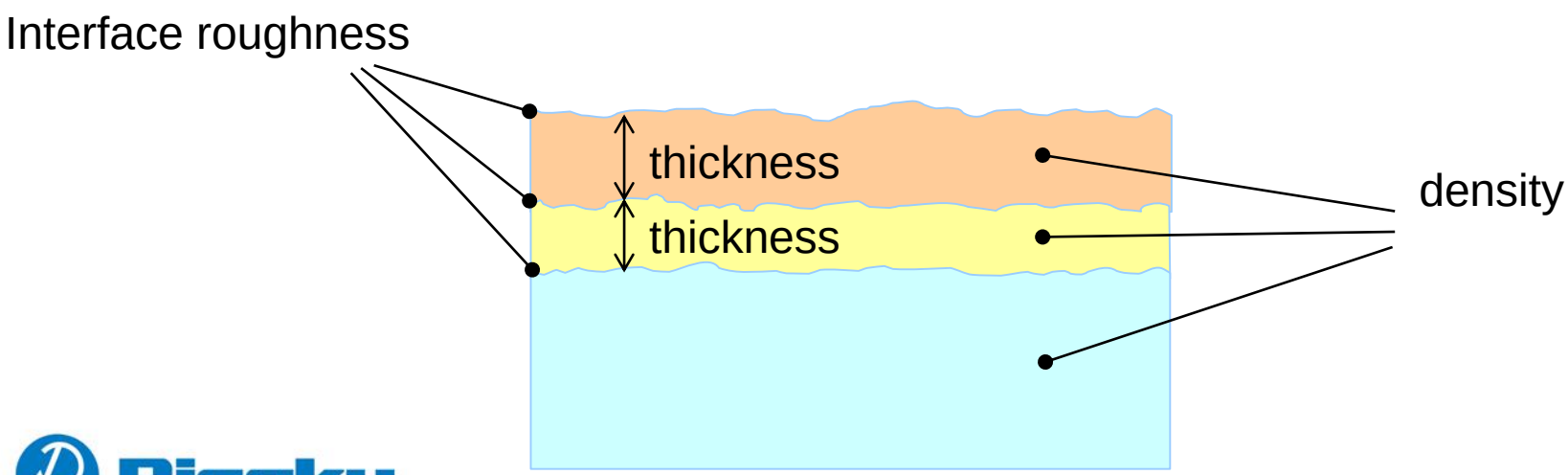
X-ray Reflectivity Analysis

What x-ray reflectivity reveals

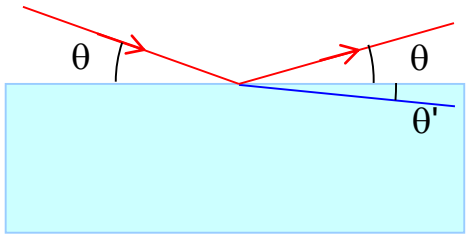
X-ray reflectivity nondestructively reveals

- layer structure of multi layers
- thickness (1 to 300 nm)
- density
- surface and interface roughness



Total reflection of x-rays

refractive index for x-rays : $n = 1 - \delta - i\beta < 1$



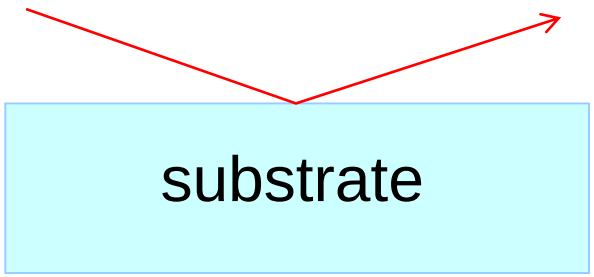
Snell's low : $n = \dfrac{\sin(\pi/2 - \theta)}{\sin(\pi/2 - \theta')}$

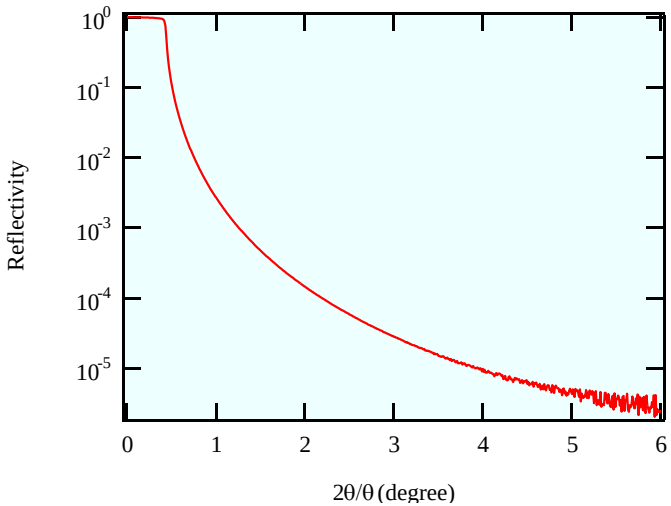
$\theta' < \theta$

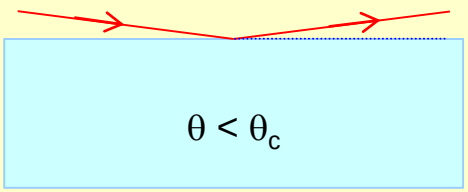
when $\theta < \theta_c \cong \sqrt{2\delta}$ x-rays are totally reflected.

(The critical angle θ_c depends on the chemical formula and density of the material.)

Reflectivity profile (substrate)

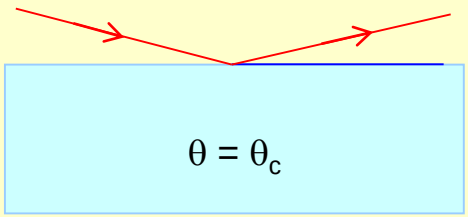






incident angle < critical angle

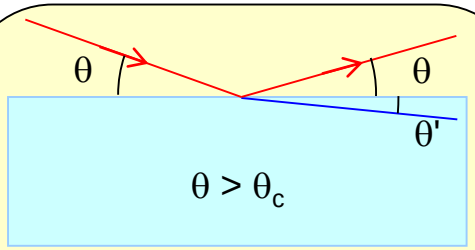
*X-rays are totally reflected.



incident angle = critical angle

*X-rays start to penetrate to the sample.

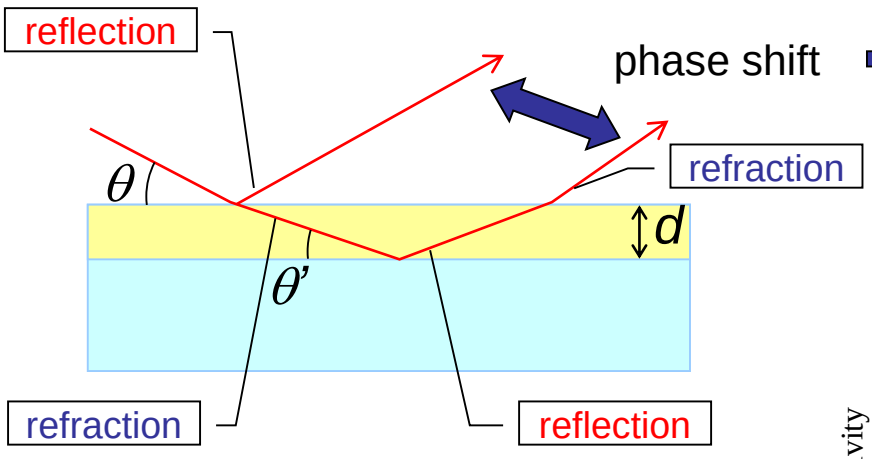
*The penetrated x-rays propagate parallel to the sample surface.



