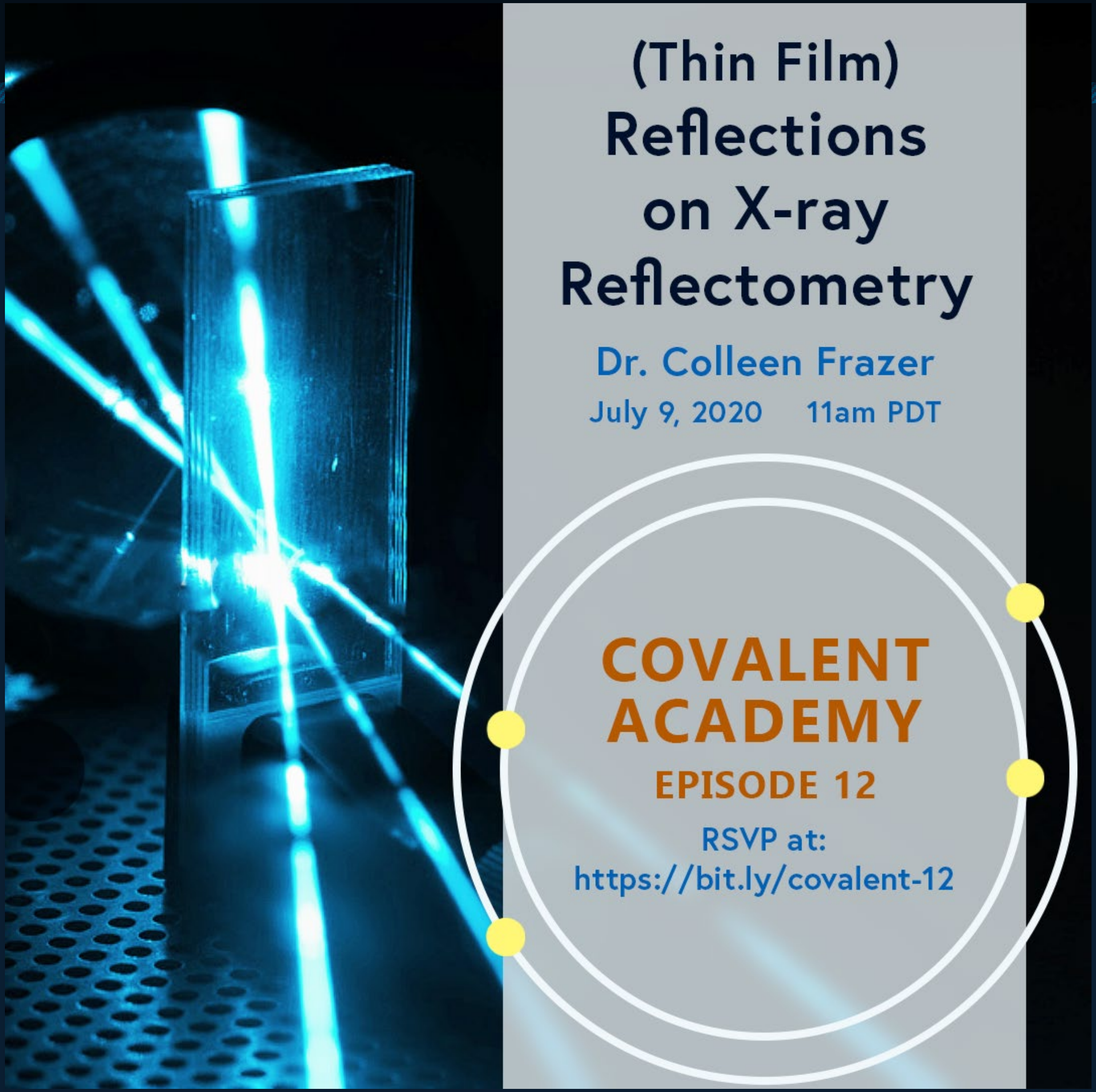
COVALENT METROLOGY

Welcome

covalentmetrology.com

COVALENT METROLOGY



Covalent Metrology

- **Founded 2016**
- Testing, measurement & characterization **Platform**
- **30 team members** (11 PhDs)
- **9,500 ft^2 lab in Sunnyvale, CA**
- **1-Stop-Shop Source for Answers**
  - 30 instruments in-house
  - 6 partnerships with instrument manufacturers
  - 11 partner labs
  - 6 corporate “tool shares”
- **More than 300 customers** (80 % repeat)

Covalent Technical Groups and Organization

COVALENT METROLOGY

hello@covalentmetrology.com

Electron & Scanning Probe Microscopy

SEM / TEM, AFM, EDS, EELS, Nanoindentation, Acoustic Microscopy

Surface Analysis

XPS, Auger, TOF-SIMS, d-SIMS

Optical Microscopy & Spectroscopy

Laser Confocal, White Light, Chromatic Aberration, Spectral Ellipsometry, UV-Vis-Nir Spectroscopy

Materials / PC Board Failure Analysis

Cross Sections, Inspections, Dye & Pry Testing

X-Ray Characterization

XRD / XRR, XRF, μCT

Chemical Analysis

ICPMS, GCMS, FTIR, Raman Spectroscopy, NMR

Nanoparticle Analysis

DLS, PSA, Particle Zeta Potential

Bulk Properties Characterization

DSC, TGA, DMA, Rheometry, Tensile Testing, Pencil Test, Surface Zeta Potential

Introducing Dr. Colleen Frazer

- Colleen supervises all X-ray Diffraction (XRD) and Reflectometry (XRR) operations and analysis: giving Covalent's customers access to the whole gamut of data available from this technique on their samples.
- Colleen obtained her PhD in Materials Science and Engineering from the University of Kentucky, concentrating on XRD residual stress measurements in highly textured materials.
- She has focused on XRD/XRR operation and advanced interpretation throughout her career, spanning Academic institutions, National Labs and private companies: ORNL, University of Kentucky, Penn State University, Sandia National Labs, Philips Lumileds and EAG.

