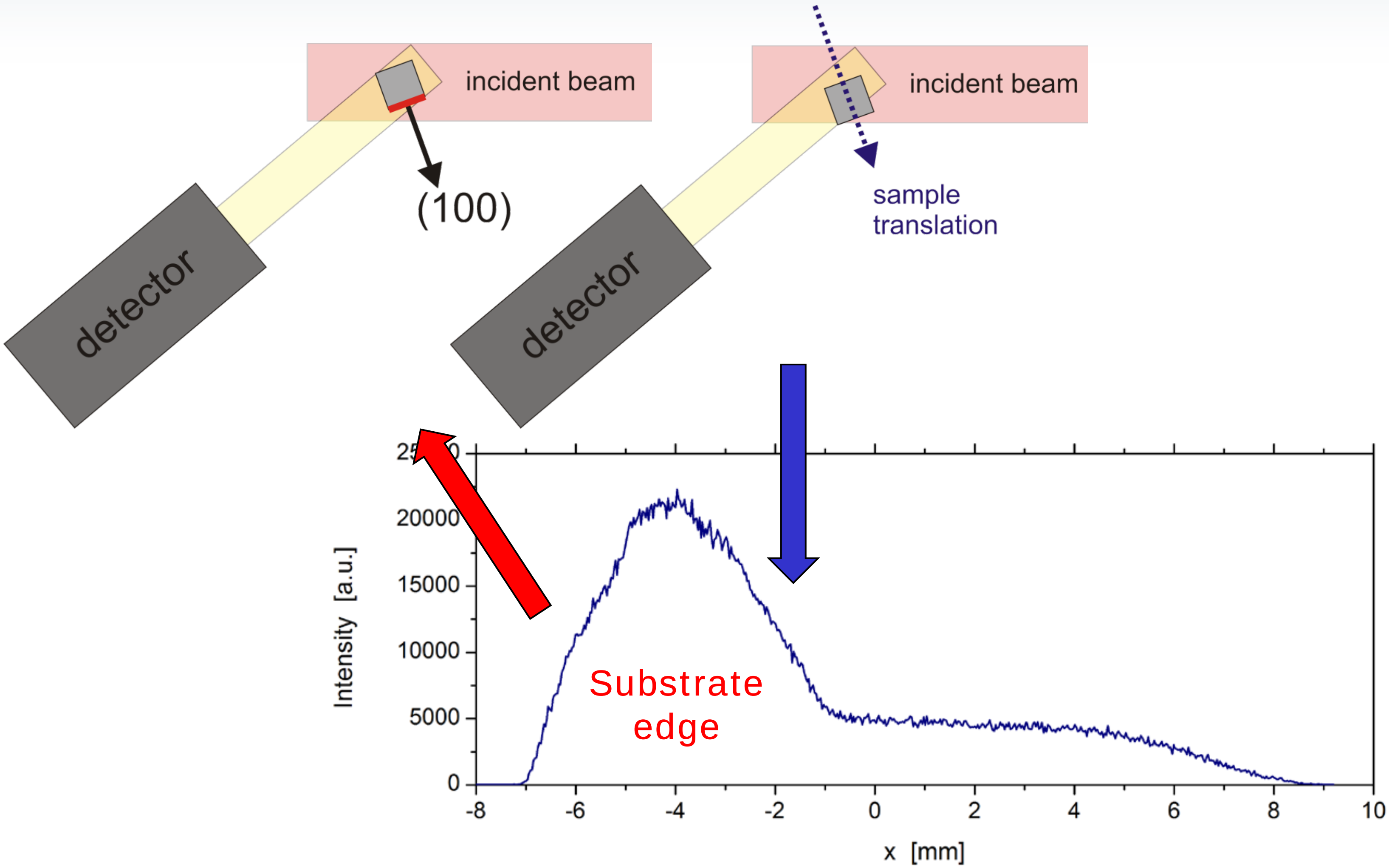
Beam width

Substrate edge

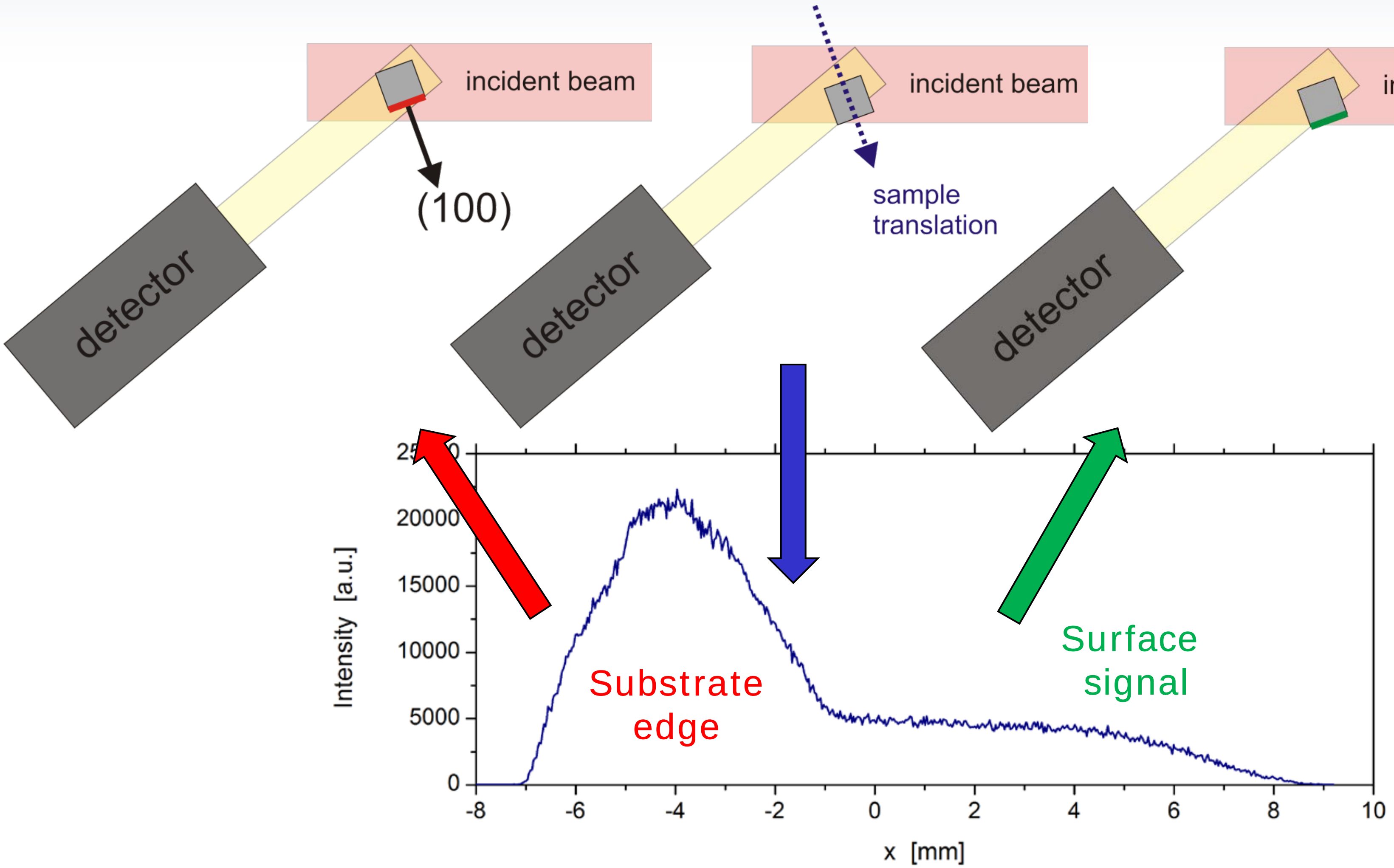
Alignment tips: Sample translation to avoid substrate scattering



Alignment tips: Sample translation to avoid substrate scattering



Alignment tips: Sample translation to avoid substrate scattering



- Introduction
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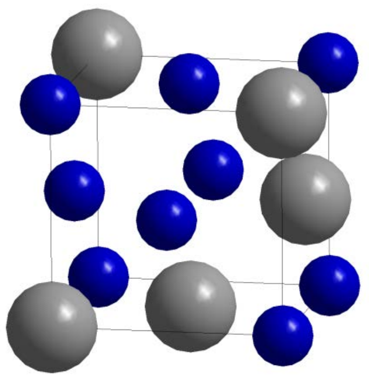
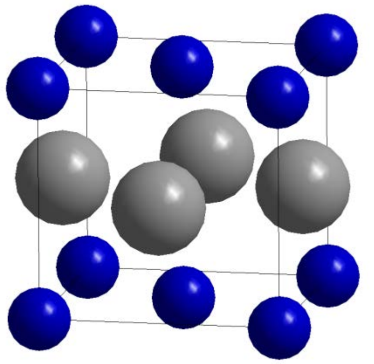
- Examples
  - Polycrystalline samples
  - Epitaxial grown samples
  - In-plane diffraction with 1D detector

Structure determination of polycrystalline FePt thin films

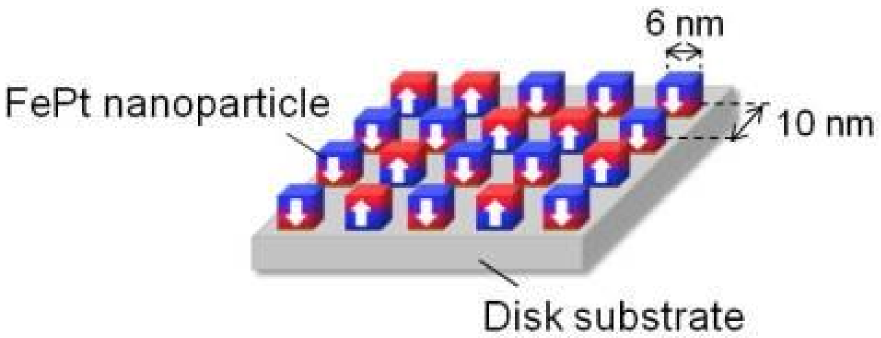
- FePt is promising material for magnetic mass storage devices
- A1 phase (face-centered cubic)
- L1$_0$ phase (face-centered tetragonal) ferromagnetic
- Thickness of the FePt film: 10 nm



