Binghamton University - ADL

Advanced XRD Considerations & Techniques

**Adapted from various sources*

Dr. Matt Wahila

5-9-2025

CONTENTS

Resolution Modes

Low (0.01)

Mostly used for texture and residual ψ-stress analysis on polycrystalline and amorphous materials. For these measurements, the X-ray tube has to be operated in point focus.

ω/2θ scans can also be performed in low resolution mode. However, it's not the best setup for this kind of measurement. It's recommended to use normal resolution mode.

Normal (0.001)

This is the mode in which most users operate the system.

Well suited for textured epitaxial and textured polycrystalline thin films and can also be used to analyze nearly perfect epitaxial layers. ω/2θ scans and reflectivity measurements can be performed. However, rocking curve (ω-scan) in this mode is reasonable only if the layer peak FWHM is wider than the instrumental resolution.

> ***Note:***
> *It's a good idea to check the instrumental resolution with your installed optics and slits by measuring the FWHM of a high-quality, single crystal diffraction peak.*

Reciprocal space mapping can also be done in this mode. Again, it is only feasible for layers that exhibit relatively wide diffraction peaks or sufficient peak separation.

High (0.0001)

Mainly suited for single crystals, nearly perfect epitaxial layers, as well as textured epitaxial layers. This mode can be used for ω/2θ scans, rocking curve (ω-scan), and reciprocal space mapping. However, using this mode drastically increases measurement time due to the more protracted motor movement, so it is not recommended unless truly necessary.

Effects of Slits

Vertical Beam Mask

The vertical beam mask defines the vertical height of the line focused beam. Smaller masks cut the beam down, resulting in less beam hitting your sample, and thus less signal. Therefore, use the largest mask you can get away with based on the height of your sample.

Divergence Slit

The divergence slit defines the equatorial (i.e., horizontal / in-plane) divergence of the beam, which affects the width of the beam irradiating your sample. Narrow slits cut the beam down, resulting in less beam hitting your sample, thus reducing signal intensity. Therefore, it's a good idea to use the widest slit you can for your application. The width of the area on the sample that is irradiated by the incident beam can be calculated as follows:

$$L = \frac{R \times (\sin\omega \times \sin\delta)}{\sin^2\omega - \sin^2(\delta/2)}$$

where L = irradiated length of the sample,
R = radius of the goniometer (R = 320 mm),
δ = divergence angle labelled on the divergence slit,
ω = angle between incident beam and sample surface.

Parallel Plate Collimator Slit

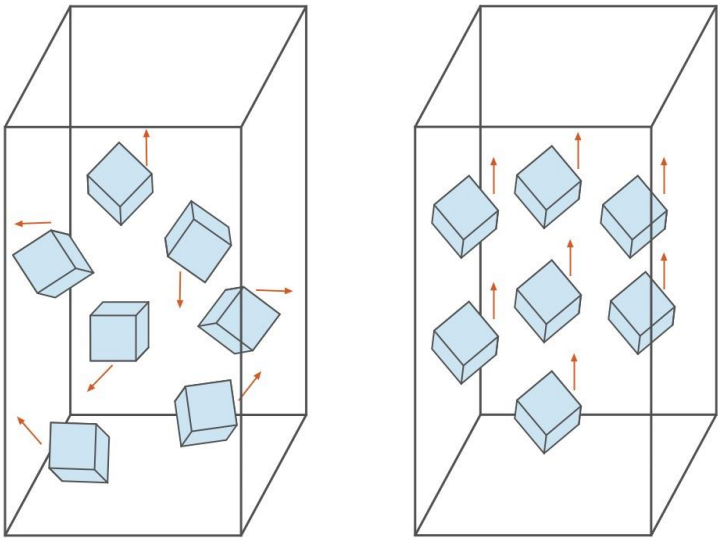
Putting a parallel plate collimator in place of basic receiving slit optics enables the use of asymmetric diffraction geometries. Properties such as twinning, texture, and stress may be studied. However, using the 0.18° parallel plate collimator slit drastically cuts down the beam entering the detector, thus reducing the signal intensity. This slit is mainly used for reflectivity measurements to enhance the resolution at very low 2θ angles (< 4°).

