

Philips X'pert XRD User Training

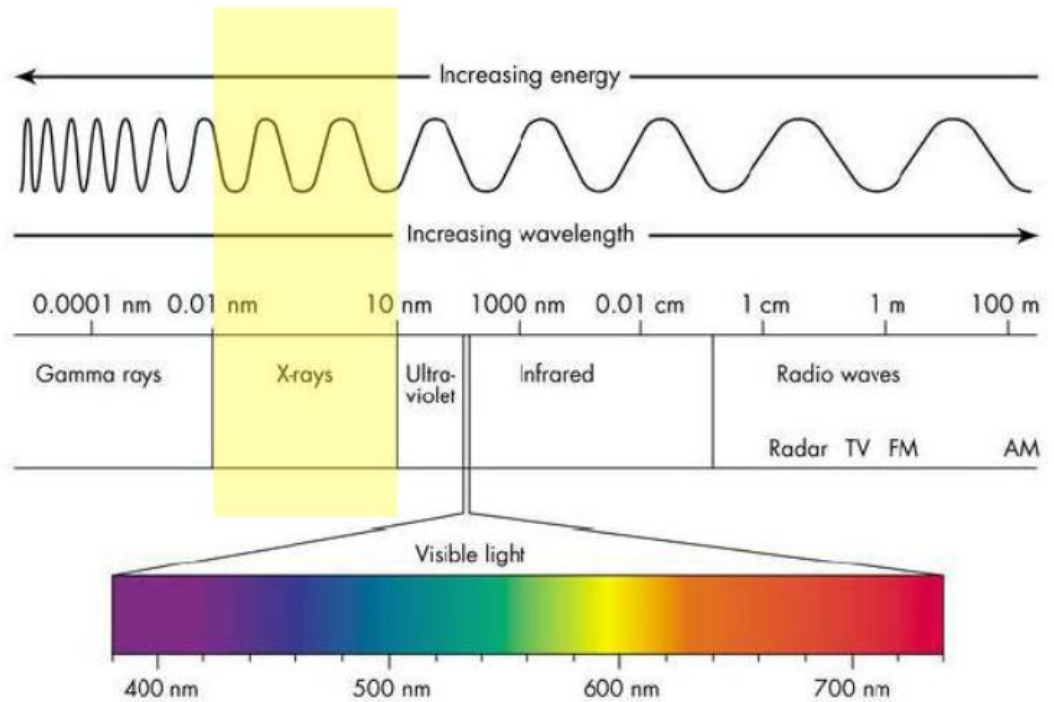
Basics of XRD and XRR

MINT Center

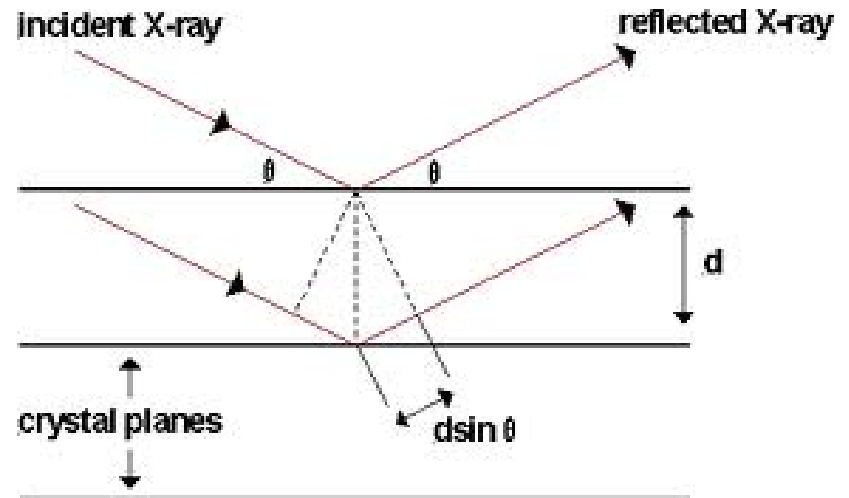
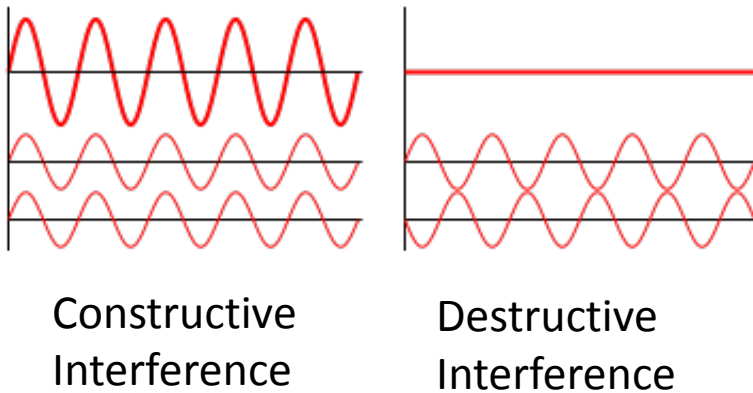
X-Rays