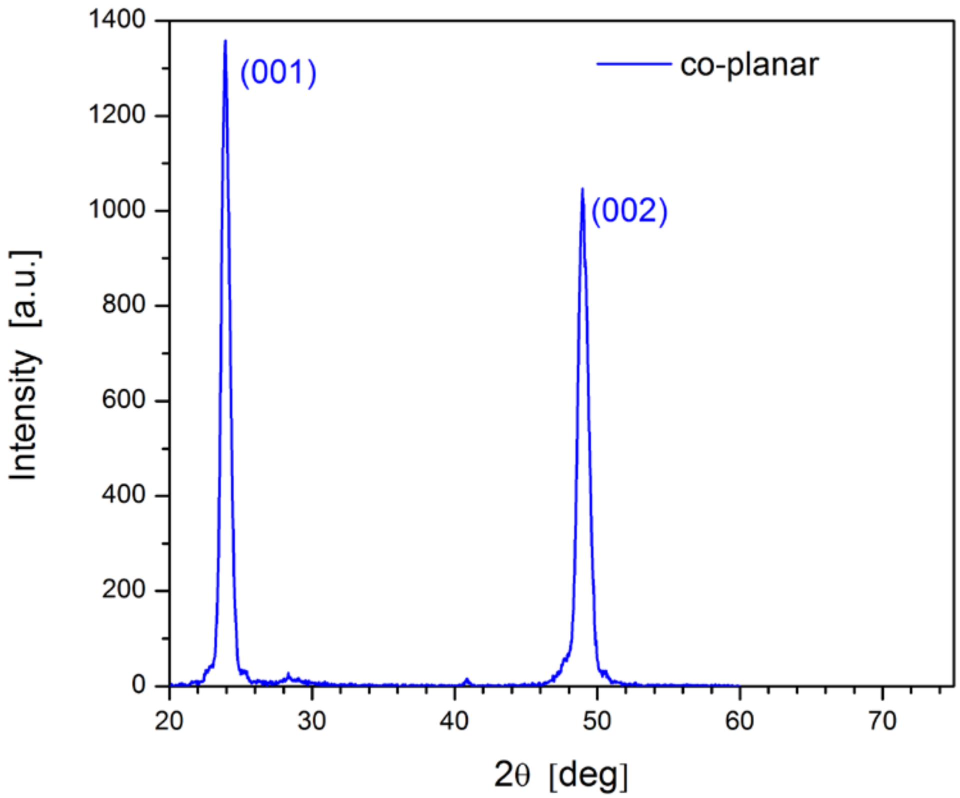
FePt - A1 phase



FePt - L1$_0$ phase

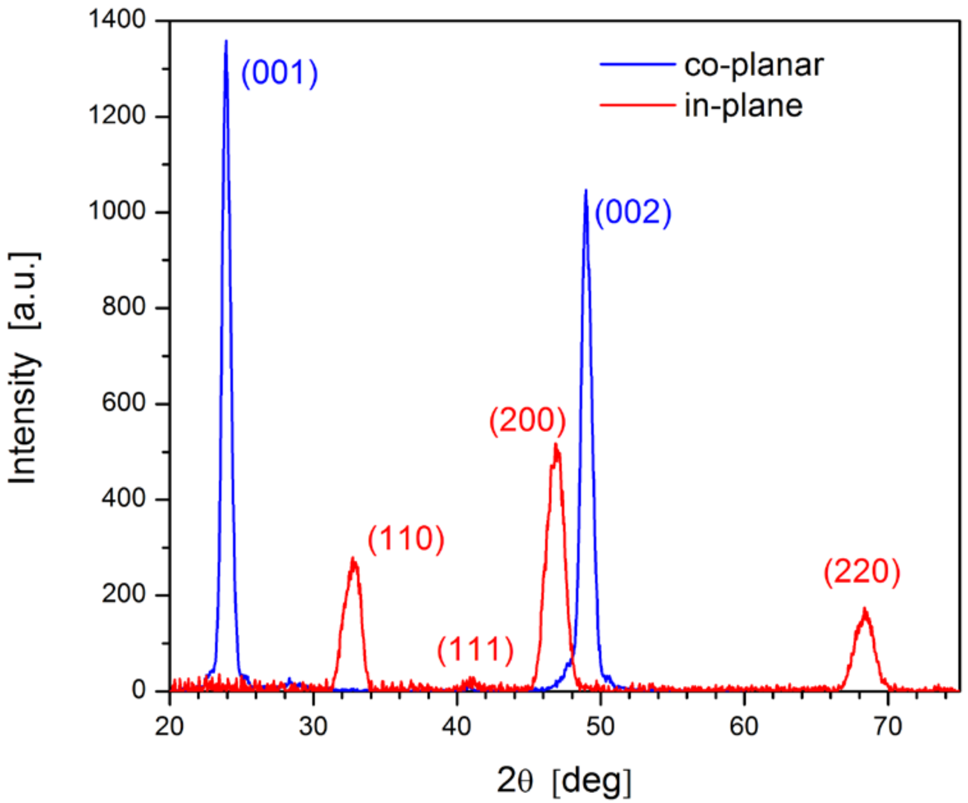


Structure determination of polycrystalline FePt thin films



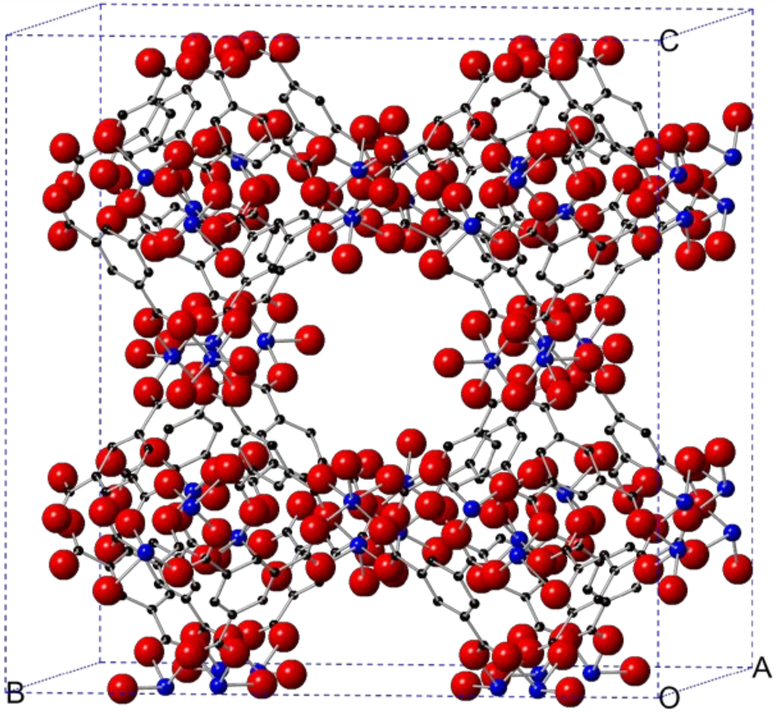
- Crystallite size in the surface normal direction is about the film thickness.

Structure determination of polycrystalline FePt thin films



- Crystallite size in the surface normal direction is about the film thickness.
- In-plane crystallite size is about 6.5 nm
- In-plane fiber textured around (001)

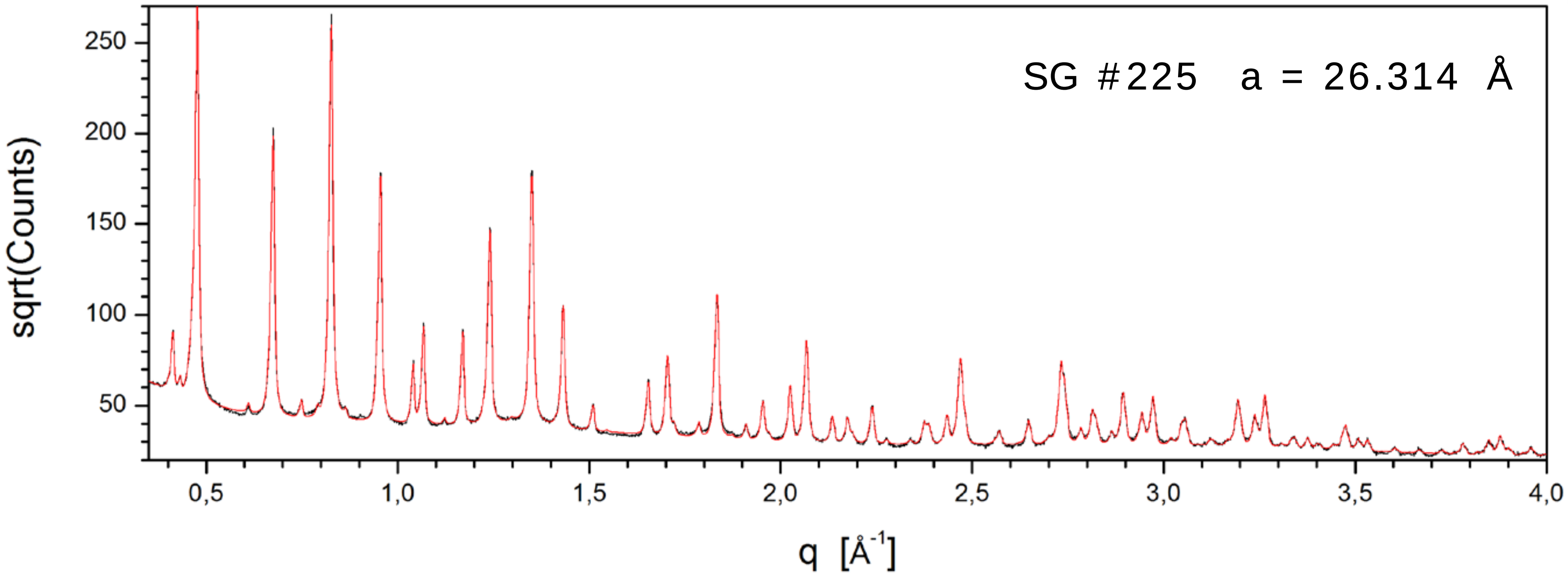
In-plane GID on Metal-organic frameworks (MOF‘s)



MOF samples kindly provided by P. Weidler, IFG, KIT / Germany

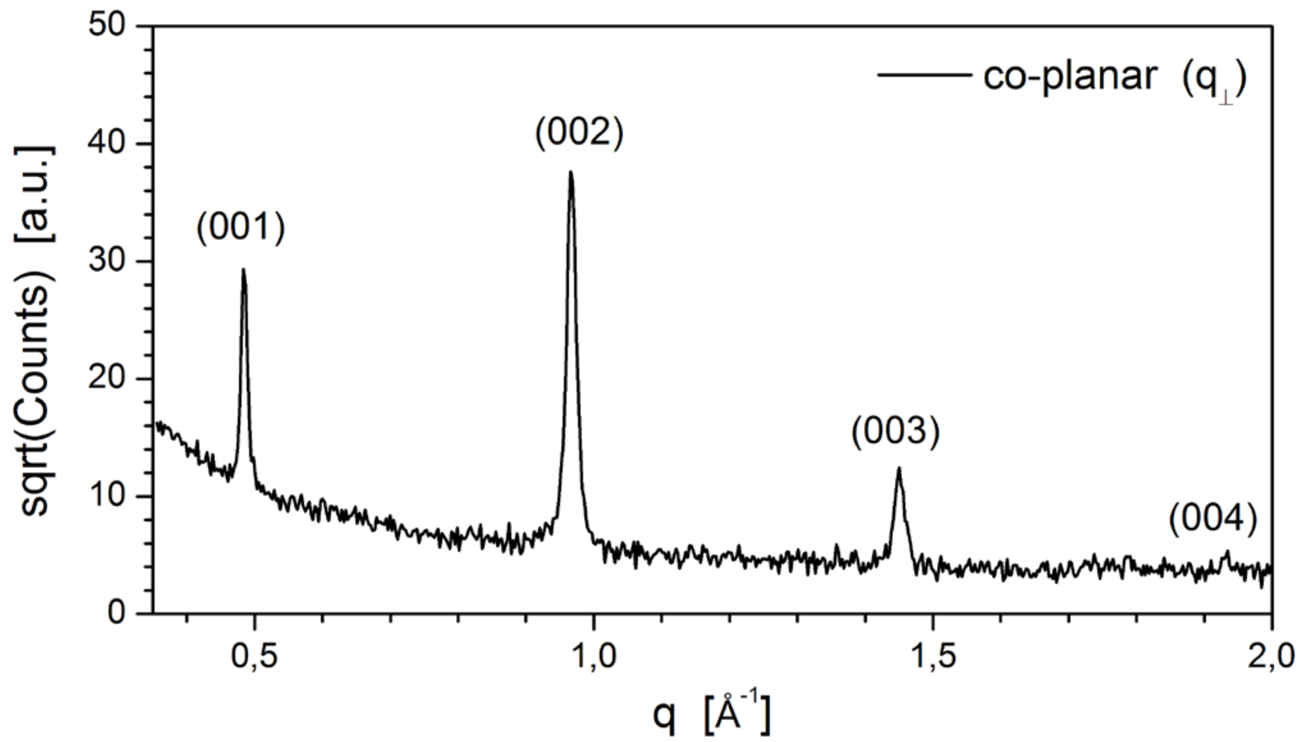
- Topic of current research
- Incorporation of nanoparticles, e.g. Au_9or Au_{55} : controlling refractive index
- Drug carrier and release systems
- HKUST-1 : $C_{18}H_6Cu_3O_{12}$
- Space group 225 with lattice constant a = 26.314 Å.

