Good Diffraction Practice Webinar Series

High Resolution X-ray Diffractometry 2 – Reciprocal Space Mapping

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Welcome

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Outline

- What is Reciprocal Space
- What can be measured by Reciprocal Space maps (RSMs)
- How to measure RSMs
- RSMs with 1D-detectors
- RSMs with 2D-detectors

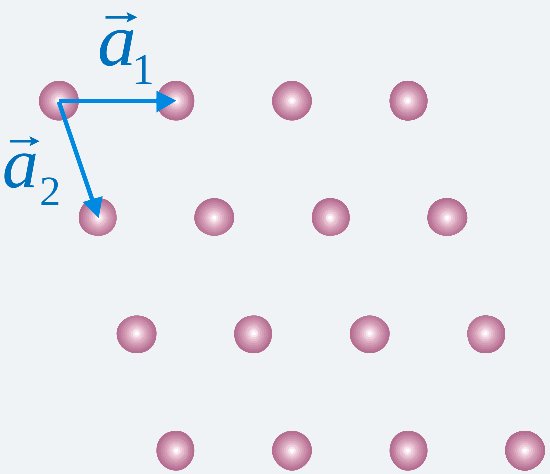
- **What is Reciprocal Space**
- What can be measured by Reciprocal Space maps (RSMs)
- How to measure RSMs
- RSMs with 1D-detectors
- RSMs with 2D-detectors

Scattering from a crystal:
The concept of reciprocal space

Real space

q-space

Crystal lattice



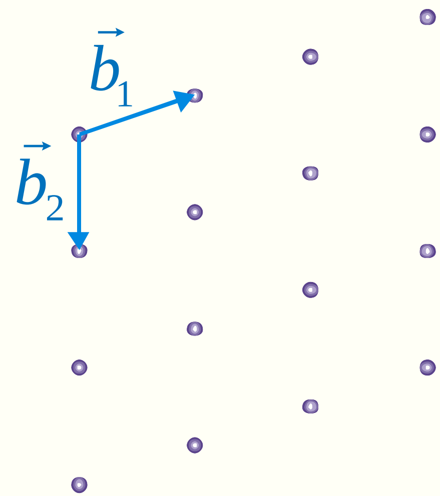
$$\vec{R} = n_1 \vec{a}_1 + n_2 \vec{a}_2 + n_3 \vec{a}_3$$

Fourier transform

$$\vec{a}_i \cdot \vec{b}_k = 2\pi\delta_{ik}$$

$$\exp\left(i \vec{G} \vec{R}\right) = 1$$

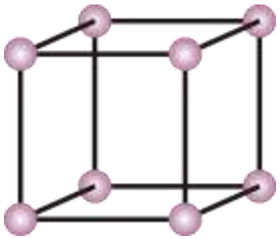
Reciprocal lattice

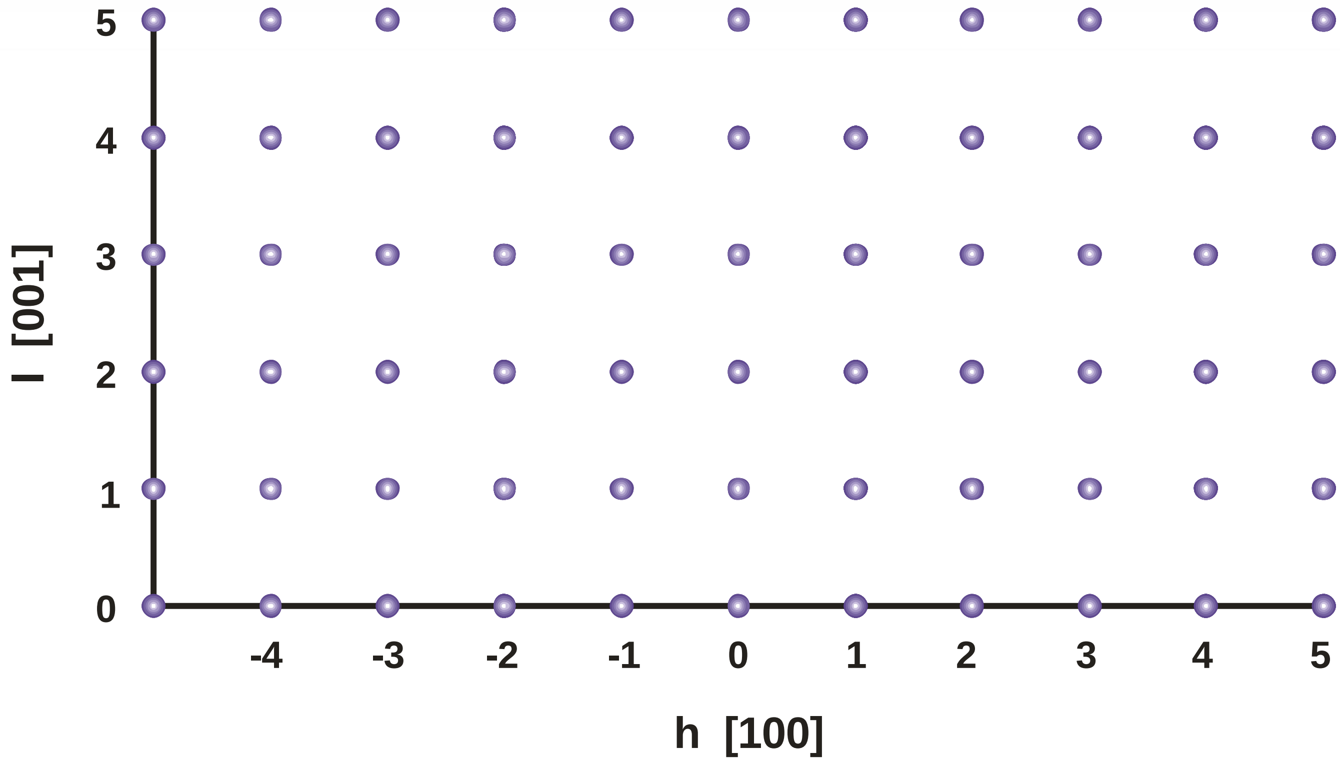


$$\vec{G} = h \vec{b}_1 + k \vec{b}_2 + l \vec{b}_3$$

Accessible region in reciprocal space – Experimental constraints

of a single atom
cubic crystal





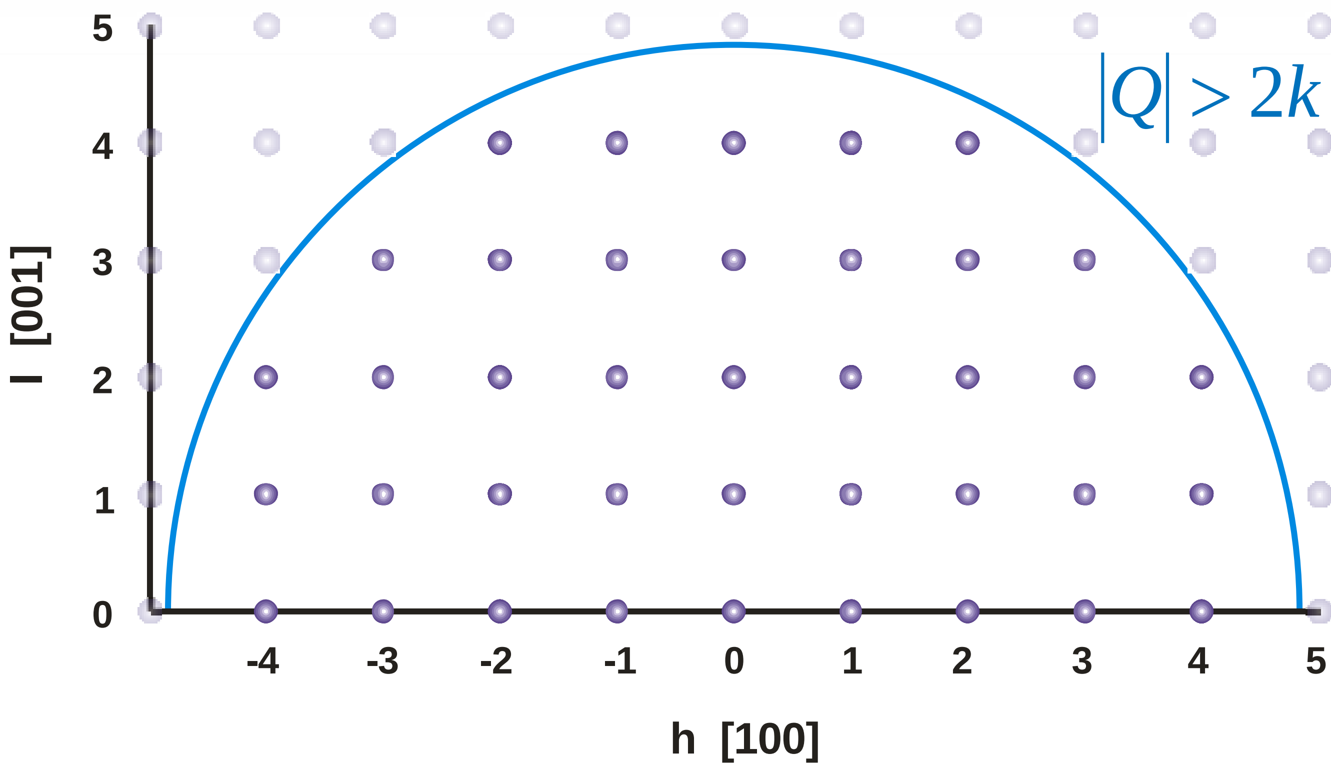
Accessible region in reciprocal space - Wavelength

Wavevector

$$k = 2\pi / \lambda$$

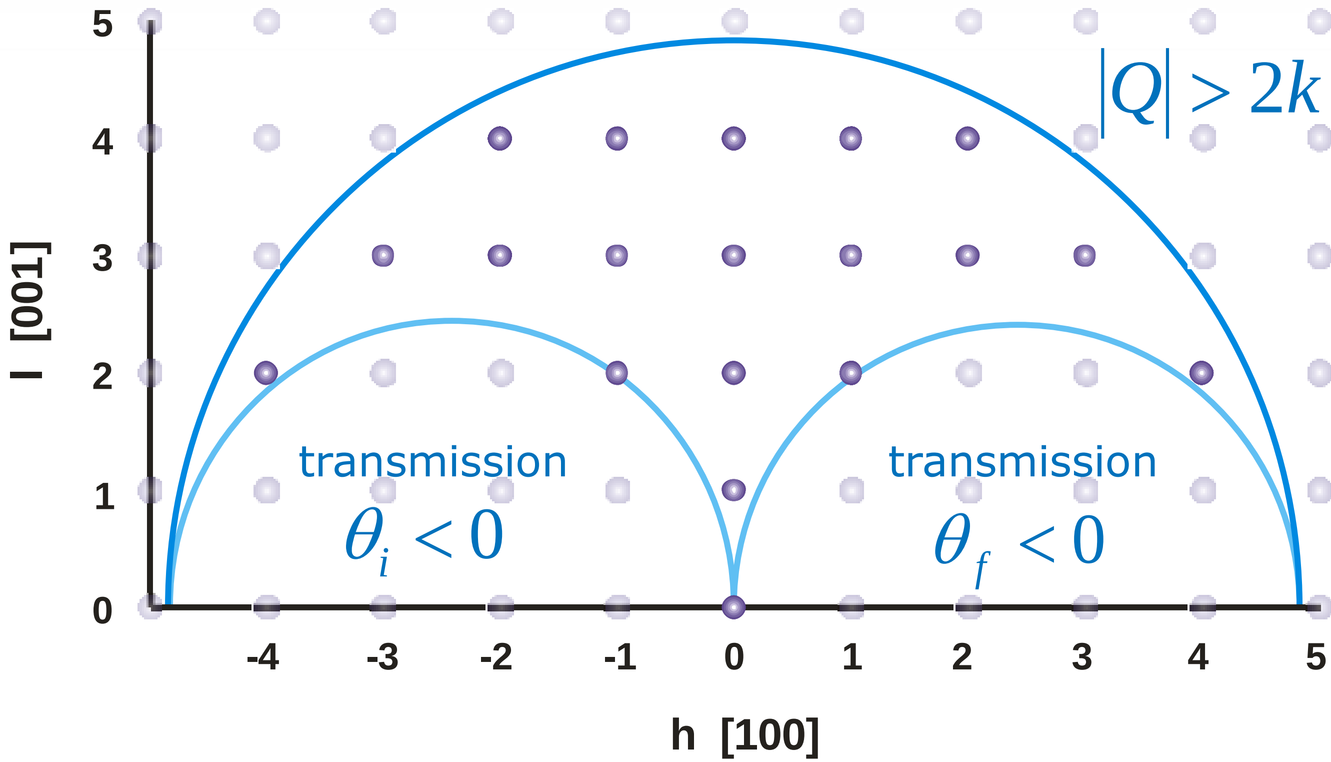
Wavelength

$$\lambda\,[nm] = \frac{1.24}{E\ [keV]}$$



The range of accessible reflections can be increased by using X-rays of a higher energy.

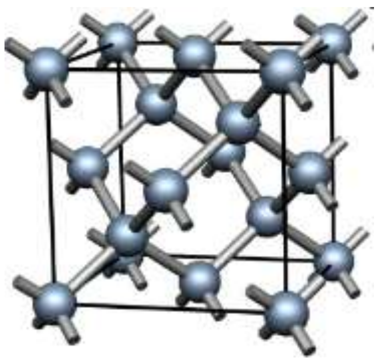
Accessible region in reciprocal space - Geometry

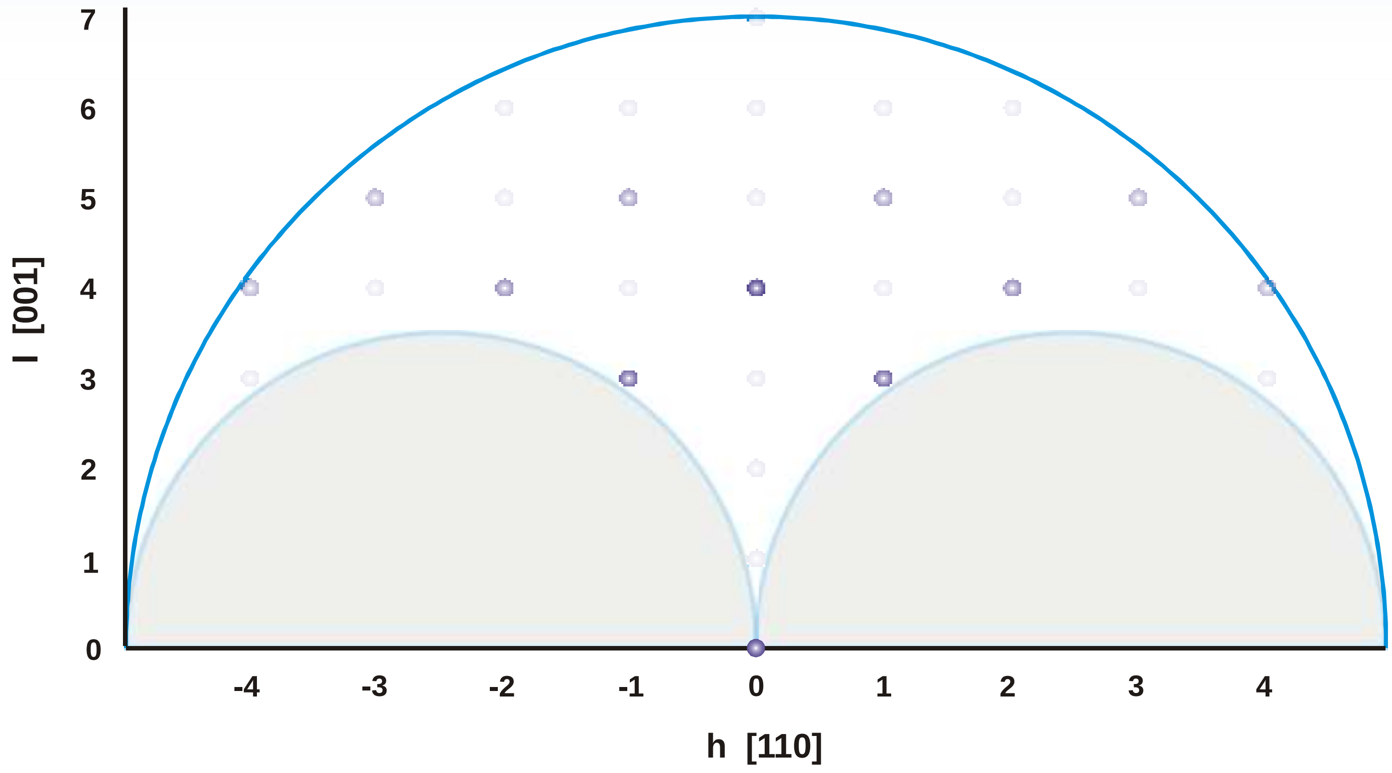


Reflections very close to the half-spheres have grazing incidence or grazing exit geometry → surface sensitivity

A silicon crystal in reciprocal space – Structure Factor

FCC-lattice with Si atoms at (0, 0, 0) and (¼, ¼, ¼)

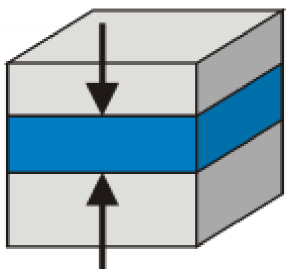




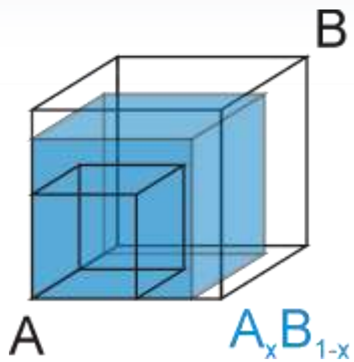
The structure factor of the crystal determines the scattered intensity.