Wilhem Conrad Röntgen

First Nobel Prize Physics 1901



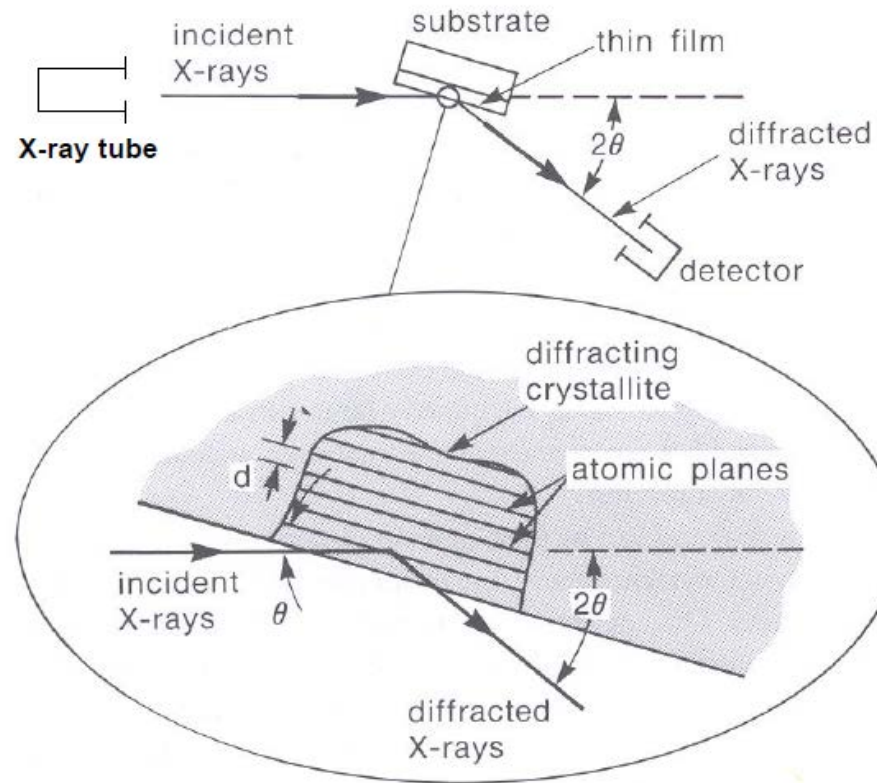
Bragg's Law



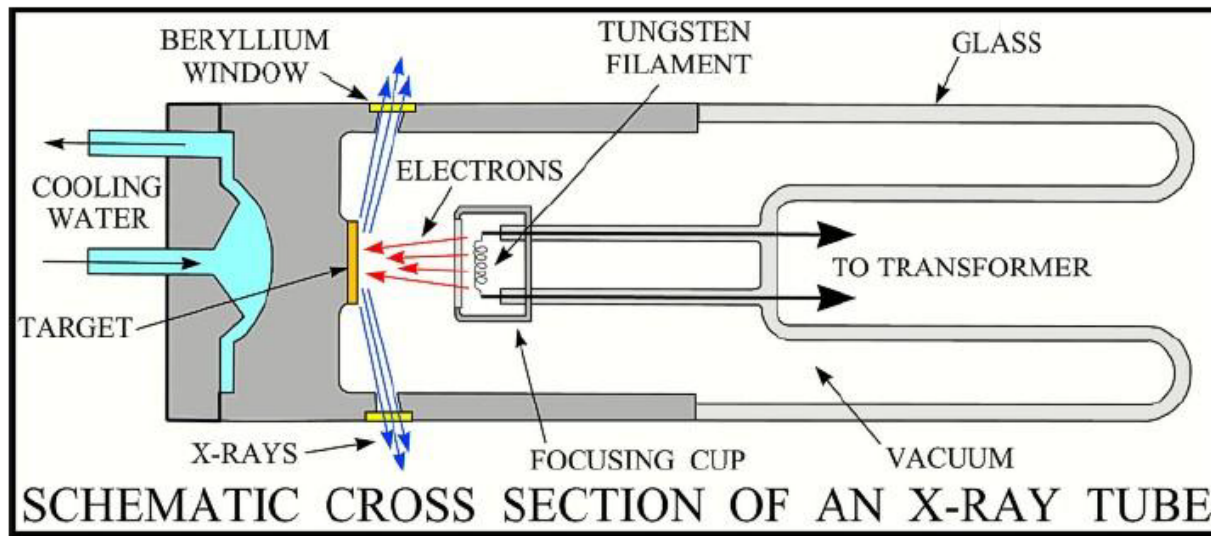
For constructive interference: $n\lambda = 2d \sin \theta$