In-plane GID on Metal-organic frameworks (MOF‘s)



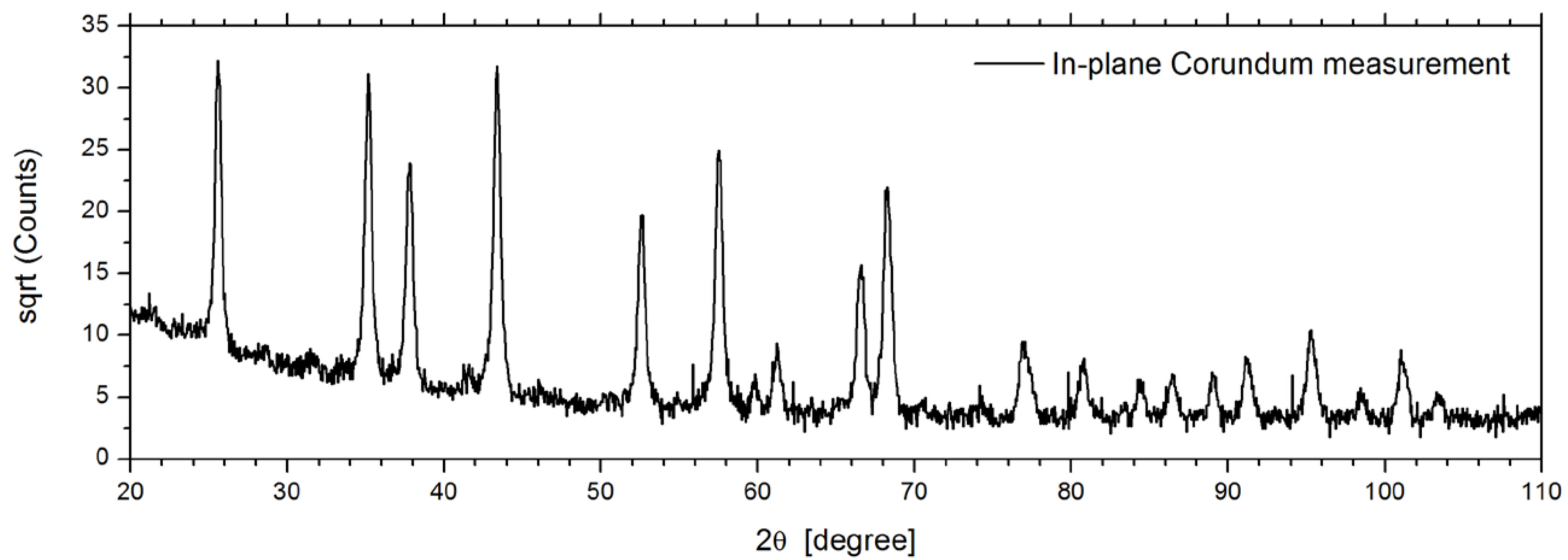
- Measurement of HKUST-1 powder provides structure information.
- Crystallite size is about 195 nm.

In-plane GID on Metal-organic frameworks (MOF‘s)



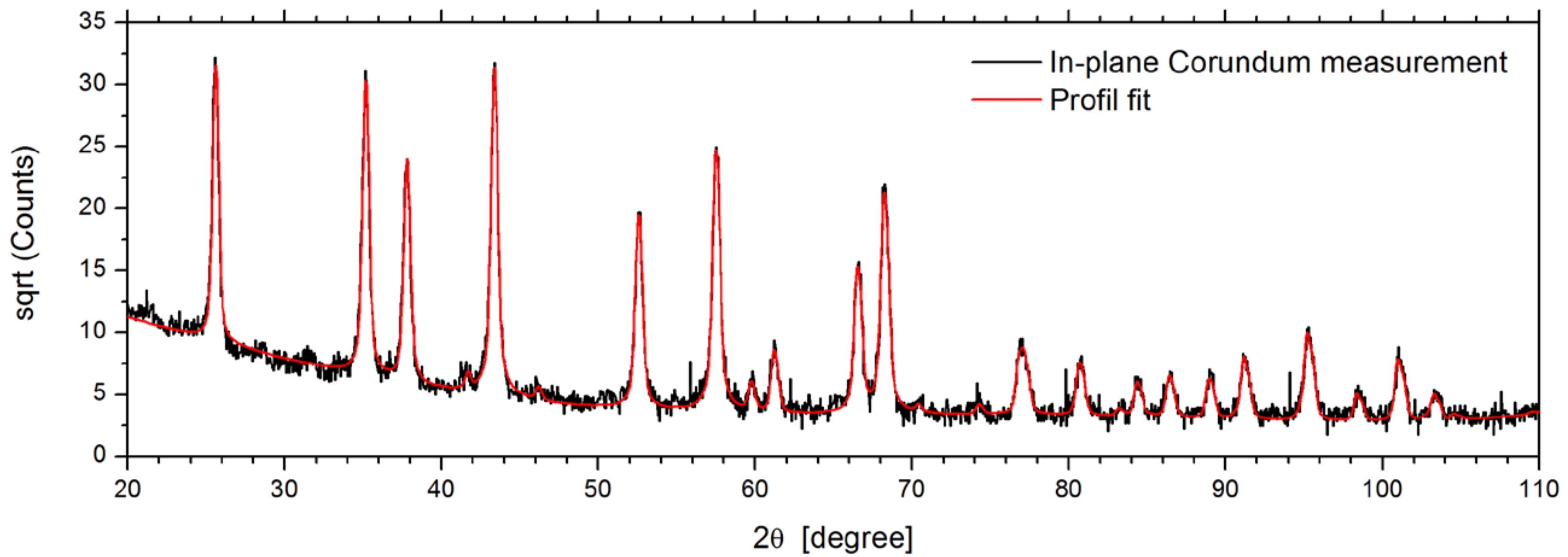
- MOF crystallites with (001) orientation and size of about 90 nm.

Determination of the in-plane resolution function

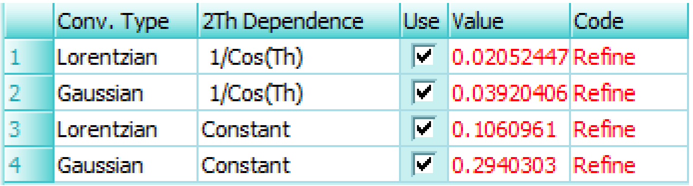


- Precise determination of the crystallite size requires knowledge of the resolution function for the used experimental setup.
- Use polycrystalline sample with high crystallite size, e.g. NIST SRM 1976 (Corundum).

Determination of the in-plane resolution function

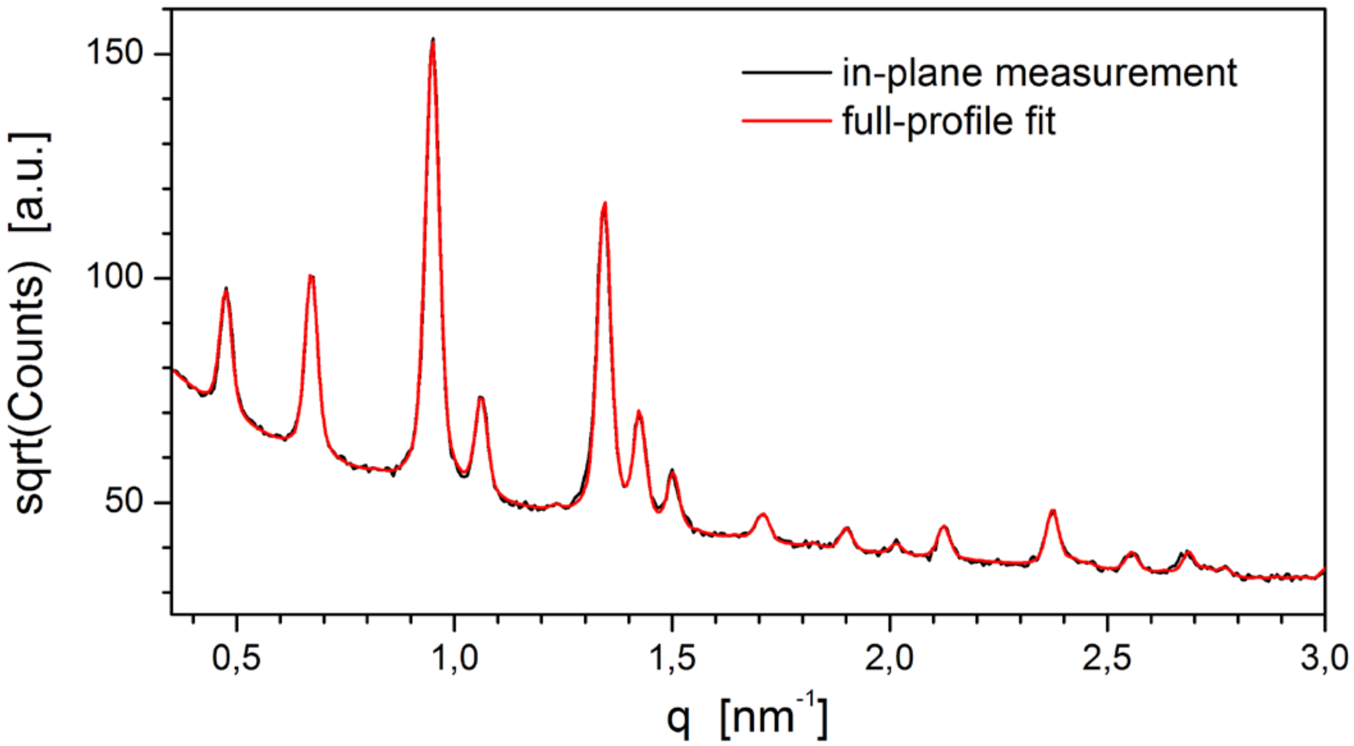


- Full profile fit provides the resolution function.