Soller Slits

Soller slits limit the axial (i.e., vertical / out-of-plane) divergence of the beam. A larger Soller slit provides more intensity, while a smaller slit produces better peak shapes at lower 2θ angles. The 0.04 radian slit is the most commonly used. The 0.02 or 0.01 radian Soller slits are typically used only for Rietveld quality data or for samples with diffraction peaks where 2θ < 20°, as using them will reduce the signal intensity.

TEXTURE

Texture is the word materials scientists use to describe the crystallographic preferred orientation that develops in many materials. This simply means that some crystals prefer to point in a certain direction, for example, if a plate is rolled, some planes might prefer to be aligned with the rolling direction. In geology, the term fiber is sometimes used instead of texture. Texture is a statistical measure of the way in which the crystals are oriented, so care must be taken to ensure many crystals are measured. Many diffraction techniques can be used to measure texture, X-ray diffraction, neutron diffraction, and electron diffraction using the technique called electron backscattered diffraction EBSD.



Random orientations

Preferred orientations

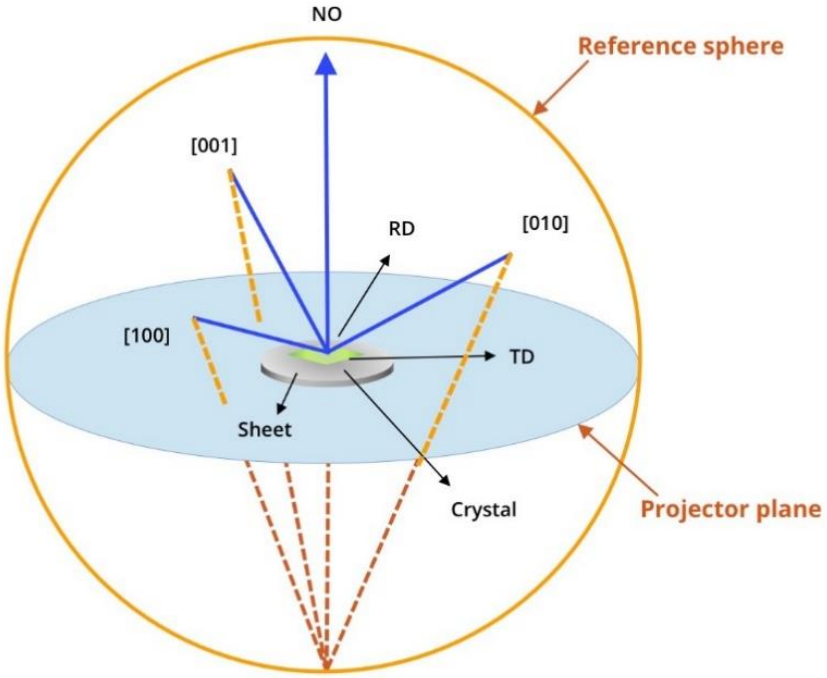
Displaying texture

There are three main ways in which we can display texture: pole figures, inverse pole figures, and orientation distribution functions.

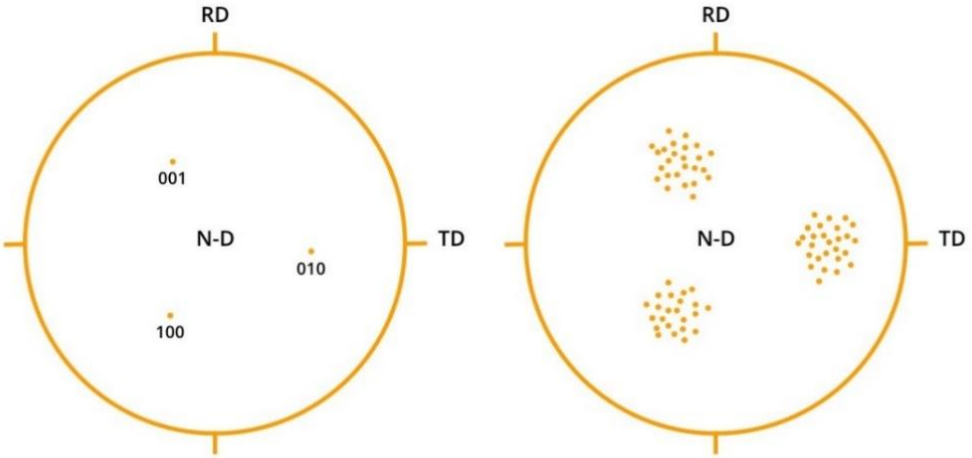
1. Pole Figures

Pole figures are probably the easiest for people to visualize and the most common way of displaying both texture and individual orientations from EBSD and XRD data. The pole figure is based on a spherical reference frame. An imaginary line is drawn perpendicular