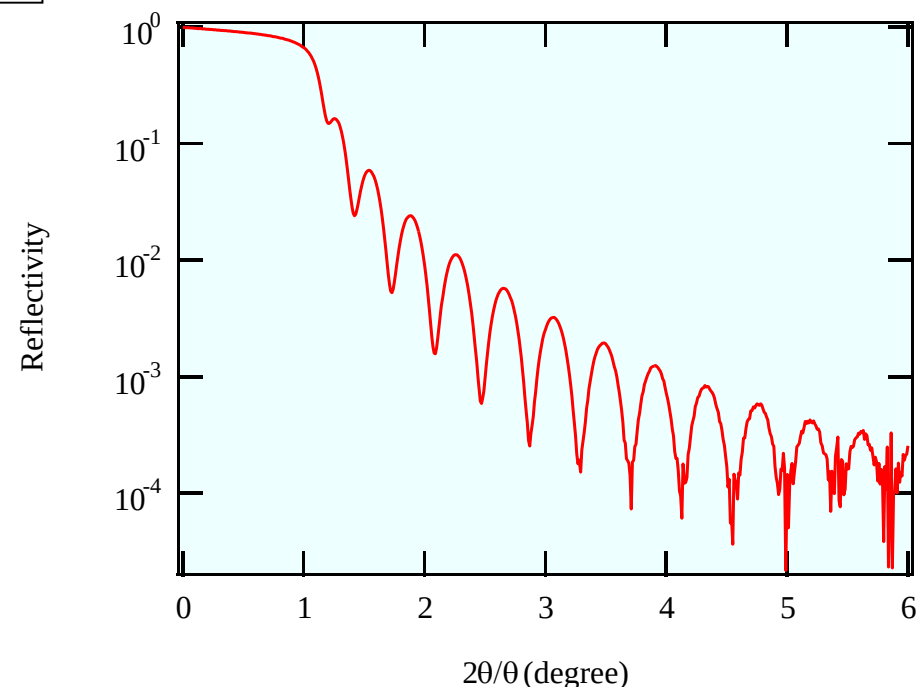
incident angle > critical angle

*The reflected intensity decays by x-ray penetration to the sample.

Reflectivity profile (substrate + thin film)



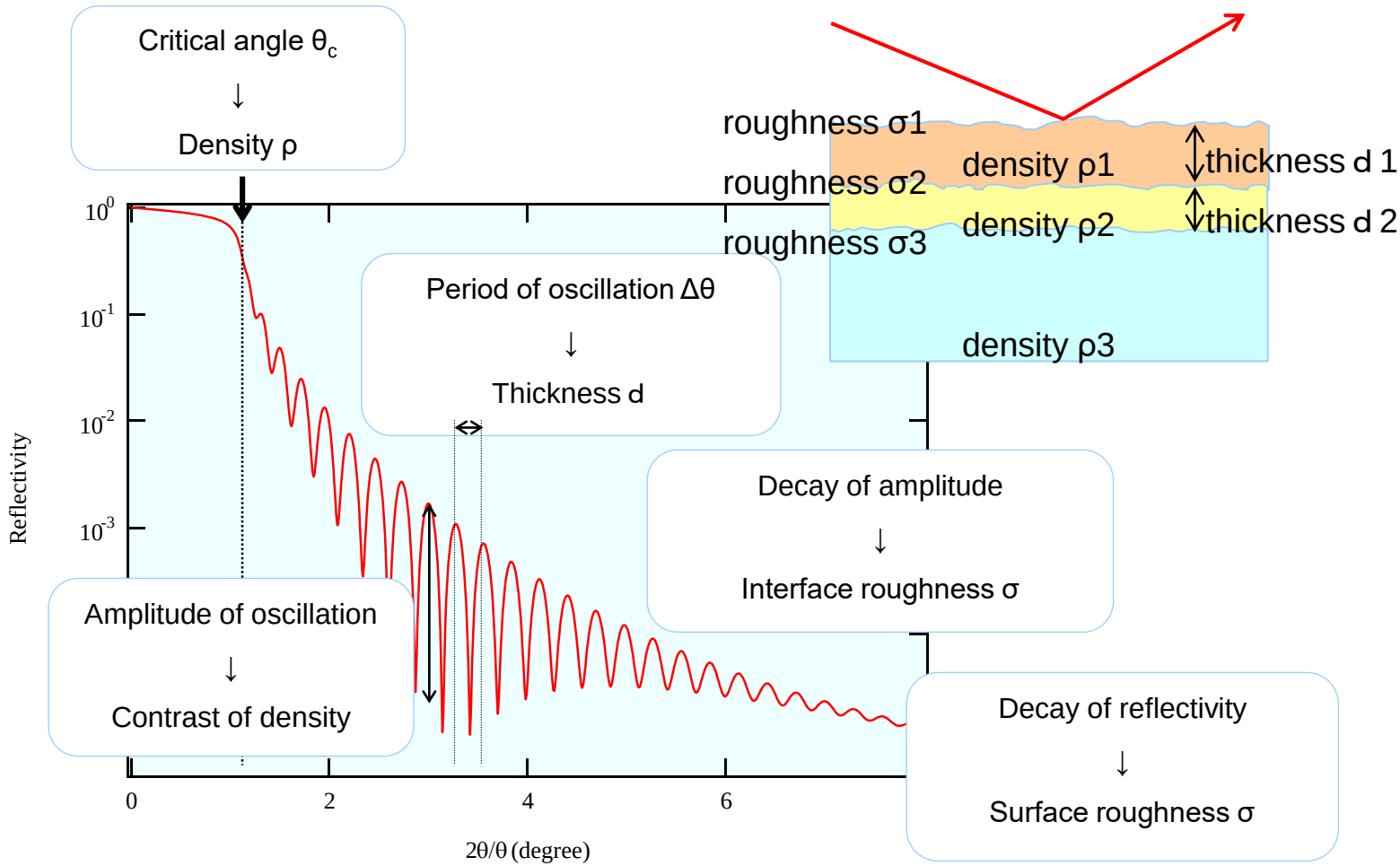
phase shift → interfere each other



Two beams reflected by the surface and the interface interfere each other.

Oscillation frequency $\Delta = 2d \cdot \sin\theta' + 2d\dfrac{1-n}{\sin\theta'}$

How to interpret the profile



XRR measurement

Optic used in reflectivity measurements

Monochromator	None	Ge (220) double-crystal	Ge (400) double-crystal	Ge (220) four-crystal
Relative intensity (Reference estimation)	200	10		1
Resolution (Angular divergence)	up to 0.04°	up to 0.02°		up to 0.003°
Wavelength monochromaticity	$K\alpha_1$+$K\alpha_2$ (+$K\beta$)	$K\alpha_1$	$K\alpha_1$	Part of $K\alpha_1$
Applicable film thickness	0.5 to 100nm	50 to 200nm	50 to 200nm	200nm or greater
RINT III series	○	○		
SmartLab	○	○	○	○

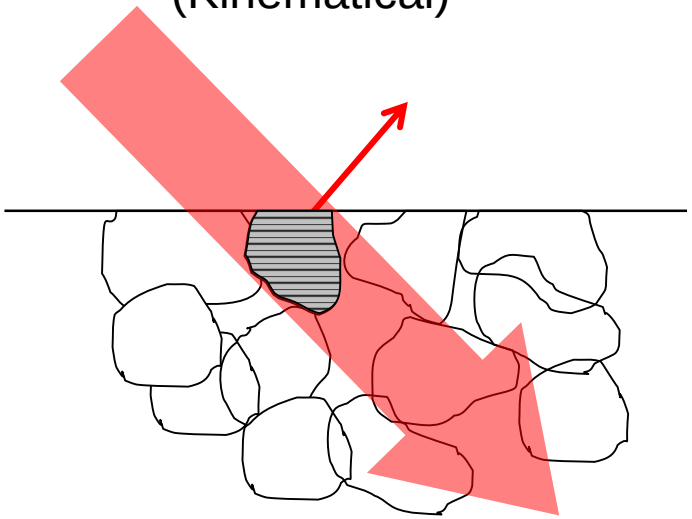
High-resolution X-ray diffraction (HRXRD)

Kinematical & Dynamical theory of diffraction

- Multiple diffraction occurs in large volume perfect crystal
- Multiple diffraction is considered in dynamical theory of diffraction