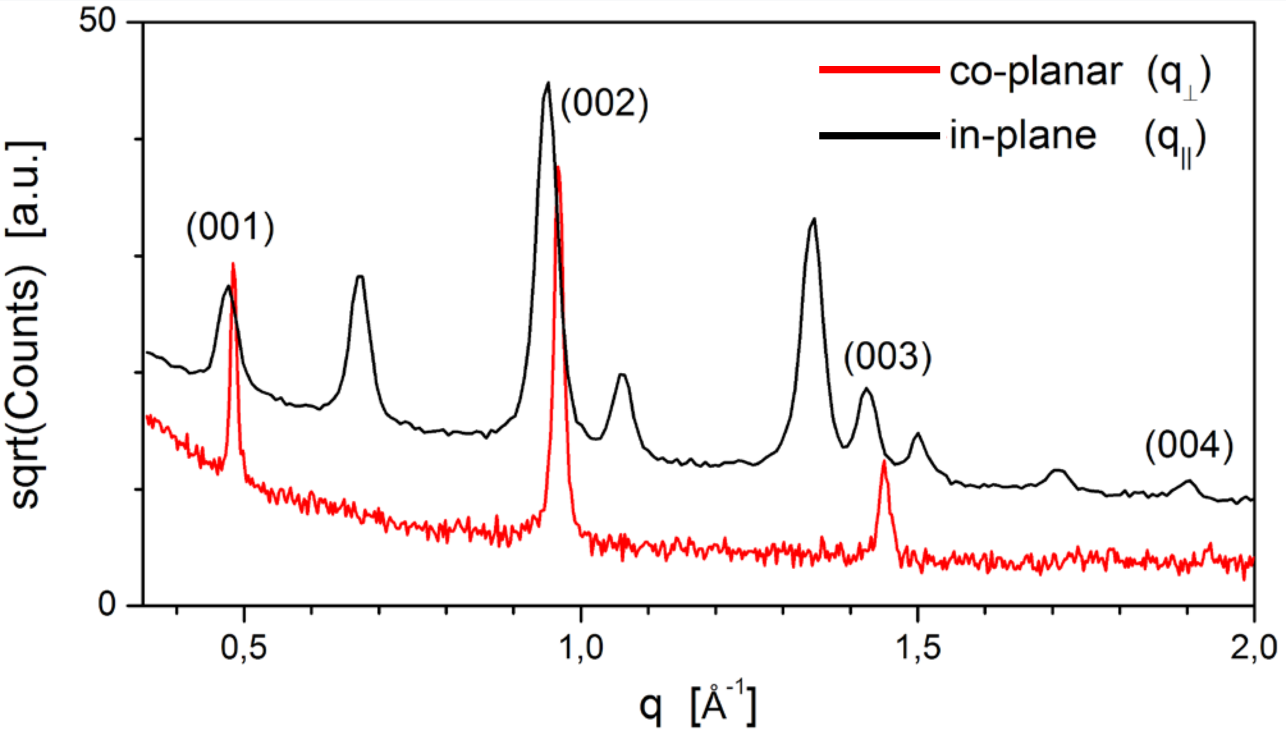
	Conv. Type	2Th Dependence	Use	Value	Code
1	Lorentzian	1/Cos(Th)	☑	0.02052447	Refine
2	Gaussian	1/Cos(Th)	☑	0.03920406	Refine
3	Lorentzian	Constant	☑	0.1060961	Refine
4	Gaussian	Constant	☑	0.2940303	Refine

Determination of the in-plane crystallite size



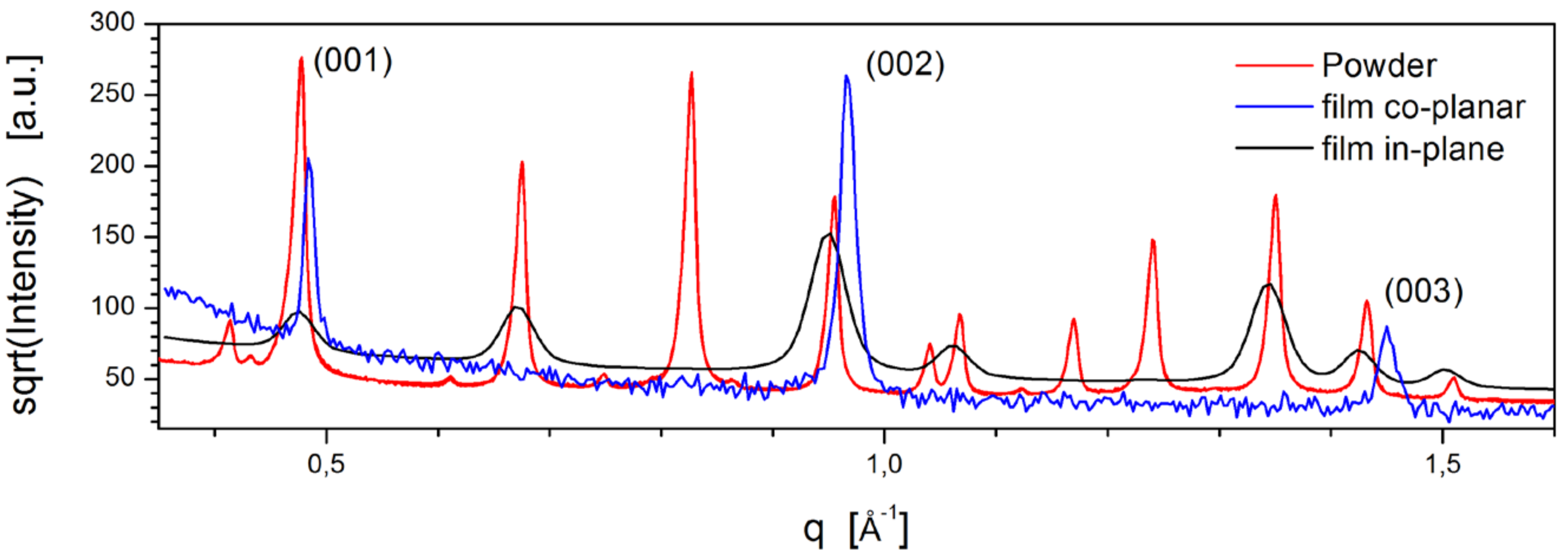
- Use the known resolution function
- Full profile fit yields 120-nm crystallite size parallel to the surface.

In-plane GID on Metal-organic frameworks (MOF‘s): crystallite size



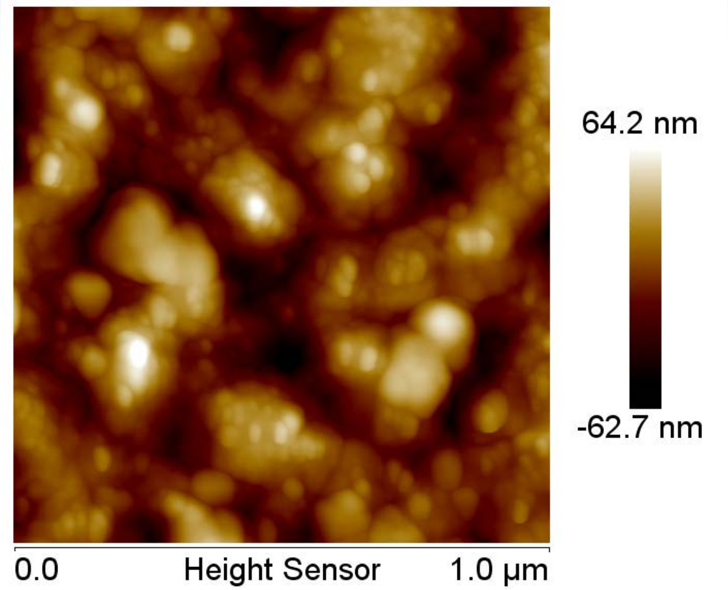
- MOF crystallites with (001) surface normal and size of about 90 nm.
- Fiber textured with 120 nm crystallite size parallel to the surface.

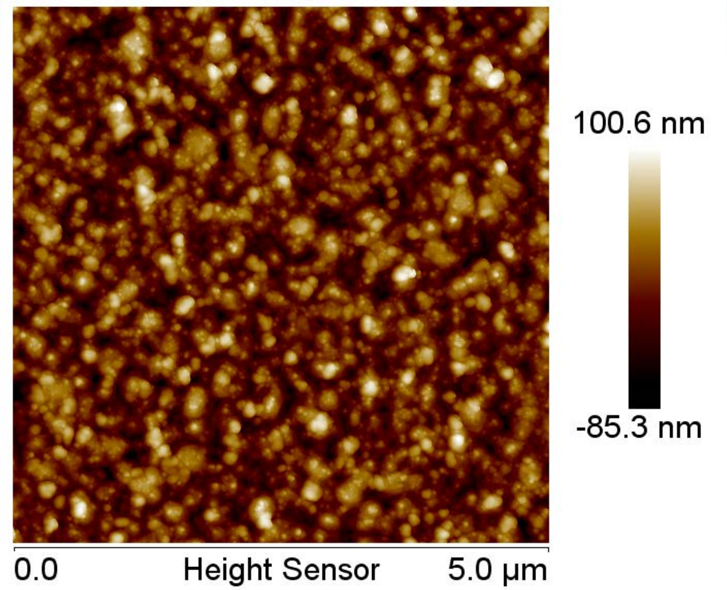
In-plane GID on Metal-organic frameworks (MOF‘s): lattice constants



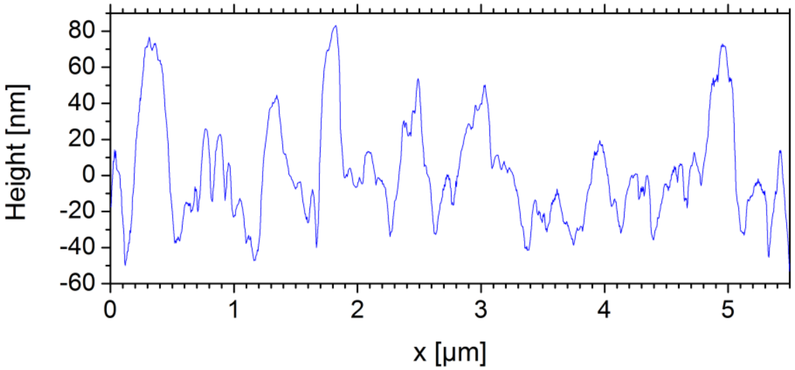
- MOF crystallites with (001) surface normal and size of about 90 nm.
- In-plane lattice parameter: 26.482 Å   Crystallite size about 120 Å
- Co-planar lattice parameter: 26.0055 Å

In-plane GID on Metal-organic frameworks (MOF‘s): AFM pictures





- The AFM pictures yield particles with size of 250-350 nm.
- This is not the crystallite size.



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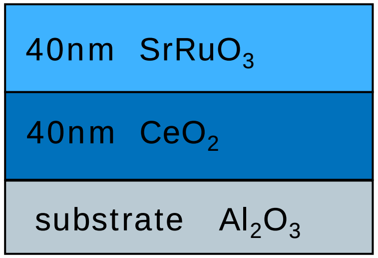
Application 3

Probing in-plane symmetry directly

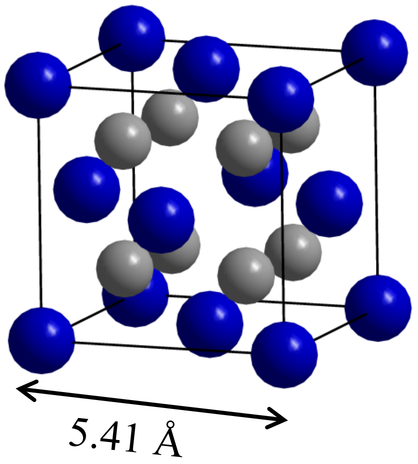


40nm $SrRuO_3$

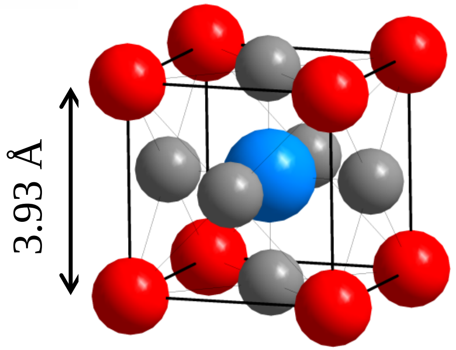
40nm CeO_2

substrate Al_2O_3

By Madhana Sunder,
Bruker AXS, Madison(WI) *



CeO_2



$SrRuO_3$

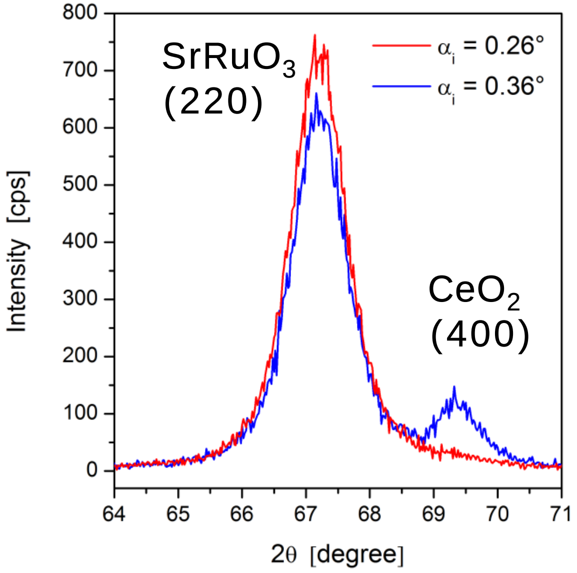
- Determine the epitaxial relationship.
- Based on lattice mismatch one would expect the unit cells to exhibit a twisted cube on cube epitaxy.

