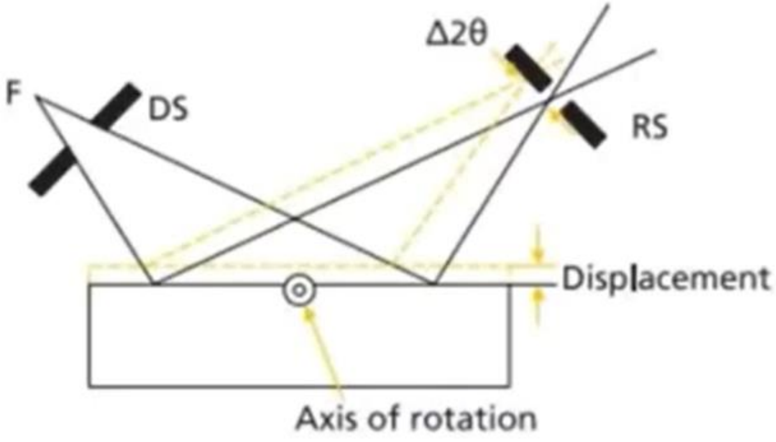
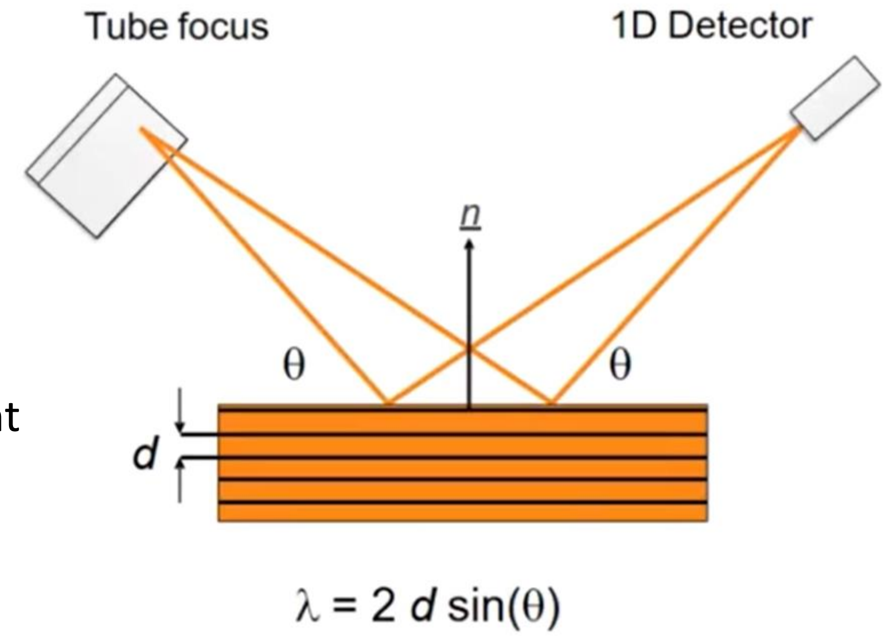
Diffraction from thin films

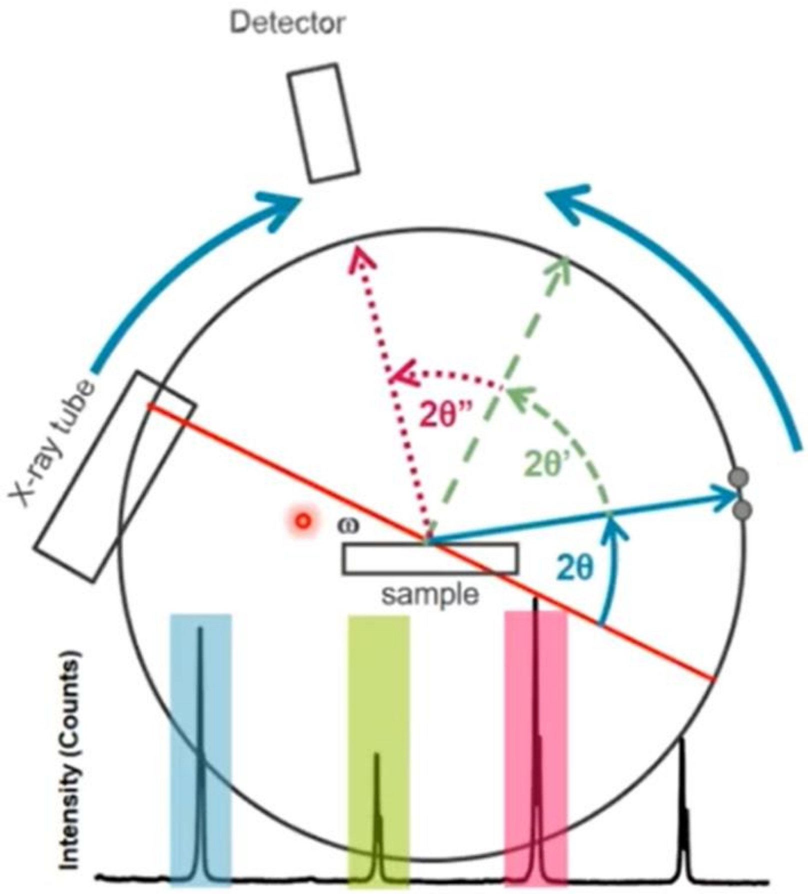
- Very tiny volume of material confined at nanoscopic dimension
- Conventional diffraction geometries limited or even fail
- Set–up more appropriate scattering geometries
- Special care in sample alignment