XRR Webinar Overview

XRR = *X-Ray Reflectometry* or *X-ray Reflectivity*

- Used to measure thin film properties:
  - Thickness
  - Density
  - Interfacial / surface roughness
- Nondestructive analysis
- Works well on crystalline, amorphous, and combination film stacks
- Sensitive to electron density perpendicular to surface

This webinar will cover:

- Indispensable sample alignment strategies
- Interpreting results without overinterpreting
- Working in the real world
- Troubleshooting with confidence

Notable Events and People

Heinz Kiessig

Published paper noting X-Ray oscillations from smooth interfaces (1931), still referred to as “Kiessig fringes”

Wilhelm Röntgen

Discovered X-Rays (1895), received first Nobel Prize in Physics

Lyman Parratt

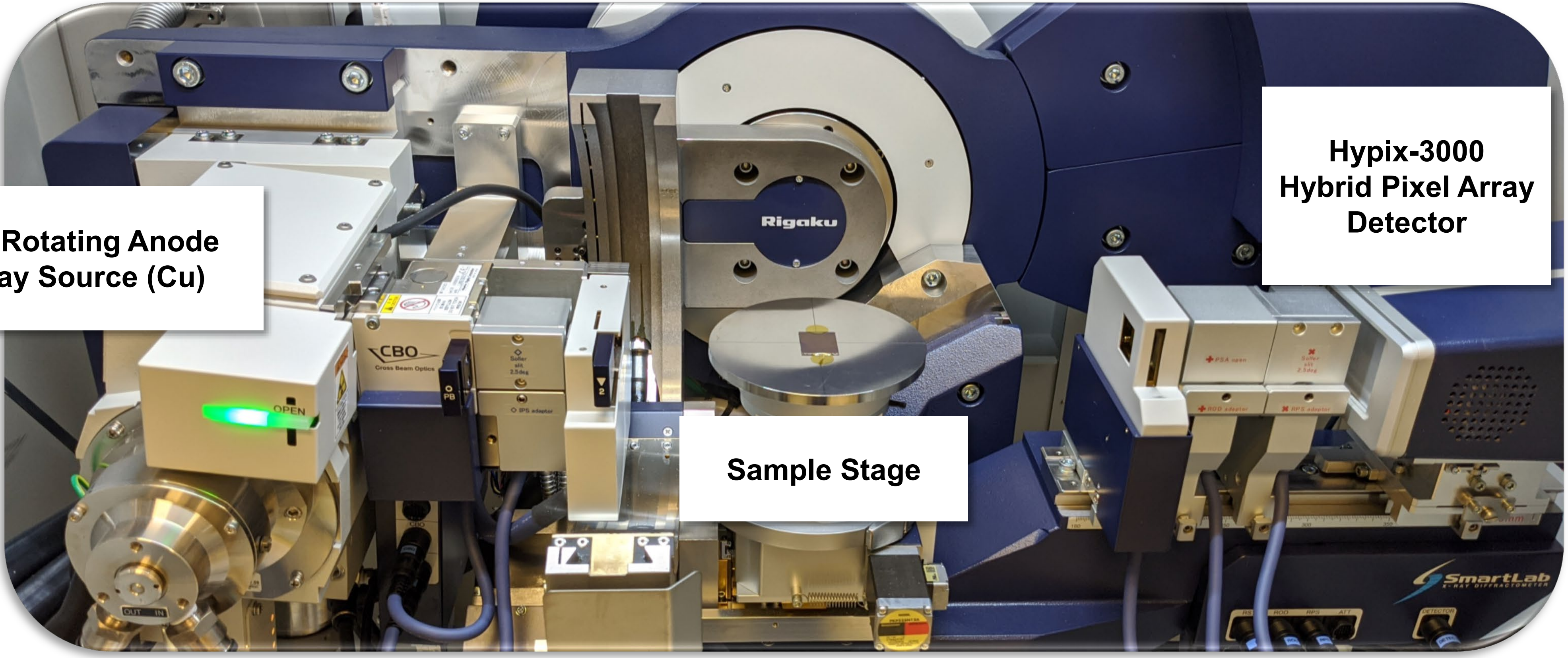
Formalized XRR (1954), Group Leader in Manhattan Project, Head of Physics Department at Cornell University

Rigaku XRR Equipment for the Lab and In-Line Metrology

- Ongoing partnership with Rigaku Corporation
- Latest-generation **SmartLab Multi-Purpose** X-Ray Diffractometer in our lab
- 9kW Rotating Anode X-Ray Source + Hypix-3000 Hybrid Pixel Array Detector in 0D mode
  - **very fast, accurate XRR measurements**
- **Rigaku SmartLab Studio II XRR** software
  - Analyze multi-layer, complex thin films stacks of various materials with minimal uncertainty
- In-line XRR metrology is also available through Rigaku Corporation: we will be happy to put you in touch with the representative if you would like to learn more