- What is Reciprocal Space
- **What can be measured by Reciprocal Space maps (RSMs)**
- How to measure RSMs
- RSMs with 1D-detectors
- RSMs with 2D-detectors

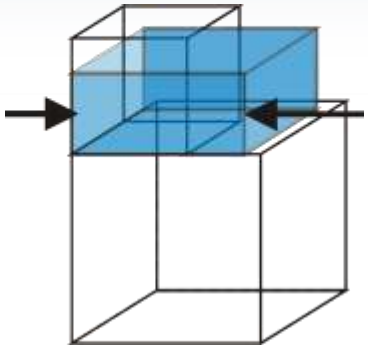
Analytical tasks



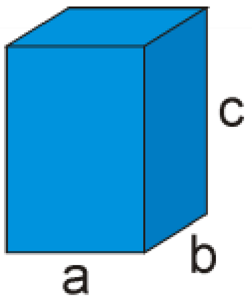
Layer thickness



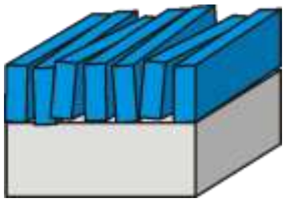
Chemical composition



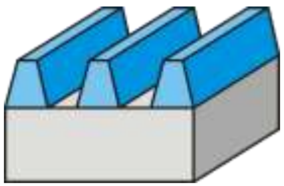
Mismatch & relaxation



Lattice parameters



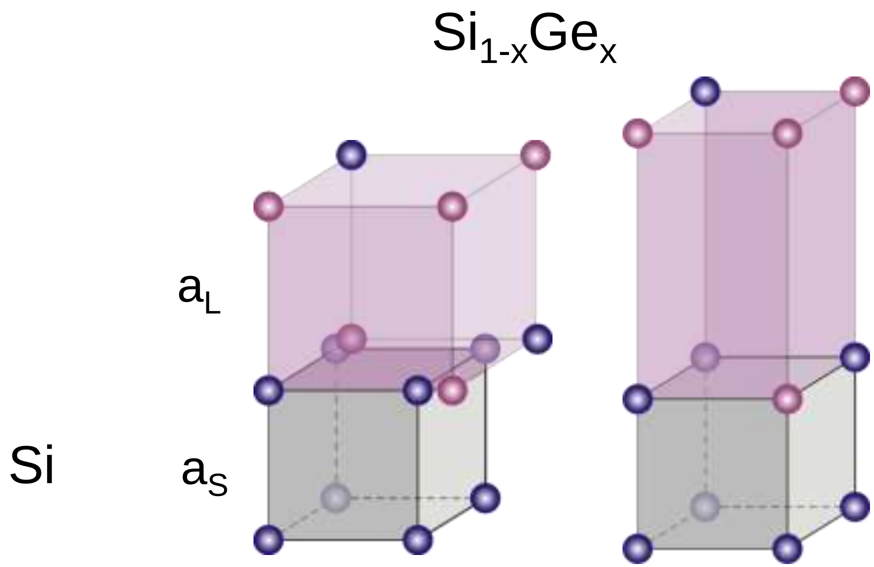
Defects & Crystal size



Lateral structure

Pseudomorphic and relaxed strain state

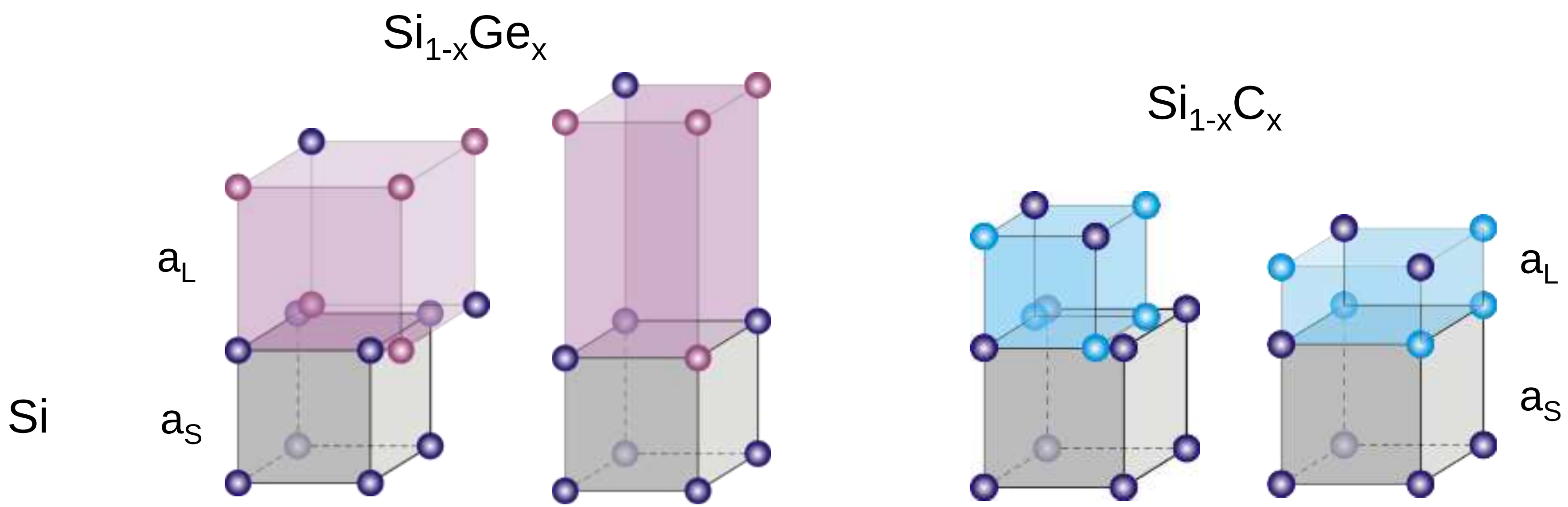
- Relaxed layer lattice mismatch : $\frac{\Delta a}{a}_{rel} = \frac{a_L - a_S}{a_S}$



- Compressive strain : $\frac{\Delta a}{a}_{rel} > 0$

Pseudomorphic and relaxed strain state

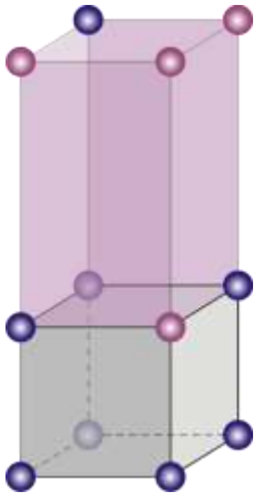
- Relaxed layer lattice mismatch : $\frac{\Delta a}{a}_{rel} = \frac{a_L - a_S}{a_S}$

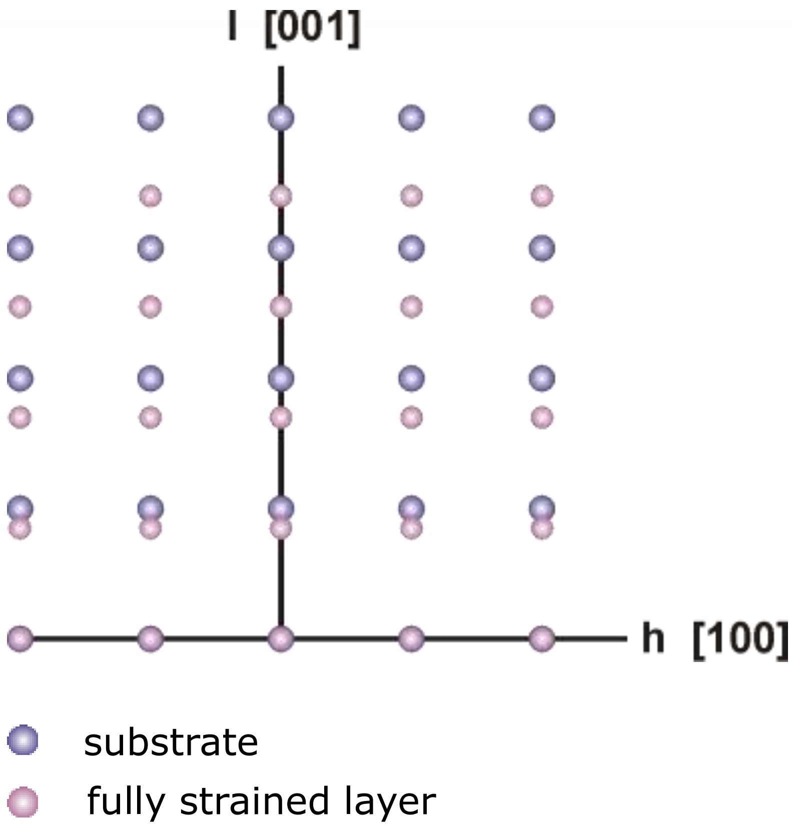


- Compressive strain : $\frac{\Delta a}{a}_{rel} > 0$
- Tensile strain : $\frac{\Delta a}{a}_{rel} < 0$

Epitaxial Layers in Reciprocal Space

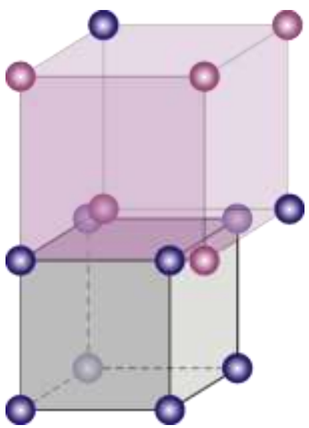
Pseudomorphic Layer

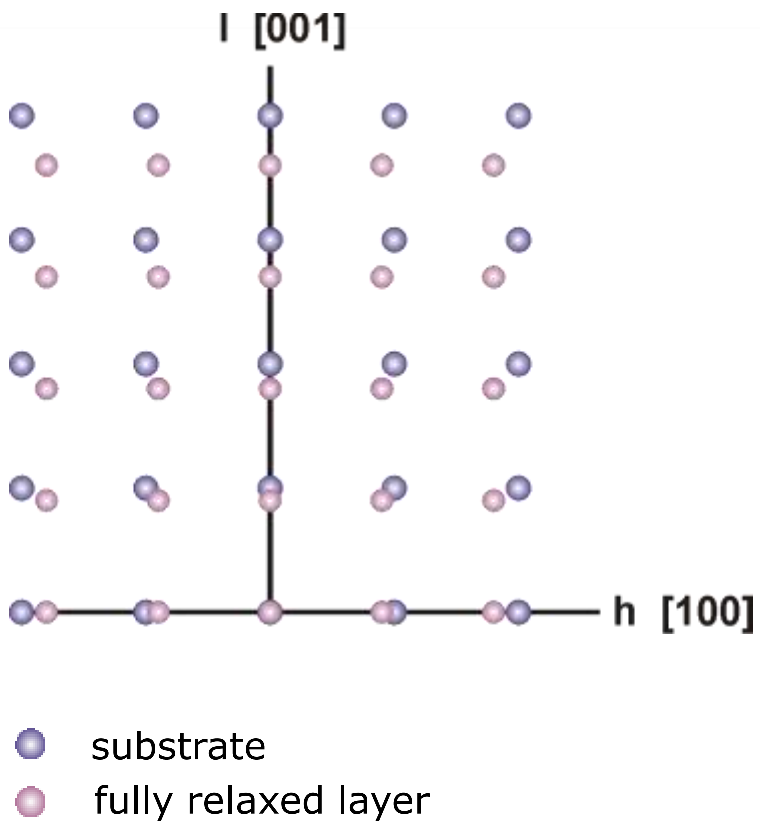




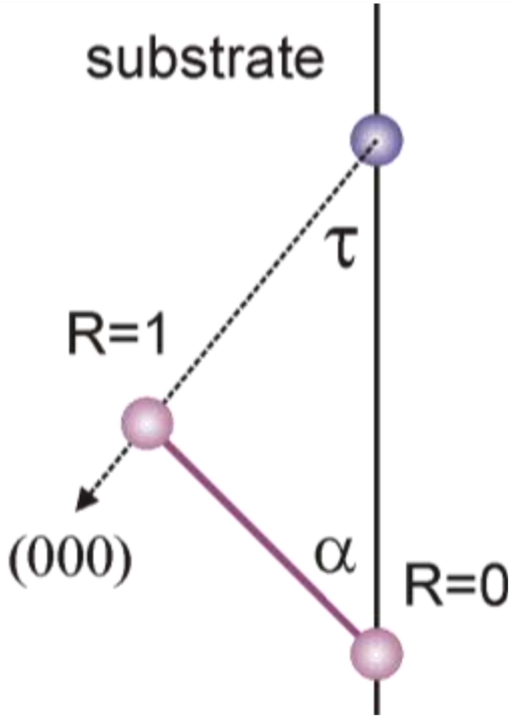
Epitaxial Layers in Reciprocal Space

Completely relaxed layer





The relaxation line



- The reflection of a fully strained layer is located on a perpendicular line.
- A reflection of a fully relaxed layer is on a line through the substrate reflection and (000).
- Reflections of partly relaxed layers are on the relaxation line.

$$\tan\alpha = -\tan\tau / D$$

Theory of elasticity: $D \approx 0.5, \ldots, 1$

- What is Reciprocal Space
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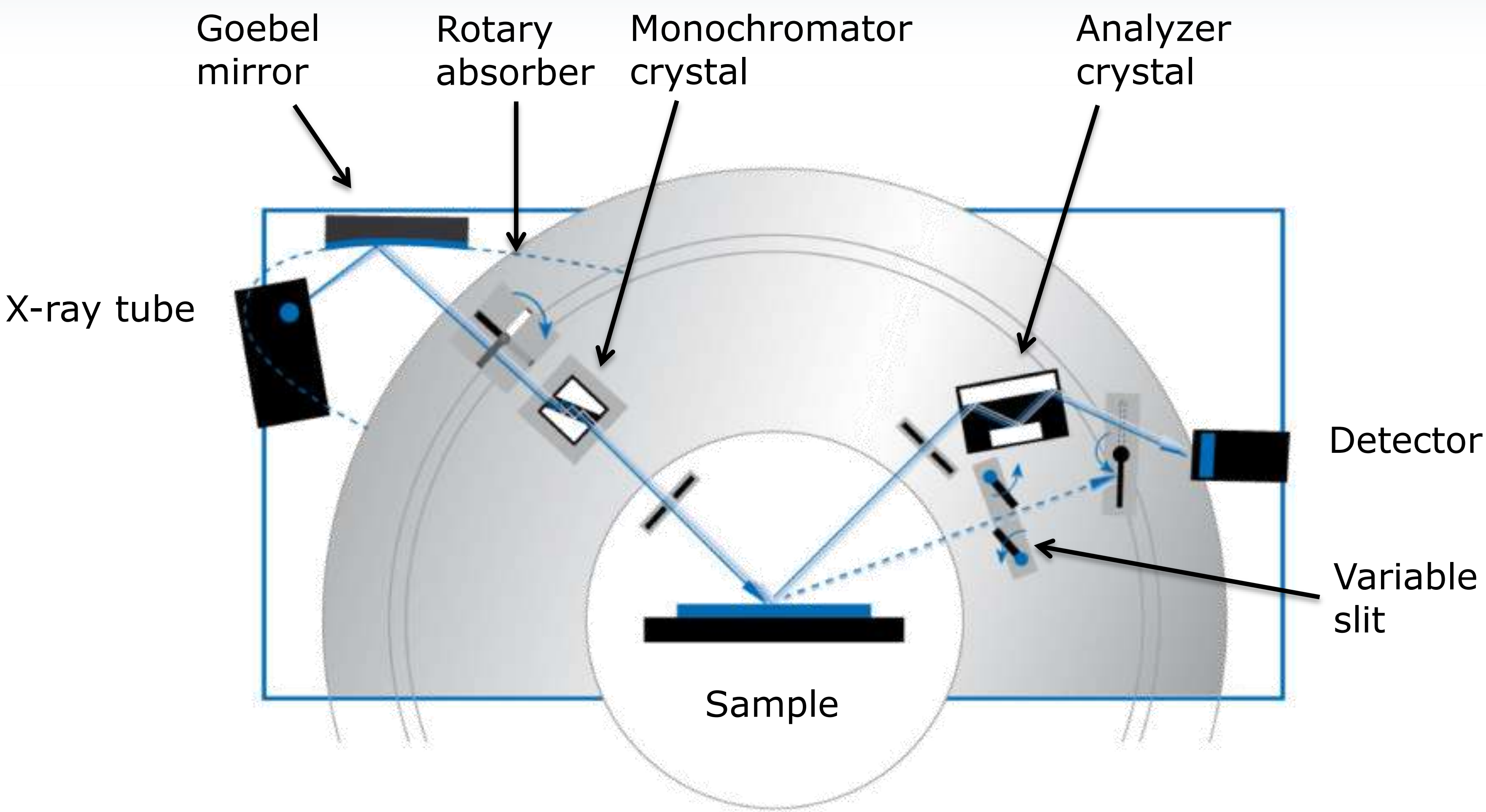
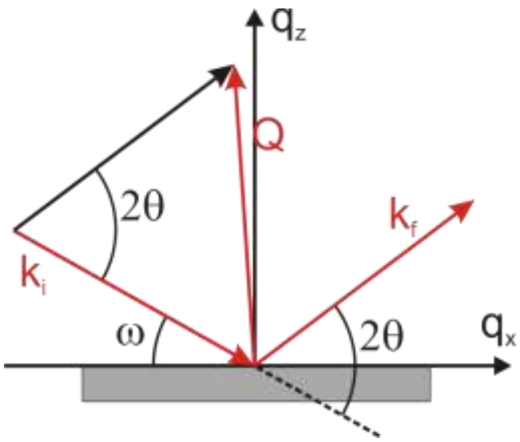
- **How to measure RSMs**
- RSMs with 1D-detectors
- RSMs with 2D-detectors