Bragg-Brentano geometry
Very sensitive to sample displacement

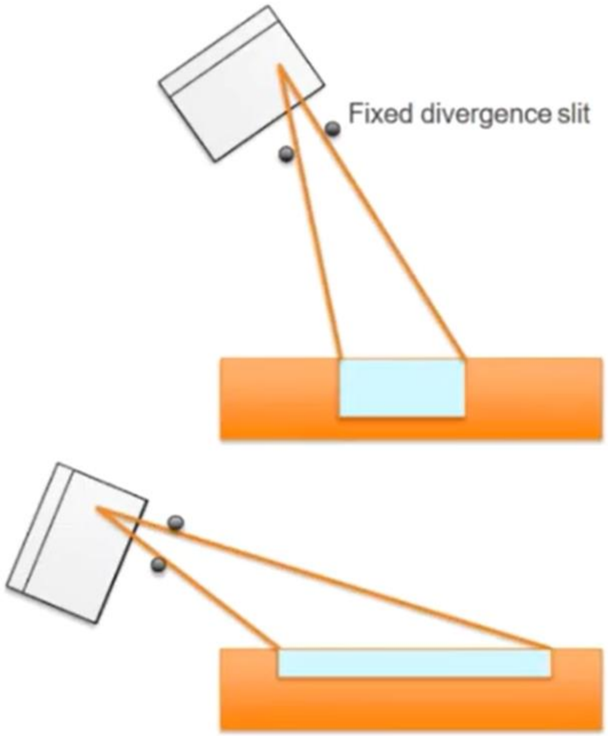


Diffraction from thin films

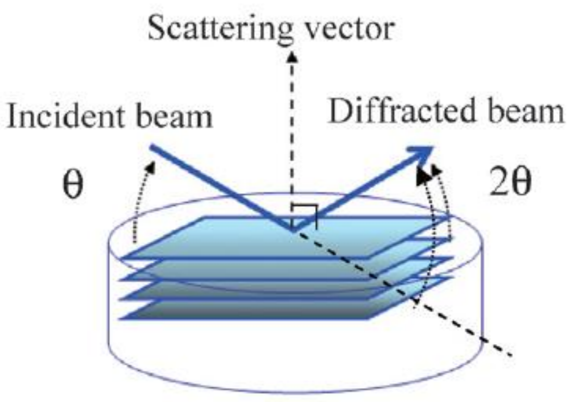


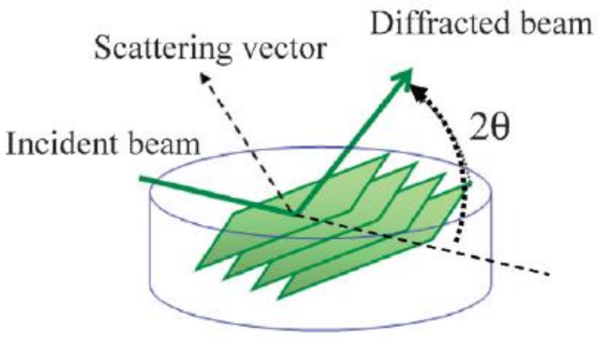
- The divergence slit defines the irradiated length on the sample
- As the incident angle increases:
  - The irradiated length reduces as 1/sinθ
  - The irradiated depth increases as sinθ

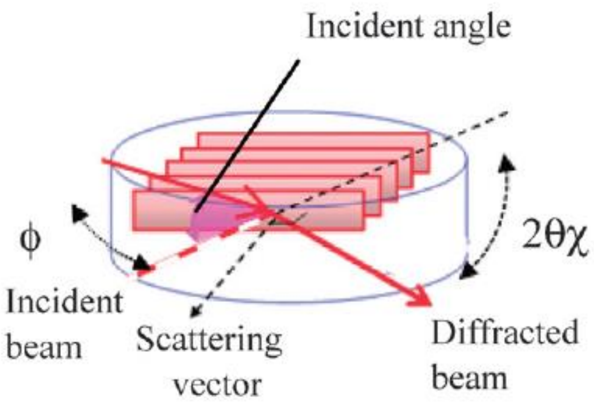
The penetration depth can be larger than the fim thickness