---

* *Growth of heteroepitaxial single crystal Lead Magnesium Niobate-Lead Titanate thin films on r-plane Sapphire substrates*, Doctoral dissertation, **Madhana Sunder**, 2009

Application 3
Probing in-plane symmetry directly

- 2θ/ω-scan at $SrRuO_3$ (220)

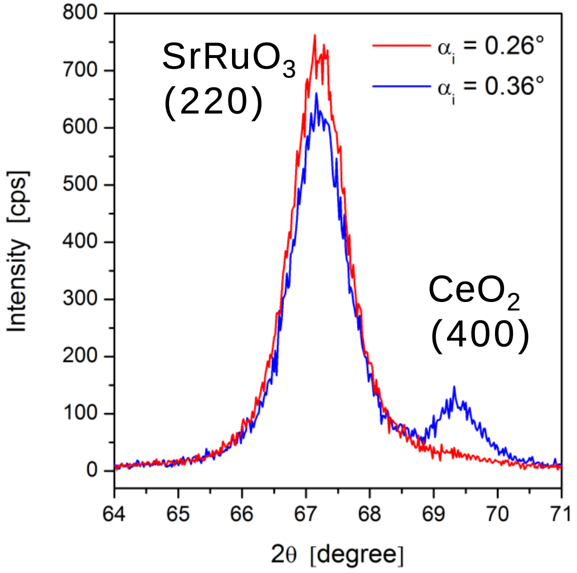


- $SrRuO_3$ (220) || CeO_2 (100)
- Clear isolation of $SrRuO_3$ (220) reflection by depth control

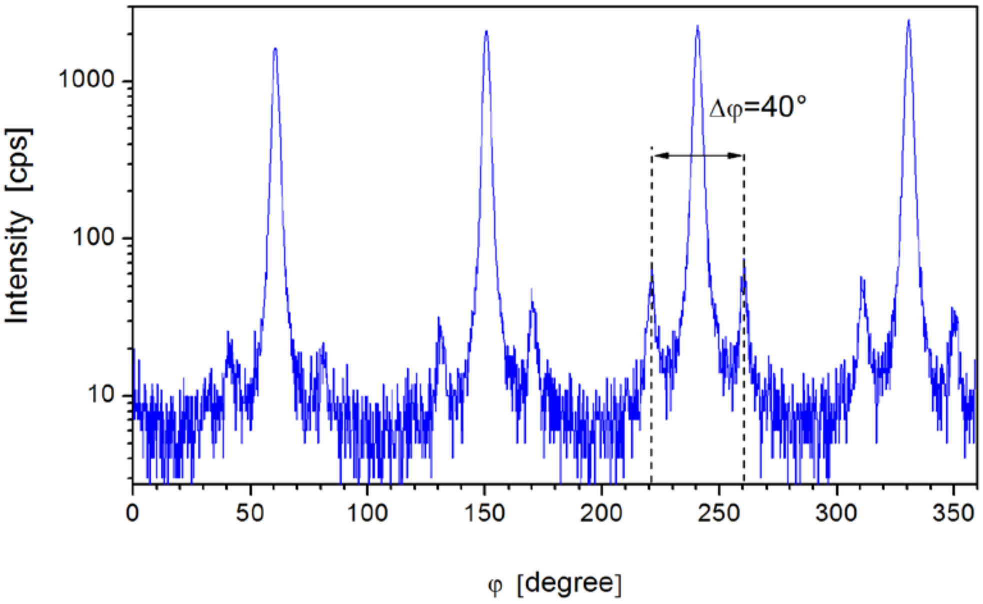
Application 3

Probing in-plane symmetry directly

- 2θ/ω-scan at $SrRuO_3$ (220)



- φ-scan at 2θ of $SrRuO_3$ (220)



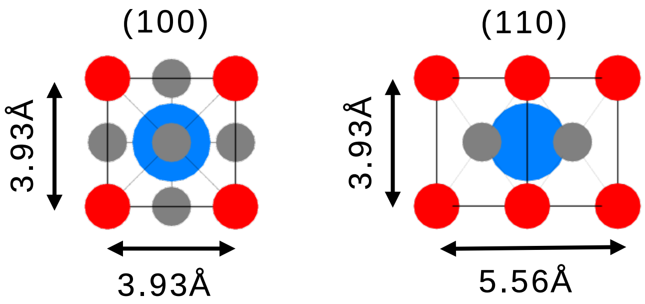
- $SrRuO_3$ (220) || CeO_2 (100)
- Clear isolation of $SrRuO_3$ (220) reflection by depth control

- A simple rotation of the sample around the surface normal directly reveals the in-plane symmetry.
- Requires surface normal || φ-axis.

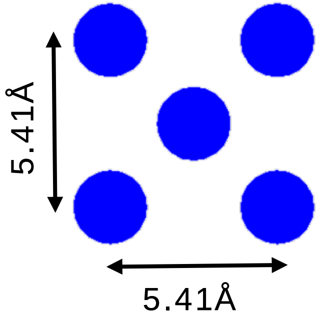
Application 3

Probing in-plane symmetry directly

SrRuO$_3$



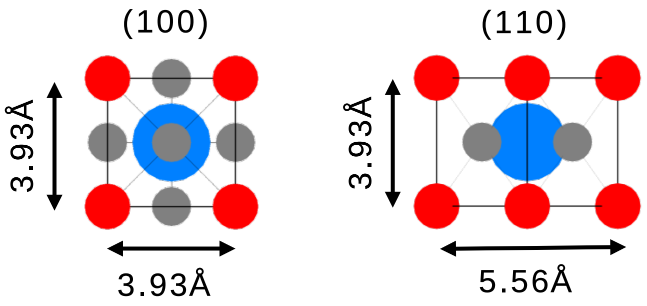
CeO$_2$



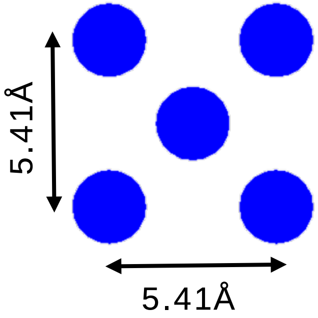
Application 3

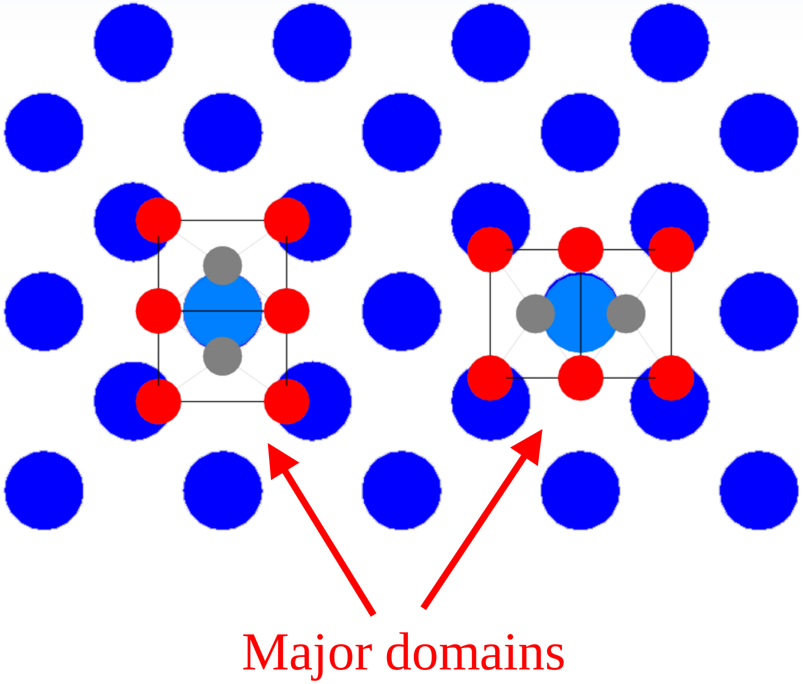
Probing in-plane symmetry directly

$SrRuO_3$



CeO_2

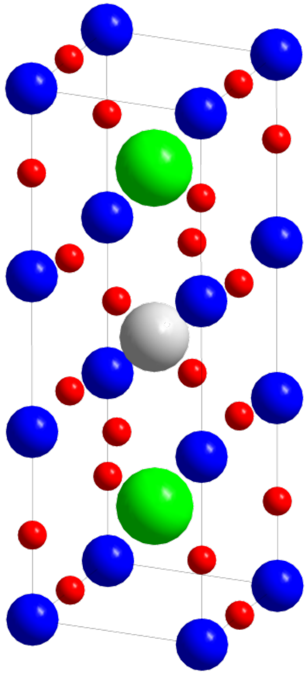




- Two domain directions explain the four main peaks.
- Smaller satellite peaks indicate additional domains.

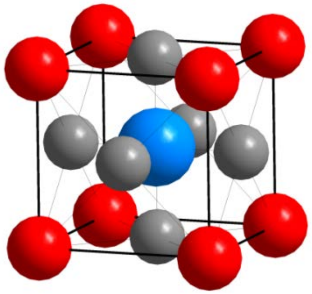
YBCO on STO
Determination of epitaxial relations