Law of Reflection: $\theta_{\text{in}} = \theta_{\text{out}}$

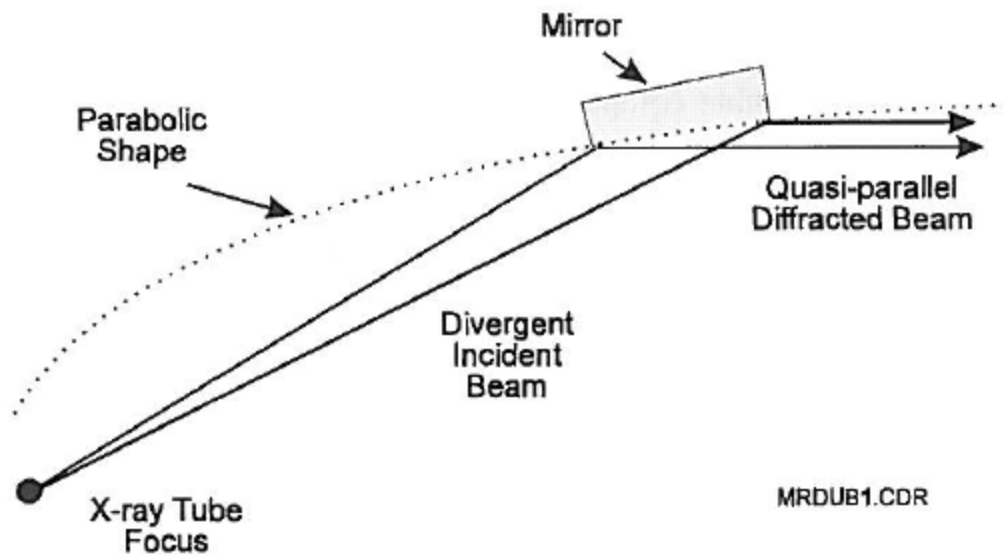
Typical XRD Experiment



X-Ray Tube

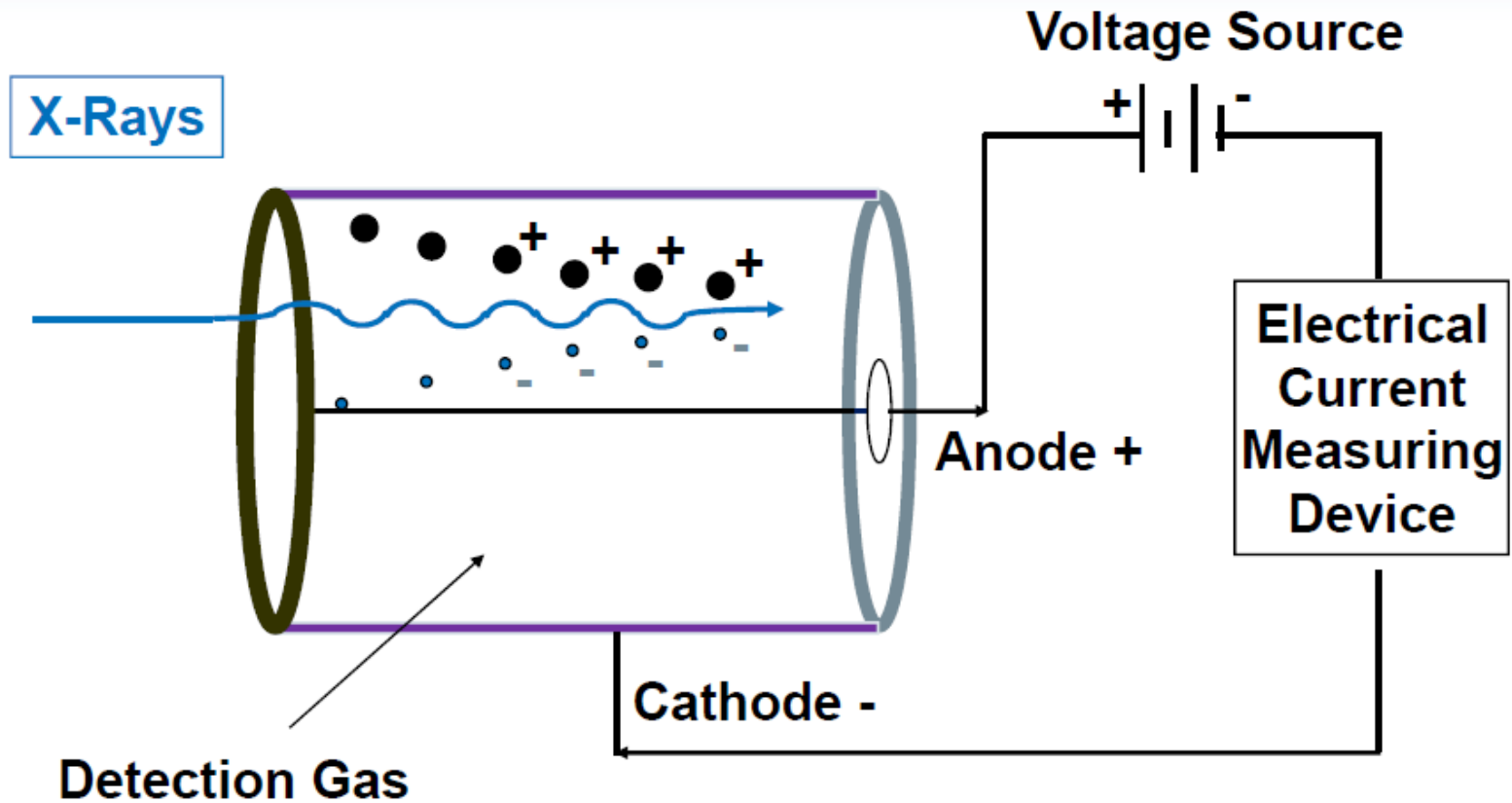


Primary Optics (X-Ray Source)