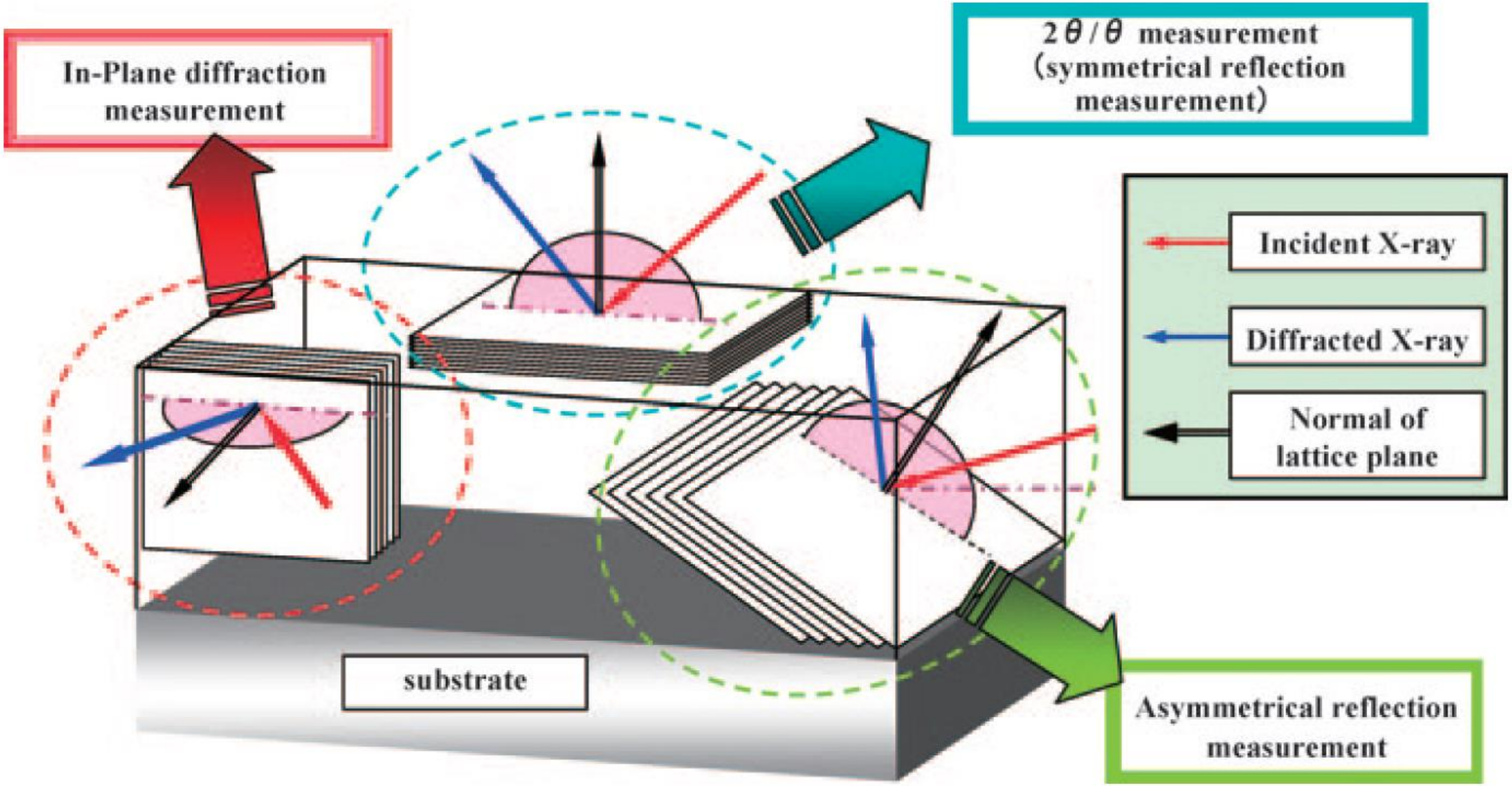
Scan geometries for thin films

Out-of-plane XRD measurement **Symmetrical reflection** **-2θ / θ scan-**	

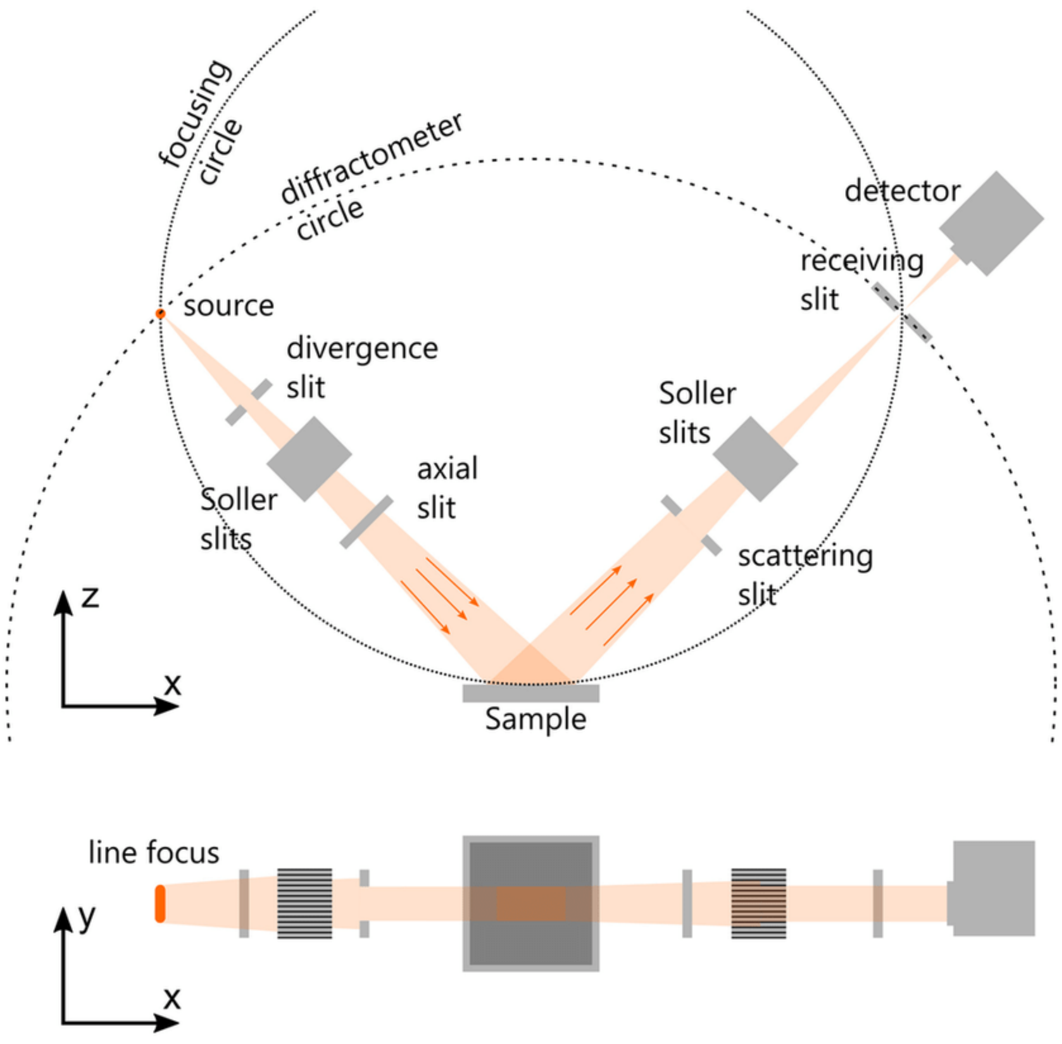
Out-of-plane XRD measurement **Asymmetrical reflection (Thin film method)** **-2θ scan-**	