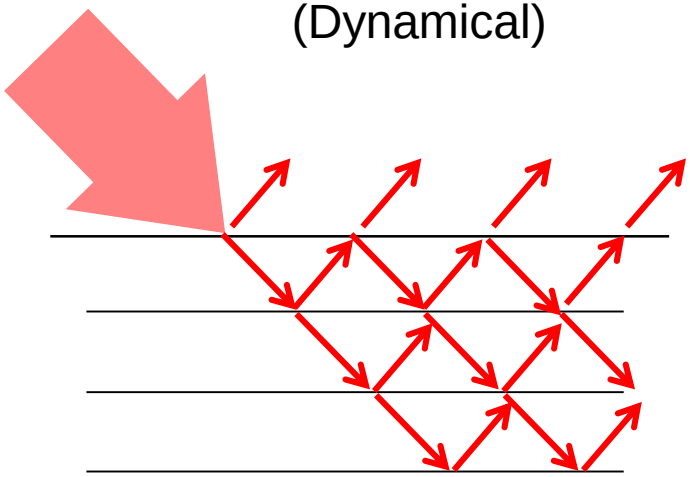
Single-scattering by mosaic crystal

(Kinematical)



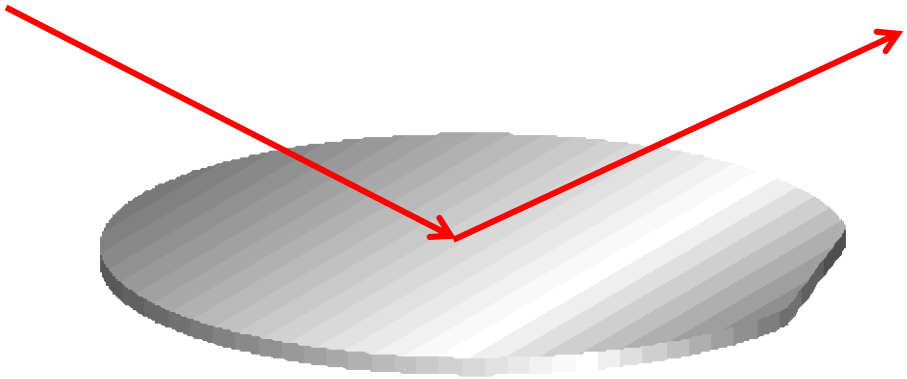
Multiple-scattering by single crystal

(Dynamical)



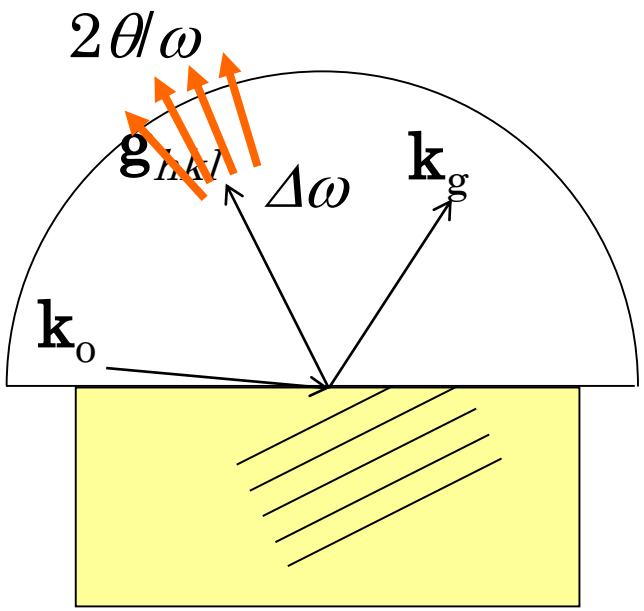
Dynamical theory is applied to …

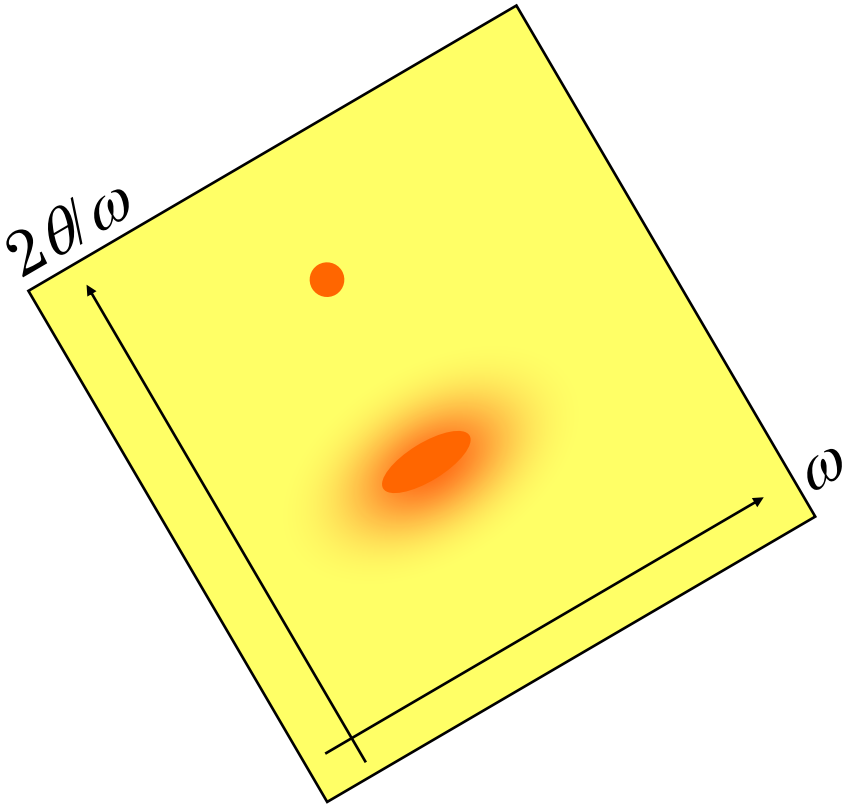
- Single crystal substrates like **Si**, **Ge**, **GaAs**, **GaN**, diamond and quarts, etc.
- Epitaxial films which is fabricated on those single crystal substrates.
  - Kinematical theory is also used when the film thickness is quite small.



Reciprocal space mapping

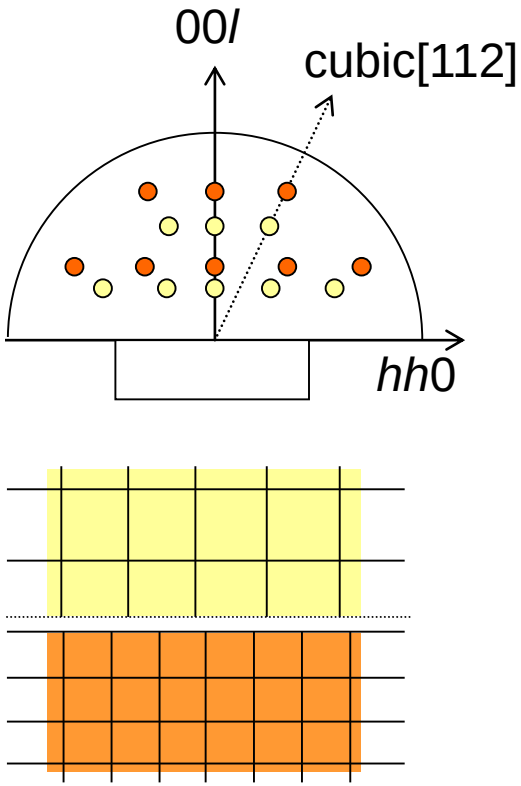
- Diffraction intensity distribution is plotted on reciprocal space