Diffractometer configuration for measuring RSMs

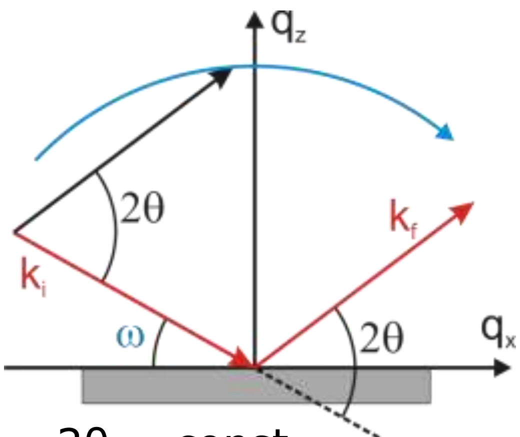


How to measure RSMs?
Scan Types



How to measure RSMs?
Scan Types

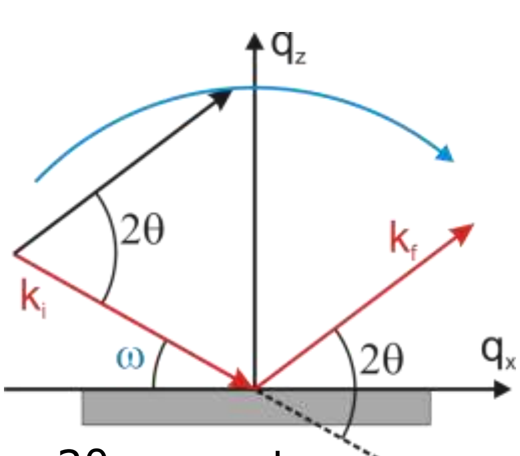
Rocking curve



2θ = const

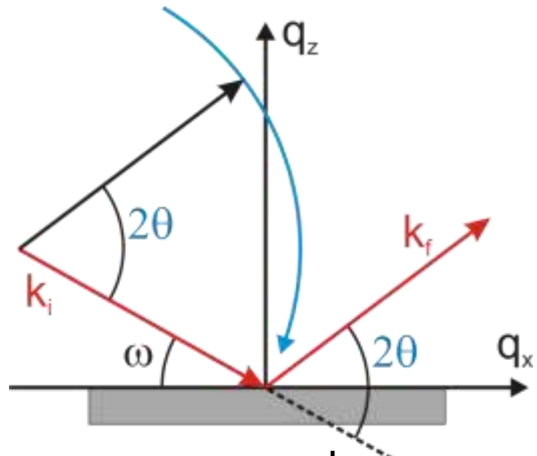
How to measure RSMs?
Scan Types

Rocking curve



2θ = const

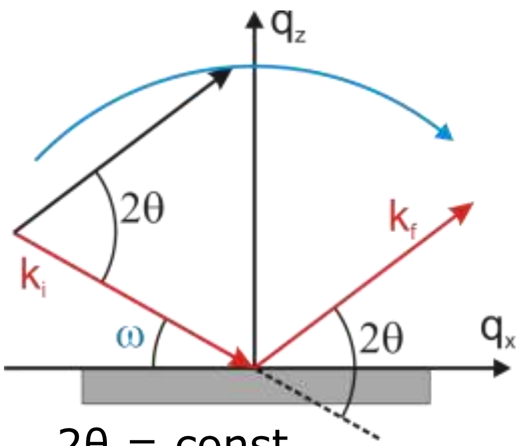
Detector scan



ω = const

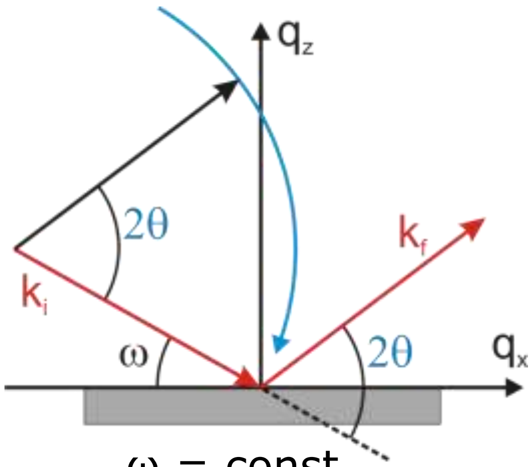
How to measure RSMs?
Scan Types

Rocking curve



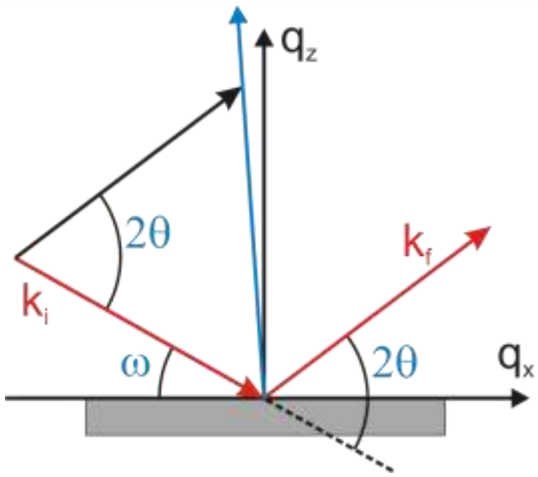
2θ = const

Detector scan



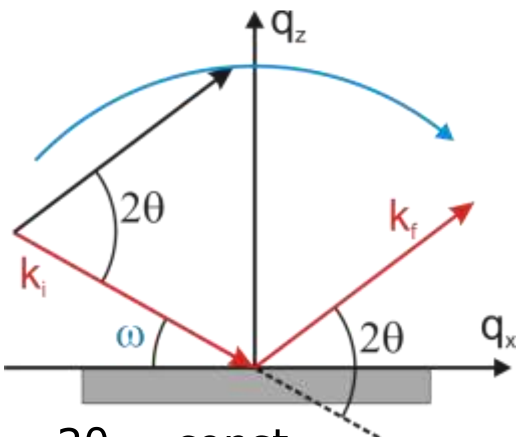
ω = const

2θ/ω , ω/2θ scan



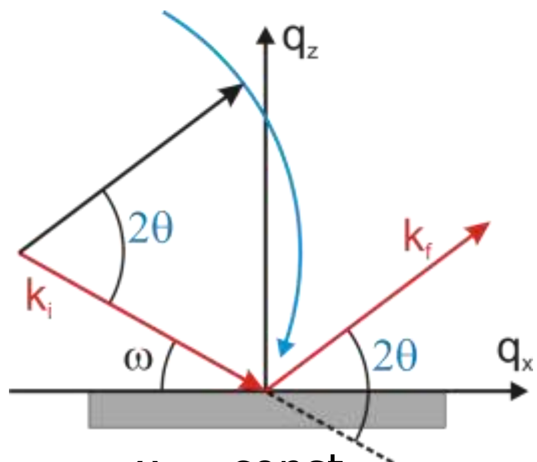
How to measure RSMs?
Scan Types

Rocking curve



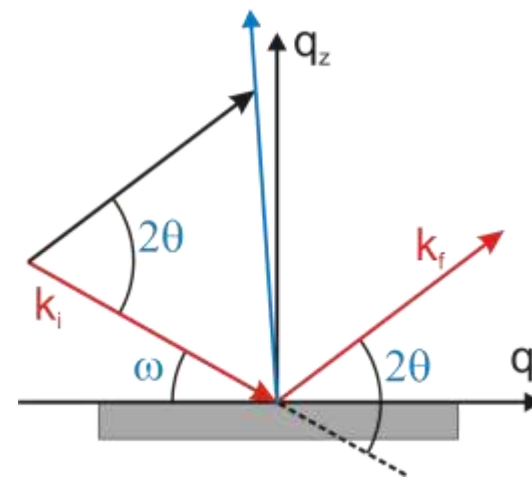
2θ = const

Detector scan



ω = const

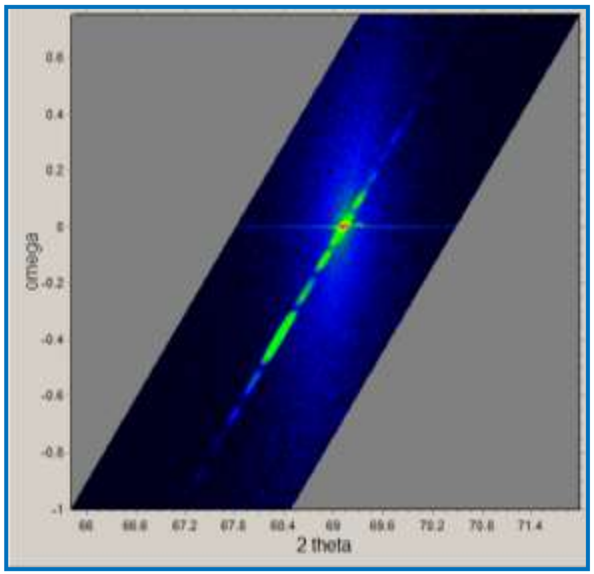
2θ/ω , ω/2θ scan



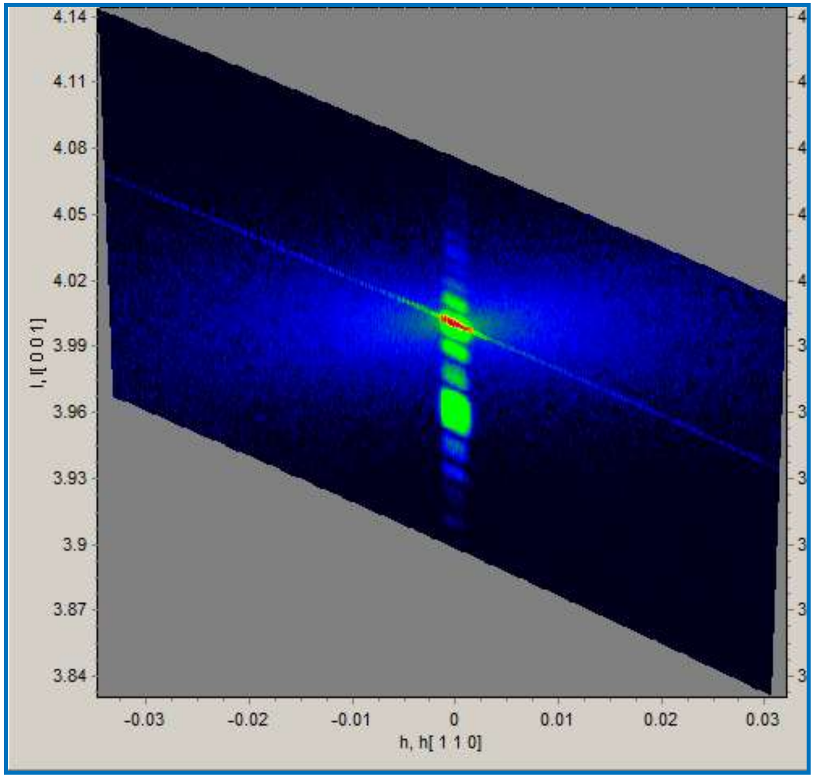
- reciprocal space scans
  - q_x scan, q_z = const
  - q_z scan, q_x = const….

Conversion from angular to reciprocal lattice units

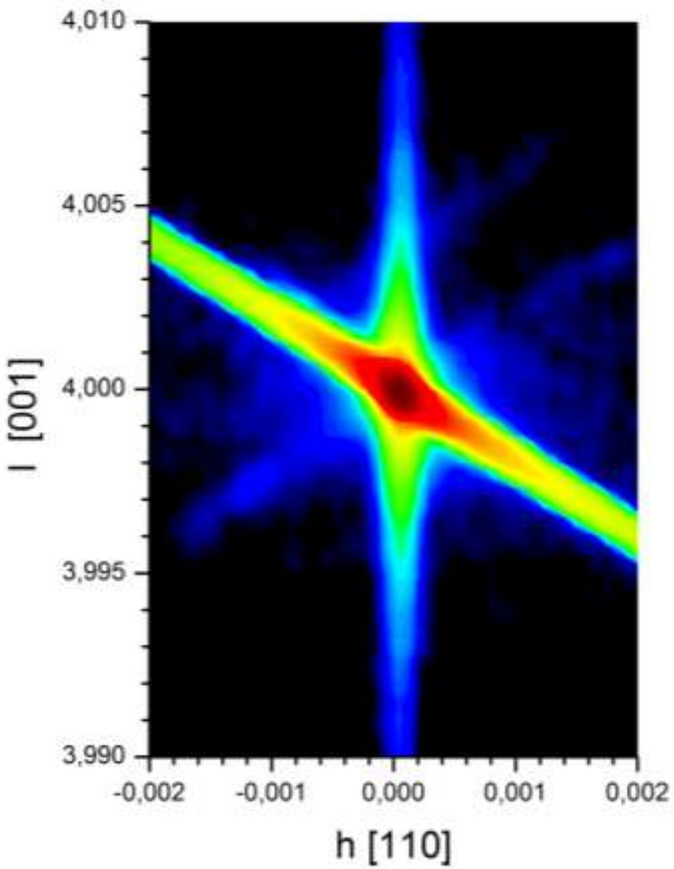
Measurement is performed
in angular space



Analyses are done
in reciprocal space



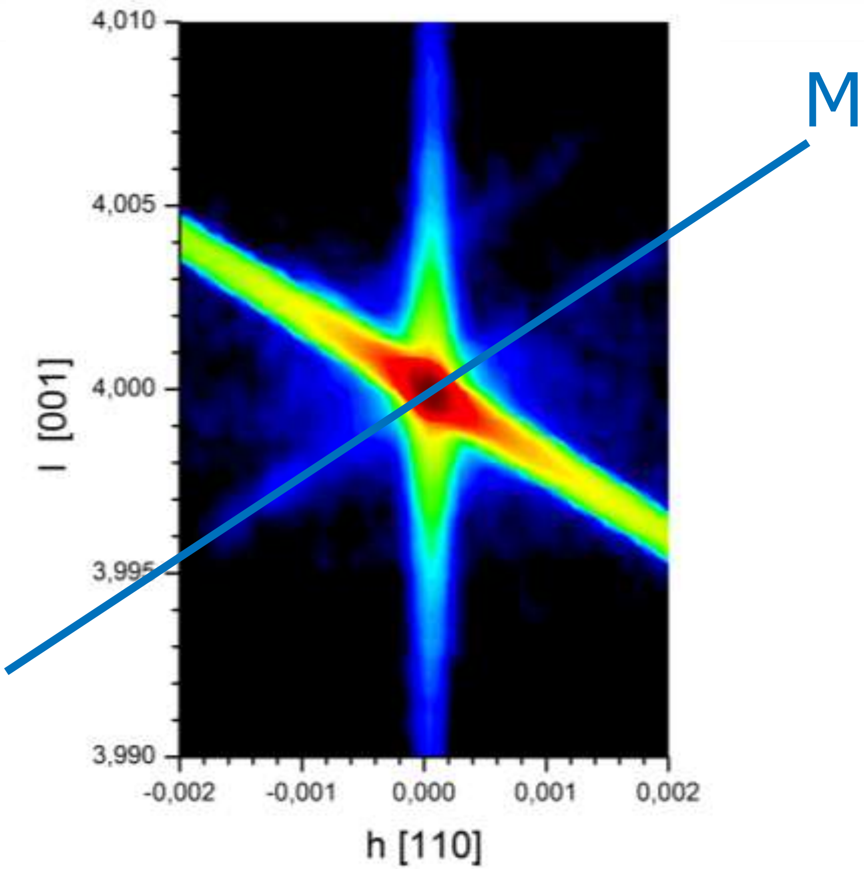
The instrumental resolution function in RSM

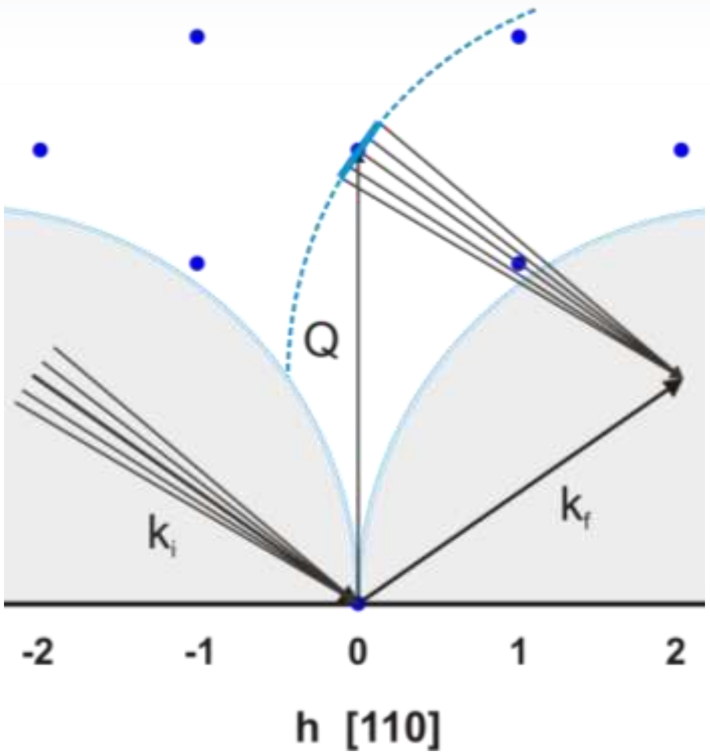


- The recorded intensity at any point in reciprocal space is an average over the respective resolution element.