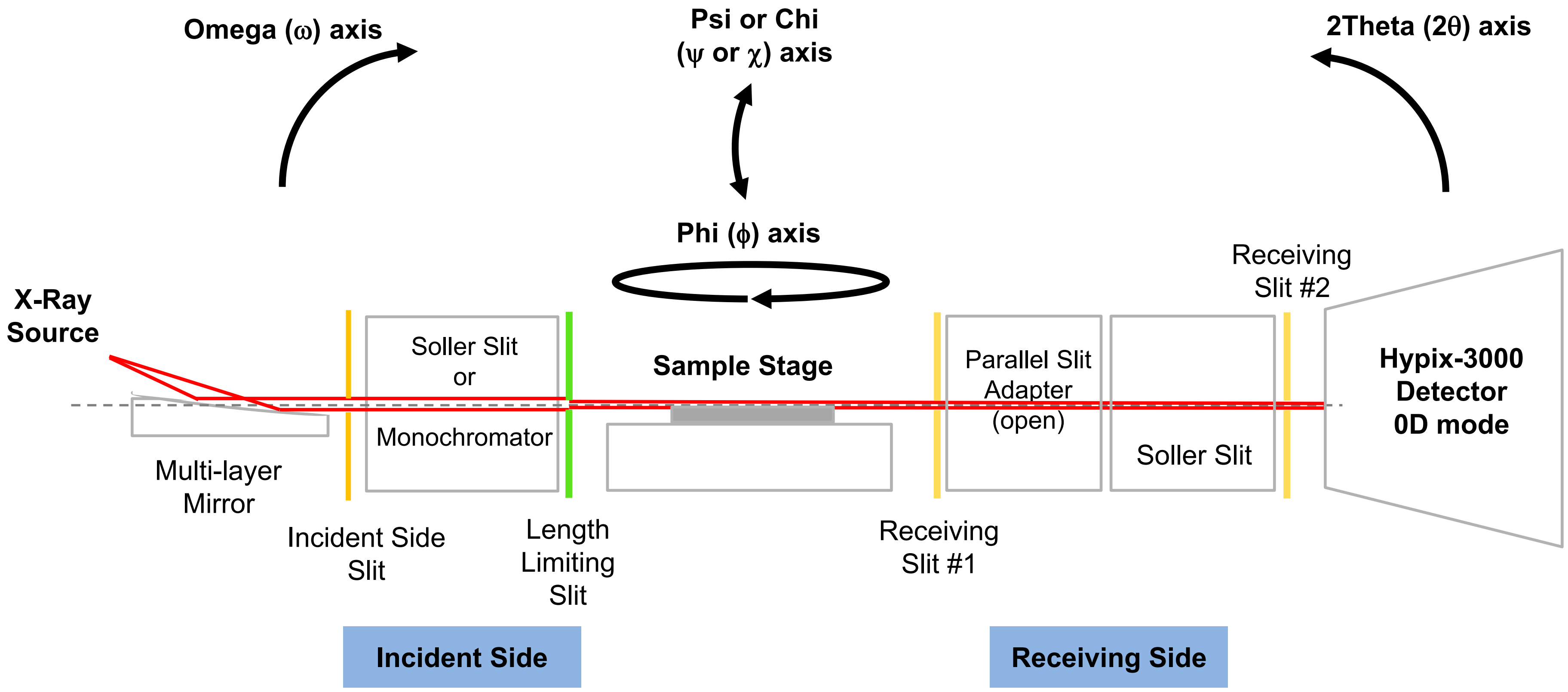
https://www.rigaku.com/techniques/x-ray-reflectometry-xrr

Covalent Metrology's Rigaku SmartLab 4-Circle Goniometer



XRR Beam Path

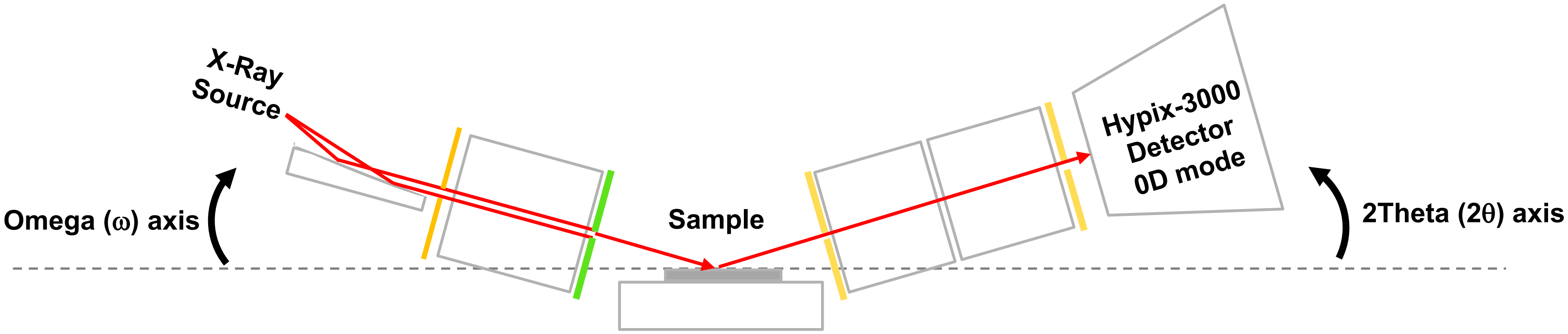
XRR scans are typically 2Theta-Omega (2θ-ω) scans