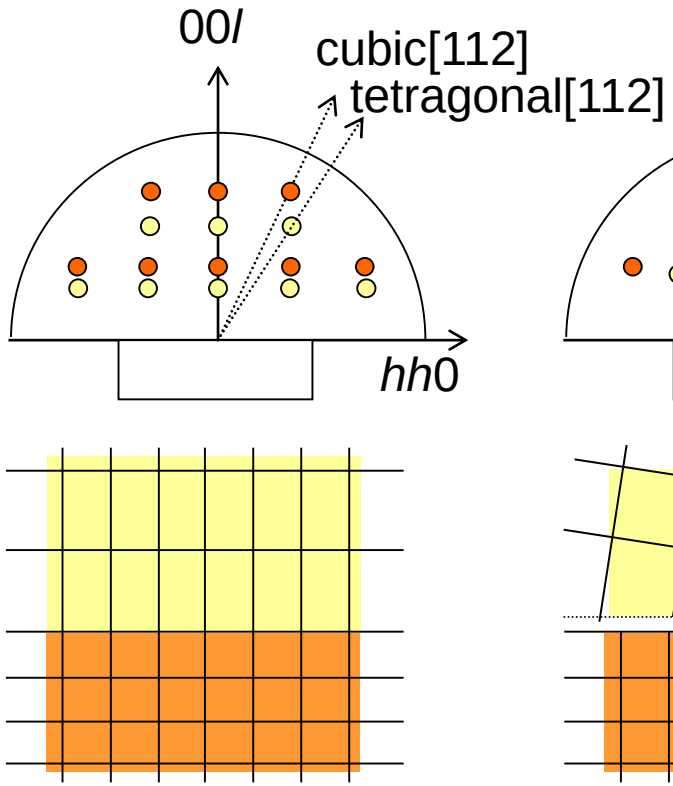


Epitaxial layer structures

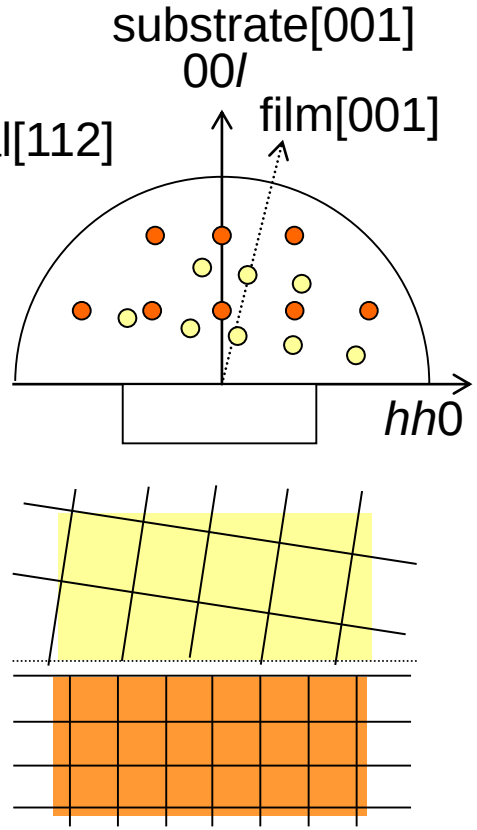
Relaxation



Strain



Misorientation

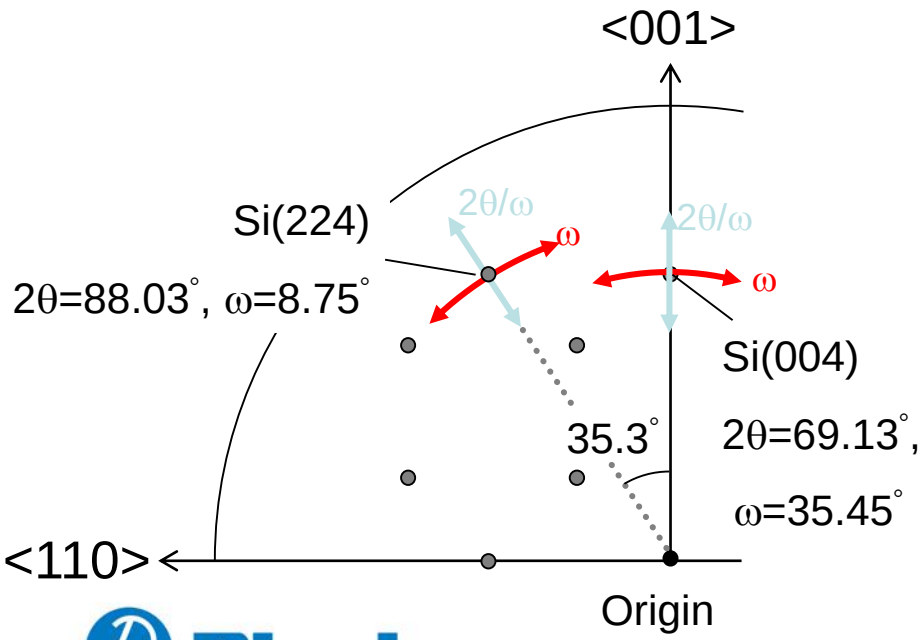


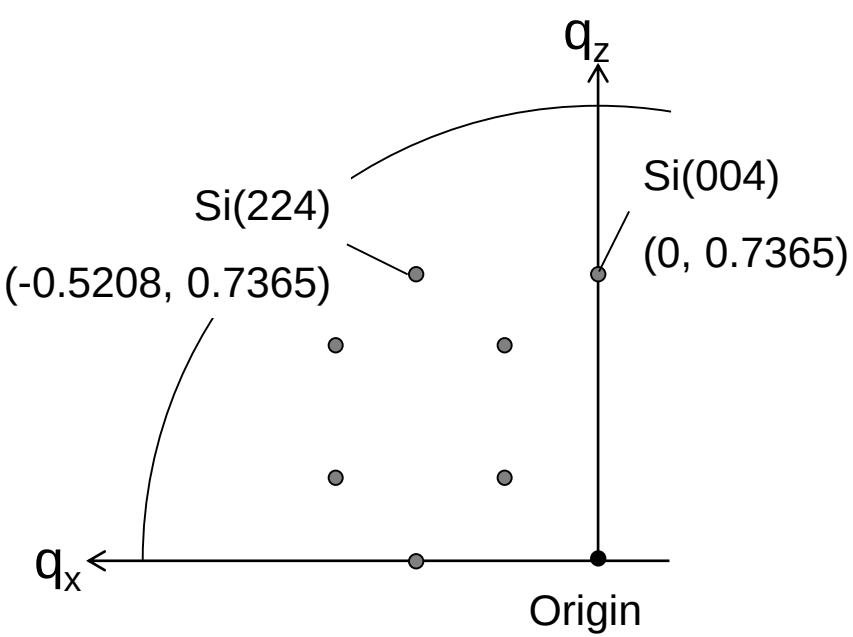
Gonio axes → Q coordinates

- Gonio coordinates (radial) → Q coordinates (orthogonal)

$$qx = \frac{2\pi}{\lambda}\sin\frac{(2\theta/\omega)}{2}\sin\left(\frac{(2\theta/\omega)}{2}-\omega\right)$$

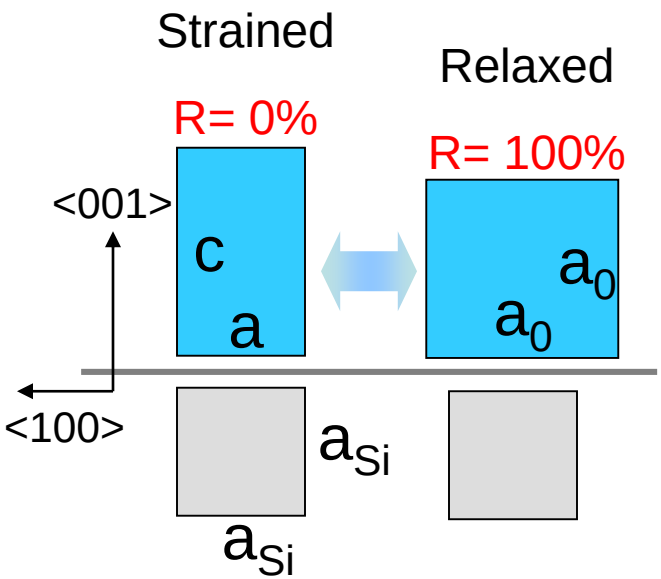
$$qz = \frac{2\pi}{\lambda}\sin\frac{(2\theta/\omega)}{2}\cos\left(\frac{(2\theta/\omega)}{2}-\omega\right)$$