- Example : Si(004) reflection measured with
  - 2xGe(004a) monochromator
  - 1xGe(002) analyzer

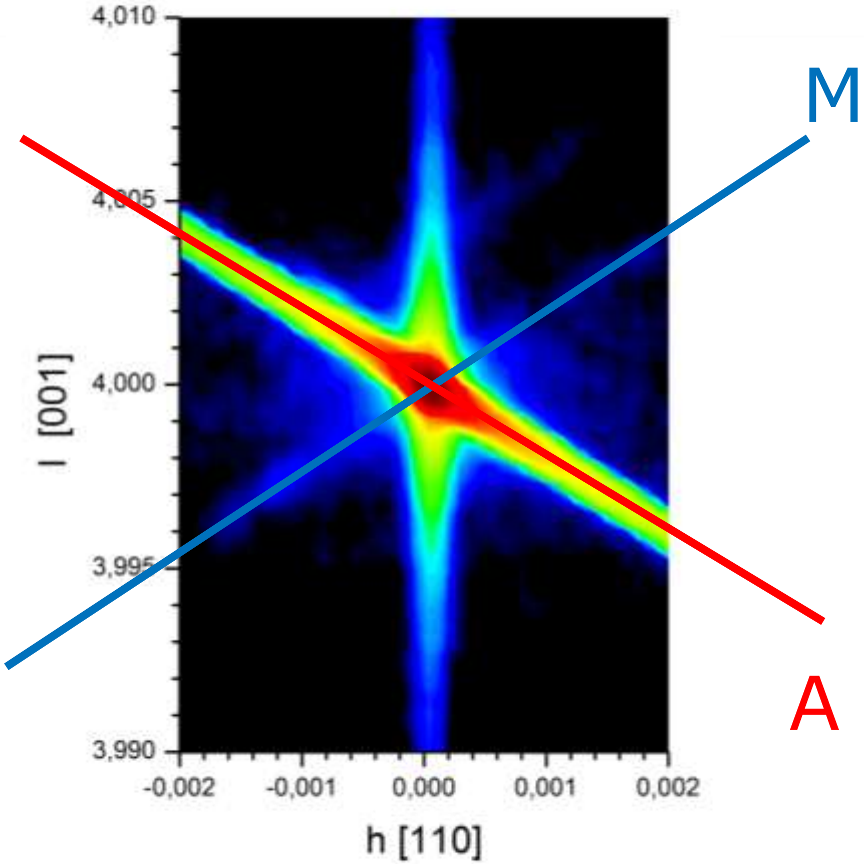
The instrumental resolution function in RSM

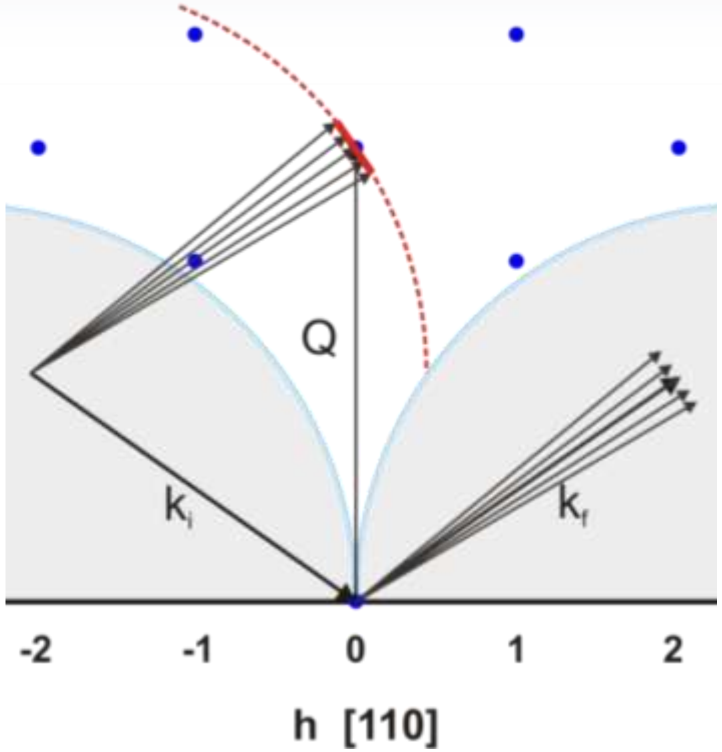




Monochromator streak is normal to incident beam

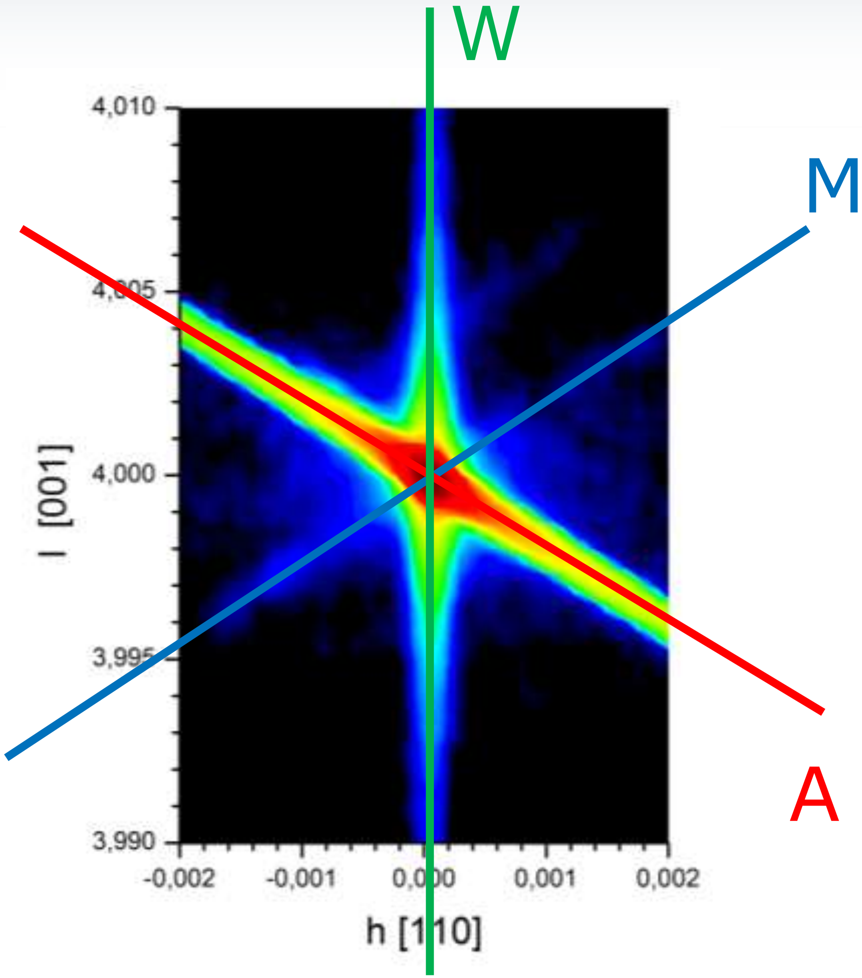
The instrumental resolution function in RSM

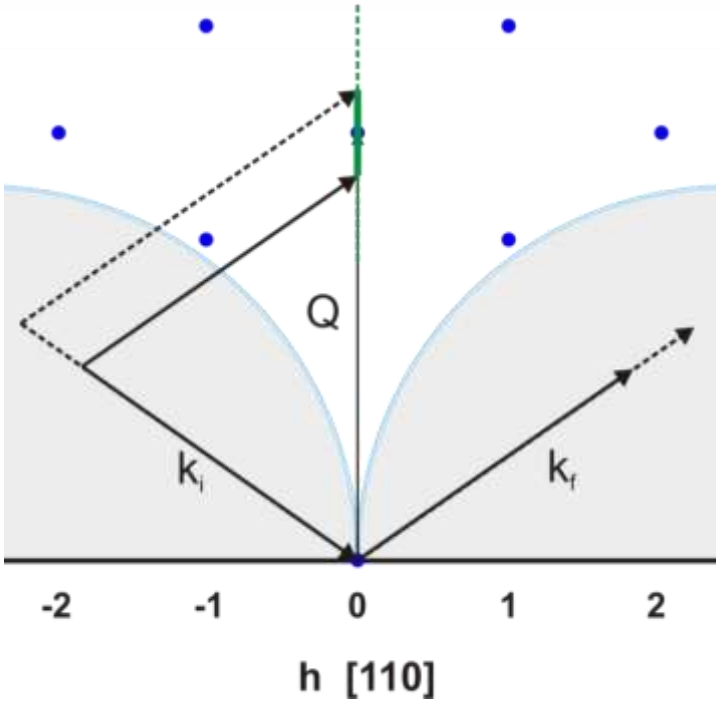




Analyzer streak is normal to exit beam

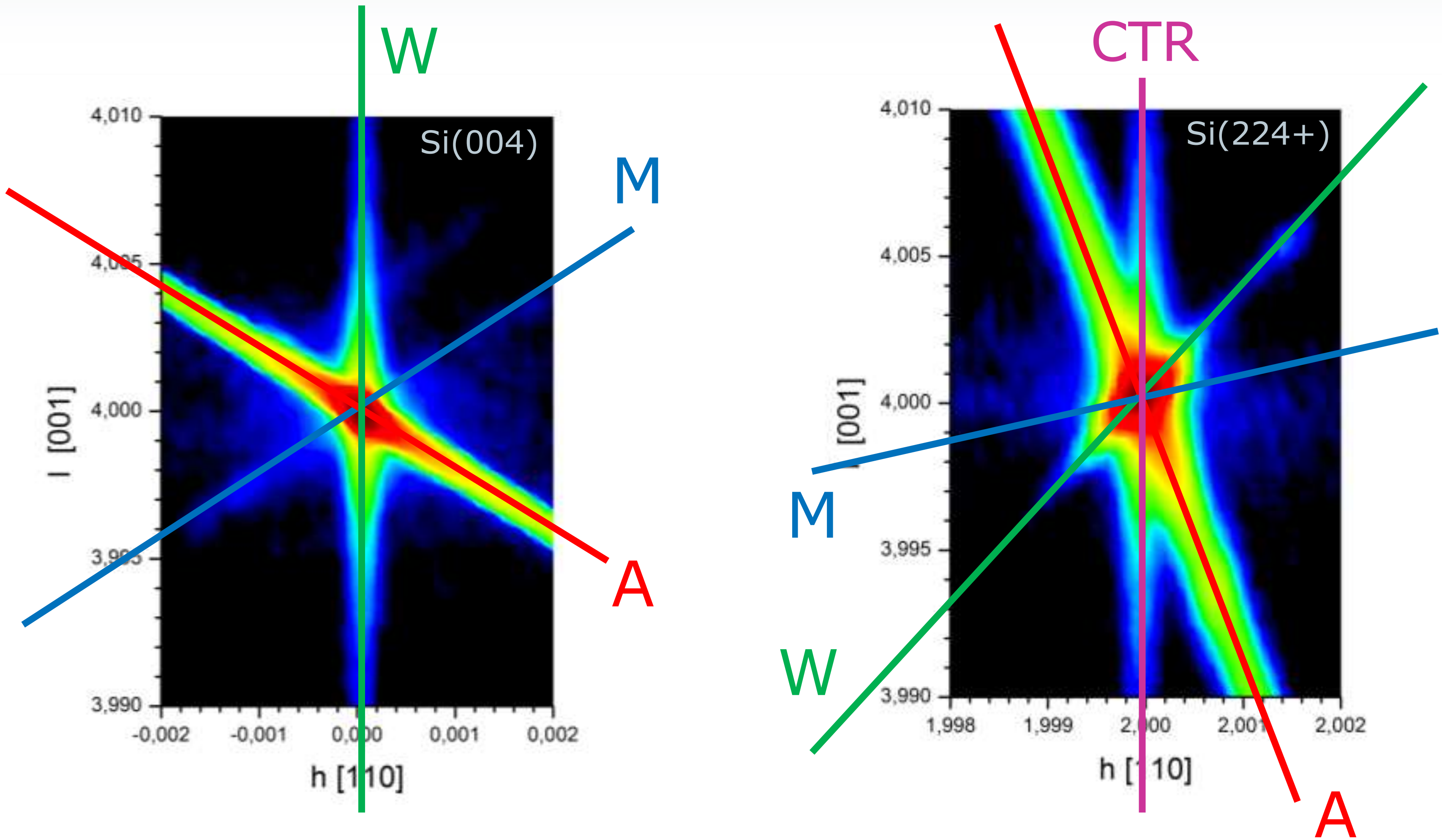
The instrumental resolution function in RSM





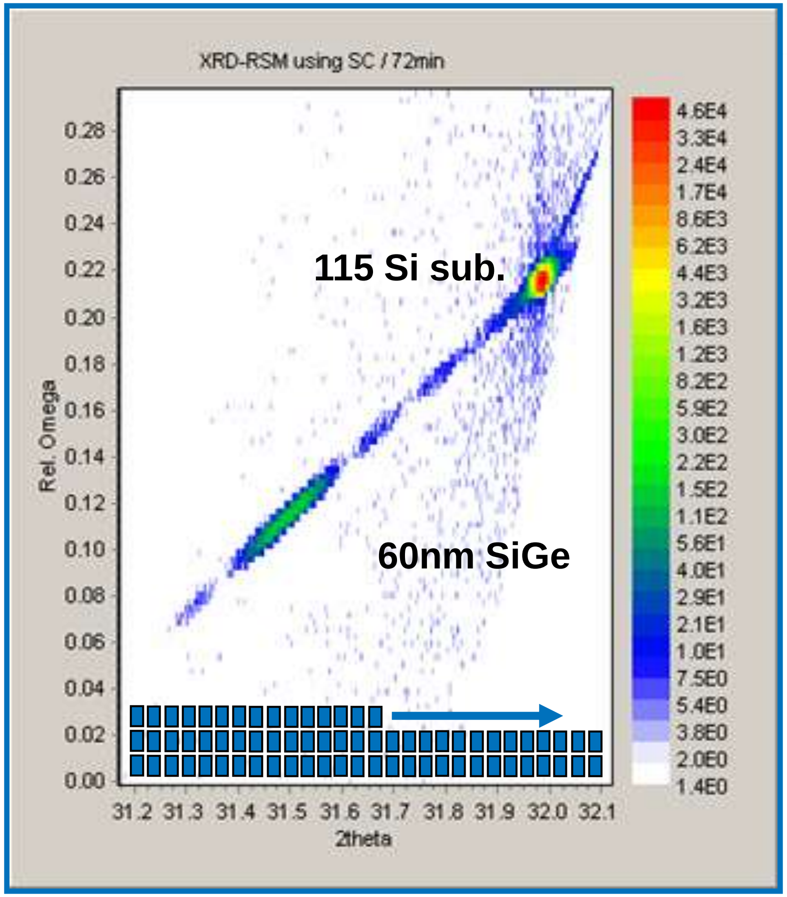
wavelength streak is along a line through (000)

The instrumental resolution function in RSM

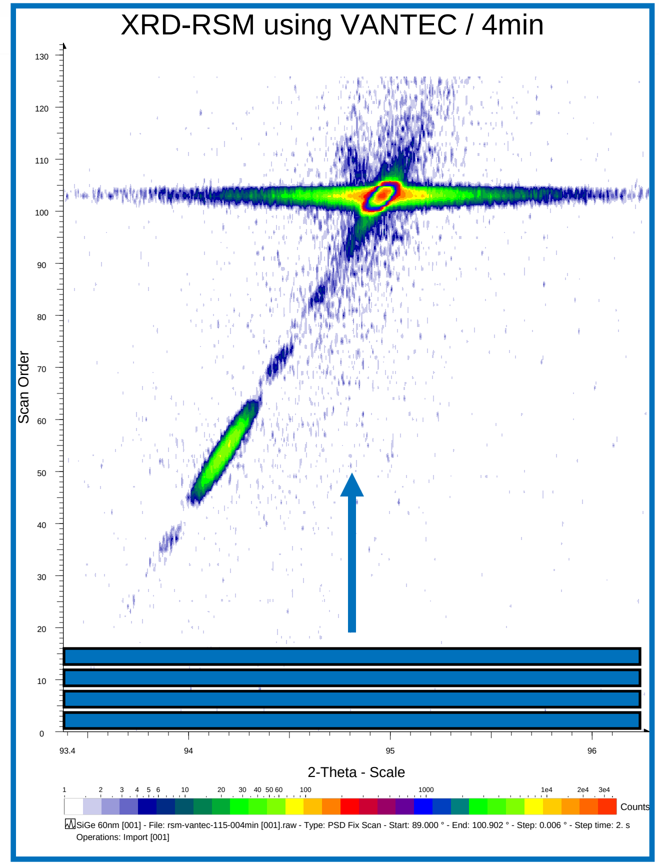


- What is Reciprocal Space
- What can be measured by Reciprocal Space maps (RSMs)
- How to measure RSMs
- **RSMs with 1D-detectors**
  - **Technique**
  - Examples
- RSMs with 2D-detectors

Reciprocal Space Maps
Measurement with 1D detectors
Scintillation Counter vs. VÅNTEC-1



Scintillation Counter: 72 min



VÅNTEC-1: 4 min

1D-detectors for fast RSM measurements: LYNXEYE

- Silicon strip detector
- 192 strips of 75 µm width
- Total window width 14.4 mm
- Resolution at 300 mm ≈ 0.012°
- Subsampling in Fast-scan mode
- Can be used as a 0D-detector