In-plane XRD measurement **-2θχ / ϕ scan-**	

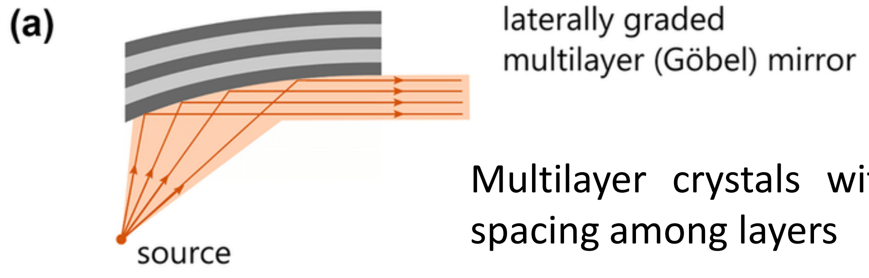
Scan geometries for thin films



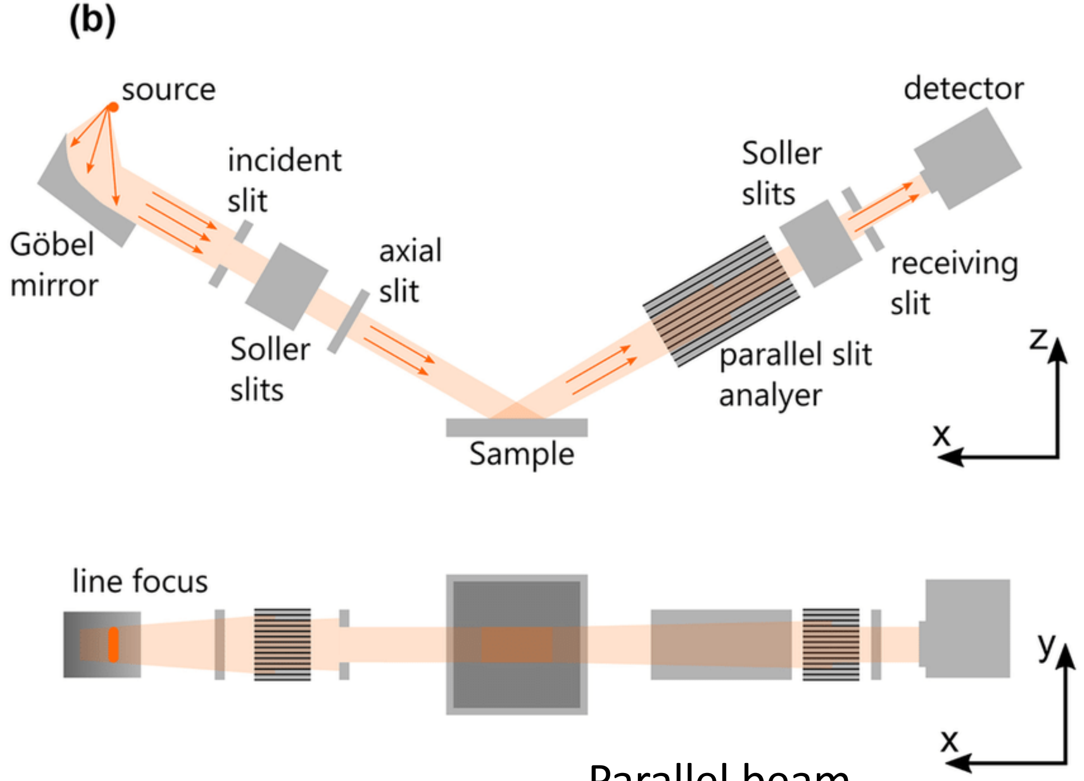
X-ray beam



Bragg-Brentano



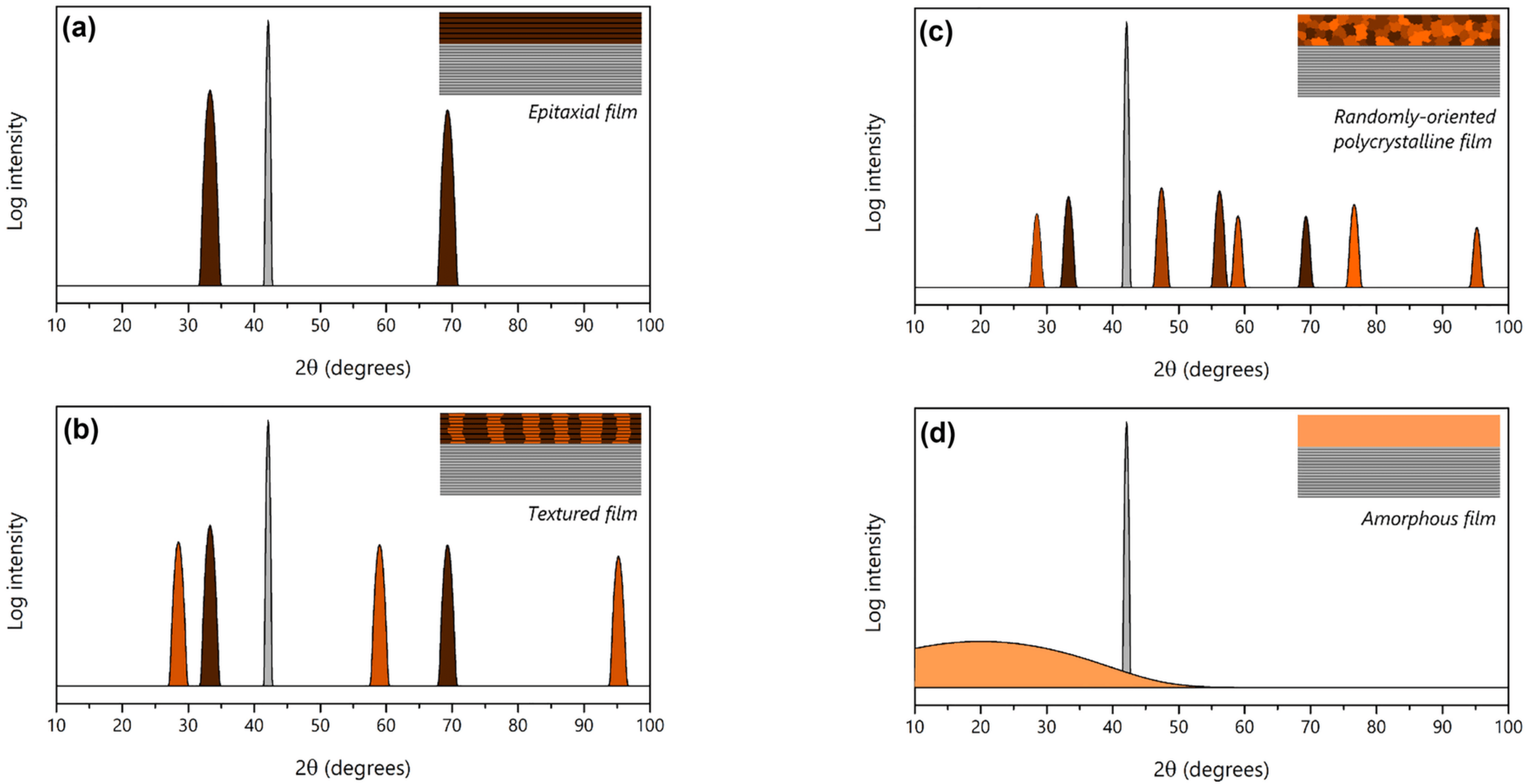
Multilayer crystals with controlled spacing among layers



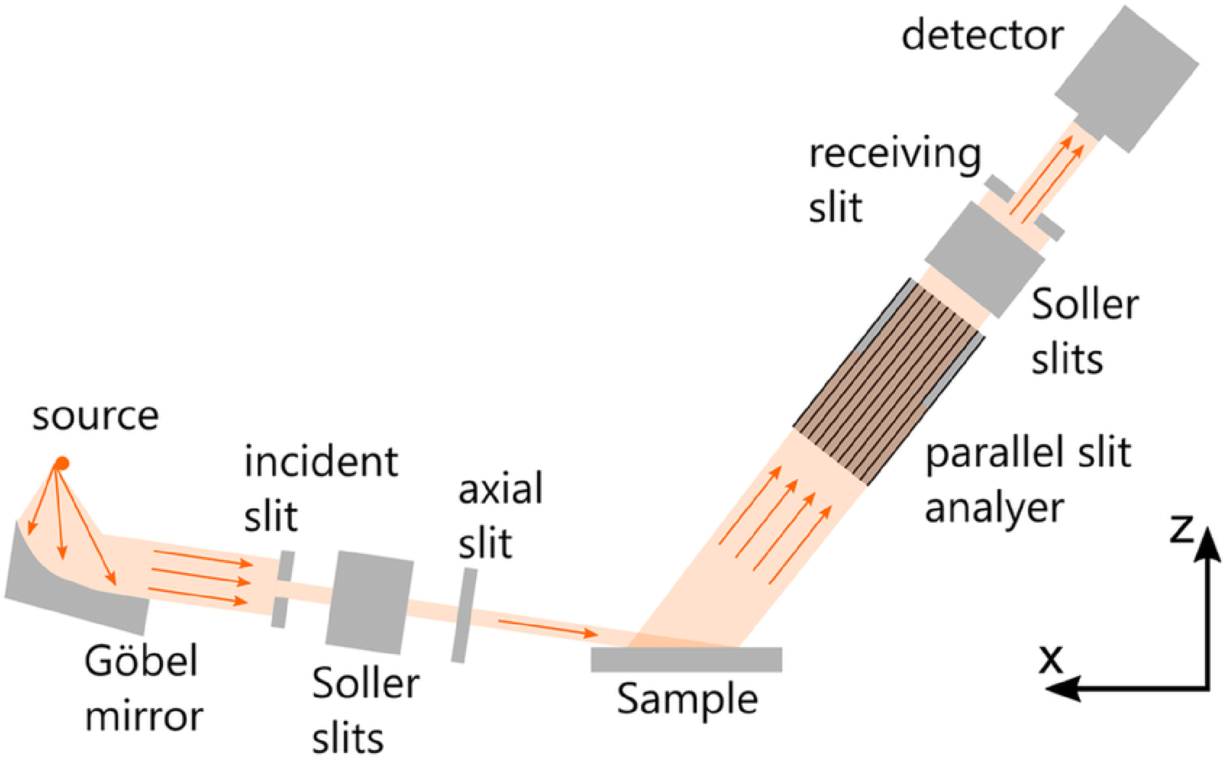
Parallel beam

Parabolically bent: a divergent beam striking the mirror at different locations and angles yields a diffracted parallel beam. In each point of the segment, Bragg's law is fulfilled for the angle of incidence.