Strain - Relaxation

ex. SiGe epitaxial film on a Si wafer



$$a_o = a + \frac{C_{11}}{C_{11} + 2C_{12}}(c - a)$$

$$a_0 = a_{Si}(1 - x) + a_{Ge}x$$: Vegard's law

$$C_{ij} = C_{ij,Si}(1 - x) + C_{ij,Ge}x$$

C_{ij} : elastic coefficients

→ Ge concentration *x*

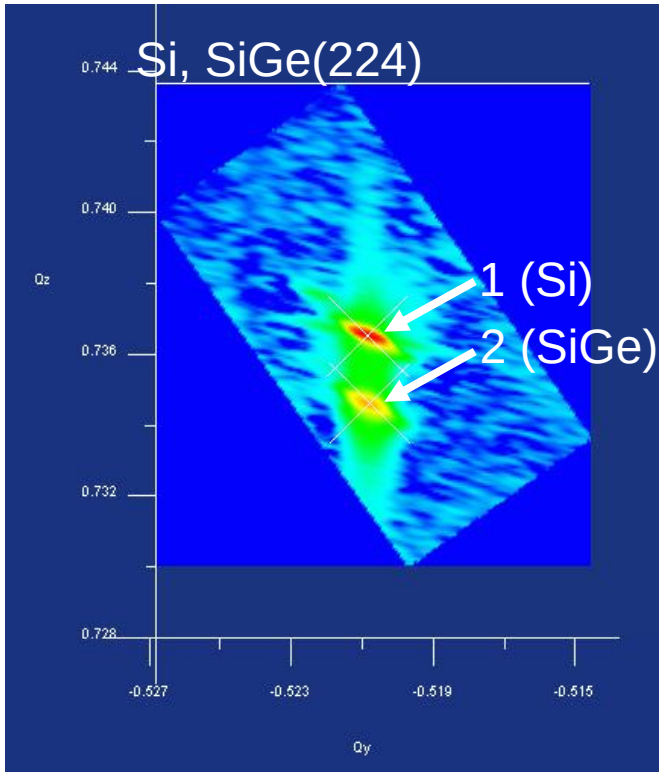
Degree of relaxation

$$R = \frac{a - a_{Si}}{a_0 - a_{Si}} \times 100(\%)$$

Calculation of lattice parameters

Designed structure : SiGe(Ge 3.8%, 400nm) / Si(100) substrate

Peak No.	X	Y	Intensity	Width(x)	Width(y)	d-value	Mismatch	Integrated Intensity
1	-0.5208	0.7365	187538.1834	0.0004	0.0001	1.1085	0.0000	16773.0267
2	-0.5208	0.7346	20051.6359	0.0005	0.0003	1.1105	0.0000	5404.9427



Si(224) (q_y, q_z)=(-0.5208,0.7365)

$$\frac{1}{d_{220}} = 0.5208 \Rightarrow d_{004} = \frac{1}{0.5208} = 1.920 \Rightarrow a,b = 1.920 \times \sqrt{2^2 + 2^2} = 5.431\ \text{Å}$$

$$\frac{1}{d_{004}} = 0.7365 \Rightarrow d_{004} = \frac{1}{0.7365} = 1.358 \Rightarrow c = 1.357 \times \sqrt{4^2} = 5.432\ \text{Å}$$

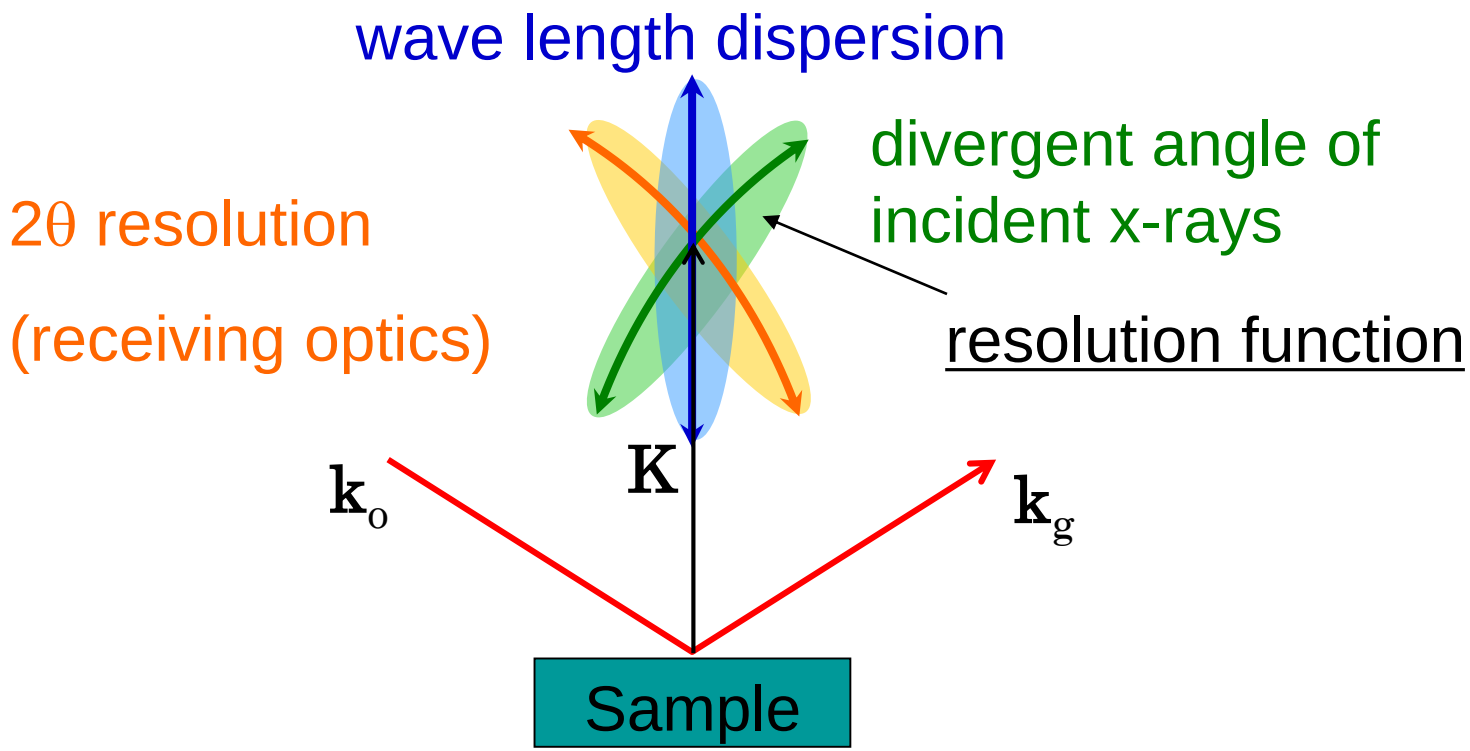
SiGe(224) (q_y, q_z)=(-0.5208, 0.7346)

$$\frac{1}{d_{220}} = 0.5208 \Rightarrow d_{004} = \frac{1}{0.5208} = 1.920 \Rightarrow a,b = 1.920 \times \sqrt{2^2 + 2^2} = 5.431\ \text{Å}$$

$$\frac{1}{d_{004}} = 0.7346 \Rightarrow d_{004} = \frac{1}{0.7346} = 1.361 \Rightarrow c = 1.361 \times \sqrt{4^2} = 5.444\ \text{Å}$$

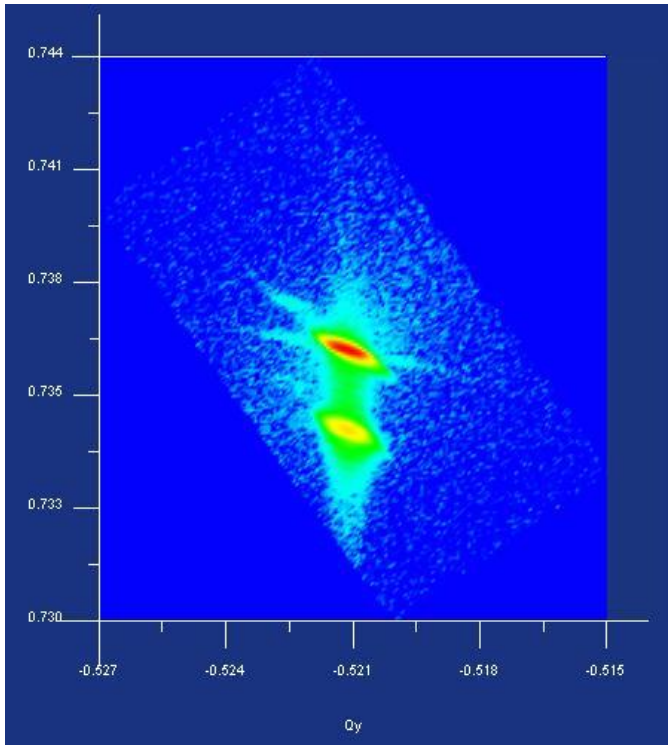
Optical resolution in reciprocal space

Think about the resolution function!