1D-detectors for fast RSM measurements: LYNXEYE and VÅNTEC-1

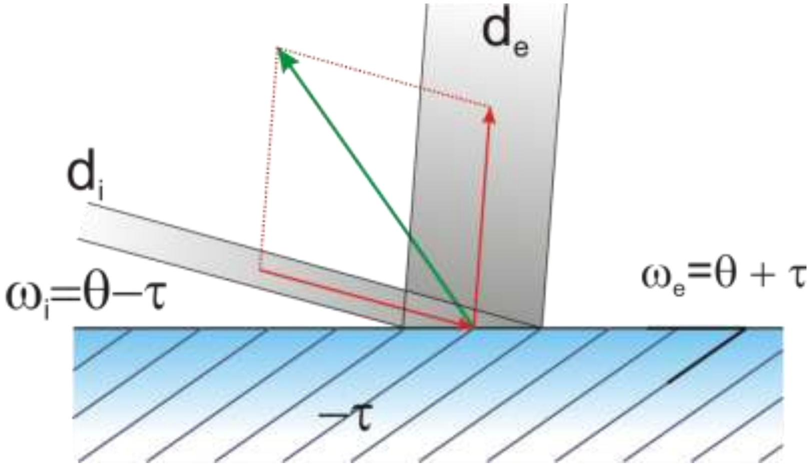
BRUKER

- Silicon strip detector
- Total window width 14.4mm
- 192 strips of 75µm width.
- Resolution at 300mm ≈ 0.012°
- Subsampling in Fast-scan mode
- Can be used as a 0D-detector

- Gas detector (Xe-CO_2)
- 50x16 mm^2 active area, simultaneous up to 12° in 2θ
- 1600 electronic channels
- Resolution at 300 mm ≈ 0.006°
- Low detector noise

Influence of the measurement geometry on the resolution

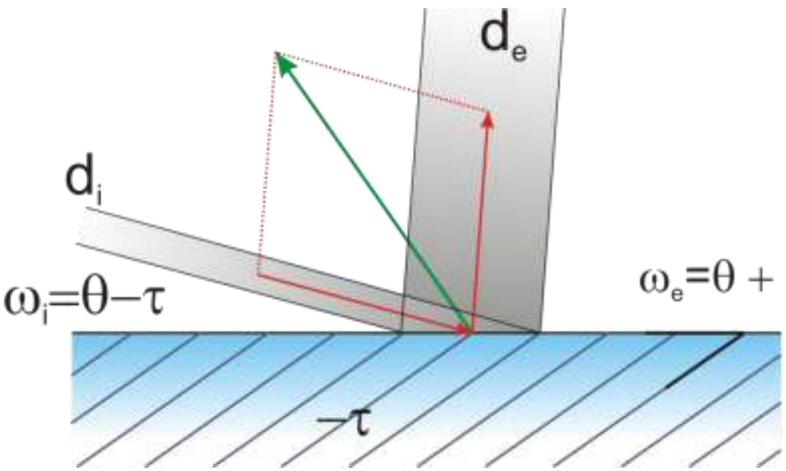
Grazing incidence geometry



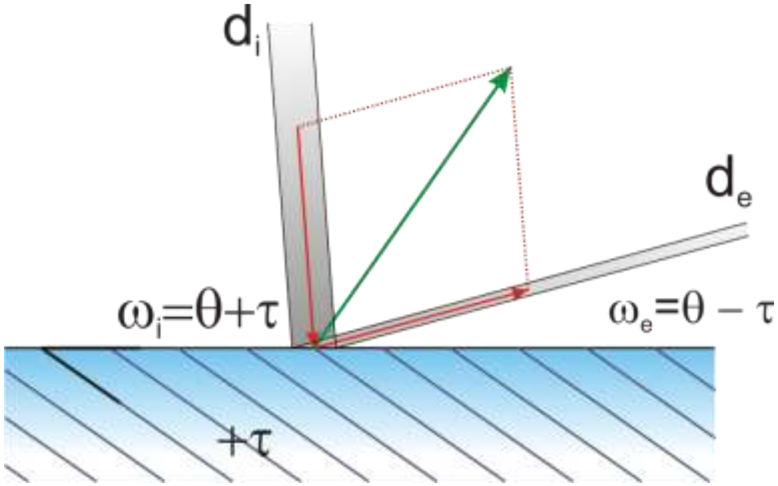
- Asymmetry factor $b = \frac{\sin(\omega_i)}{\sin(\omega_e)}$
- Exit beam width $d_e = \frac{d_i}{b}$

Influence of the measurement geometry on the resolution

Grazing incidence geometry



Grazing exit geometry



- Asymmetry factor $b = \frac{\sin(\omega_i)}{\sin(\omega_e)}$
- Exit beam width $d_e = \frac{d_i}{b}$

Influence of the measurement geometry on the resolution (2)

- When using a 1D-detector for the RSM, the reflection should be chosen such that the beam compression is high.
- The incident beam should be adapted to achieve the highest possible resolution.

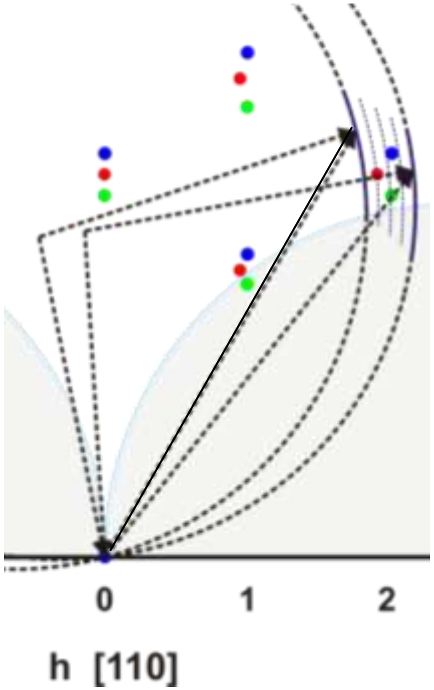
hkl	ω_i	ω_e	b	d_{inc} (75 µm)
113+	56.2°	2.9°	15.9	1.1 mm
224+	79.2°	8.8°	6.42	0.5 mm
115+	63.3°	32.7°	1.7	0.1 mm

Si reflections for Cu-Ka radiation

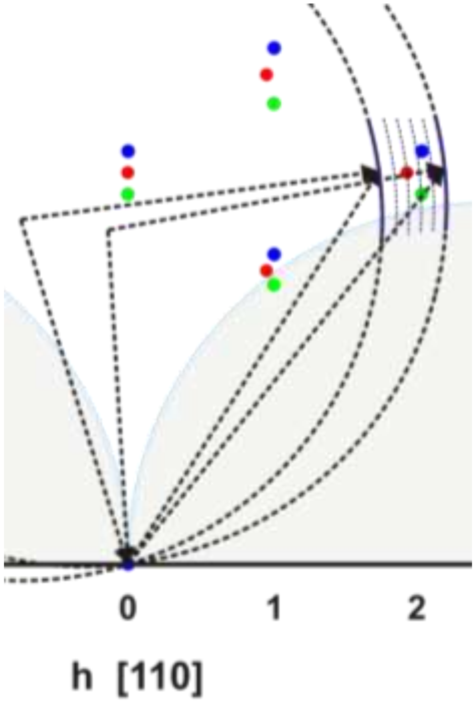
- When RSMs are measured using an analyzer crystal, the full incident beam can be used and the geometry is not critical.

RSMs with a 1D detector:
Choice of the loop scan

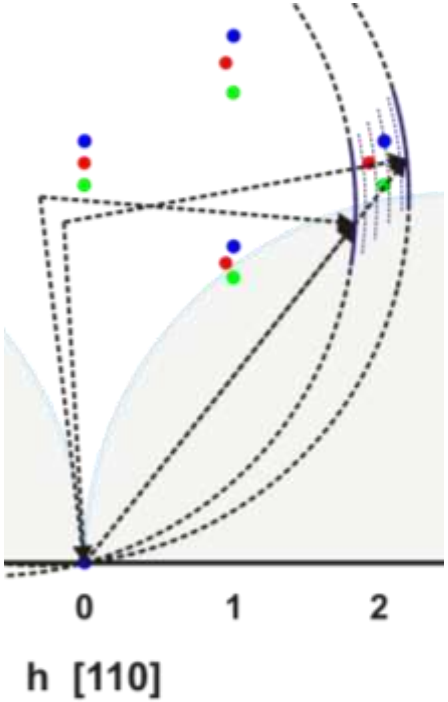
Looped over rocking curve



Looped over h-scan



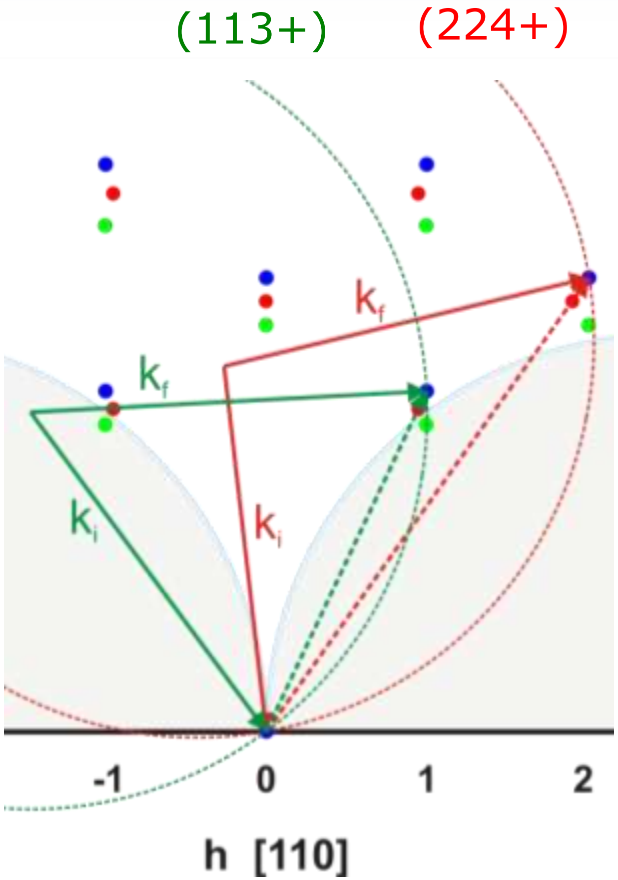
Looped over 2θ/ω scan



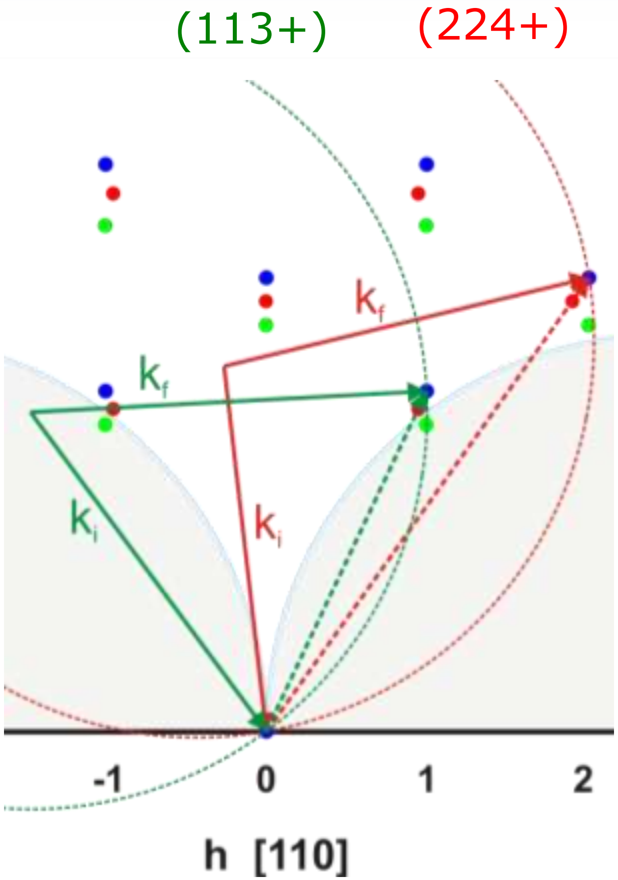
Pseudomorphic layers

Fully relaxed layers

Choosing the appropriate reflection for the RSM: (113+) vs. (224+)



Choosing the appropriate reflection for the RSM: (113+) vs. (224+)



- \+ Structures are more compact. Faster RSM.
- \+ Higher structure factor. More intensity.
- \+ Detector Snapshot is almost like a l-scan. Faster RSM.
- \+ Beam Compression factor is higher. Better resolution.
- – Penetration depth decreases at grazing angles. Deeper layers of the sample may not show up.
- – Less precision in lattice parameter determination.

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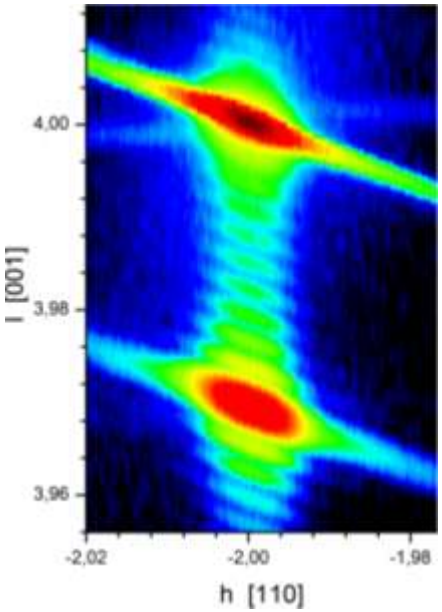
  - **Examples**
- RSMs with 2D-detectors

RSMs from $In_{0.06}Ga_{0.96}As$ films on GaAs with different layer thicknesses

d = 200 nm

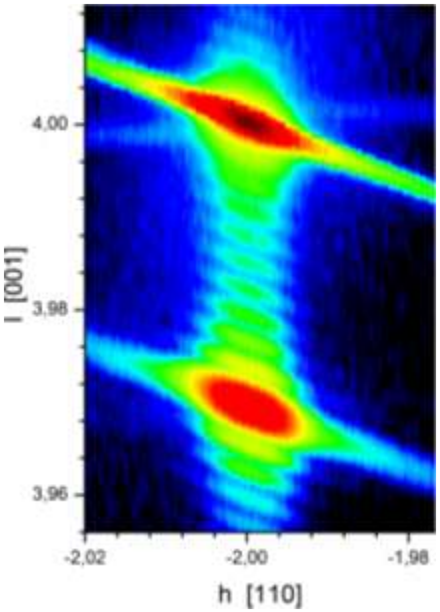


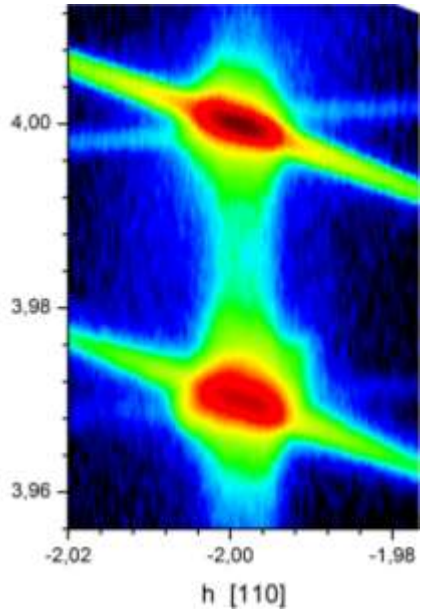
Samples provided by F. Rinaldi (Uni Ulm / Bruker AXS)

RSMs from $In_{0.06}Ga_{0.96}As$ films on GaAs with different layer thicknesses

d = 200 nm

d = 400 nm

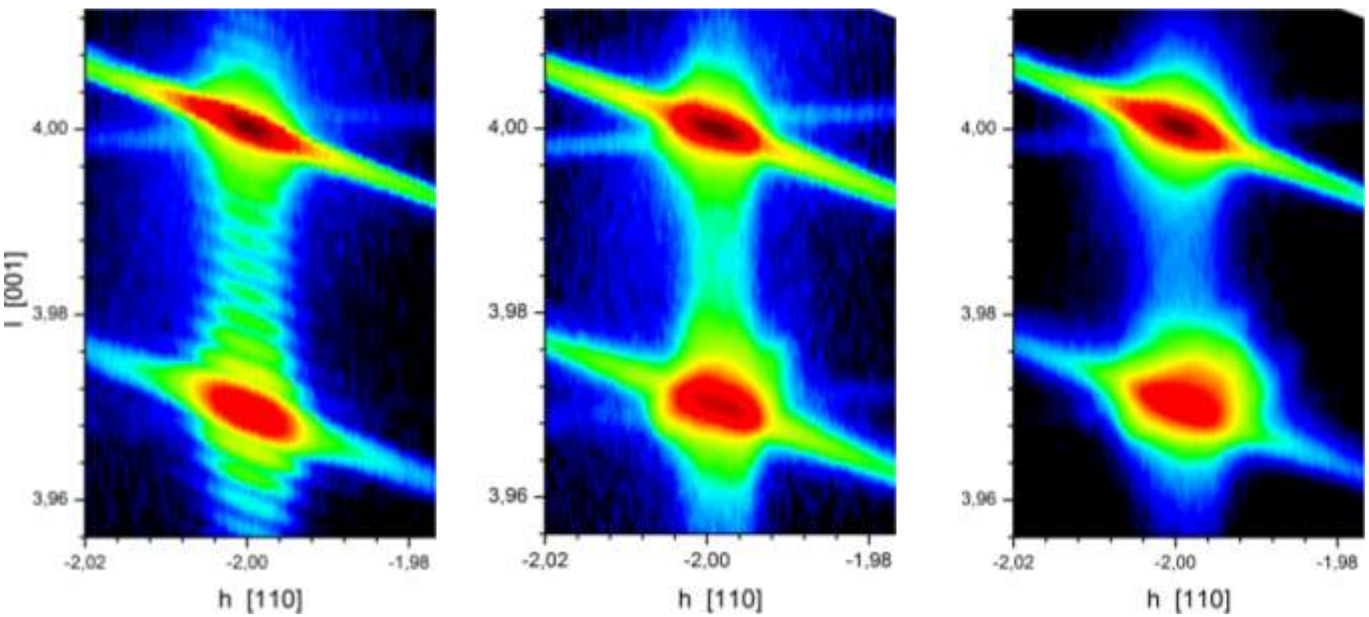




Samples provided by F. Rinaldi (Uni Ulm / Bruker AXS)