Thin films


(a)
Epitaxial film
(b)
Textured film
(c)
Randomly-oriented polycrystalline film
(d)
Amorphous film
Log intensity
2θ (degrees)
10 20 30 40 50 60 70 80 90 100

Grazing incidence diffraction GID



Benefits of grazing incidence geometry:

- Confines X-ray beam within a given depth (depth resolution information)
- Emphasized diffraction signal from thin coatings

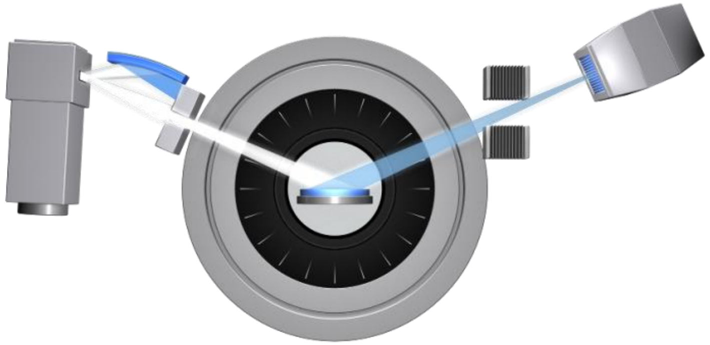
GID is suited for samples without a preferential out-of-plane orientation

For highly-oriented samples, peaks will often not be observed,

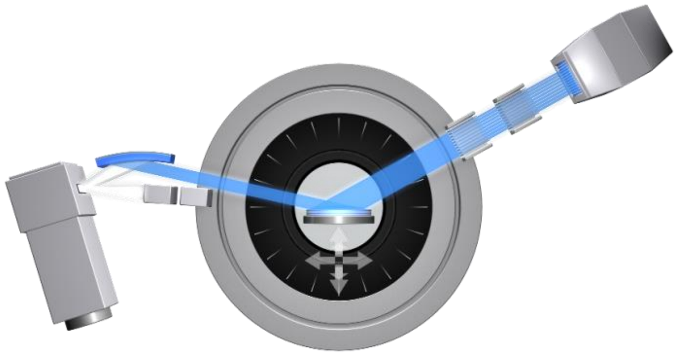
Fixed incident angle

Bragg conditions collected by moving detector only (2θ scan)

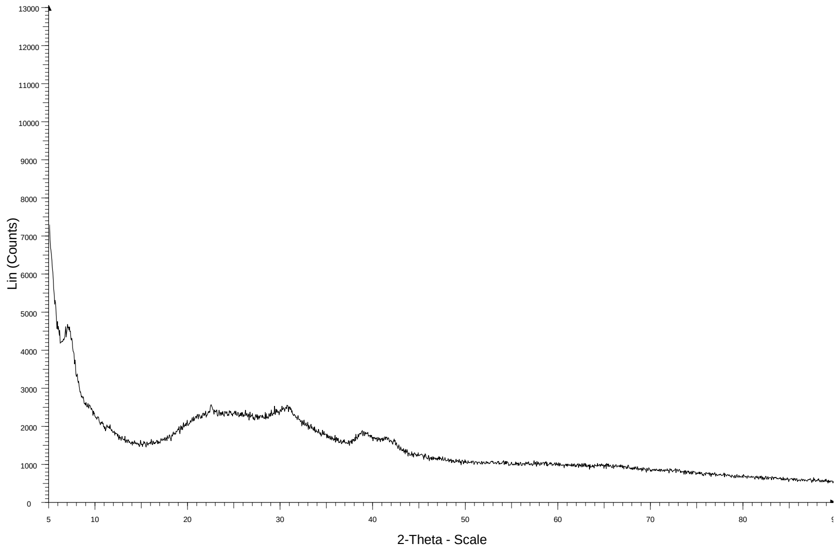
Bragg-Brentano or GID?

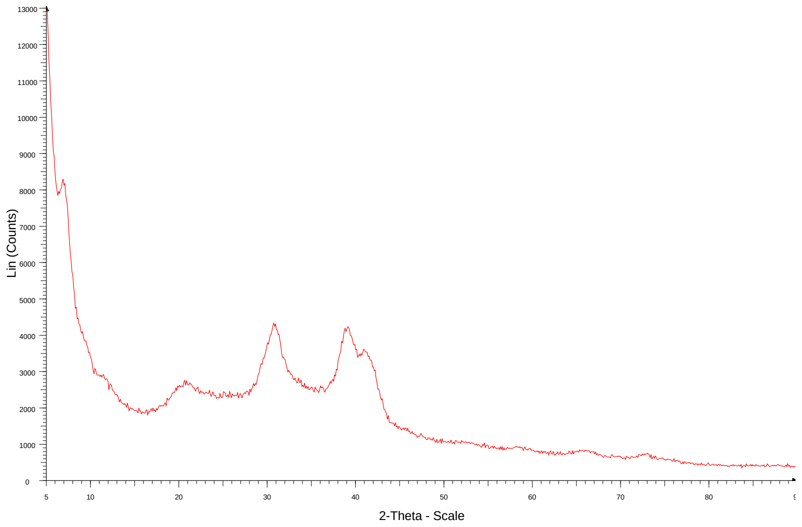


Bragg-Brentano geometry



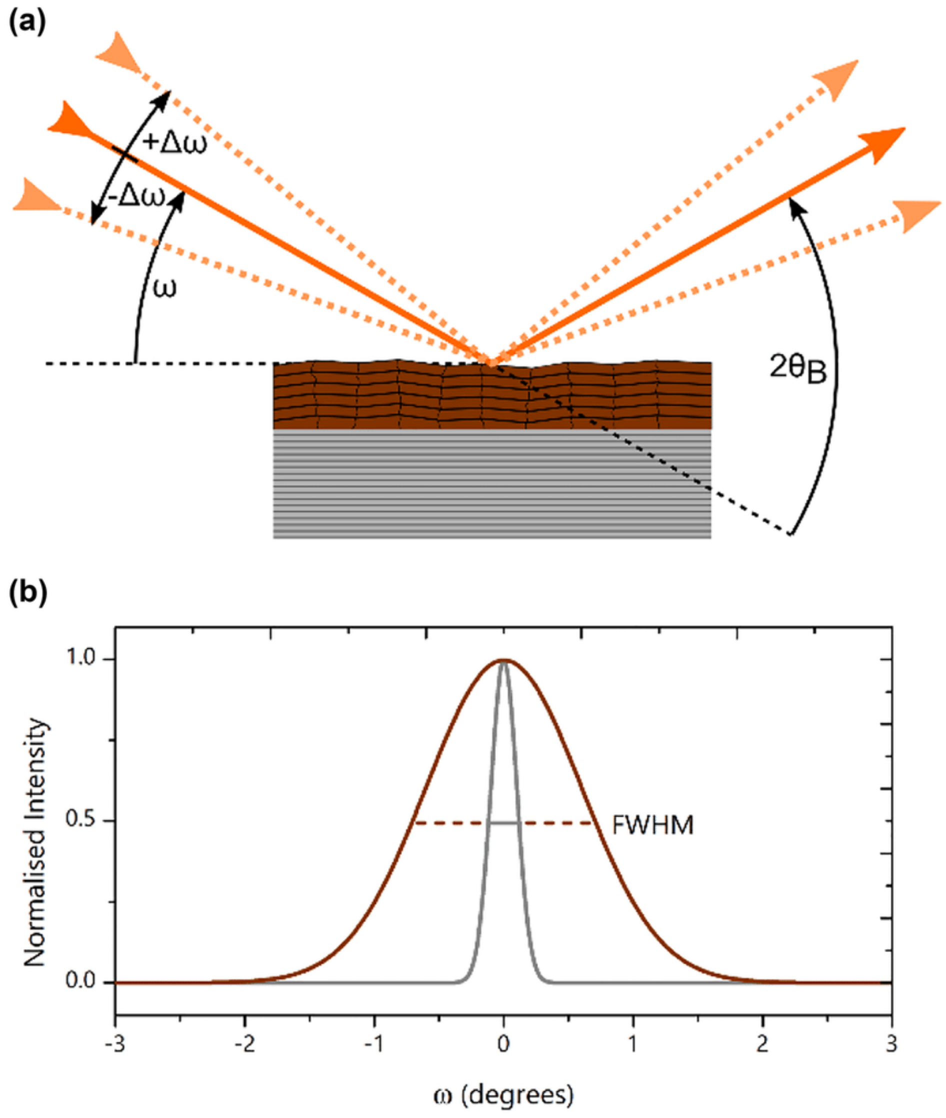
Grazing incidence geometry





GID emphasizes the signal of the Ag_2Te nanocrystallites and the glass substrate signal is reduced

Rocking curve



The width of the rocking curve depends upon the mosaic spread of the grains, density of dislocations, and substrate curvature, which disrupt the parallel nature of the lattice planes.