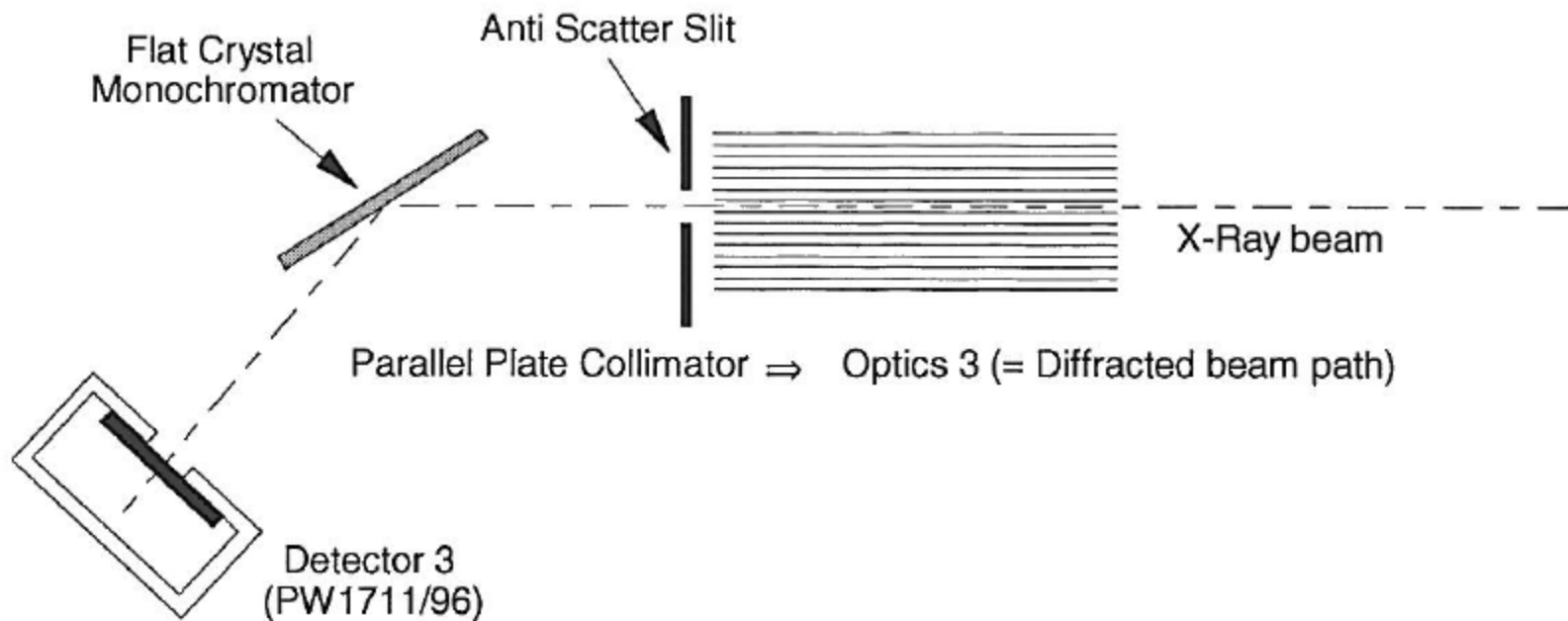
Beam Dimensions: 1.2 mm x 20 mm

Detector

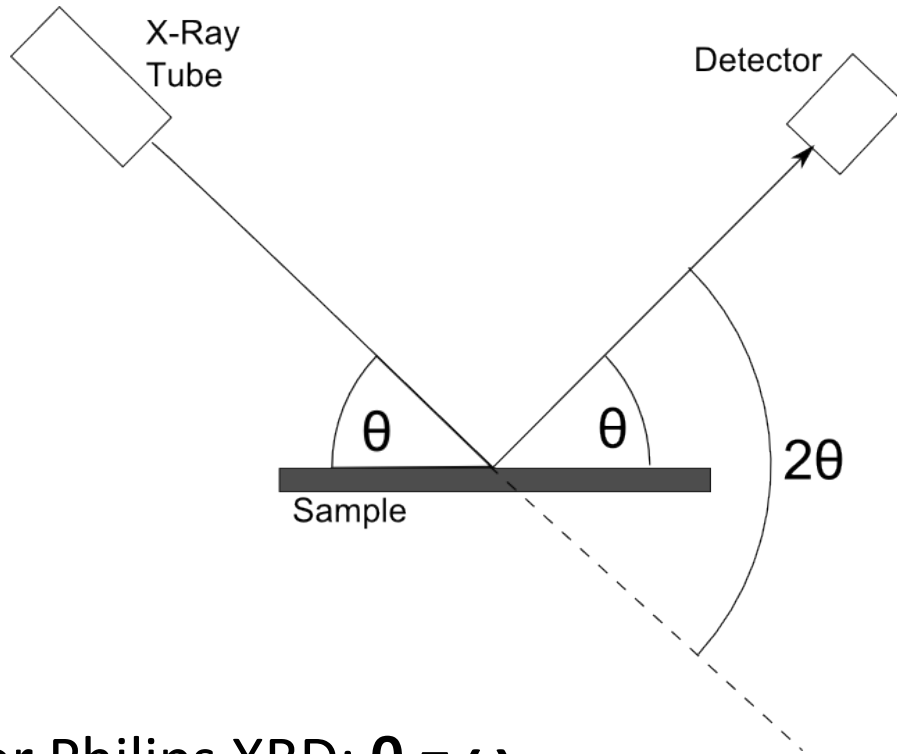


Xe-filled Proportional Detector

Detector Thin Film Setup



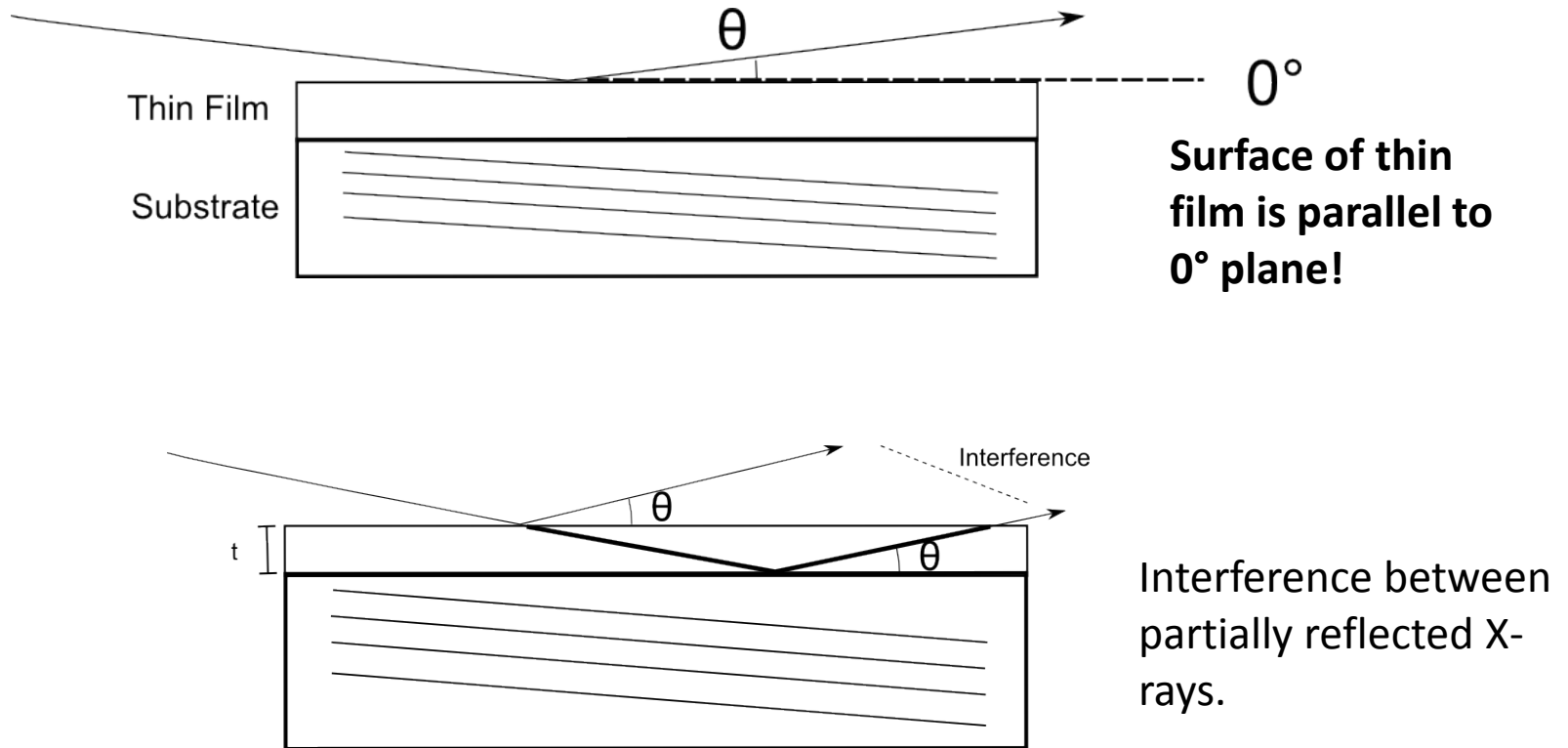
Angles in XRD



For Philips XRD: $\theta = \omega$.

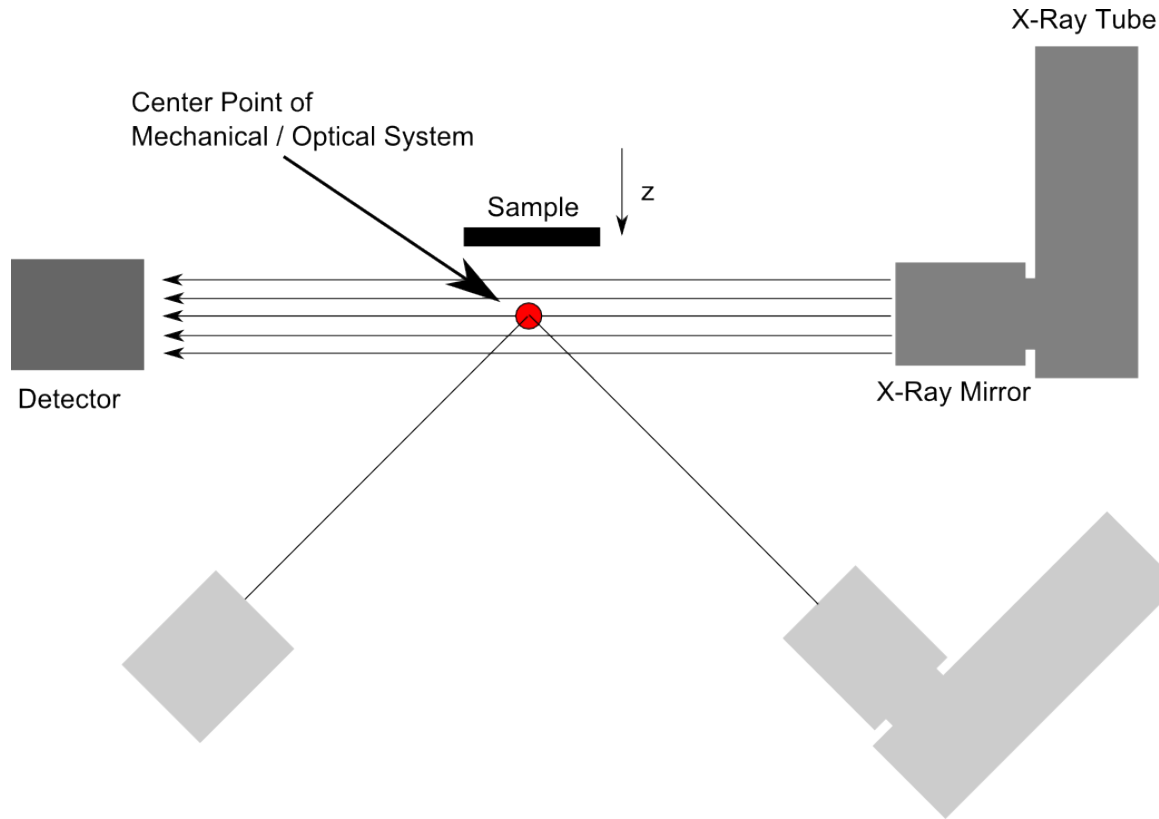
For mechanical reasons, the X-ray tube is stationary. The detector and the sample are rotated to satisfy the Bragg condition.