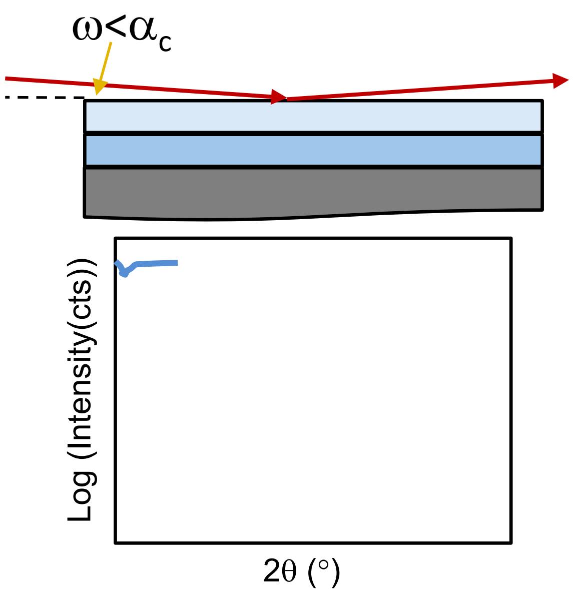


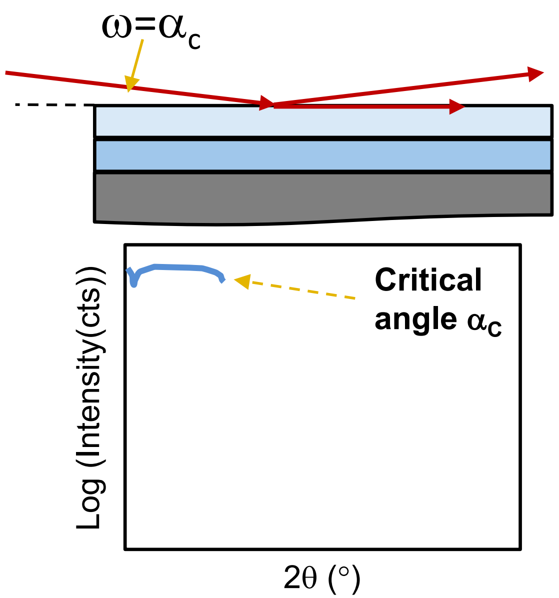
Based on Rigaku schematic
Not to scale

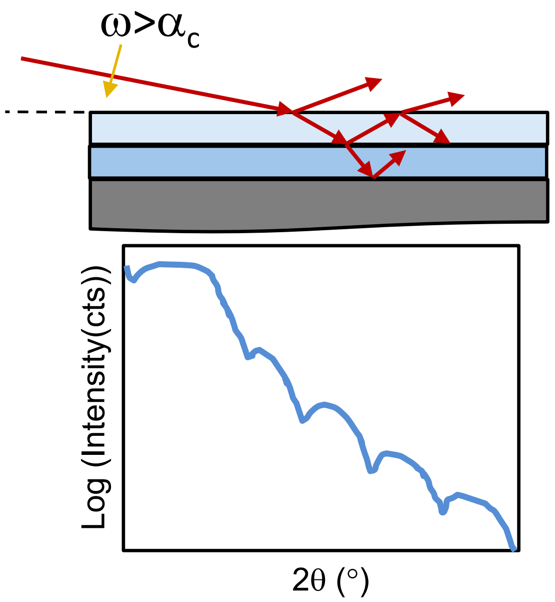
XRR Scans With Measurement Axes

XRR scans are collected at very low incident and reflected angles, almost parallel to the sample surface (schematic angles exaggerated for clarity)

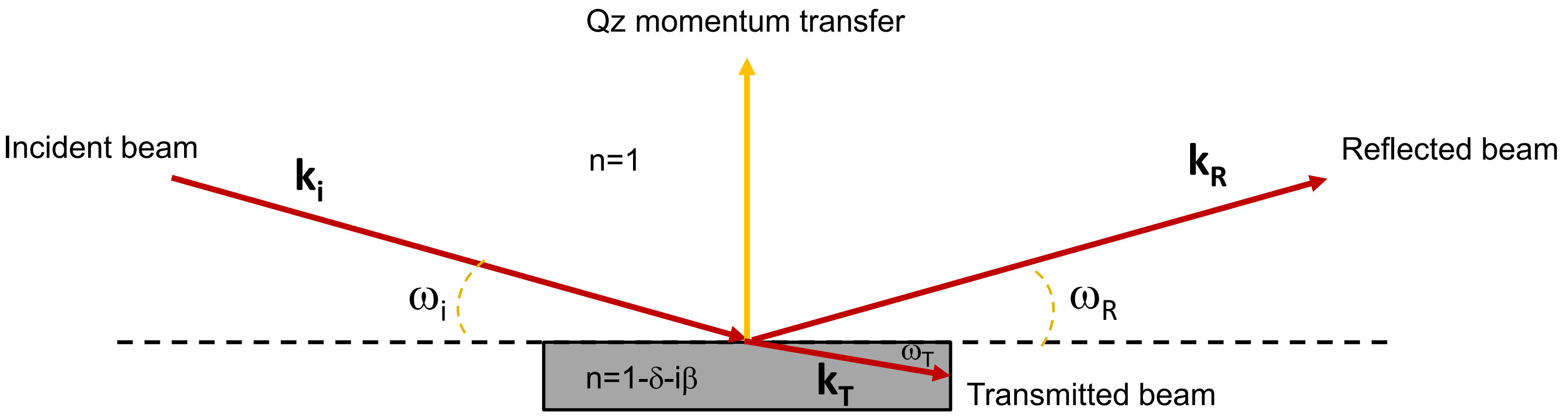


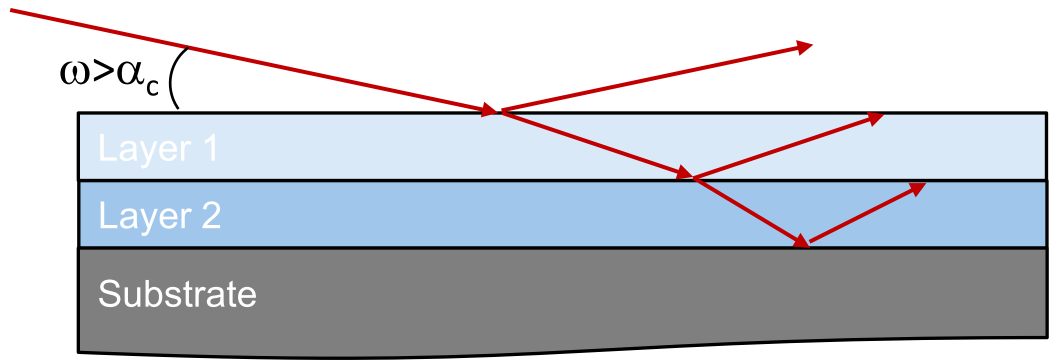






Specular reflectivity uses symmetric incident and reflected angles and represents a combination of Fresnel reflectivity and interference patterns