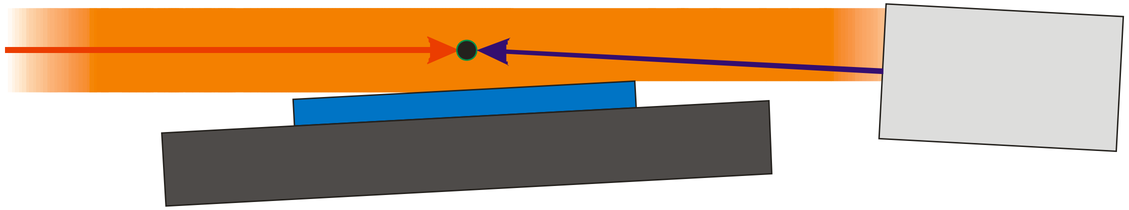
The full-width half-maximum (FWHM) of the rocking curve is generally used as an indication of preferential orientation.

A randomly oriented film would show a constant intensity independent of the ω angle simply because there is the same probability of finding crystallites in any direction.

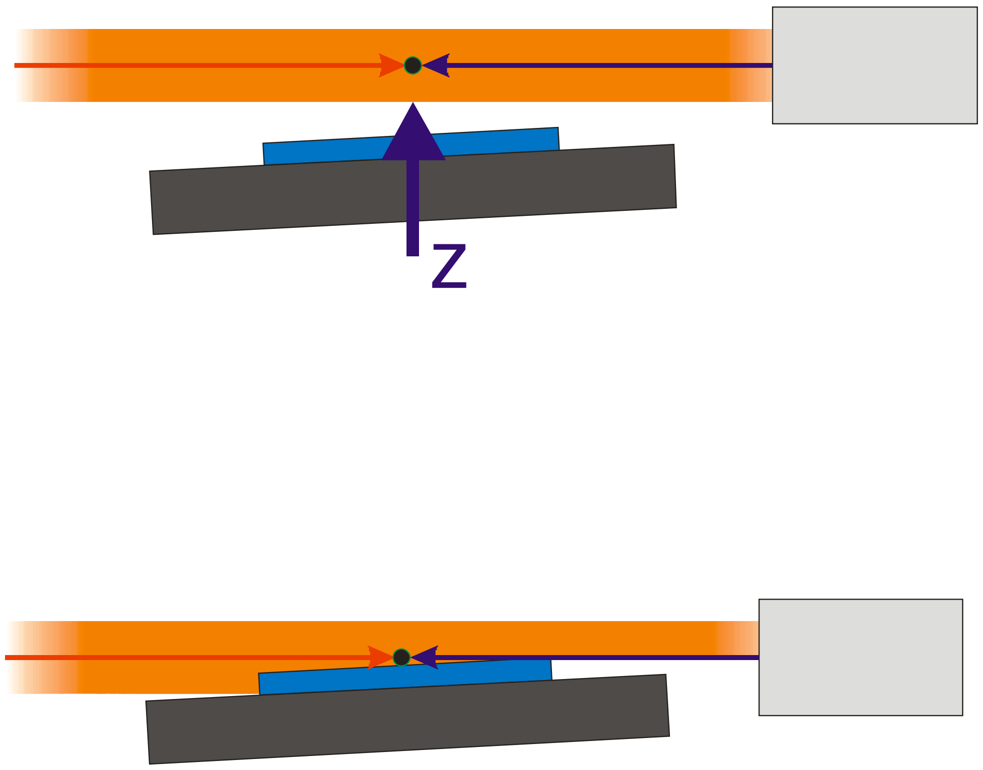
Sample alignment

Ideal sample alignment

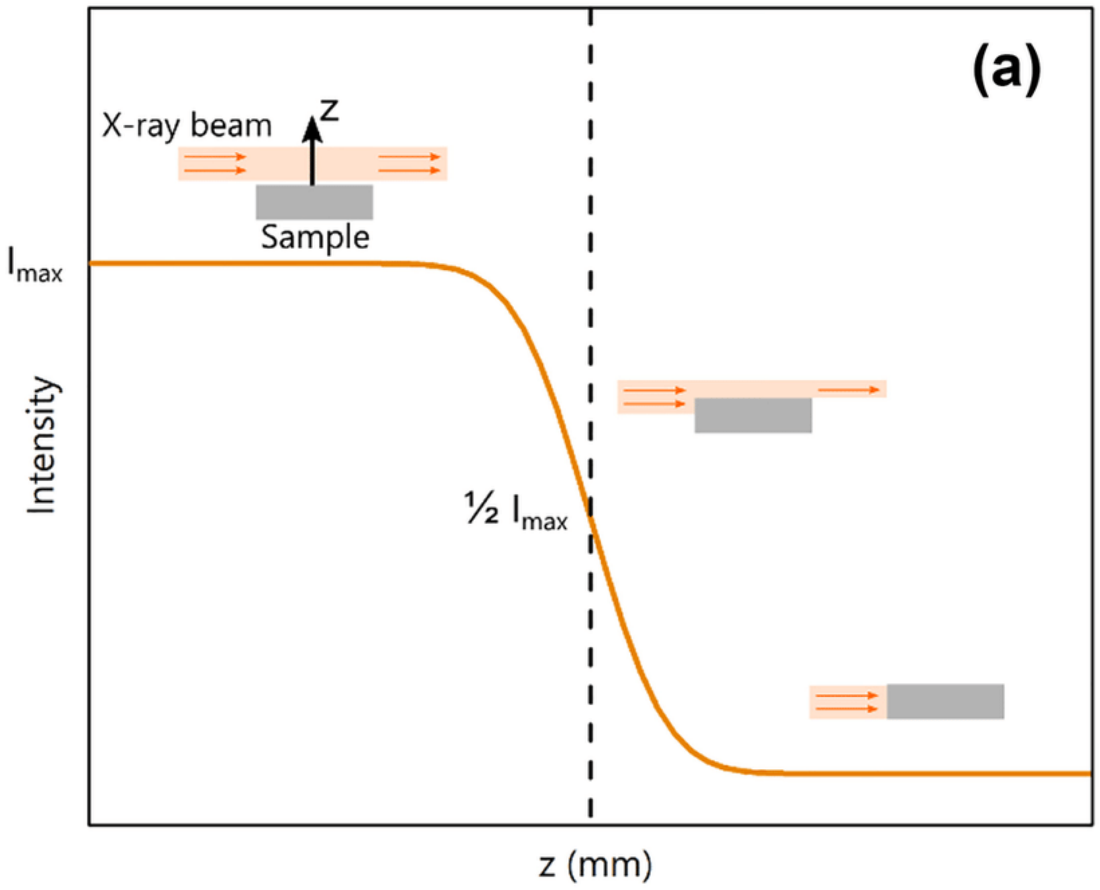