RSMs from $In_{0.06}Ga_{0.96}As$ films on GaAs with different layer thicknesses

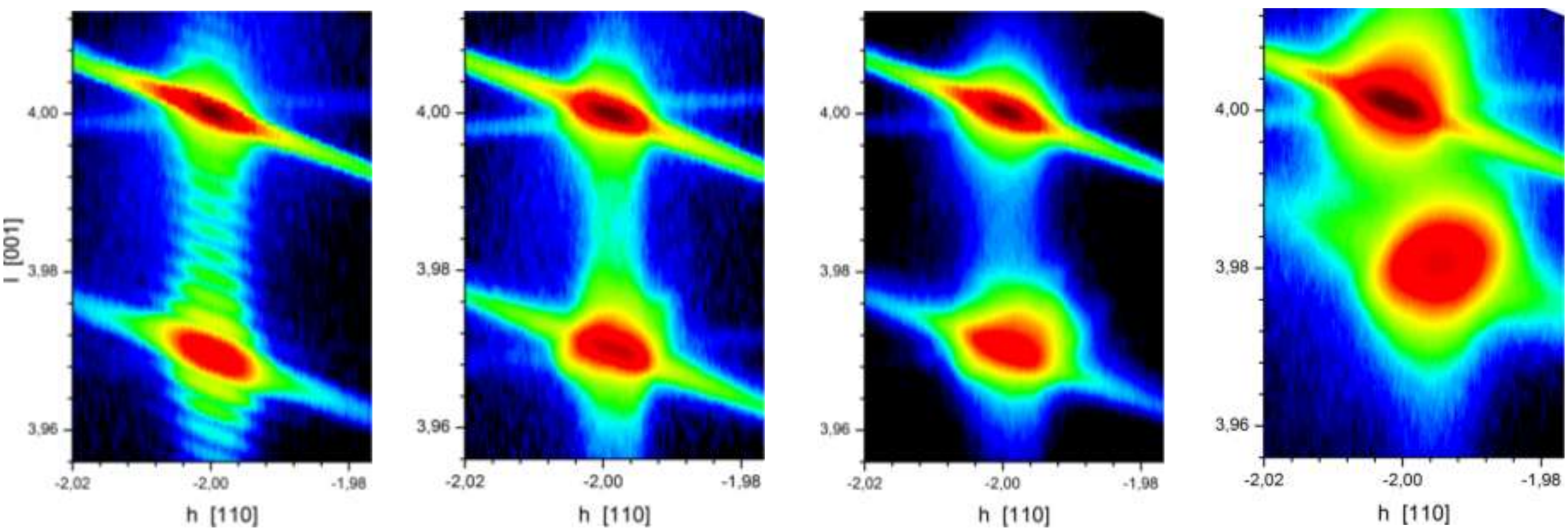
d = 200 nm d = 400 nm d = 450 nm



Samples provided by F. Rinaldi (Uni Ulm / Bruker AXS)

RSMs from $In_{0.06}Ga_{0.96}As$ films on GaAs with different layer thicknesses

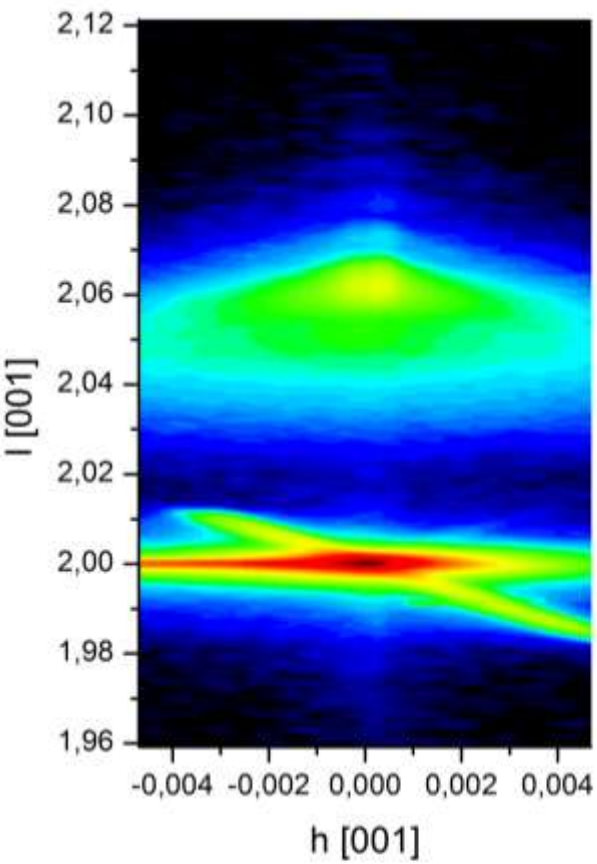
d = 200 nm d = 400 nm d = 450 nm d = 800 nm



Samples provided by F. Rinaldi (Uni Ulm / Bruker AXS)

Example: $LaAlO_3$ on STO

STO(002)



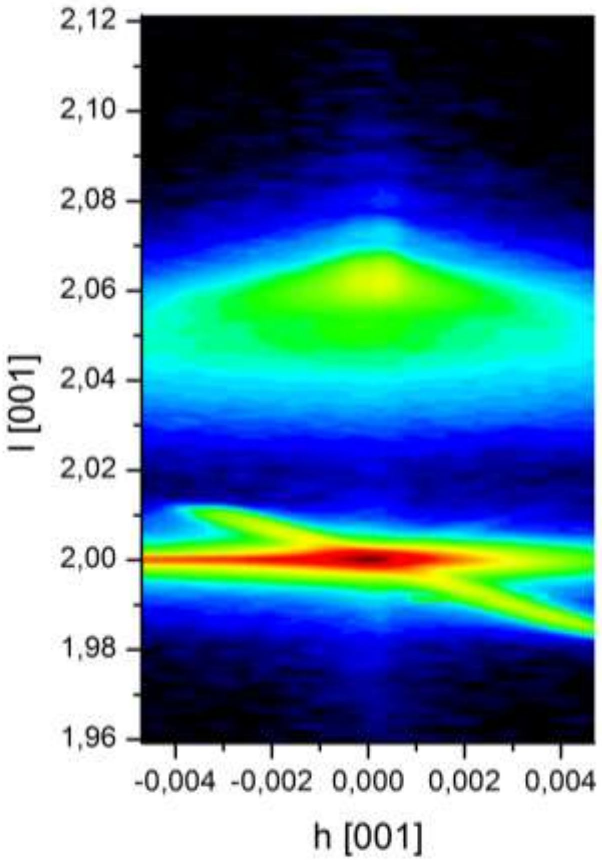
50 nm $LaAlO_3$

substrate STO

Sample courtesy of Dirk Fuchs (Institute of solid state physics, KIT)

Example: $LaAlO_3$ on STO

STO(002)



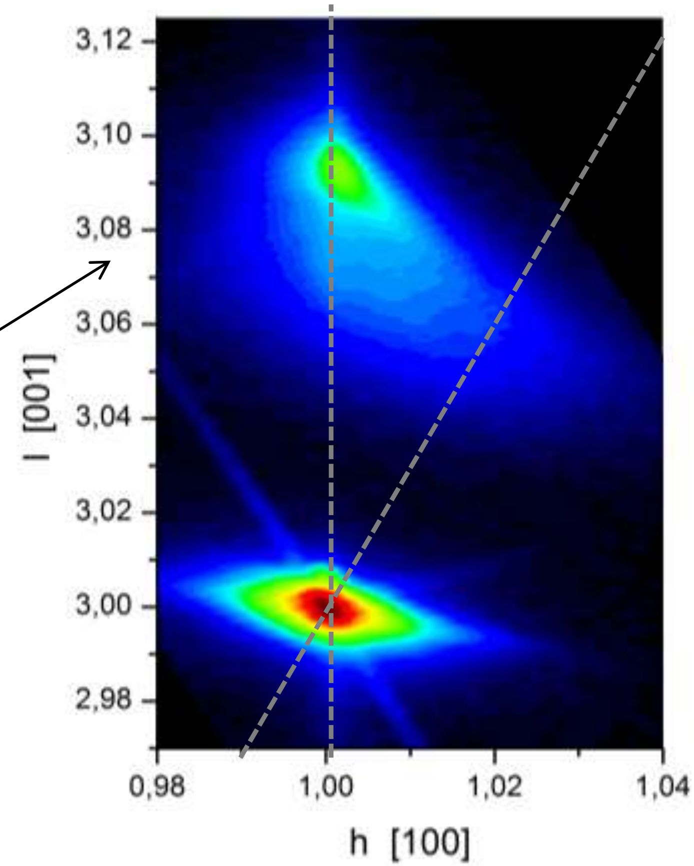
50 nm $LaAlO_3$

substrate STO

No miscut

Relaxation

STO(103+)



Sample courtesy of Dirk Fuchs (Institute of solid state physics, KIT)

Example: GaN-based HEMT structure

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

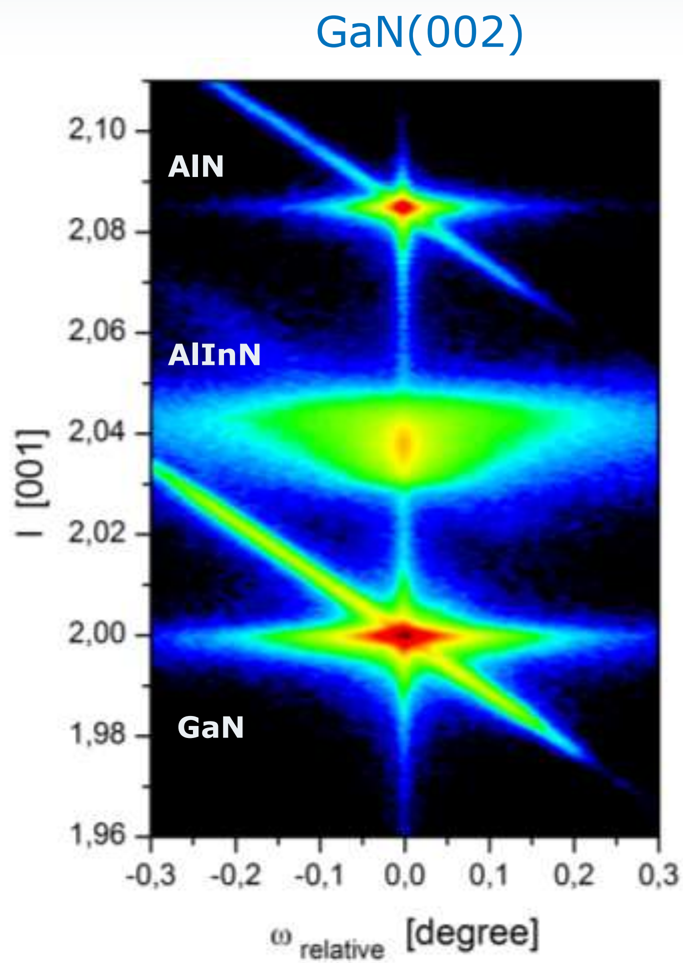
1 nm AlN

1000 nm GaN

350 nm AlN

substrate Al_2O_3

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



Example: GaN-based HEMT structure

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

1 nm AlN

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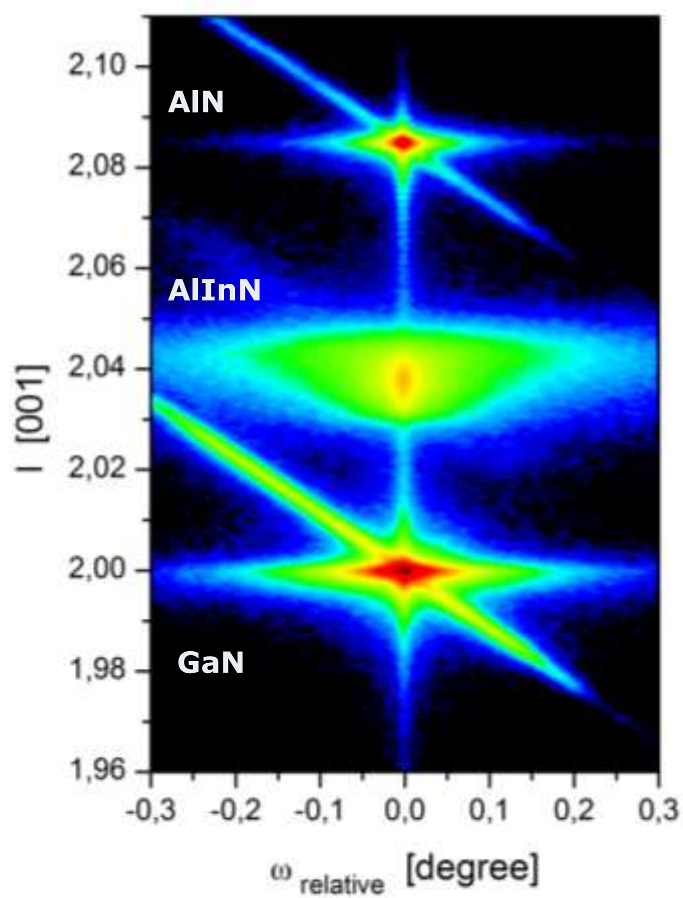
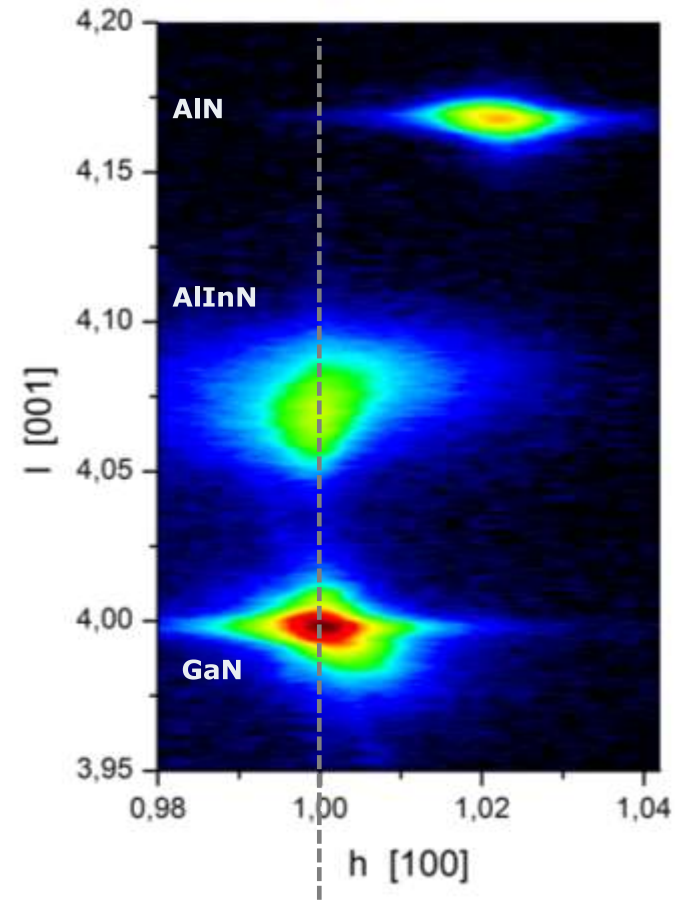
Sample courtesy of L. R. Khoshroo (RWTH Aachen)

GaN(002)



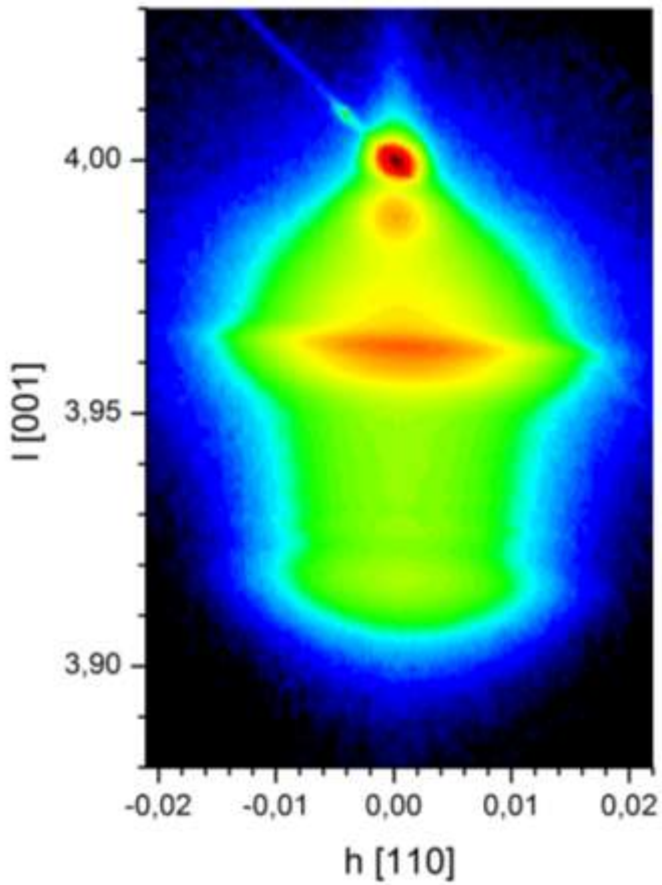
GaN(104+)



Example: HEMT structure with graded buffer layers

Layer
10 nm $In_{0.65}Ga_{0.35}As$
120 nm $In_{0.65}Al_{0.35}As$
30 nm $In_{0.65}Ga_{0.35}As$
50 nm $In_{0.65}Al_{0.35}As$
200 nm $In_xAl_{1-x}As$ (x=0.75 top, x=0.65 bottom)
500 nm $In_xAl_{1-x}As$ (x=0.65 top, x=0.52 bottom)
50 nm $In_{0.52}Al_{0.48}As$
substrate InP