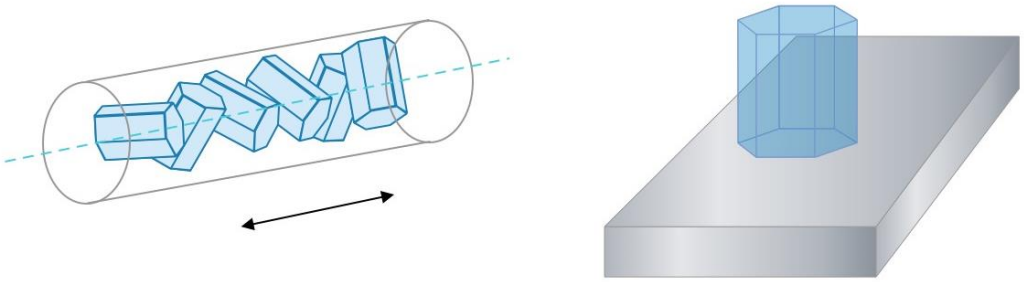
from the crystal plane onto the sphere. A line from this point on the sphere is then made down to the bottom of the sphere though the flat projection plane, as shown:

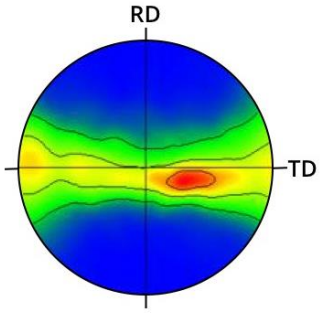


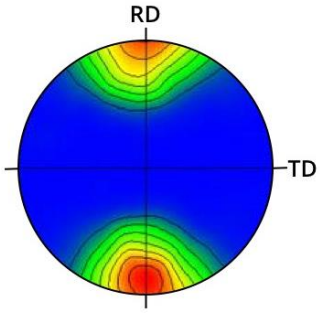
If many points are added to a pole figure, a preference for certain locations can be seen:



If we measured sufficient data, a mathematical method known as spherical harmonics can be used to create a statistical fit of the individual points into a statistically correct curve. A pole figure can be considered a map of crystal orientations with reference to the sample geometry. It shows how a crystal plane prefers to be aligned within the specimen geometry.





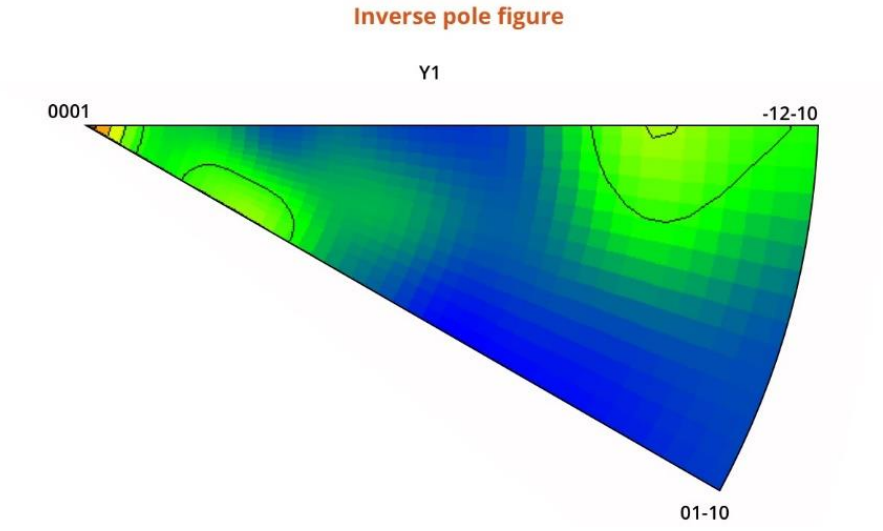


<0001> ⊥ extrusion dirn.