$YBa_2Cu_3O_7$

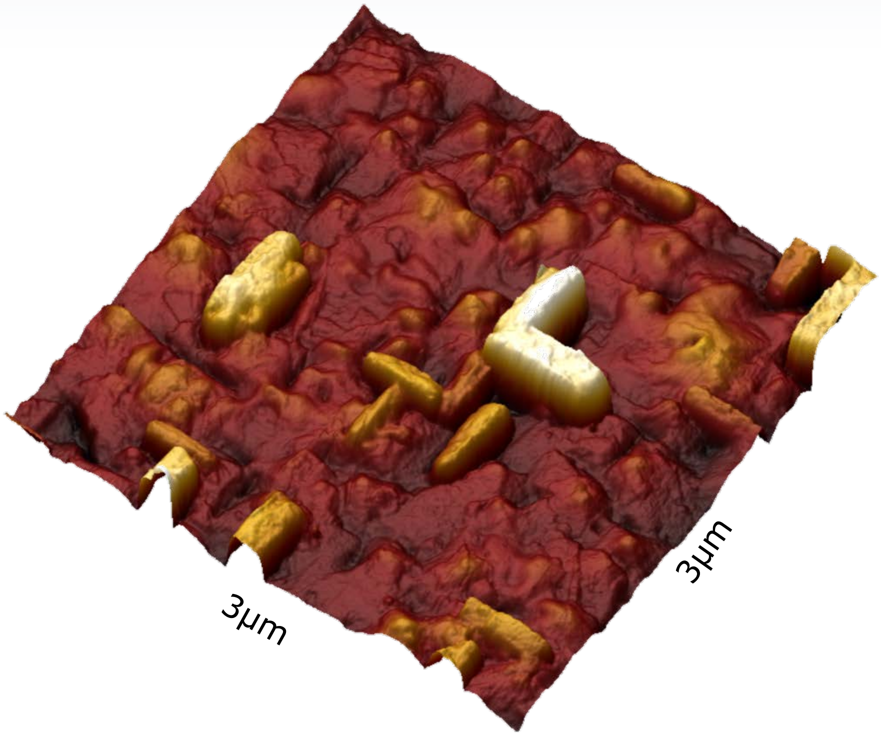


a = 3.8125(1) Å
b = 3.8750(2) Å
c = 11.6250(5) Å

$SrTiO_3$

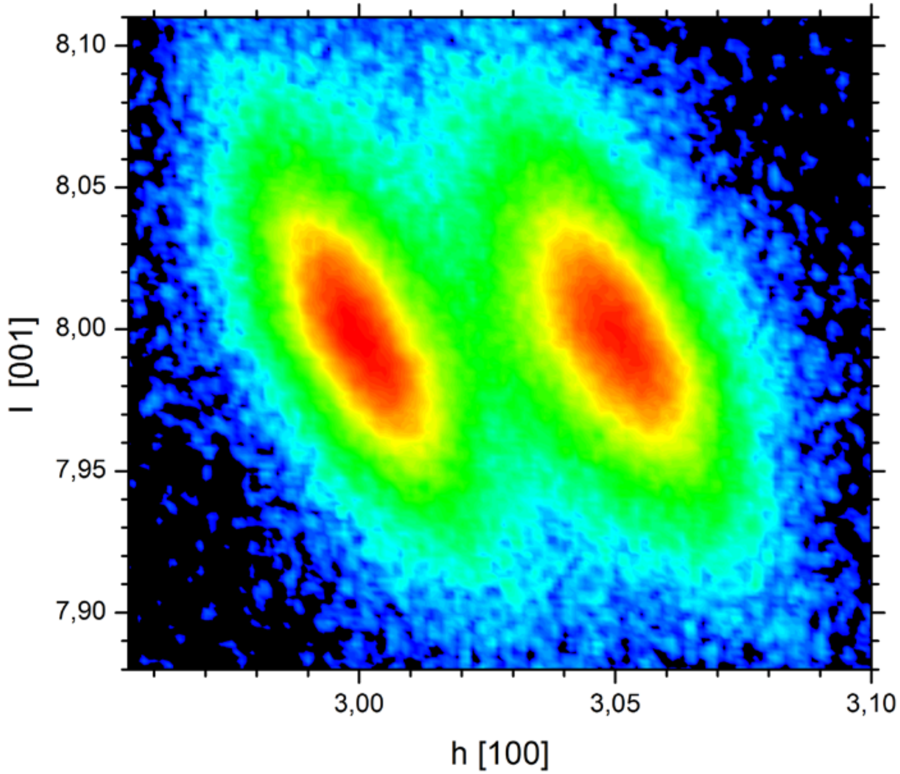


a = 3.91 Å



Co-planar reciprocal space map around YBCO(308)

YBCO(308+)



- RSM around a co-planar reflection YBCO(308+) shows 2 different in-plane lattice parameters.
- Relative lattice mismatch:

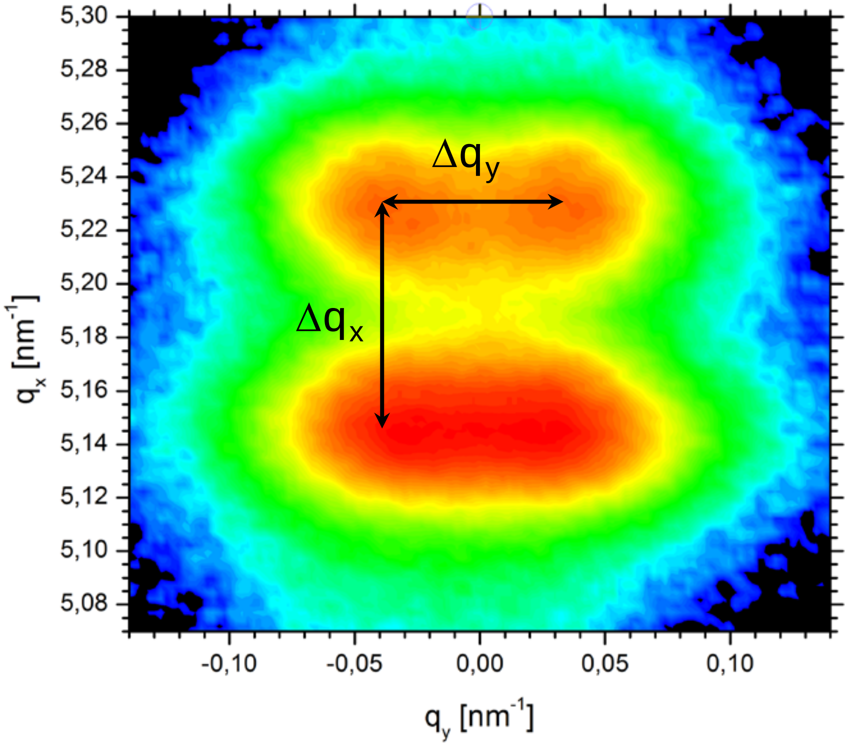
$$\frac{\Delta h}{h} = \frac{0.05}{3} \approx 1.7\%$$

- No information about domain twist.

YBCO on STO

Probing in-plane symmetry directly

In-plane RSM @ YBCO(200)



- RSM shows orthorhombic structure with 2 domain orientation.

  $\Delta q_x \approx 0.083\,\text{nm}^{-1}$

  $\Delta q_y \approx 0.072\,\text{nm}^{-1}$

- Relative lattice mismatch:

$$\frac{\Delta q_x}{q_x} = 1.63\%$$

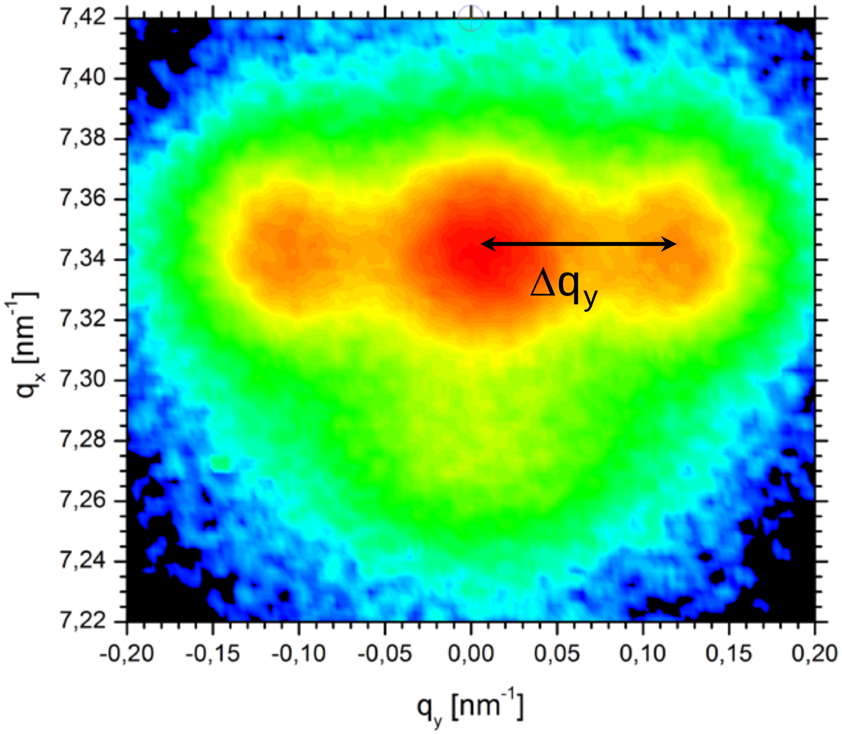
- Twist angle of domains:

$$\frac{\Delta q_y}{q_x} = 0.8°$$

YBCO on STO

Probing in-plane symmetry directly

In-plane RSM @ YBCO(220)

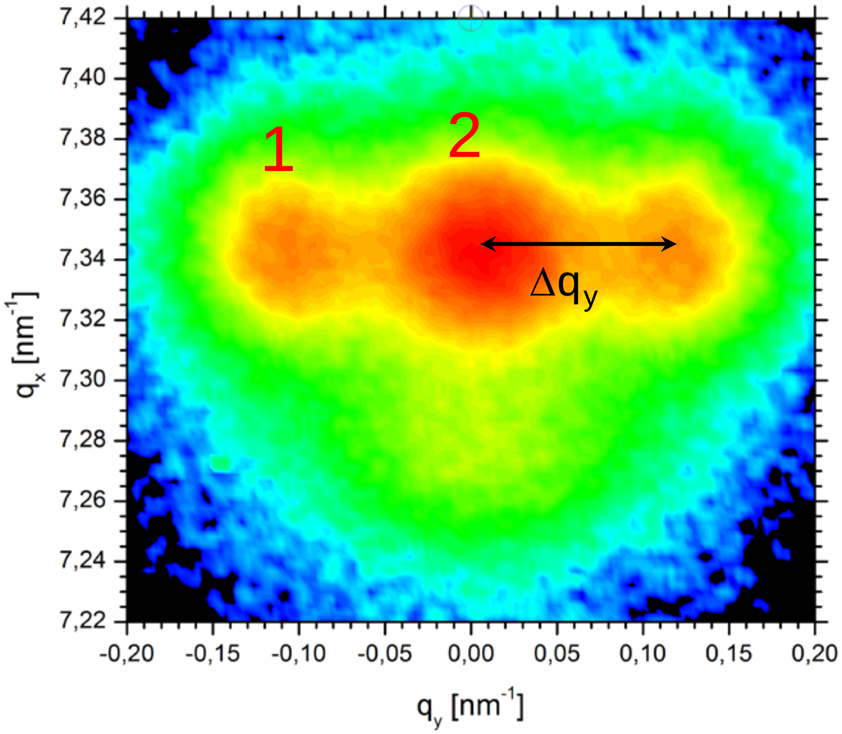


- Twist angle of domains: $\frac{\Delta q_y}{q_x} = 0.85°$

YBCO on STO

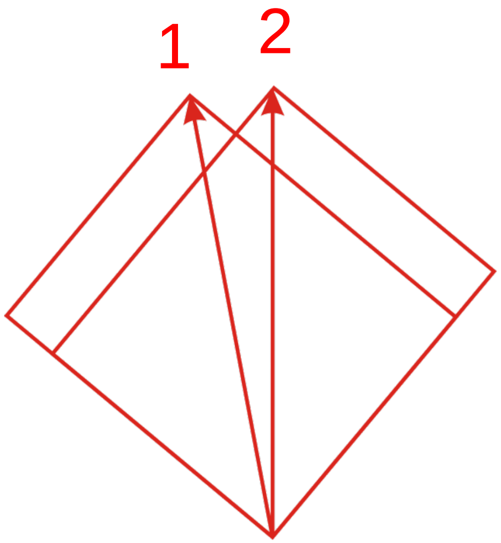
Probing in-plane symmetry directly

In-plane RSM @ YBCO(220)



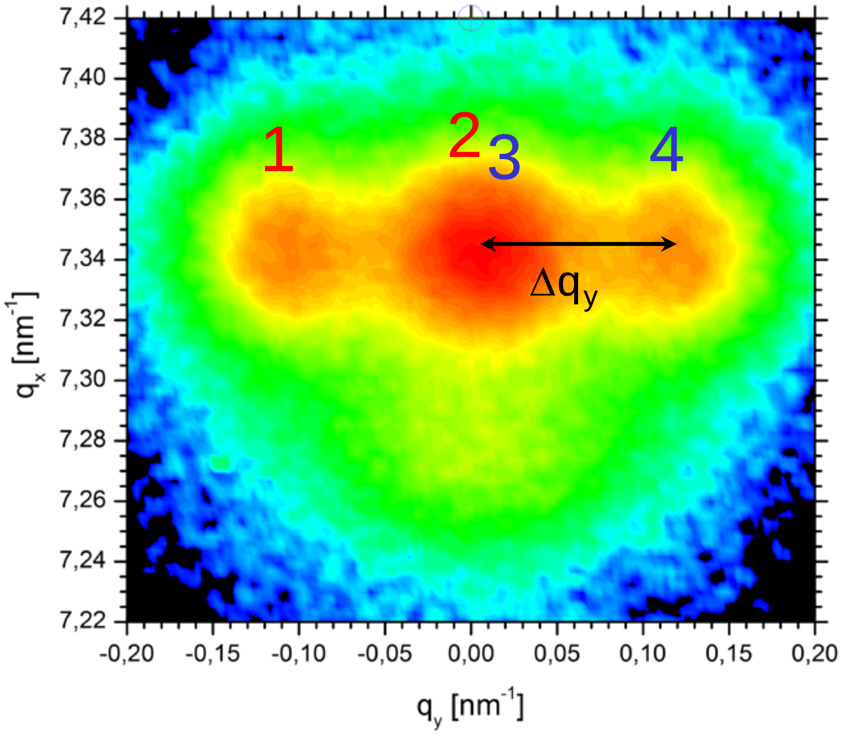
- Twist angle of domains: $\frac{\Delta q_y}{q_x} = 0.85°$