Situation after sample mounting



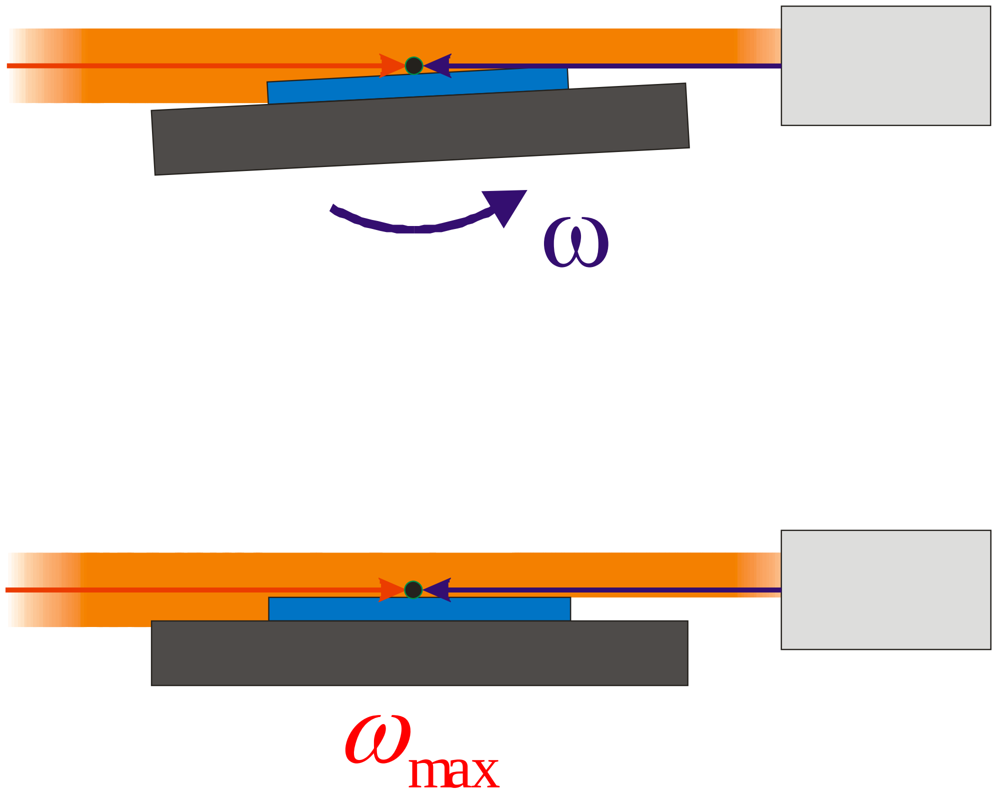
Sample alignment Z-scan

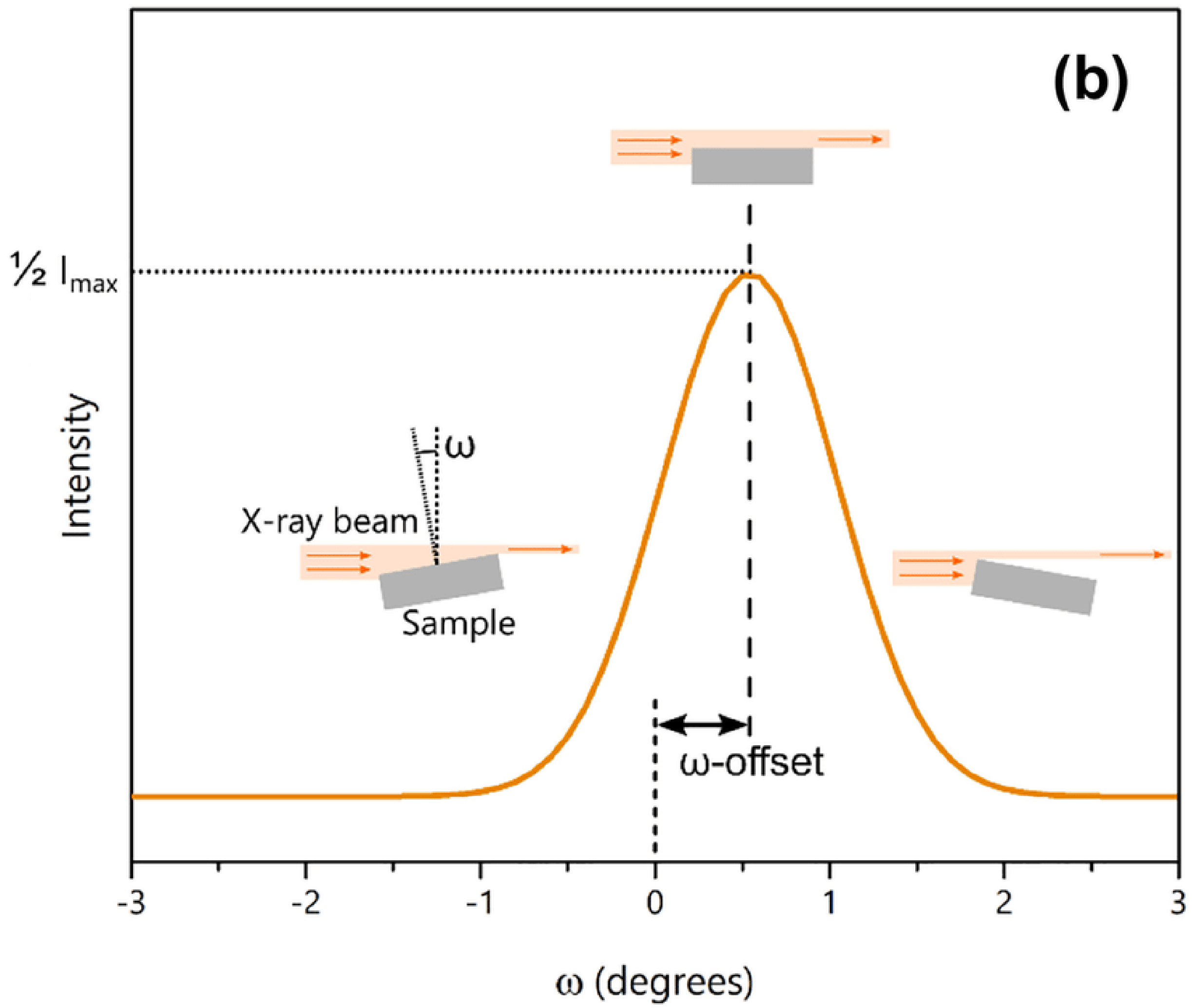


$$I(z_{1/2})=I_0/2$$



Sample alignment ω-scan





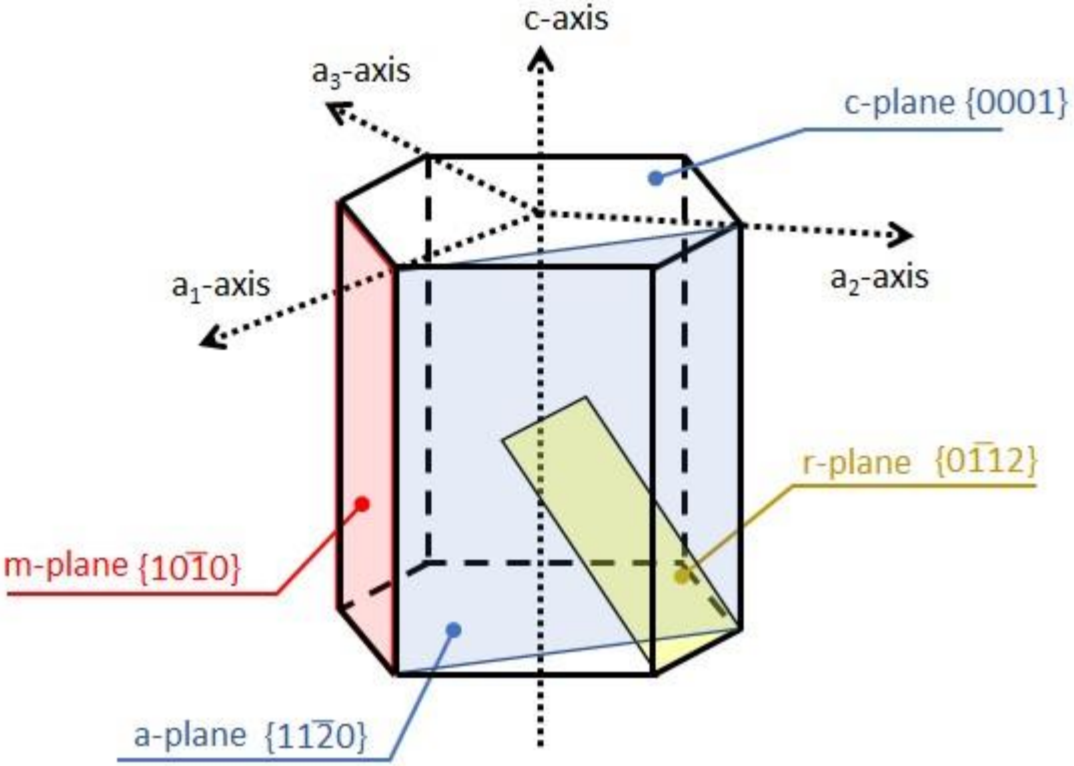
Experiment

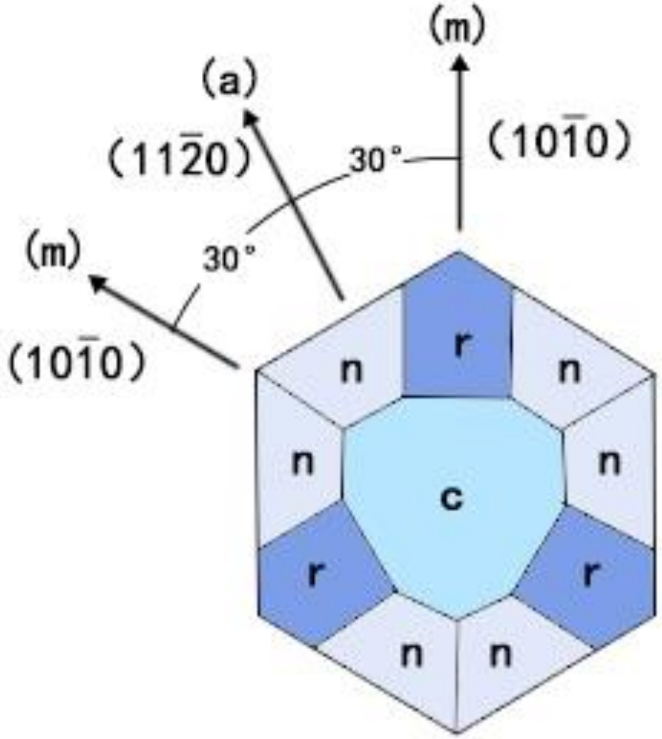
XRD on thin films (Sapphire Al_2O_3)

https://www.sapphire.lt/sapphire/

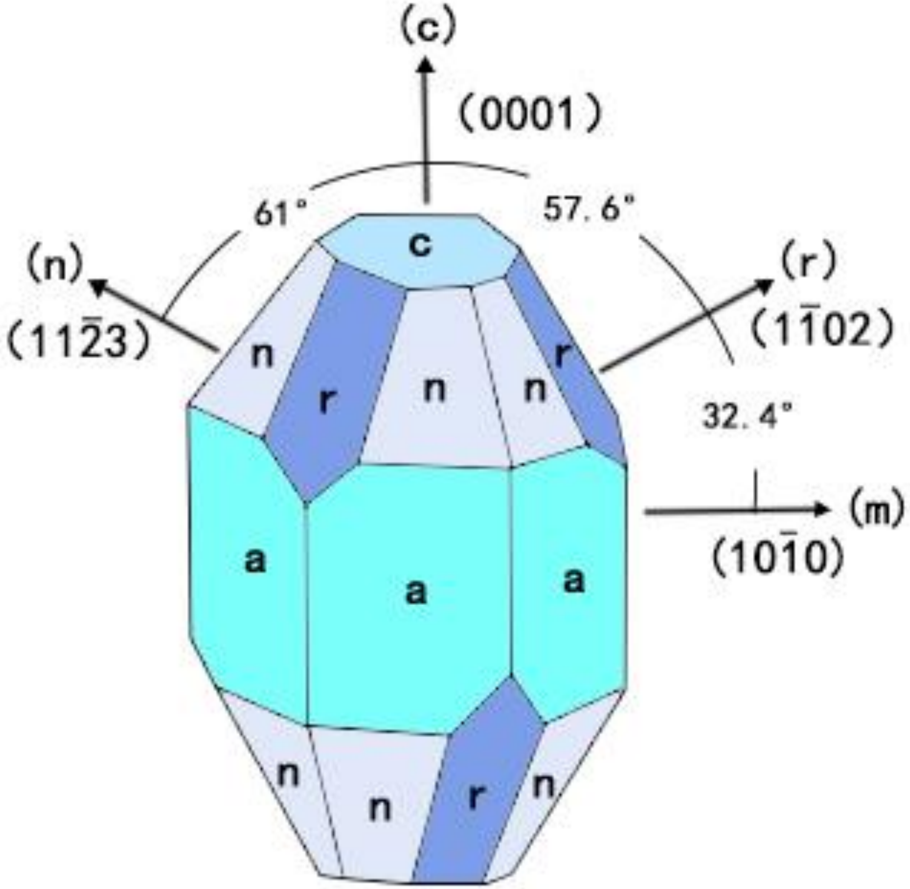
Determination of the crystal structure and possible preferred orientation (A, C, N, R)

https://ssd.phys.strath.ac.uk/resources/crystallography/crystallographic-direction-calculator/


c-axis
a_3-axis
c-plane {0001}
a_1-axis
a_2-axis
r-plane {$01\bar{1}2$}
m-plane {$10\bar{1}0$}
a-plane {$11\bar{2}0$}



(m)
(a)
(11$\bar{2}$0)
30°
(10$\bar{1}$0)
(m)
30°
(10$\bar{1}$0)
r
n
n
n
n
c
r
r
n
n



(c)
(0001)
61°
57.6°
(n)
c
(r)
(11$\bar{2}$3)
(1$\bar{1}$02)
n
r
n
r
n
32.4°
(m)
(10$\bar{1}$0)
a
a
a
n
n
r
n