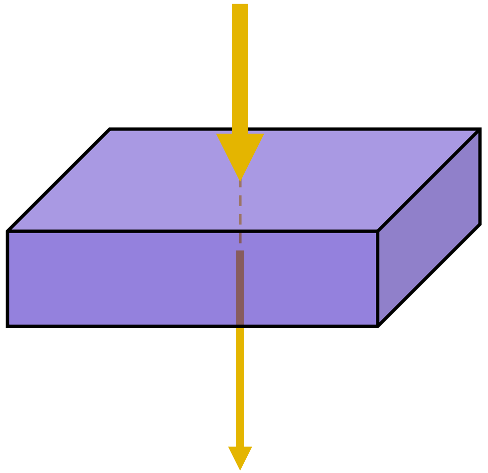




Bowen and Tanner, "X-Ray Metrology in Semiconductor Manufacturing", (2006), p112

X-Ray Penetration Depth

Bulk material, in transmission

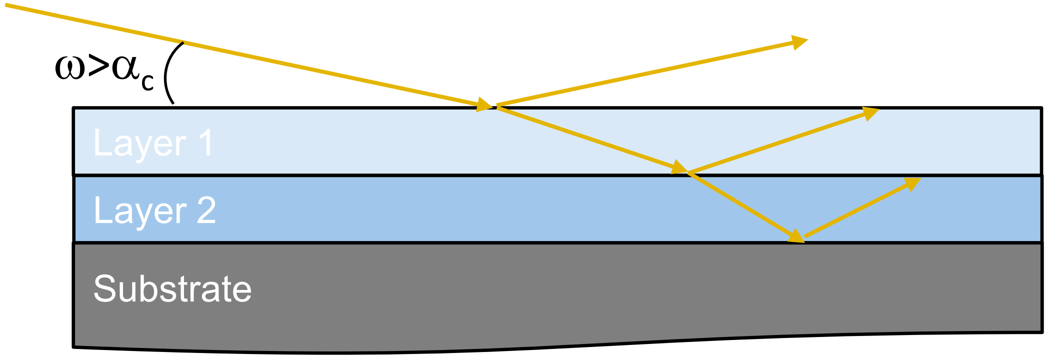


$I=I_0\exp(-\mu L)$
μ, linear absorption coefficient
L, distance

I/I_0 of ~10% is usually measurable

For Al, I/I_0 is ~76μm

Multi-layer material, in reflection



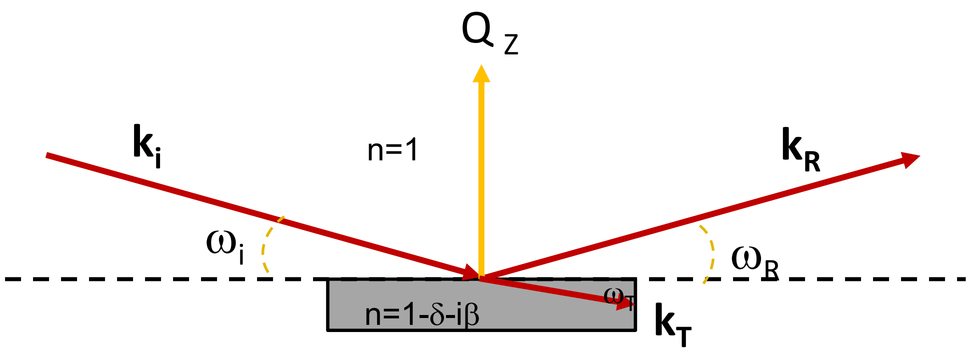
Above image shows the critical angle for total external reflection (α_c).

The beam path is affected by the incident angle, the physical properties of the layers, and interfaces between volumes of different electron densities (Snell's Law)

X-Ray Reflectivity Versus X-Ray Diffraction

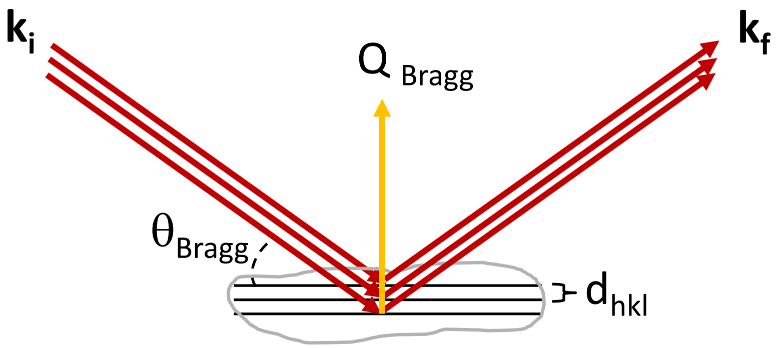
X-Ray Reflectivity is related to electron density differences at interfaces while X-Ray Diffraction is related to the interplanar spacings of crystalline materials

X-Ray Reflectivity



- Related to differences in electron density differences at interfaces
- Data collected at very low angles for the source and detector

X-Ray Diffraction

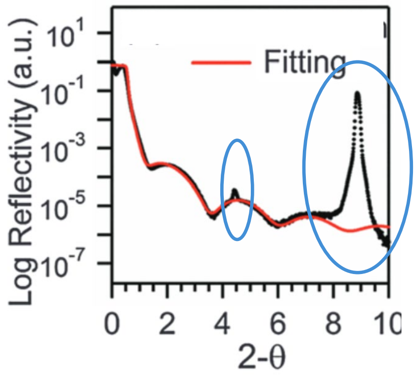


- Related to interplanar spacings of crystalline materials
- Data collected at low and high angles for the source and detector, and the axes can be uncoupled