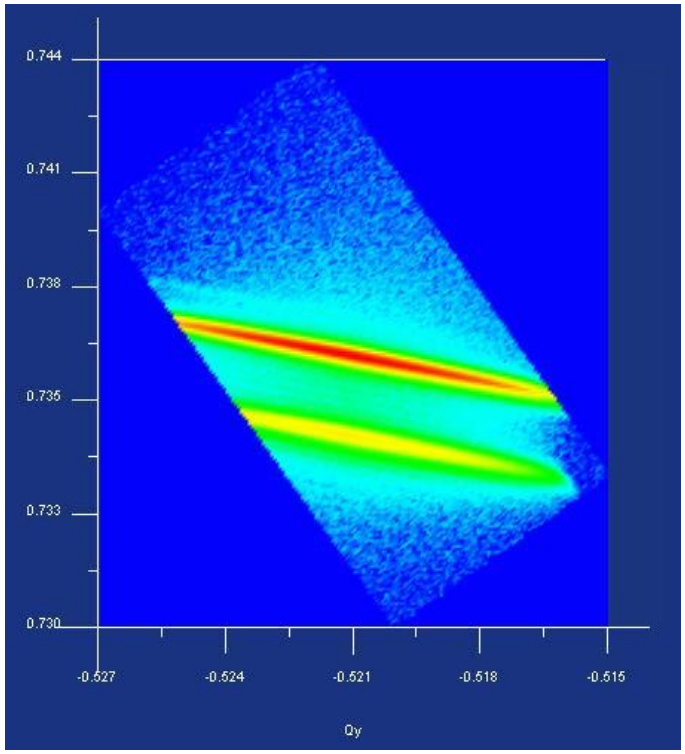


When the resolution is not appropriate to the crystal perfection…

ex. SiGe/Si (224)



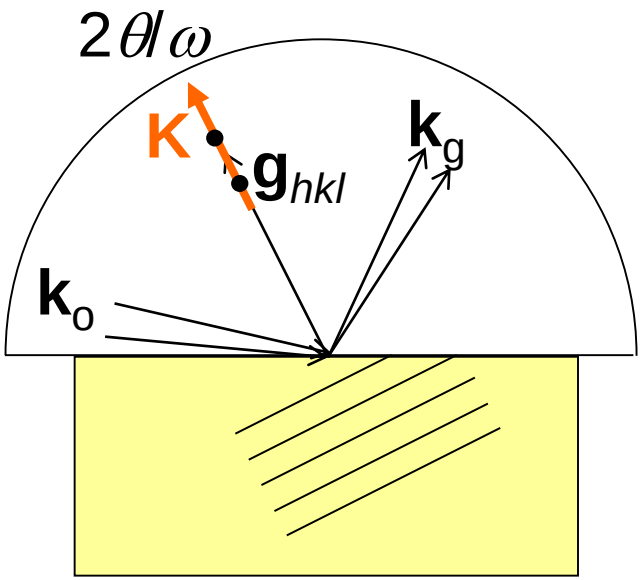
RS slit =0.1mm

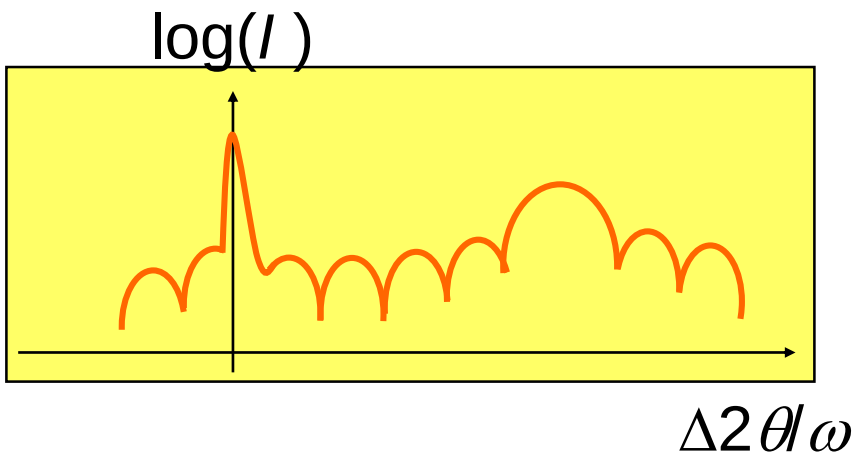


RS slit =1mm

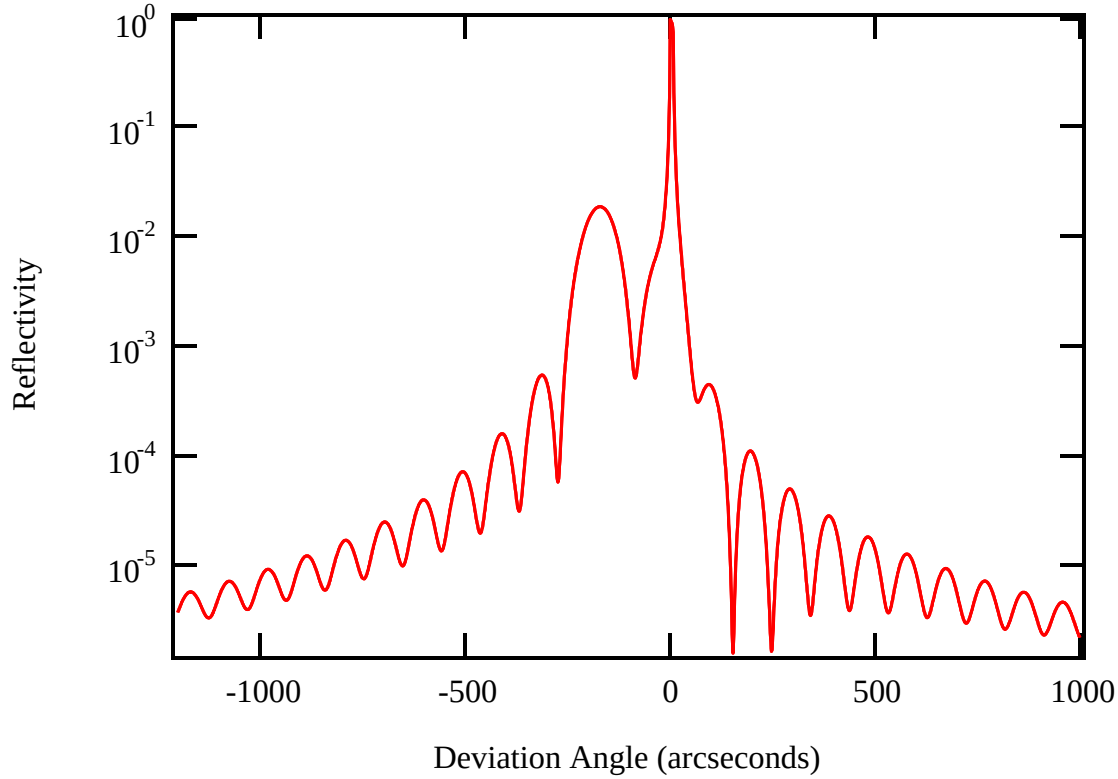
High-resolution rocking curve

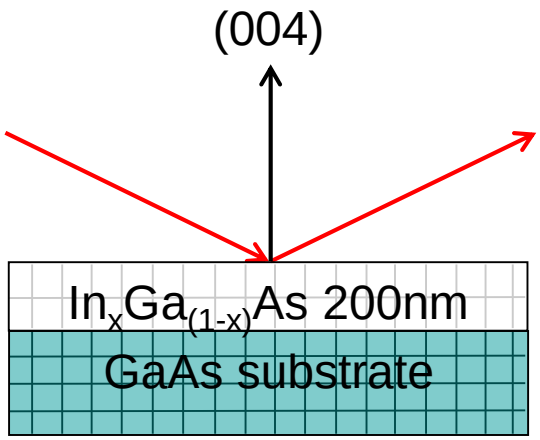
- The differences of lattice spacing between the substrate and epitaxial films are observed
  - Thickness and composition ratio of epitaxial films (when the degree of relaxation is known)