- Explanation of the RSM

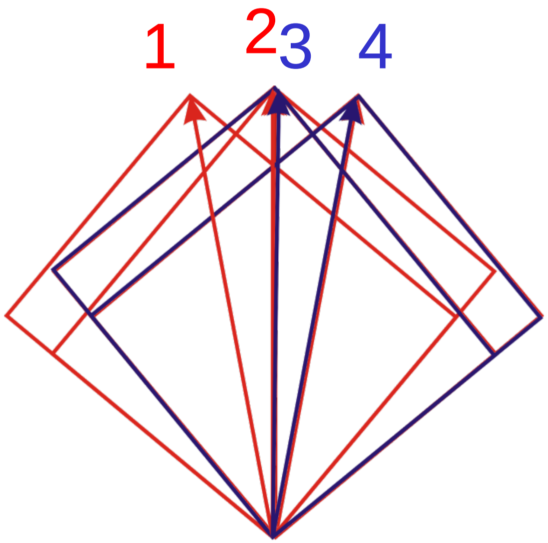


YBCO on STO
Probing in-plane symmetry directly

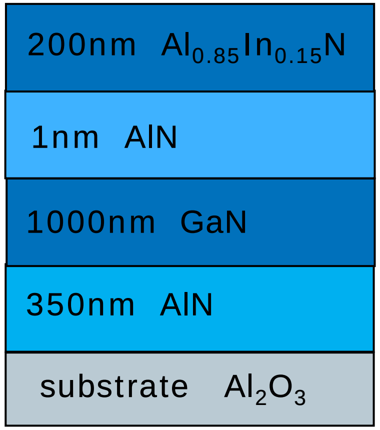
In-plane RSM @ YBCO(220)



- Twist angle of domains: $\frac{\Delta q_y}{q_x} = 0.85°$
- Explanation of the RSM



Example: GaN-based HEMT structure



200nm $Al_{0.85}In_{0.15}N$

1nm AlN

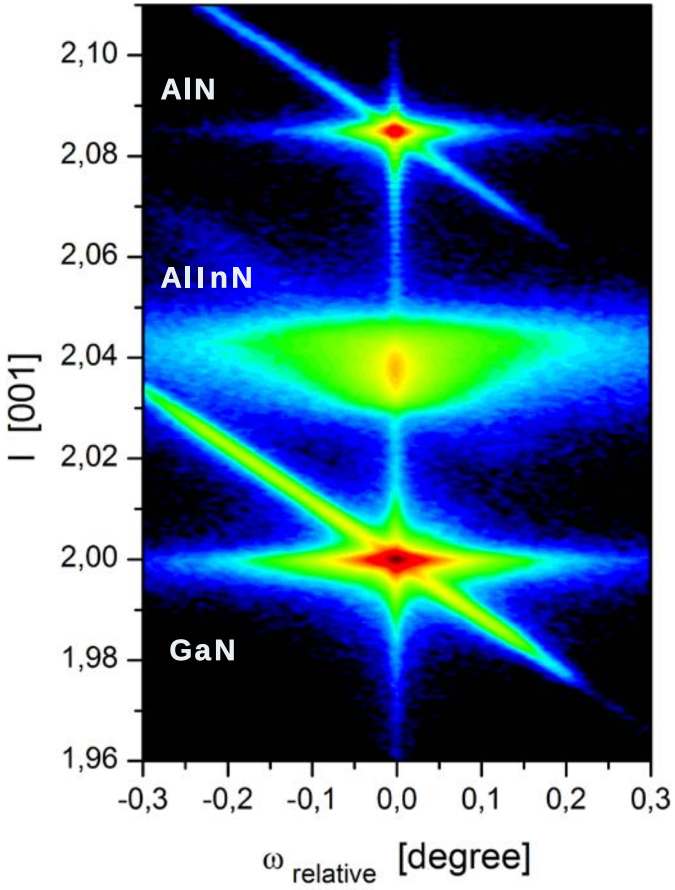
1000nm GaN

350nm AlN

substrate Al_2O_3

Sample courtesy of L. R. Khoshroo (RWTH Aaachen)

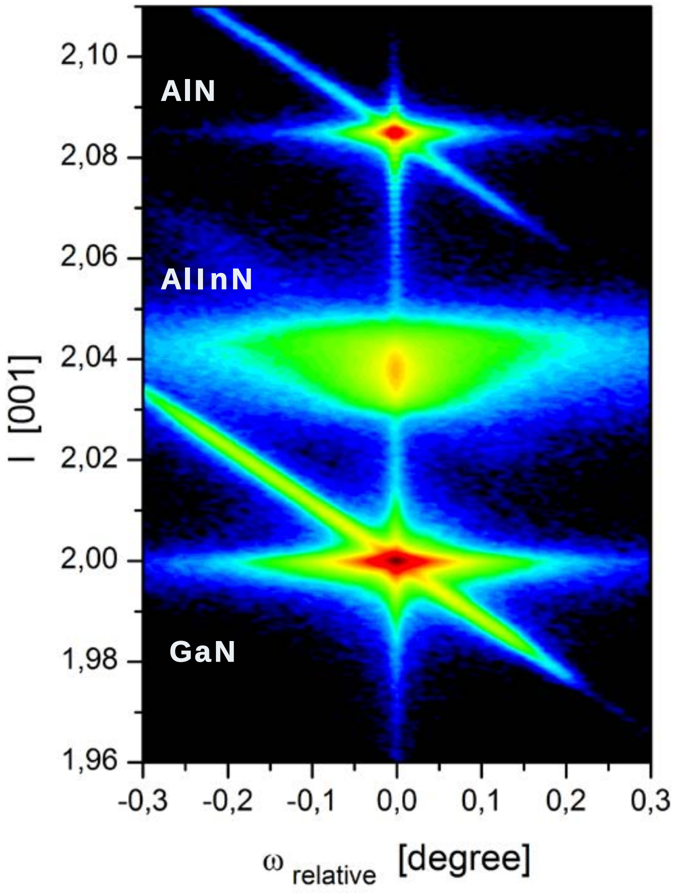
GaN(002)

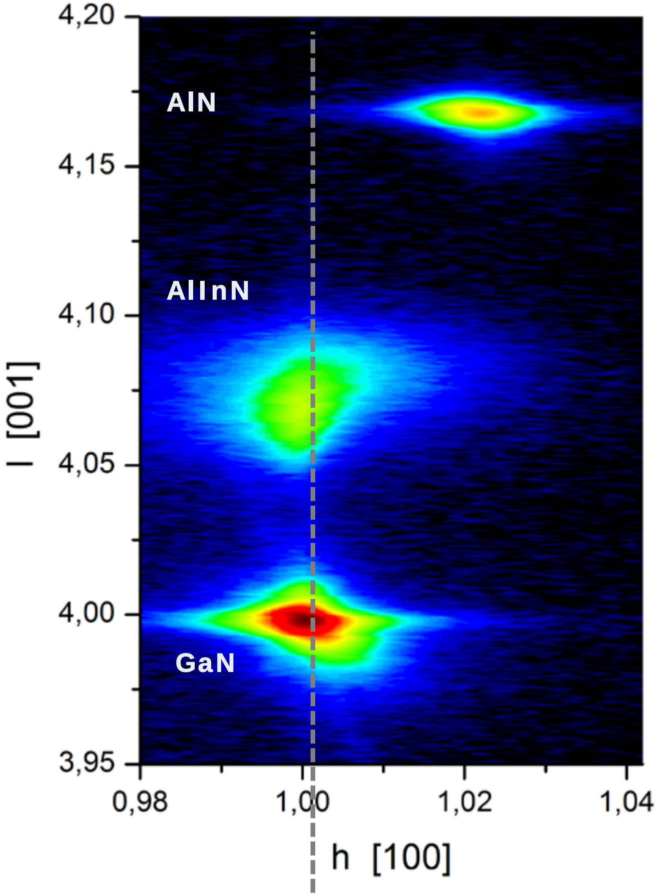


Example: GaN-based HEMT structure

200nm $Al_{0.85}In_{0.15}N$
1nm AlN
1000nm GaN
350nm AlN
substrate Al_2O_3

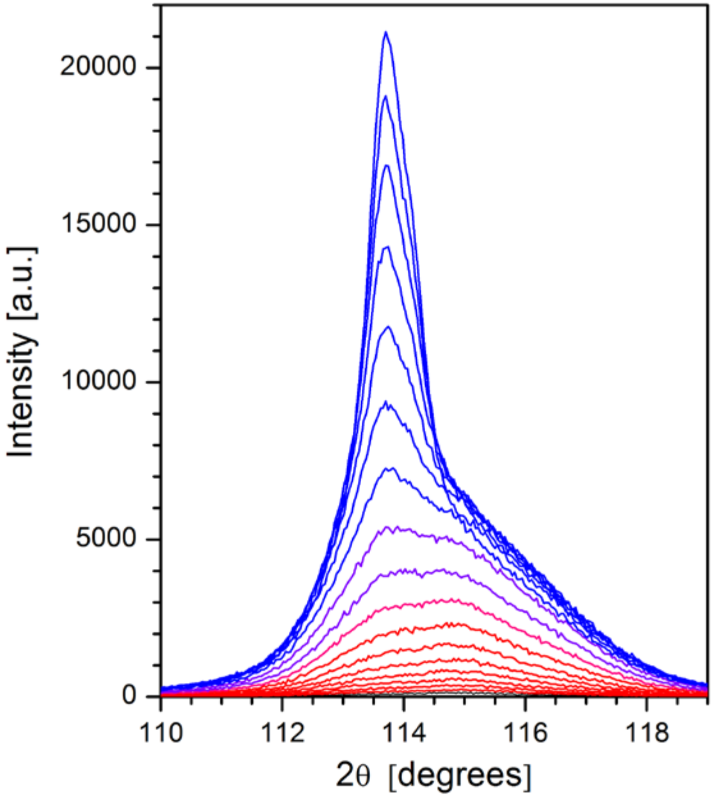
Sample courtesy of L. R. Khoshroo (RWTH Aaachen)


GaN(002)


GaN(104+)