<0001> ⊥ rolling plane

2. Inverse Pole Figure

An inverse pole figure, as the name implies, plots data as a mirror of the pole figure. The inverse pole figure is a map of the specimen direction or geometry with respect to the crystal directions. The IPF shows which of the different crystal orientations are preferred by the specimen geometry. Usually, we plot one direction like the rolling direction, or the direction of solidification. The IPF has a triangular shape with one curved edge, and each edge of the IPF represents one of the main crystal planes, as shown:

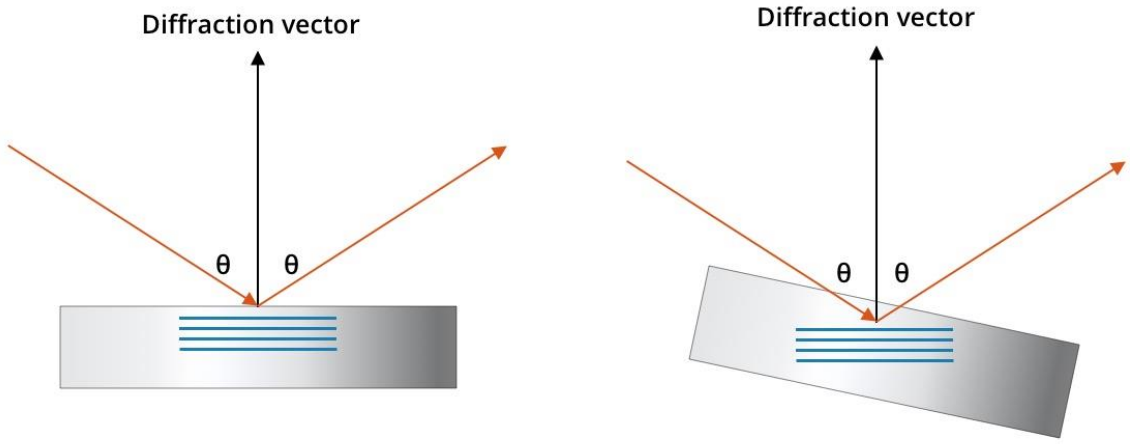


3. Orientation Distribution Functions

Another method of describing the directionality of a crystal is to use a coordinate system known as Euler angles. This is also used in other fields such as astronomy. The three angles, Euler 1, 2 and 3, can be plotted in the same way as we would plot any other set of three Cartesian coordinates. Just like the pole figure, the regions where there are many points in a cluster indicate those regions that are preferred in the specimen. The data can be contoured by spherical harmonics, just like the other two methods of showing texture. Some researchers prefer this description, but it is apparent that obtaining any reference between the ODF and the specimen geometry requires extensive experience.

Residual Stress

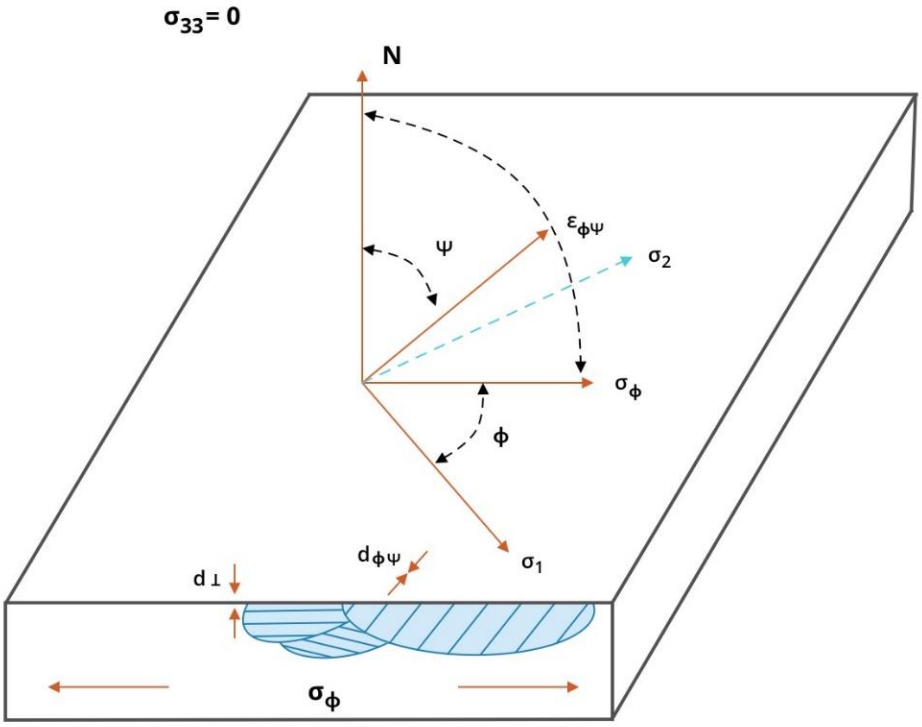
Residual stress is usually measured on the same instrument as a texture measurement (i.e., those that can allow tilting of the specimen). The residual stress measurement is based on the notion that the crystal planes parallel to the free surface (i.e., the one being measured) are free of stress and therefore have a d-spacing indicative of zero stress. Therefore, usually we measure diffracted peaks parallel to the specimen surface for XRD.