Sample Alignment for Low Angle (XRR)



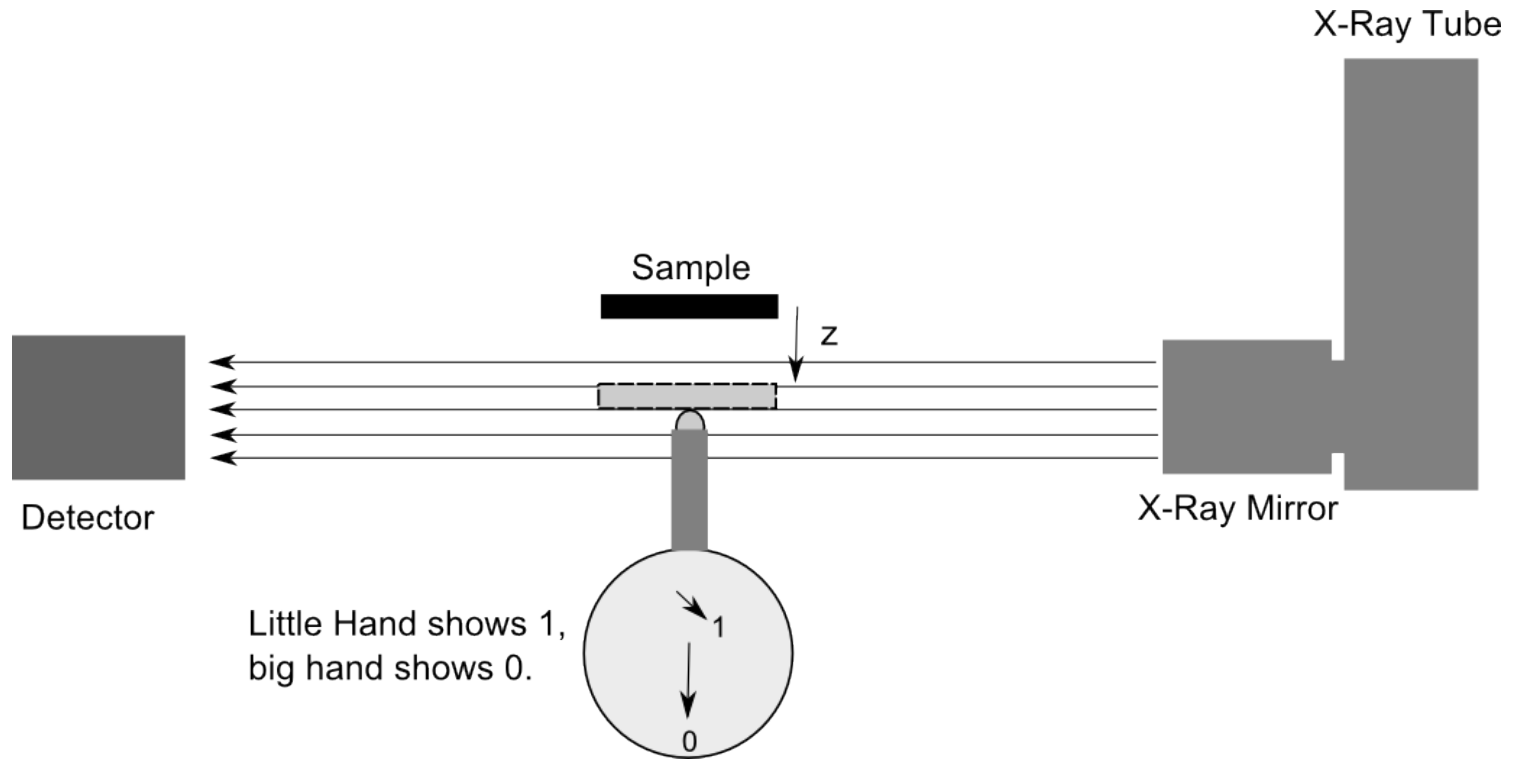
For constructive interference: $n\lambda = 2t \sin \theta$

Sample Alignment



Task: Bring surface of sample to **center point of system** and make **parallel** to X-ray beam!