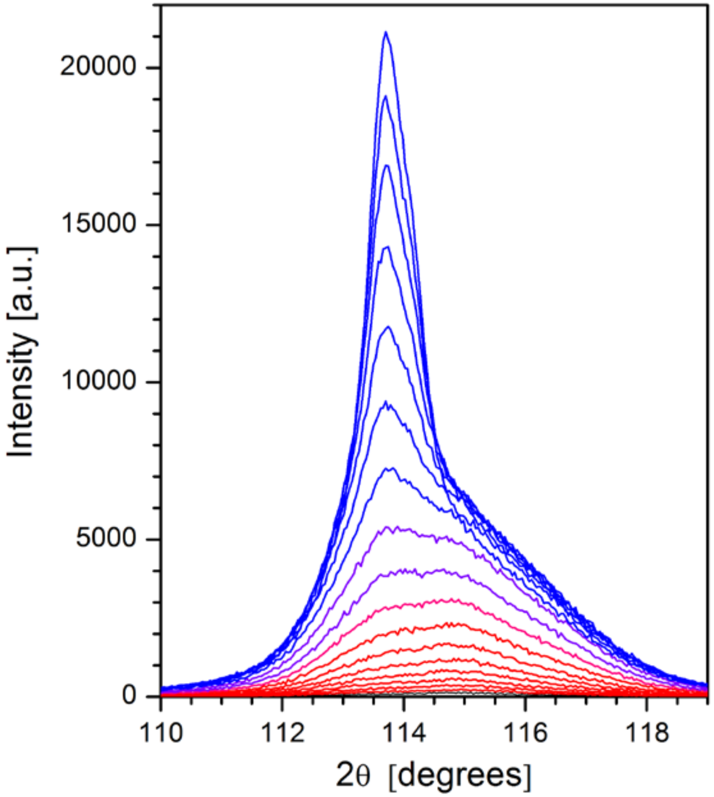
Depth-dependent in-plane GID

- $2\theta/\omega$ scans at different α_i around $Al_xIn_{1-x}N(300)$ reflection



Depth-dependent in-plane GID

- 2θ/ω scans at different α_i around $Al_xIn_{1-x}N(300)$ reflection

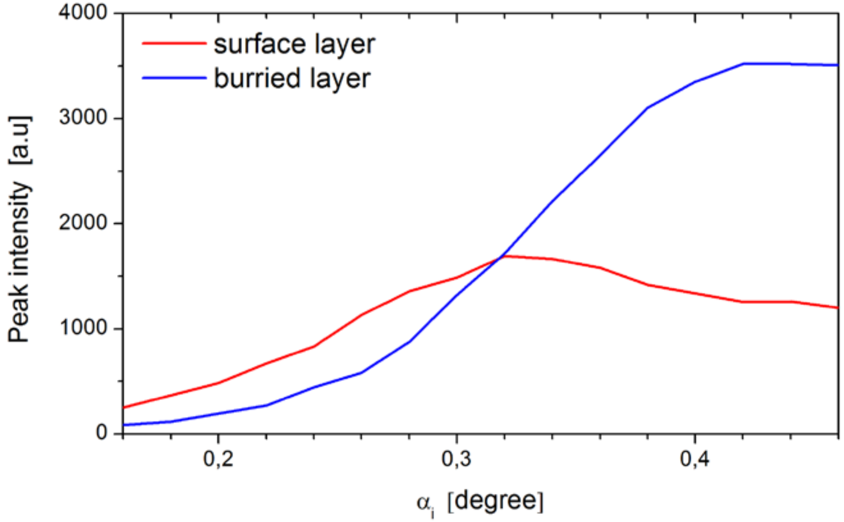


- Peak position obtained with

115.04° ± 0.2° a = 3,162 Å

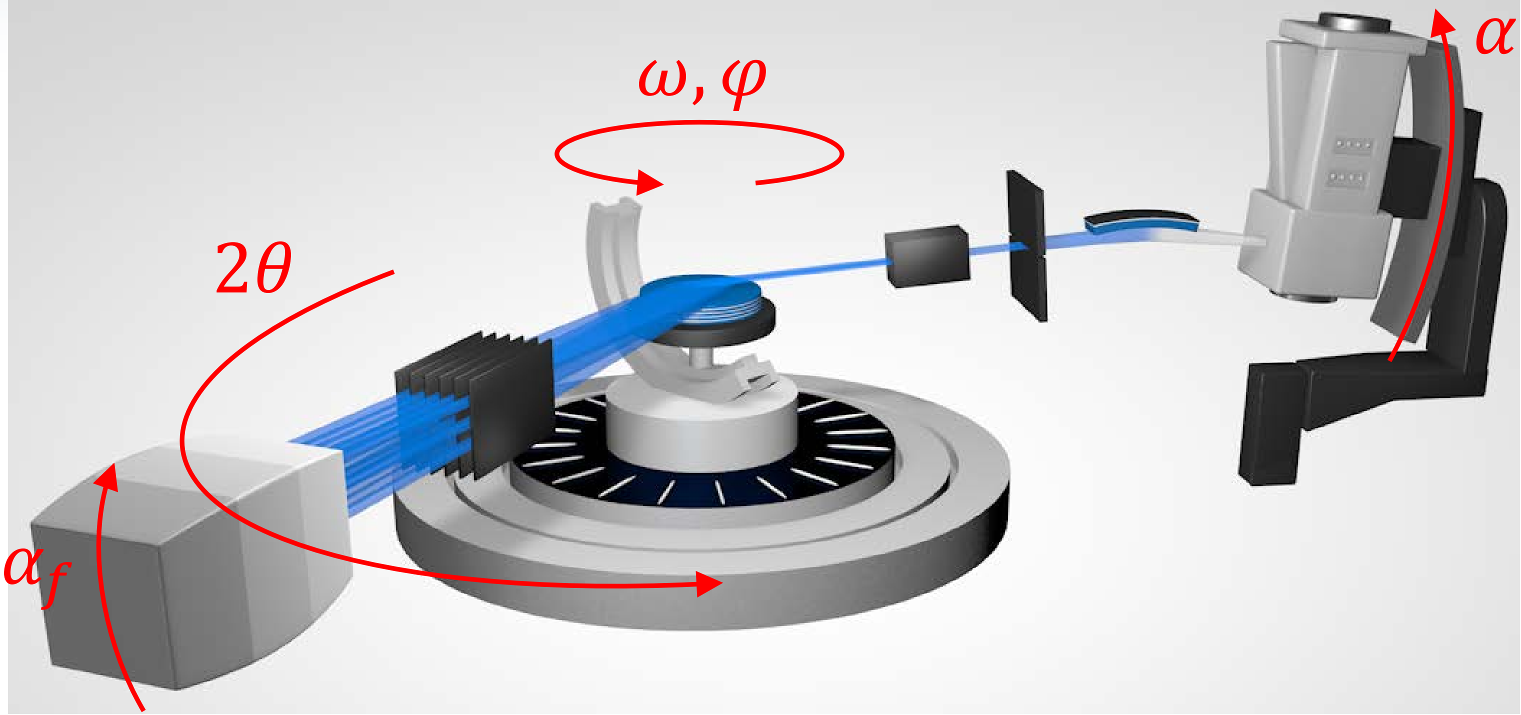
113.65° ± 0.1° a = 3,188 Å

- Integrated peak intensities



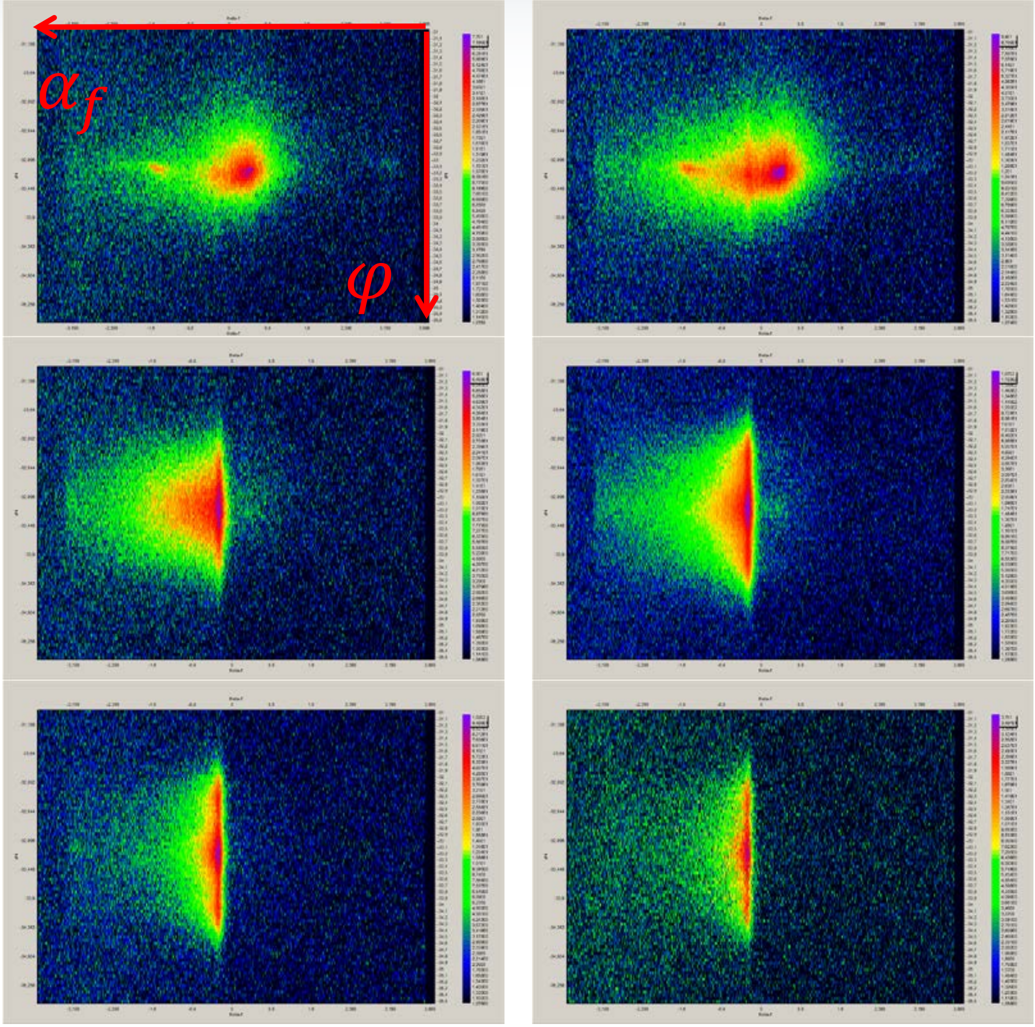
- Introduction
- Experimental configurations
- Performing an experiment
- Examples
  - Polycrystalline samples
  - Epitaxial grown samples
  - In-plane diffraction with 1D detector

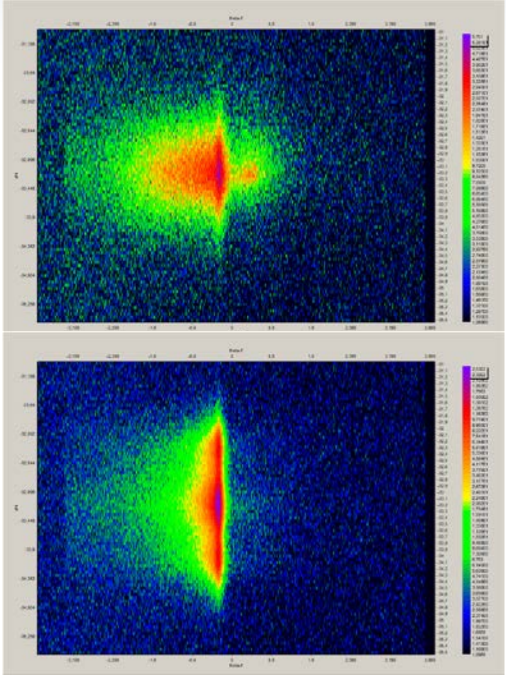
Ultra-GID configuration



- Use of a 1D-detector rotated by 90° provides resolution perpendicular to the sample surface.

3D reciprocal space mapping in IPGID geometry : YBCO(220) on STO





- RSM looped over 2θ/ω.

IPGID provides information about

- Crystallite size
- In-plane texture
- In-plane lattice parameter
- Epitaxial relation
- Domain formation and twist
- Depth-dependent information
- Micro strain

Thank you for your attention…

Any Questions?

Please type any questions you may have in the Q&A panel and then click Send.

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