However, if the specimen is now tilted, although the same family of planes is still being measured (because we are still at the same Bragg angle), these planes are now no longer parallel to the free surface and may therefore be under stress.

The complex geometry of the residual stress measurement necessitates more complex nomenclature than the figure shown above, and the stress normal to the plane is denoted σ_{33}, the rotation around this plane is termed ϕ, and the specimen tilt termed ψ.

The strain in the lattice is determined by the usual way we determine strain in any system, simply the change in length divided by initial length, but in the case of this measurement we use the lattice spacing as our measurements: ε



The full 3-dimensional description of residual stress can be derived as:

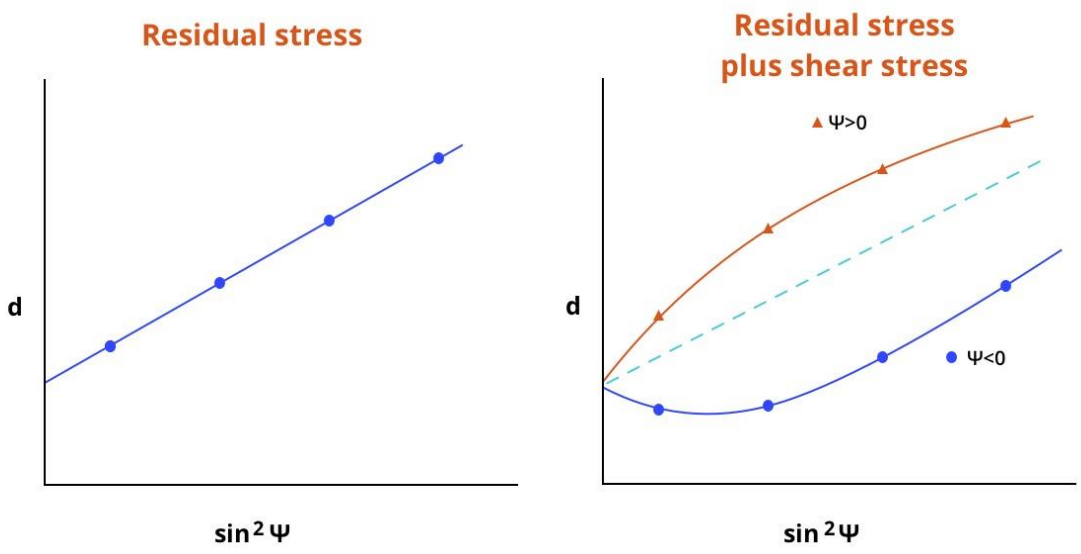
$$\varepsilon_{\psi\phi} = \varepsilon_{33} + [\varepsilon_{11}\cos^2\phi + \varepsilon_{12}\sin 2\phi + \varepsilon_{22}\sin^2\phi - \varepsilon_{33}]\sin^2\psi + [\varepsilon_{13}\cos\phi + \varepsilon_{23}\sin\phi]\sin 2\psi$$

Where ε_{33} = 0 because the free surface is not constrained
ϕ = 0° (unless multiple measurements are made at different sample rotations)

Therefore, the equation reduces to:

$$\varepsilon_{\psi\phi} = [\varepsilon_{11}]\sin^2\psi + [\varepsilon_{13}]\sin(2\psi)$$

If we assume there is no shear stress, we can simply plot $\sin^2\varepsilon$ as a function of d-spacing. And if this is a straight line, the slope = the residual strain. To convert to stress, multiply by Young's modulus. Use a modulus from literature. Don't try and measure it yourself, because elastic moduli are notoriously difficult to measure and need special equipment.