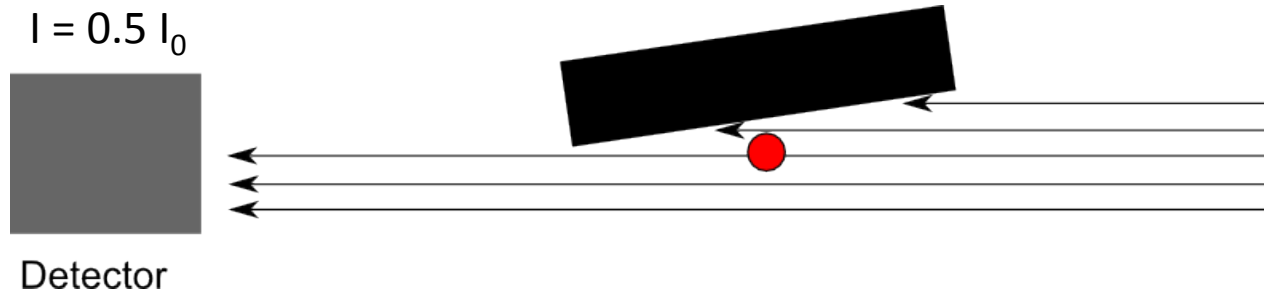
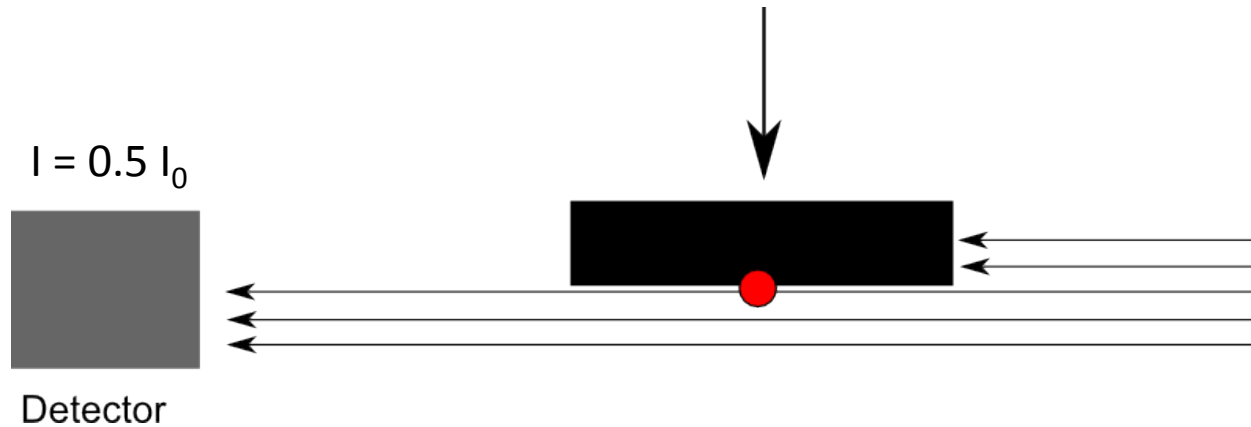
Sample Alignment – Dial Gauge



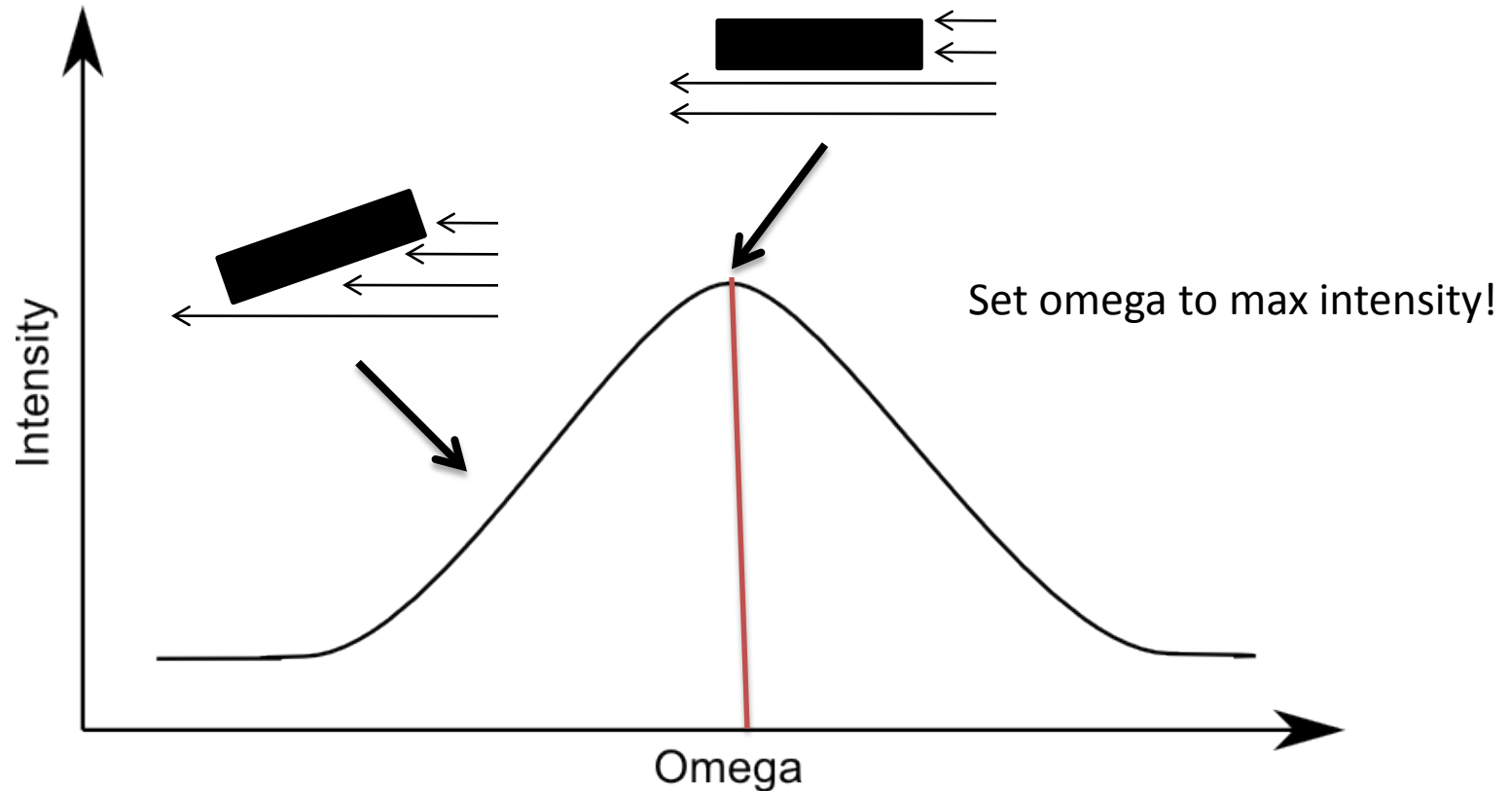
Sample Alignment – Use X-ray Intensity

- The X-ray beam is 1.2 mm wide and ~ 20 mm tall.
- If your sample is **larger than 20 mm** you can use the following method to align the sample.