Rocking curve profile of an epitaxial sample



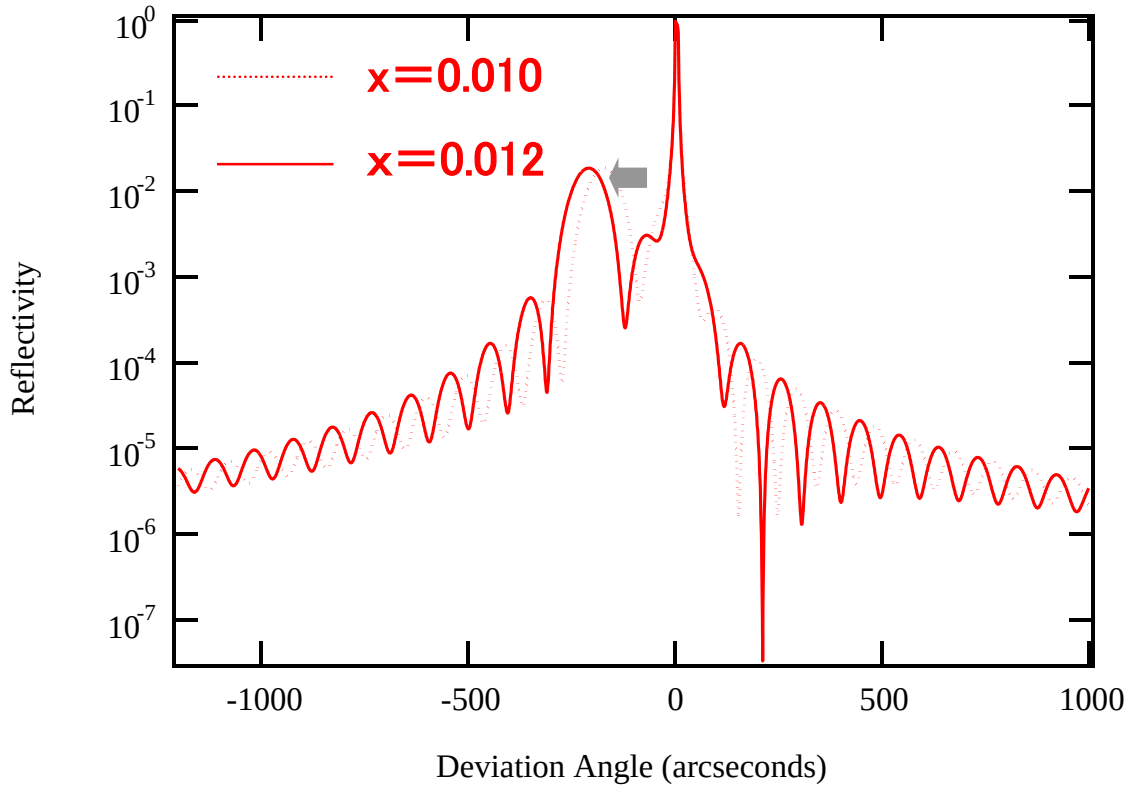


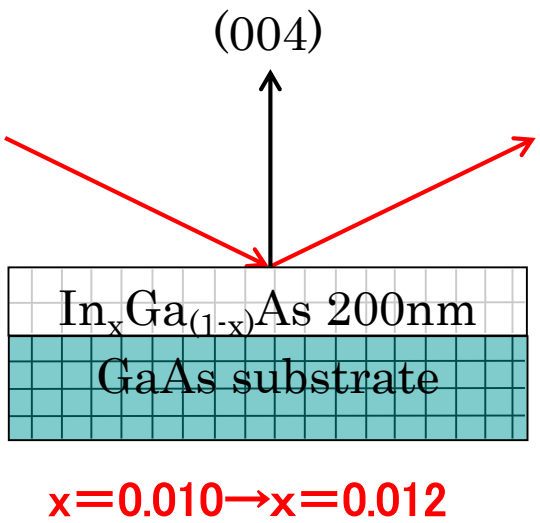
x=0.010

Relax : 0%

When d-spacing changes…

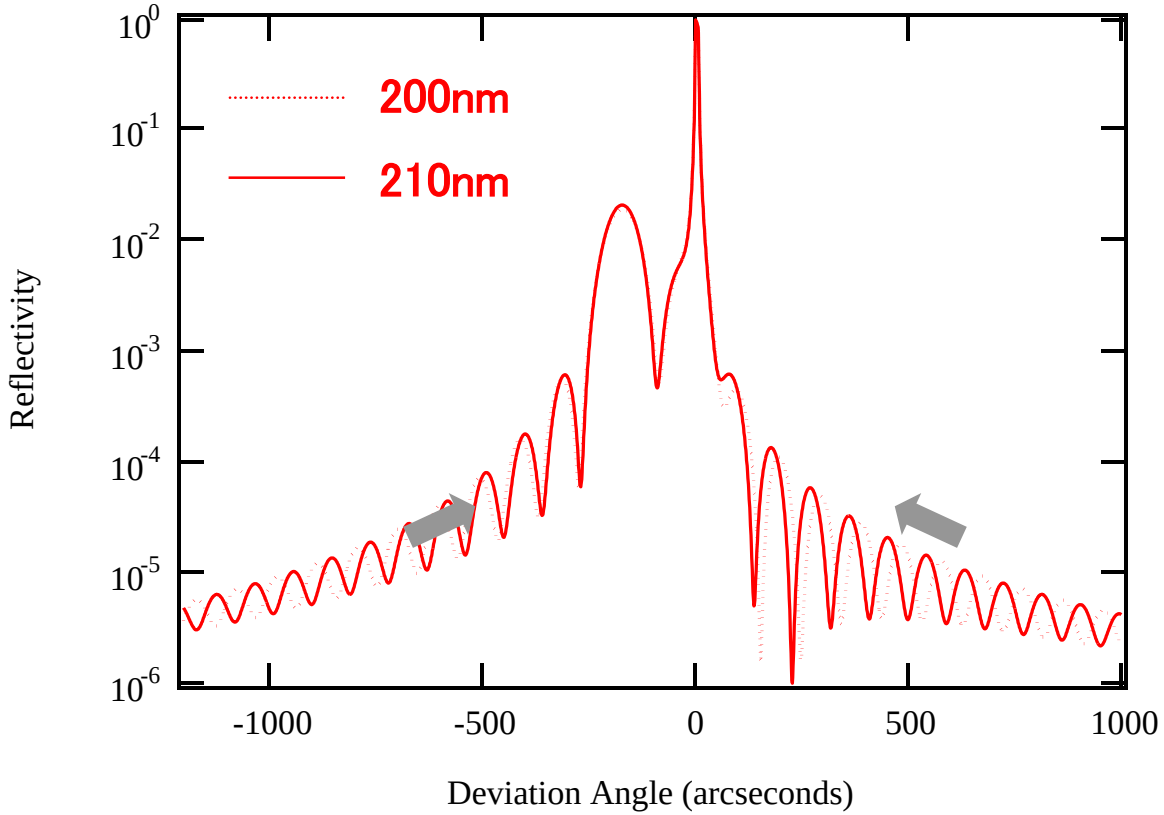
- Peak position difference between the substrate and the films change