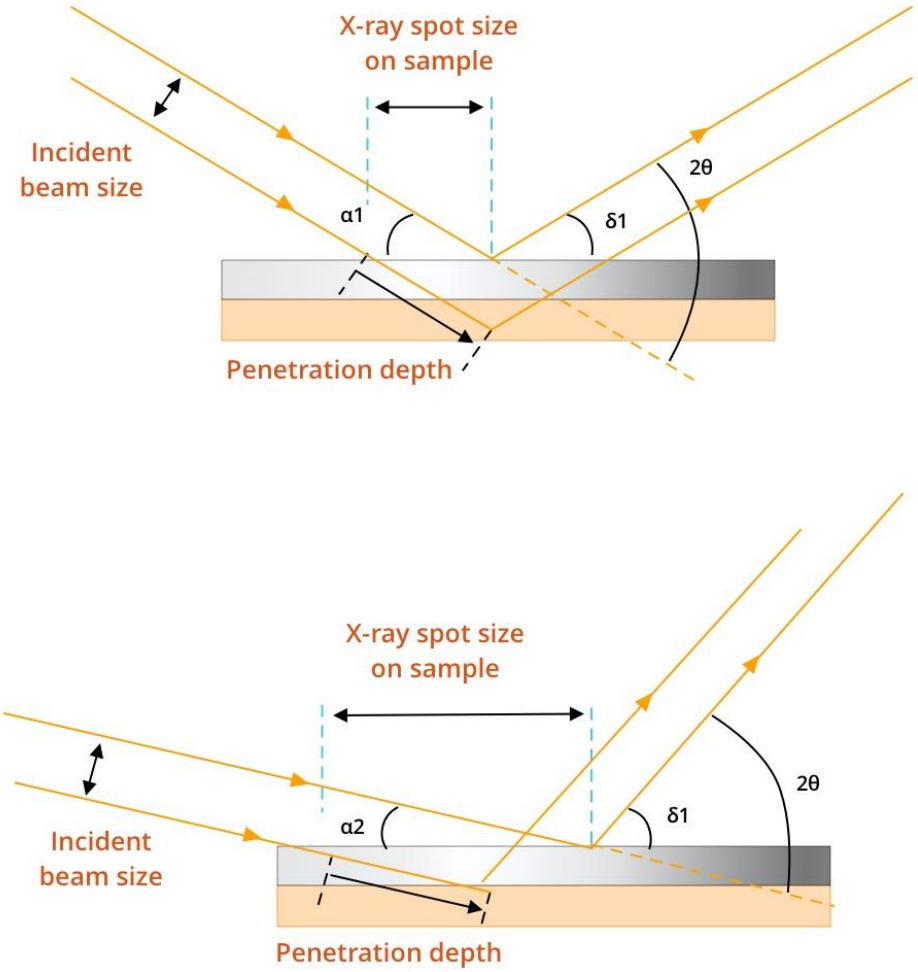
If your data shows ψ splitting it means you have a shear component. You will not get both sides of the split, just one, but you can measure the other by rotating your sample by 180° and measuring the residual stress again (However, this is typically not required). Using the same plot, you will be able to solve for ε_{11} and ε_{13}.

You will notice that at high tilt angles the intensity of the peak decreases due to the defocusing issued discussed in the texture section. This is not a concern of residual stress measurement because it is only the peak position that is of interest here.

GLANCING ANGLE XRD

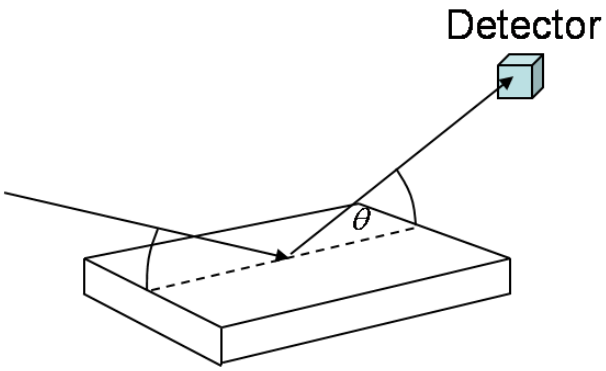
At higher angles of 2θ, the X-ray diffraction signal comes from deeper within the specimen. In these cases where we have a thin film such as a coating, it is advantageous to remain at a low angle so that more of the diffracted signal comes from the surface.