Sample Alignment – X-ray Intensity

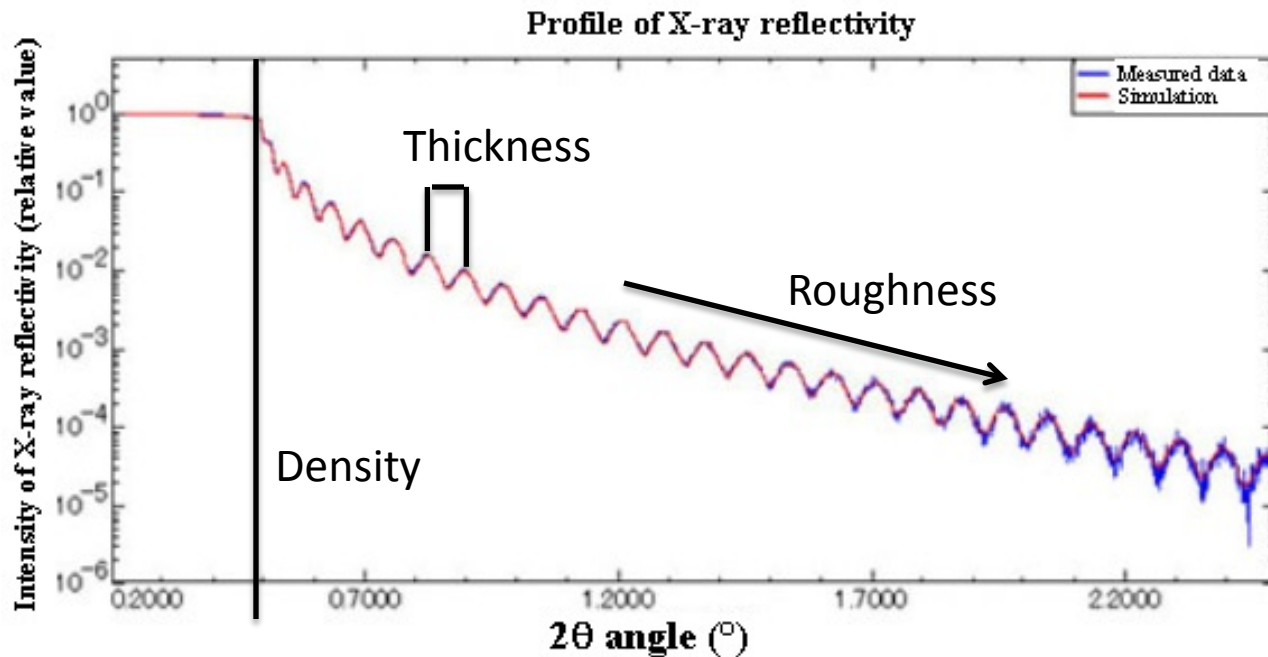


Sample Alignment – Rocking Curve



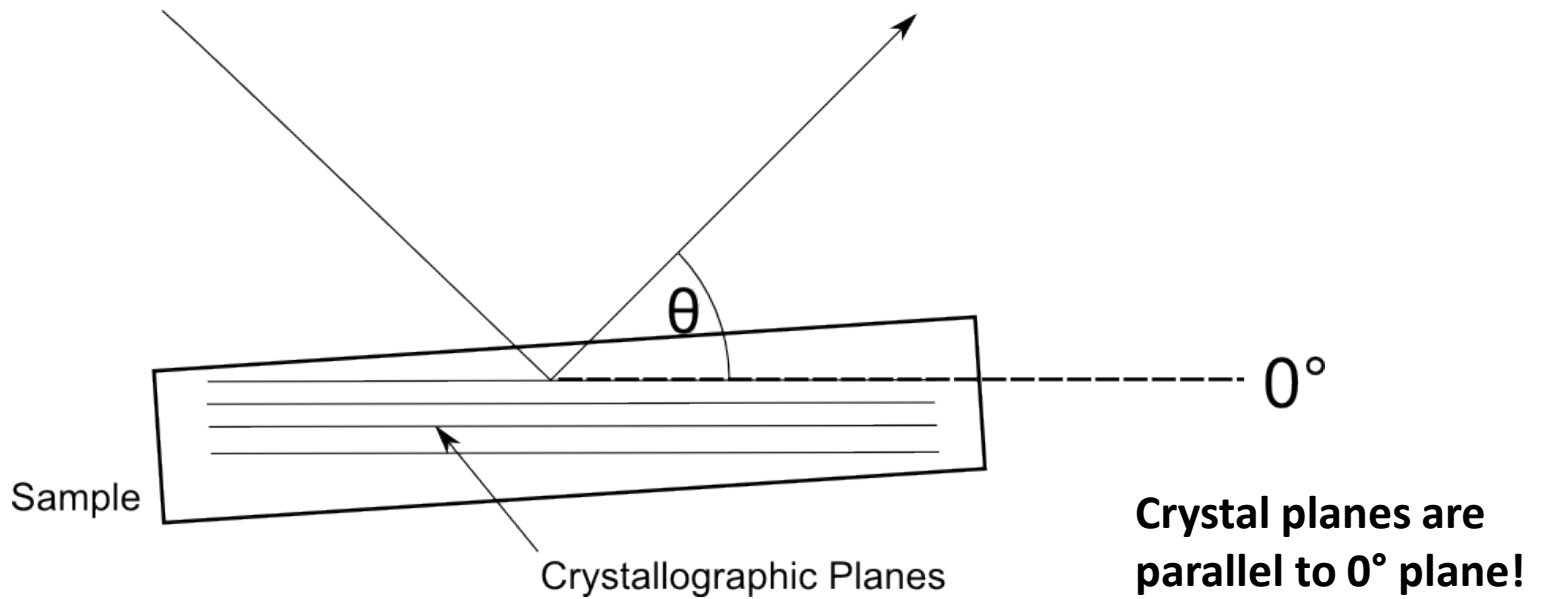
This omega (or theta) scan is called a '**rocking curve**'!
Now we know the surface is parallel to the X-ray beams.