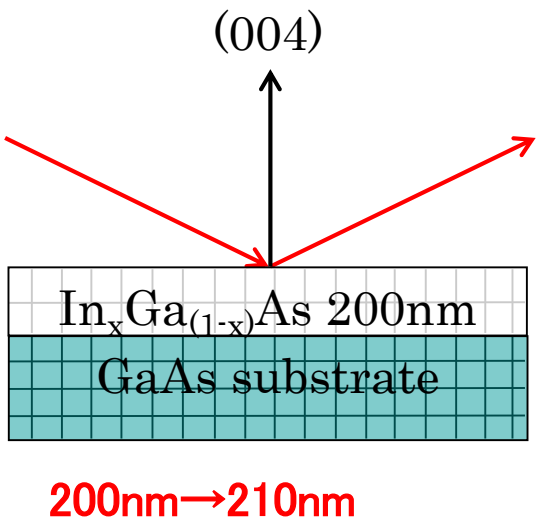




When film thickness changes…

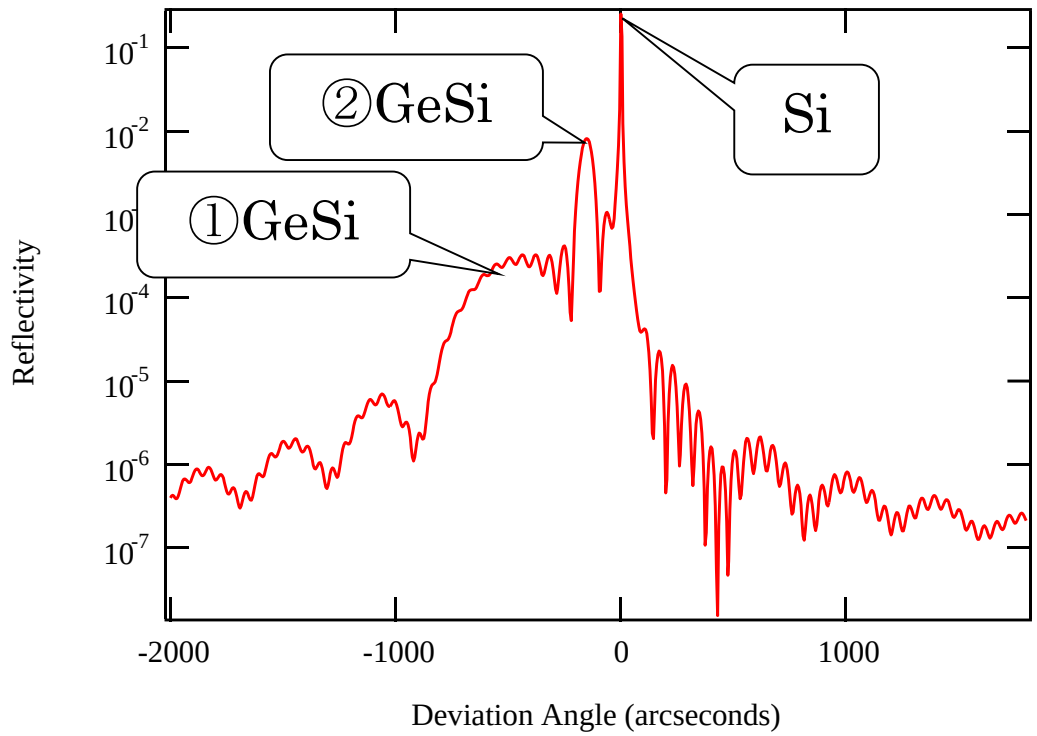
- The period of oscillation changes

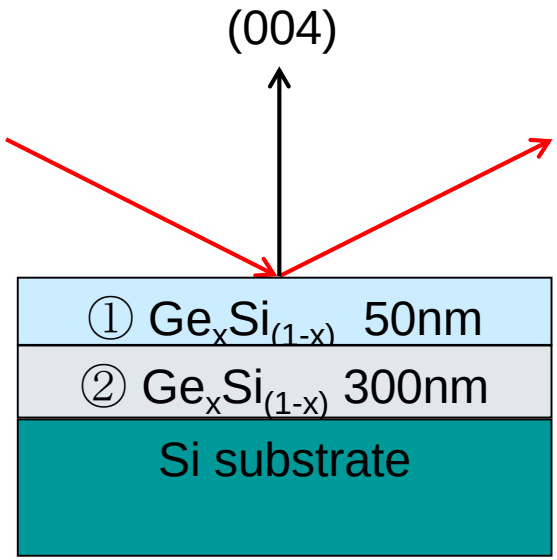




When the sample has multilayer structure…

- Complicated oscillation composed of oscillation from each layer is observed

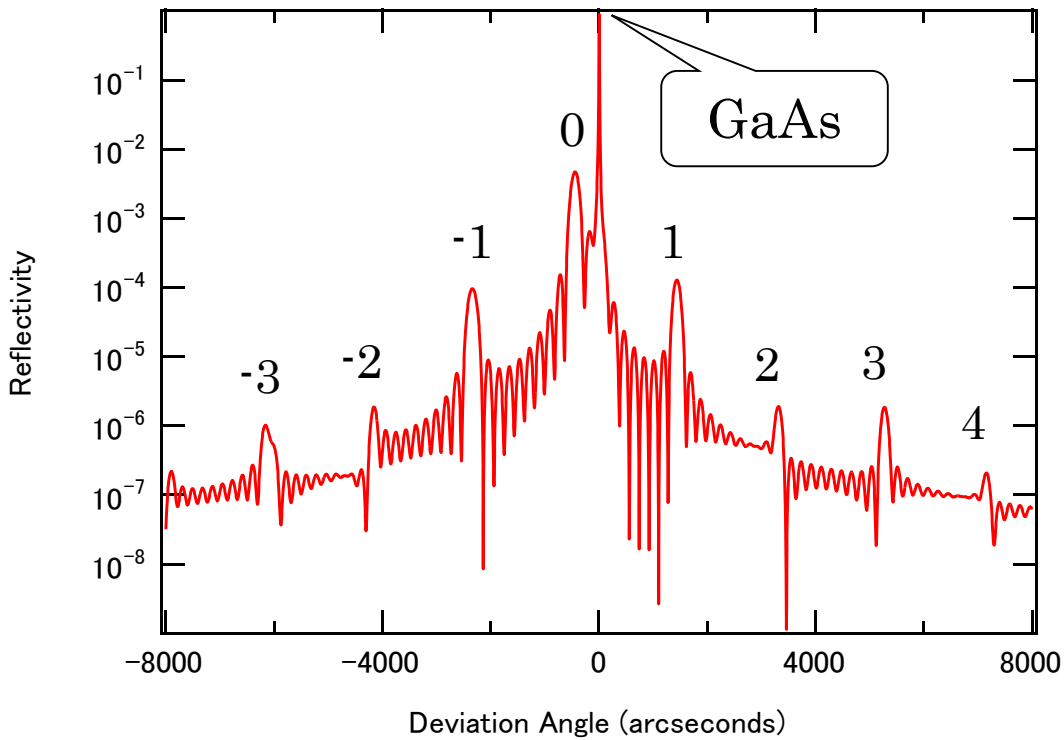


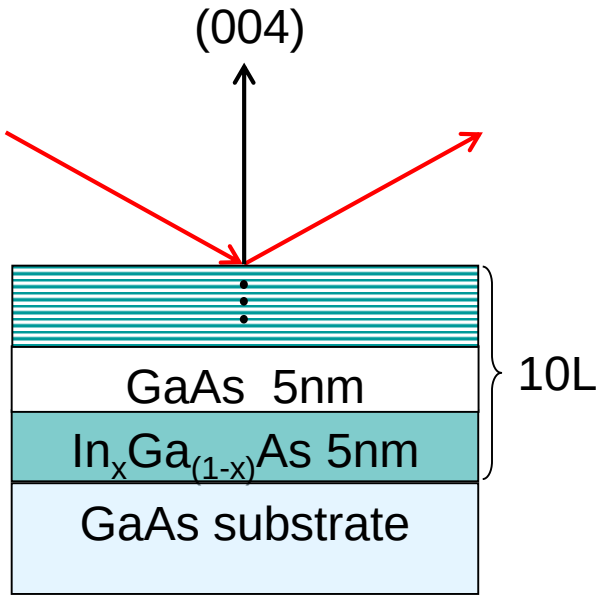


①x=0.050 ②x=0.015

When the sample has superlattice structure…

- Satellite peaks are observed





x=0.200

How to interpret the profile