This is called glancing angle XRD and can be carried out on some lab instruments, although most of this work is increasingly being carried out at synchrotrons. Regardless, the physics of the experiment is the same: the incident beam is kept at a constant low (glancing) angle, and only the detector is moved through the full range of 2θ required for the experiment. For instruments with a 2D detector, the measurement is simply a case of setting up the beam at a glancing angle with the sample then measuring the full 2D diffractogram.

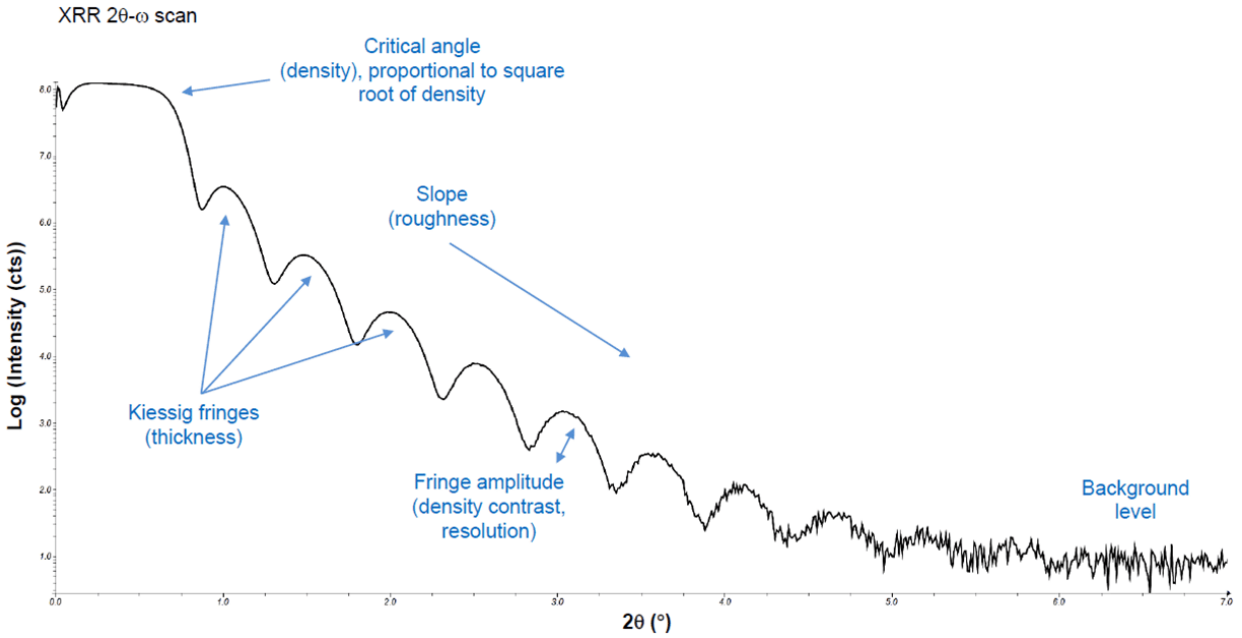


X-RAY REFLECTIVITY/REFLECTOMETRY (XRR)

X-ray reflectivity can be used to characterize surfaces, thin films, and multilayers. The basic principle of X-ray reflectivity is to reflect a beam of X-rays off a flat surface and to then measure the intensity of reflected X-rays in the specular direction (reflected angle equal to incident angle). This can be accomplished by measuring XRD at very grazing 2θ angles.



For films with thin layers of uniform thickness, X-ray reflectivity may display oscillations, analogous to the Fabry-Pérot effect, here called Kiessig fringes. The period of these oscillations can be used to infer layer thicknesses, interlayer roughness, electron densities and their contrasts, and complex refractive indices.



References & Videos

XRD Basics

https://www.physics.upenn.edu/~heiney/datasqueeze/basics.html

Resolution

https://web.stanford.edu/group/glam/xlab/Technique/Technique.htm

Texture

https://myscope.training/XRD_What_is_texture

https://www.youtube.com/watch?v=WhYvv5eaRS0

Residual Stress

https://myscope.training/XRD_Residual_stress

Glancing Angle XRD

https://myscope.training/XRD_What_is_glancing_angle_XRD

X-ray Reflectivity

https://covalentmetrology.com/service/x-ray-reflectometry-xrr/