Typical XRR Measurement

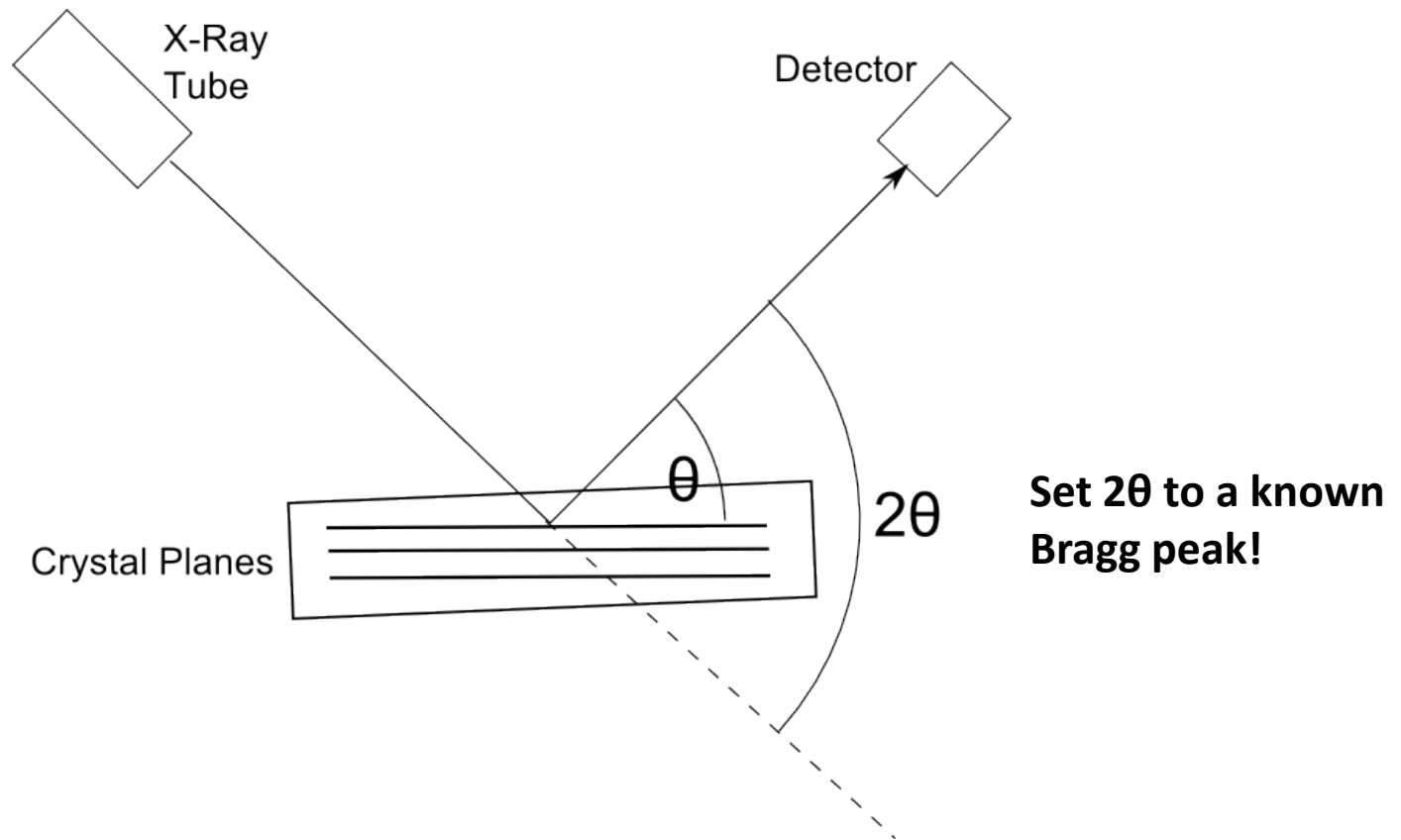


Determination of these parameters is mostly done by simulation and fitting of measured data, especially for complex thin film multilayers.

Sample Alignment for High Angle (XRD)

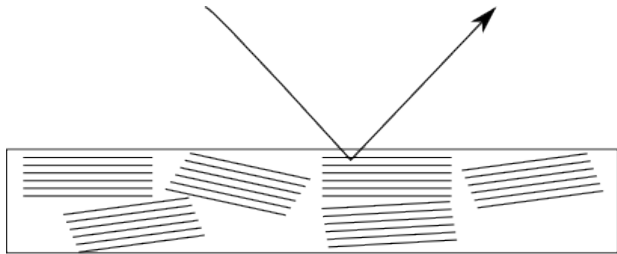


Alignment of Crystal Planes

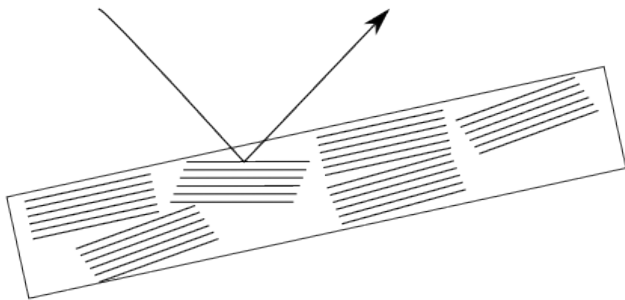


Measure a **rocking curve for θ (or ω)** and choose angle with max intensity.
Now the sample is correctly aligned for XRD measurements!

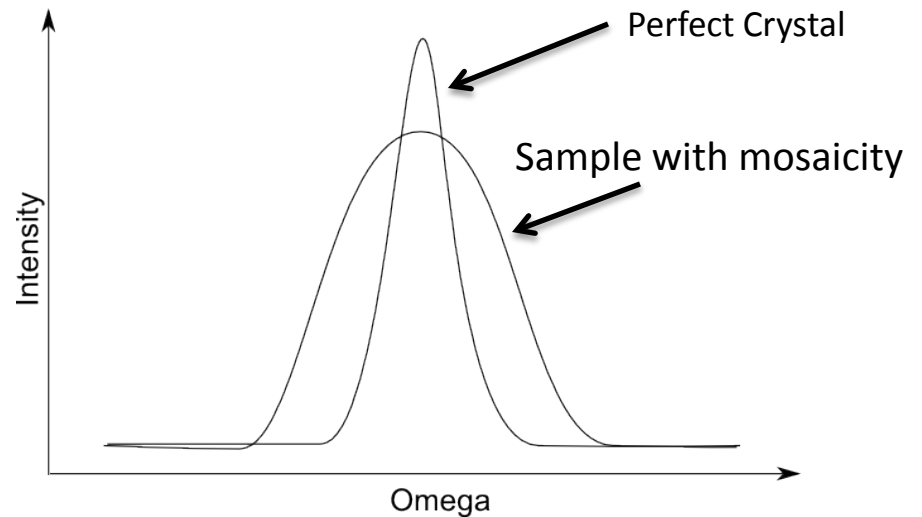
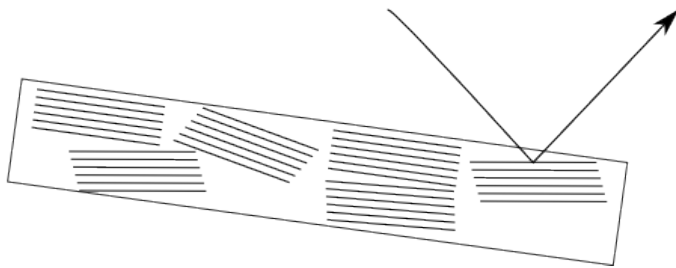
Rocking Curve and Mosaicity



Due to disorder (mosaicity) in the sample the Bragg condition can be satisfied for a range of ω values.



Compared to a perfect crystal the rocking curve is therefore broadened.



When Finished

- Set X-ray power back to 30 kV / 20 mA
- Set $z = 2$ mm
- Click Move to Rest Position
- Close control window, shut down program
- Take sample out, clean sample stage from grease
- Close sliding doors (carefully).