Unwanted XRD Peaks in XRR Patterns

COVALENT METROLOGY

XRD peaks may appear in XRR scans due to large interplanar spacings

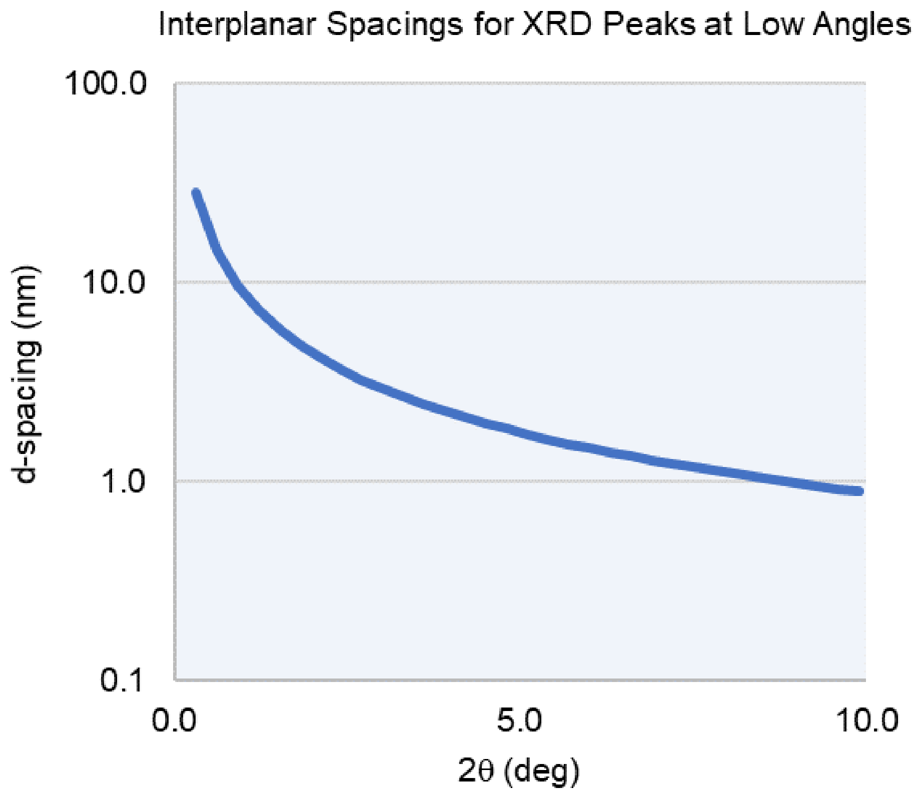


Mica substrate peaks

*Chem. Commun., 2016, 52, 2956

Bragg's Law: $n\lambda = 2d\sin\theta$

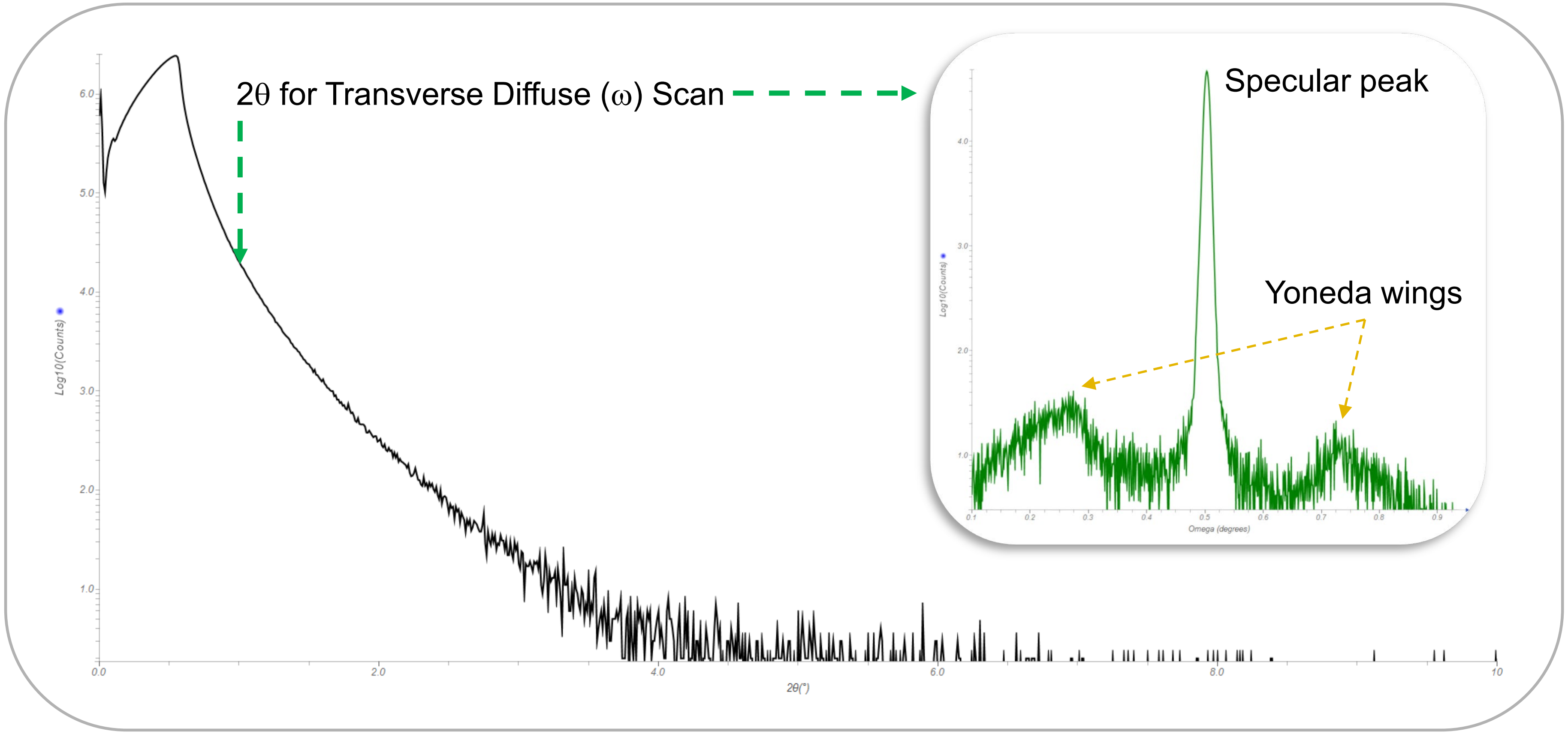
Mica peak at ~9° 2θ corresponds to interplanar spacing of ~1 nm for Cu-target X-Rays