InP(004)

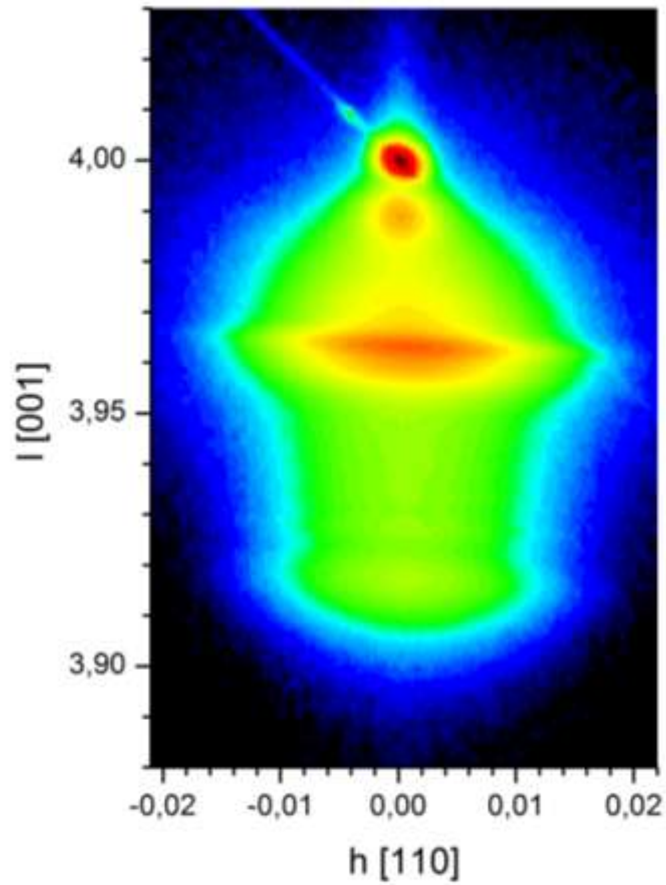


Sample courtesy of Ian Farrer (University of Cambridge)

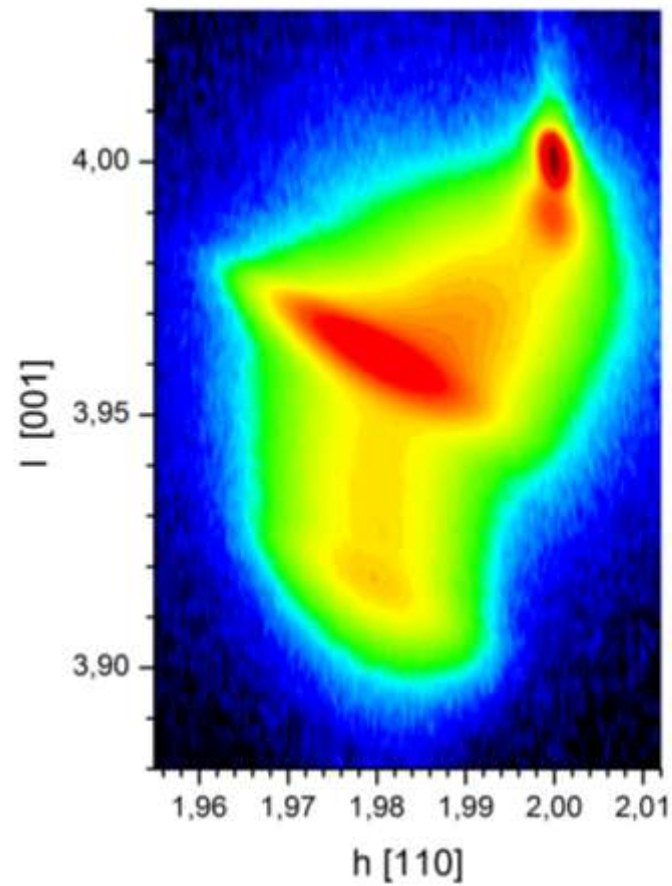
Example: HEMT structure with graded buffer layers

- 10 nm $In_{0.65}Ga_{0.35}As$
- 120 nm $In_{0.65}Al_{0.35}As$
- 30 nm $In_{0.65}Ga_{0.35}As$
- 50 nm $In_{0.65}Al_{0.35}As$
- 200 nm $In_xAl_{1-x}As$ x=0.75 / x=0.65
- 500 nm $In_xAl_{1-x}As$ x=0.65 / x=0.52
- 50 nm $In_{0.52}Al_{0.48}As$
- substrate InP

InP(004)

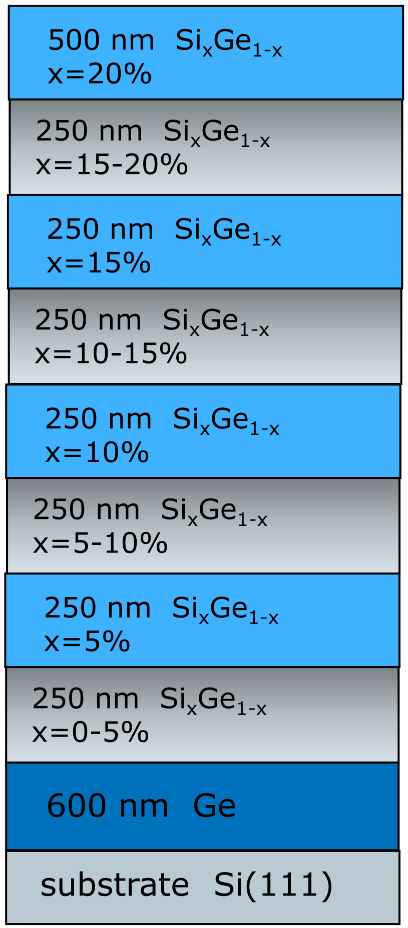


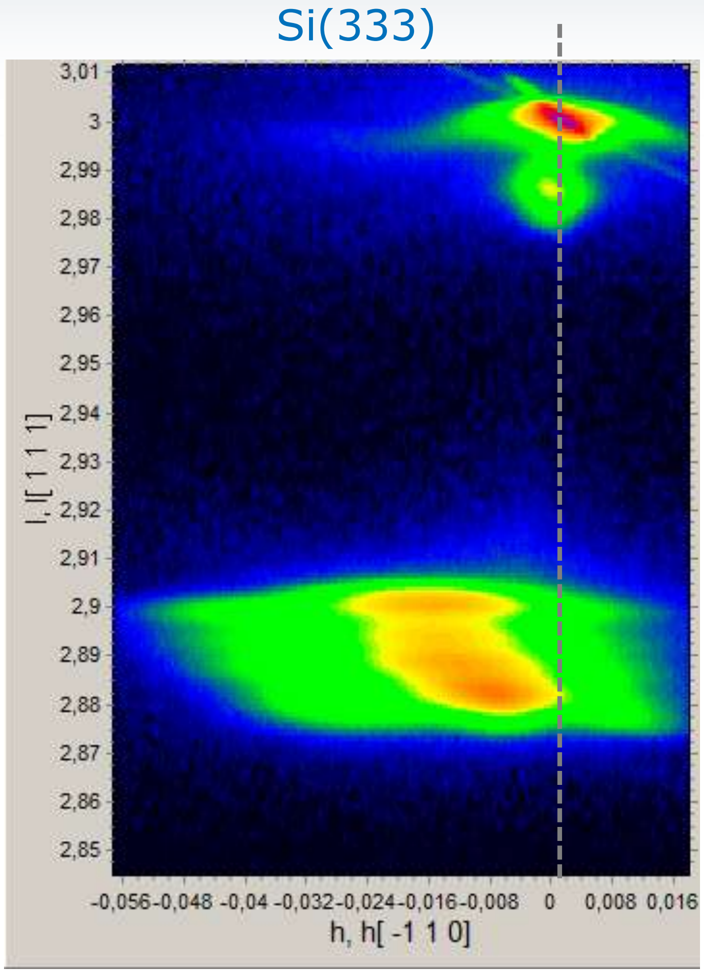
InP(224+)



Sample courtesy of Ian Farrer (University of Cambridge)

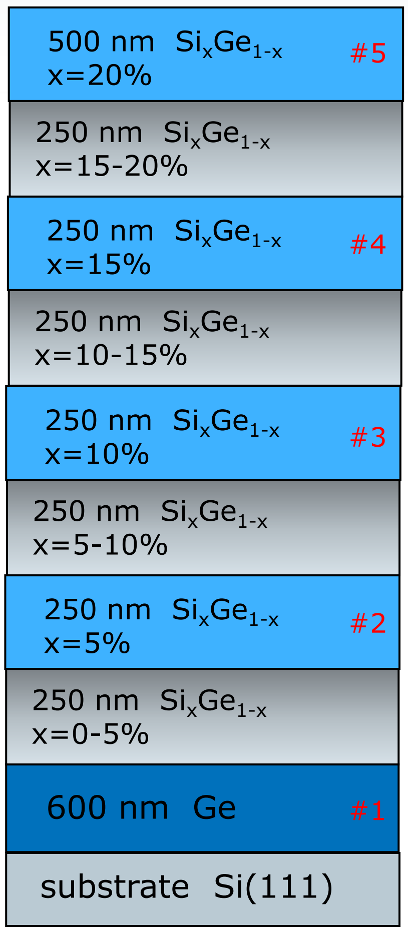
RSM from a SiGe graded heterostructure: Miscut, Relaxation and Concentration

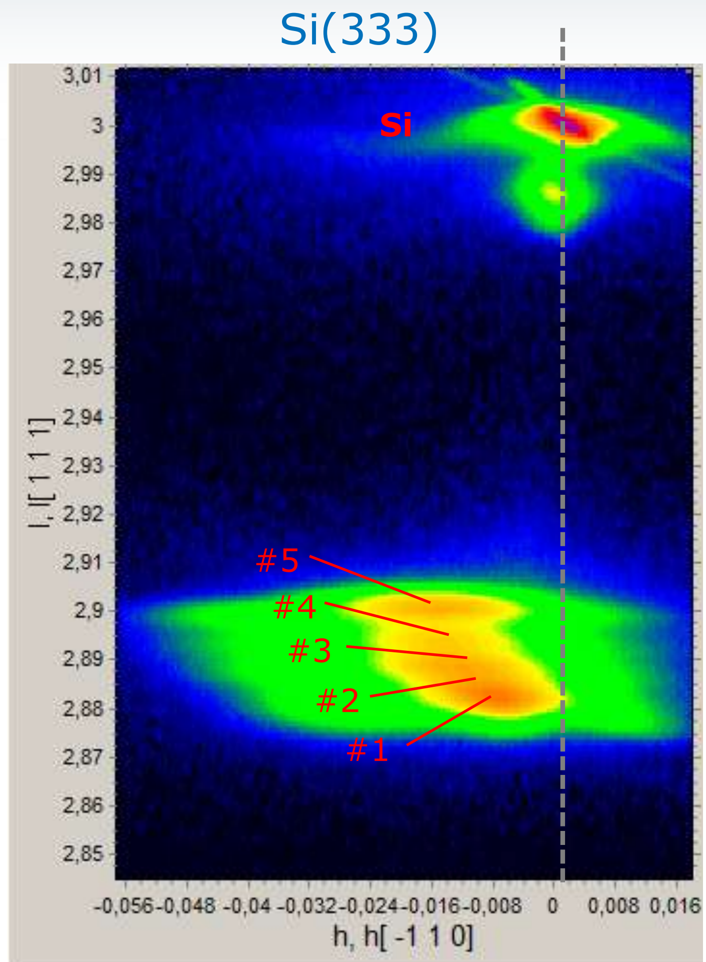




Extract miscut for each SiGe layer

RSM from a SiGe graded heterostructure: Miscut, Relaxation and Concentration





Extract miscut for each SiGe layer

RSM from a SiGe graded heterostructure: Miscut, Relaxation and Concentration

500 nm Si_xGe_{1-x}
x=20%

250 nm Si_xGe_{1-x}
x=15-20%

250 nm Si_xGe_{1-x}
x=15%

250 nm Si_xGe_{1-x}
x=10-15%

250 nm Si_xGe_{1-x}
x=10%

250 nm Si_xGe_{1-x}
x=5-10%

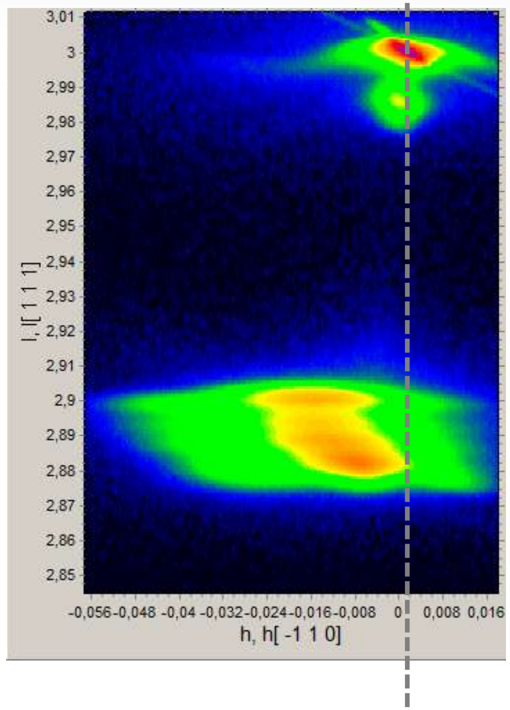
250 nm Si_xGe_{1-x}
x=5%

250 nm Si_xGe_{1-x}
x=0-5%

600 nm Ge

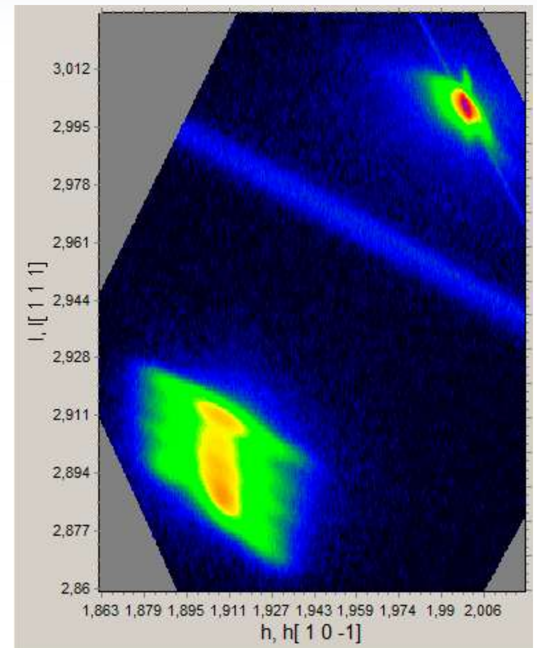
substrate Si(111)

Si(333)



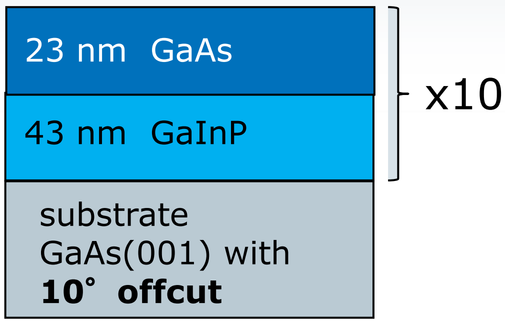
Extract miscut
for each SiGe layer

Si(531+)

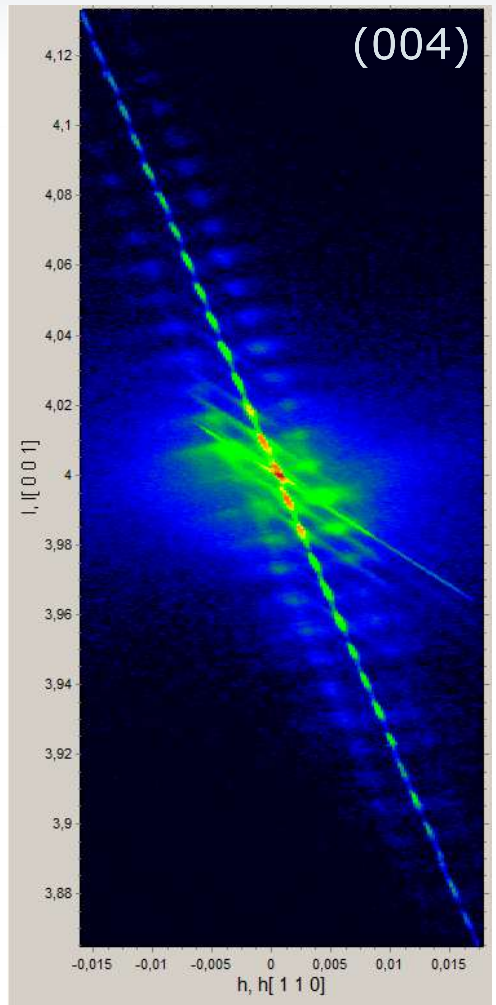


Relaxation
Concentration

RSM from superlattices with large offcut



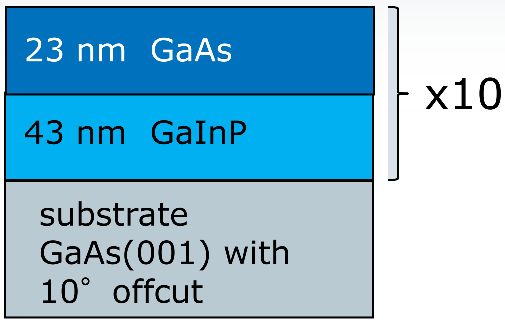
Sample courtesy of Uni Sheffield



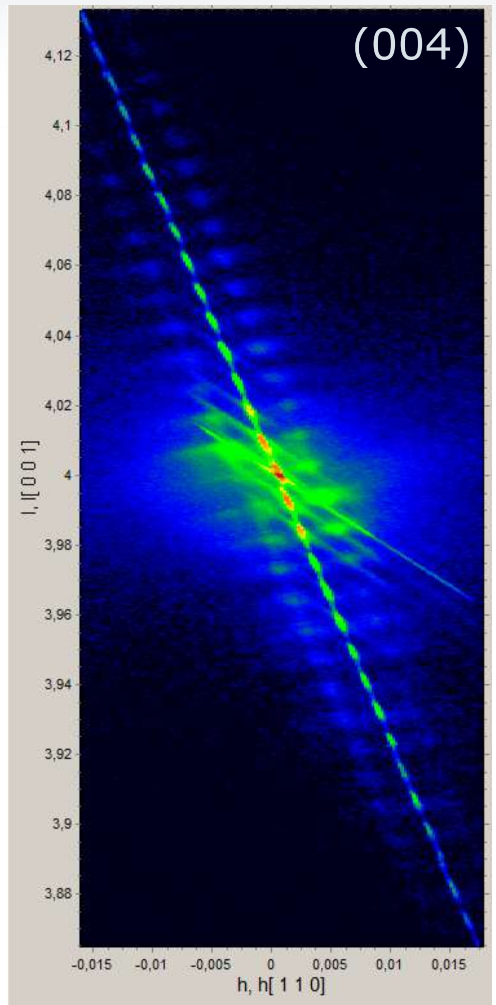
Evaluated Parameters:

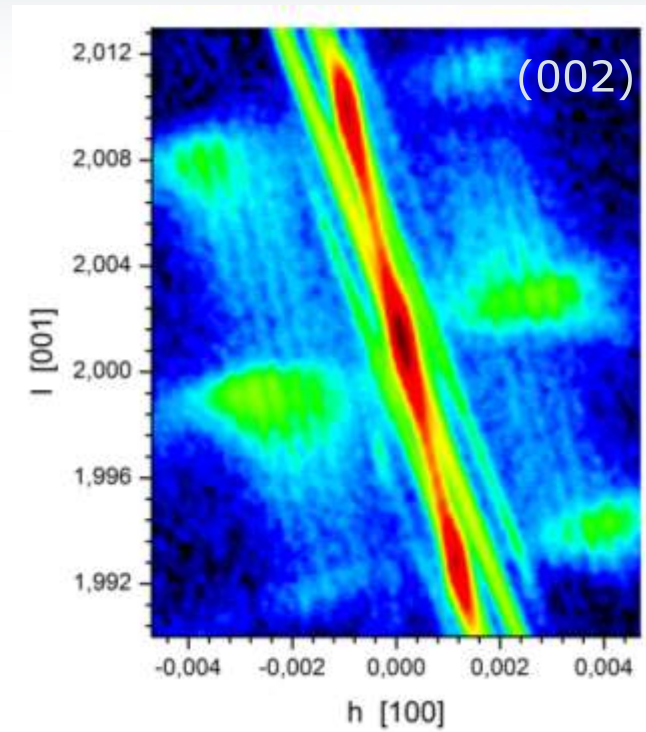
- Concentration
- Layer thickness
- SL period

RSM from superlattices with large offcut



Sample courtesy of Uni Sheffield





Evaluated Parameters:

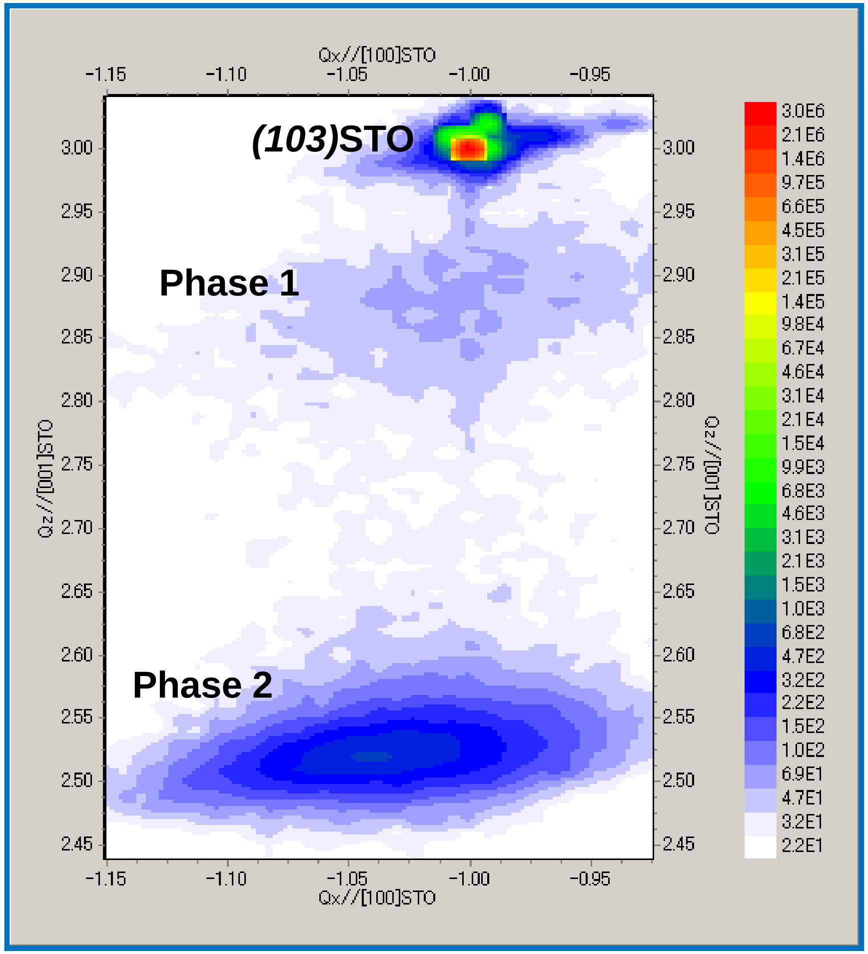
- Concentration
- Layer thickness
- SL period

Periodic lateral structure due to substrate steps

Outline

- What is Reciprocal Space
- What can be measured by Reciprocal Space maps (RSMs)
- How to measure RSMs
- RSMs with 1D-detectors
- **RSMs with 2D-detectors**

Fast RSM with a 2D-detector
Example: $BiFeO_3$(200nm)/(001)$SrTiO_3$



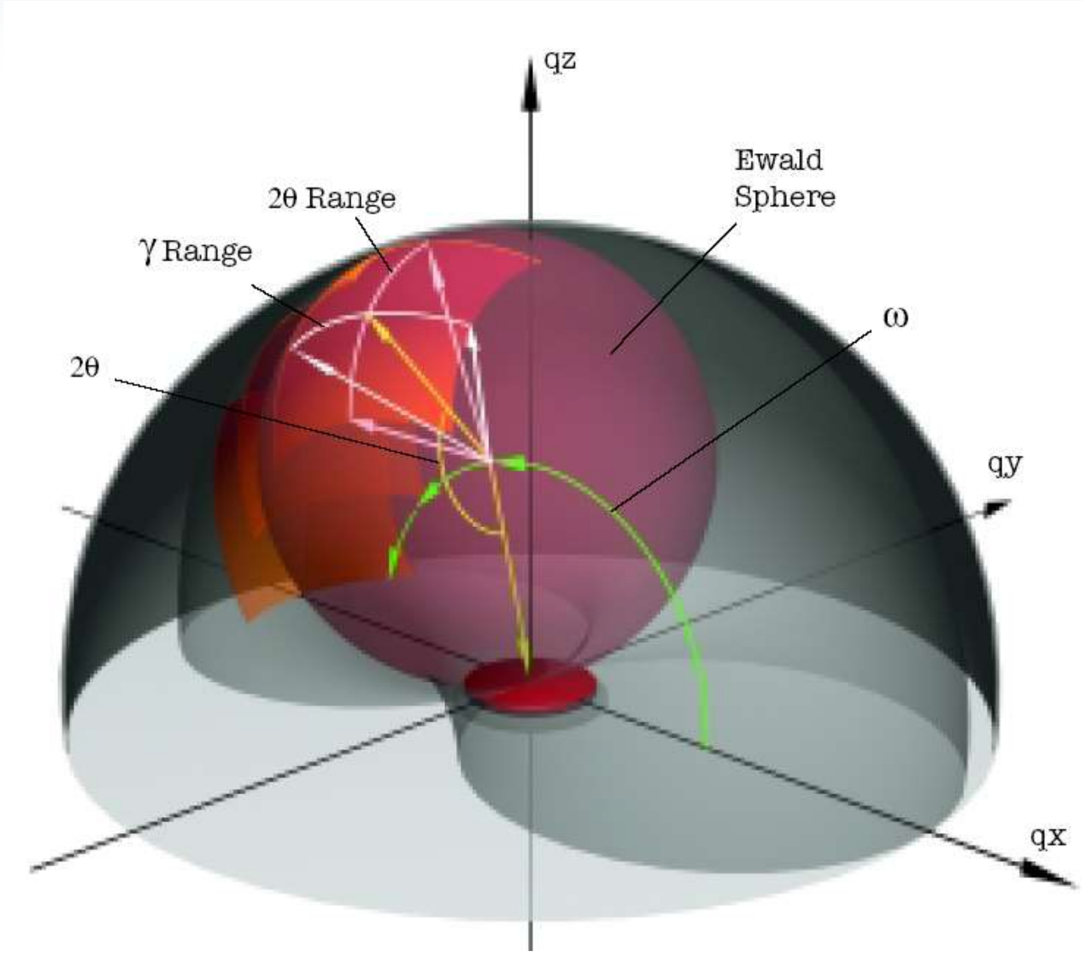
Scintillation Counter

- Measurement time: 8 hours
- Just one (103) diffraction spot
- High resolution not needed

Fast RSM with a 2D-detector:
The VÅNTEC-500

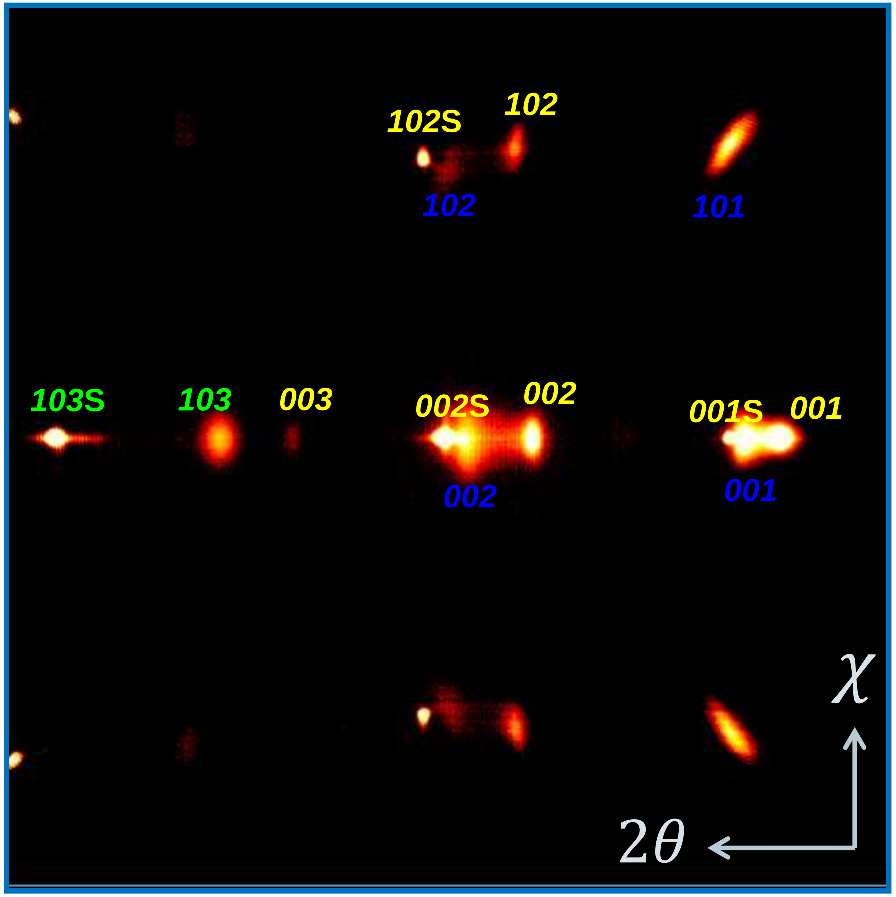
- Xe-based gas detector
- Window diameter of 140 mm
- 2048 x 2048 pixels with 68 x 68 µm size
- Detector noise <0.0005 cps/mm²
- 2θ range
  - 23° at 300 mm
  - 56° at 100 mm

Fast RSM with a 2D-detector: Measurement mode



- 2D detector used in fixed position --> fixed range in 2θ and γ is measured simultaneously
- ω scanned for one frame for half of the 2θ range of the detector --> Ewald sphere (red) is rotated to integrate the reflection

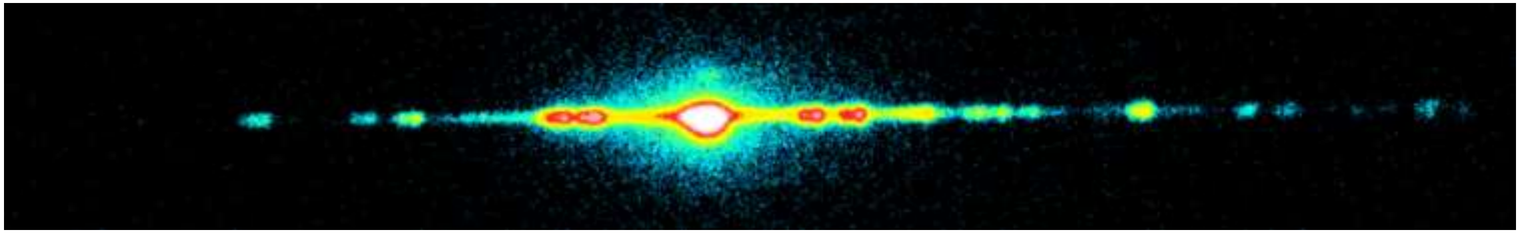
Fast RSM with a 2D-detector
Example: $BiFeO_3$(200nm)/(001)$SrTiO_3$



- Integration time ≈ 45 min
- In-plane epitaxial relation as well as exisitence of second phase (impurity phase) is clear.
- Sample from Osaka University

Benefits of RSM with 2D-detector

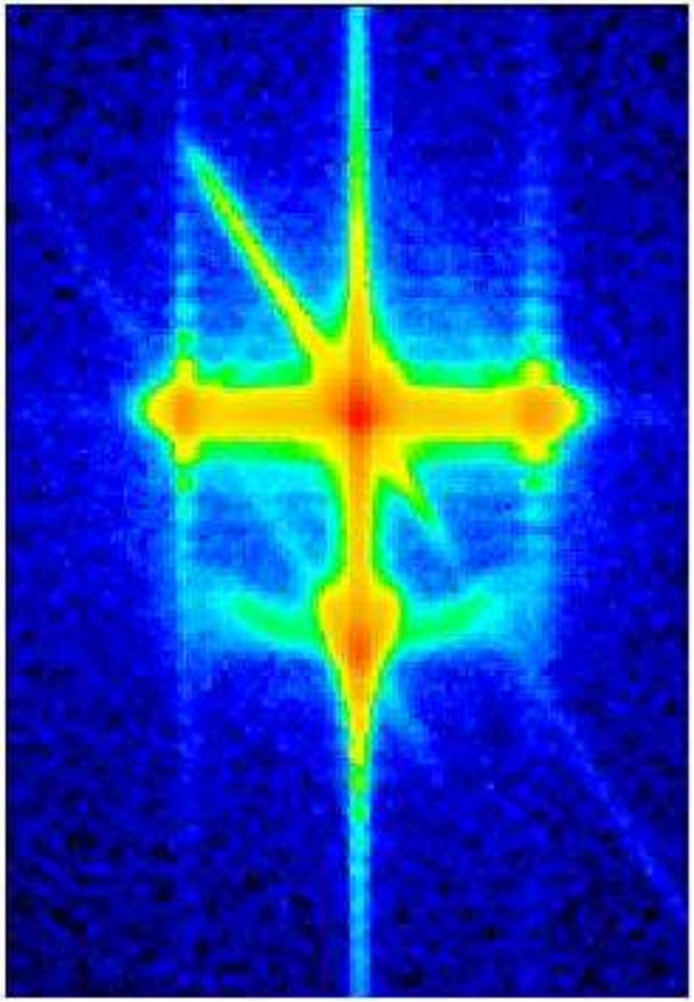
- Very fast, no alignment required
- Good indication of degree of orientation / quality of layers
- Do I have the layer structure that I want?
- Can spot certain sample-to-sample differences immediately
- Use it as a very fast feedback loop to optimize the making of your sample



Q & A

Any Questions?

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



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- Educational one-day seminars on X-ray Analysis
- April – November in a city near you!
- Check the tour schedule and register early!

April
- Vancouver, British Columbia - Monday, **April 30**

May
- Seattle, Washington - Wednesday, **May 2**
- Portland, Oregon - Friday, **May 4**
- Toronto, Ontario, Canada - Monday, **May 7**
- Ottawa, Ontario, Canada - Wednesday, **May 9**
- Montreal, Quebec, Canada - Friday, **May 11**
- Fremont, California - Monday, **May 14**
- Salt Lake City, Utah - Wednesday, **May 16**
- Denver, Colorado - Friday, **May 18**

August
- Minneapolis, Minnesota - Monday, **August 6**
- St. Louis, Missouri - Wednesday, **August 8**
- Memphis, Tennessee - Friday, **August 10**

November
- Bethesda, Maryland - Monday, **November 5**
- New Brunswick, New Jersey - Wednesday, **November 7**
- New Haven, Connecticut - Friday, **November 9**

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