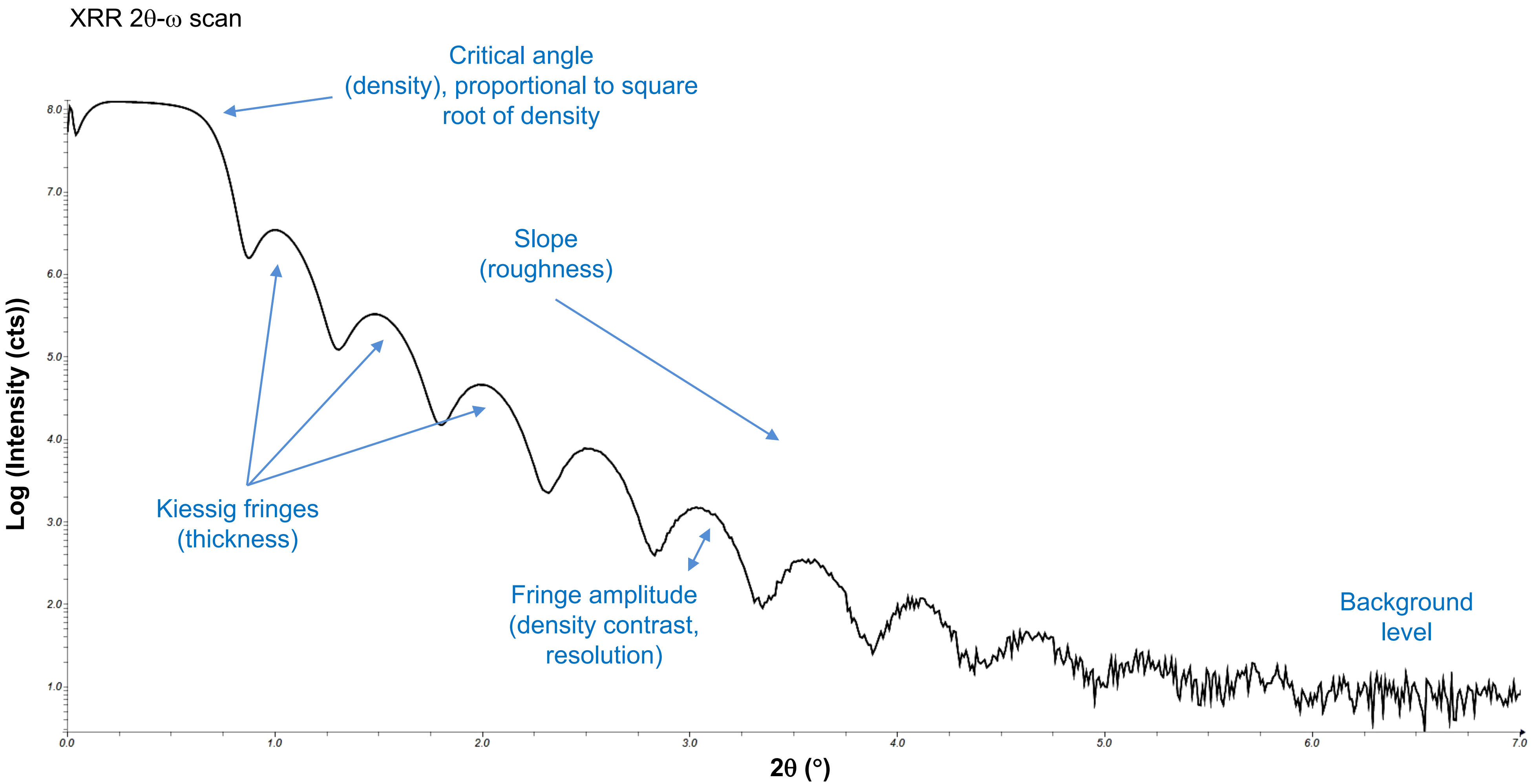
Diffuse XRR

Diffuse reflectivity = non-symmetric incident and reflected angles, relates to lateral correlation

Al_2O_3 Wafer Specular and Diffuse XRR Scans



Features of a Specular XRR Pattern



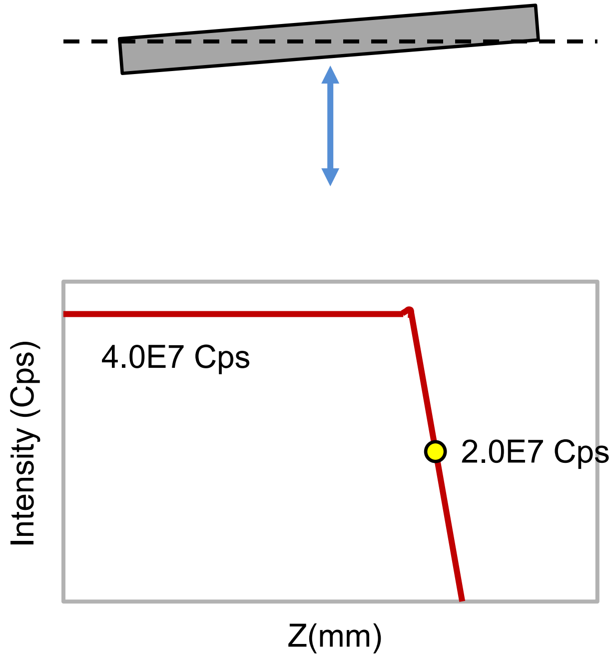
The XRR pattern is shown courtesy of **Alluxa**, manufacturer of high-performance optical filters and precision thin-film coatings

Sample Alignment Steps (1/2)

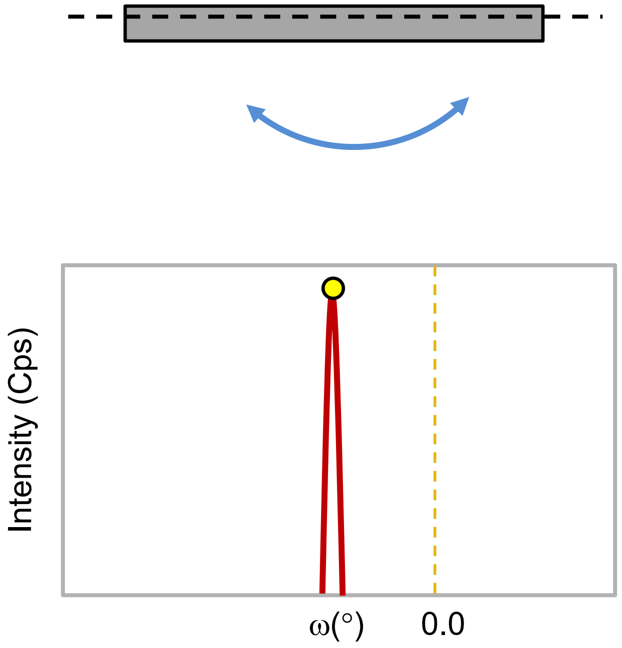
Direct X-Ray Beam

Steps are repeated until no change is observed

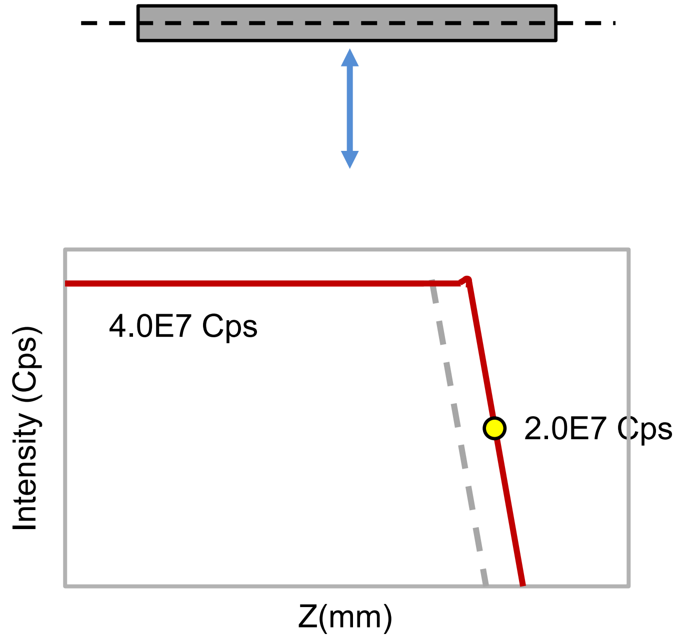
Z-Axis Optimization #1



Omega-Axis Optimization #1



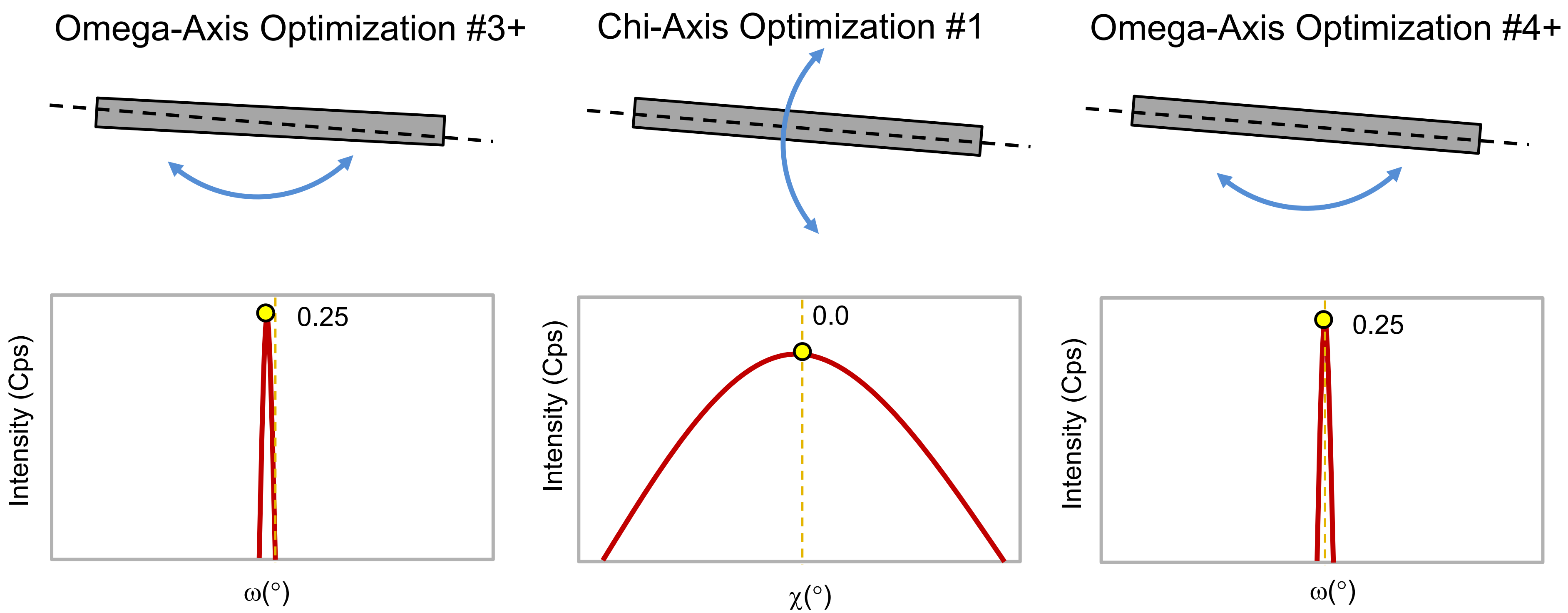
Z-Axis Optimization #2



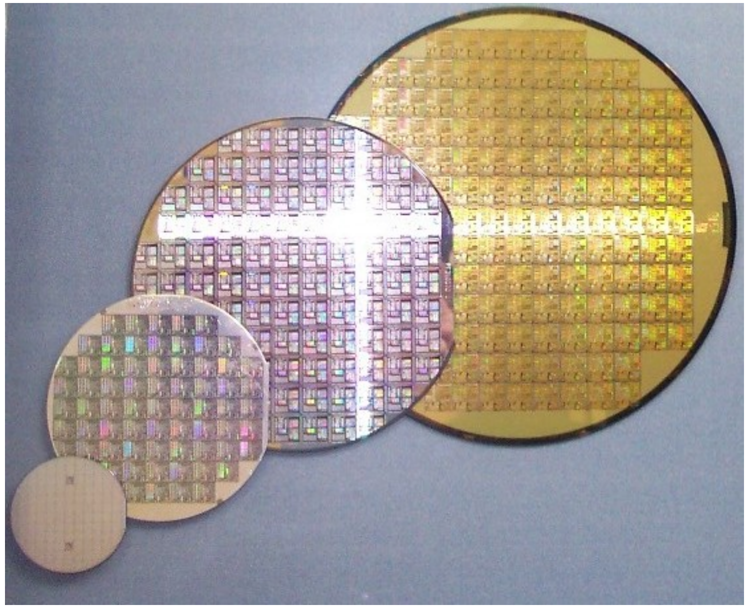
Sample Alignment Steps (2/2)

Alignment on XRR Fringe

Steps are repeated until no change is observed



Likelihood of XRR Success Due to Roughness



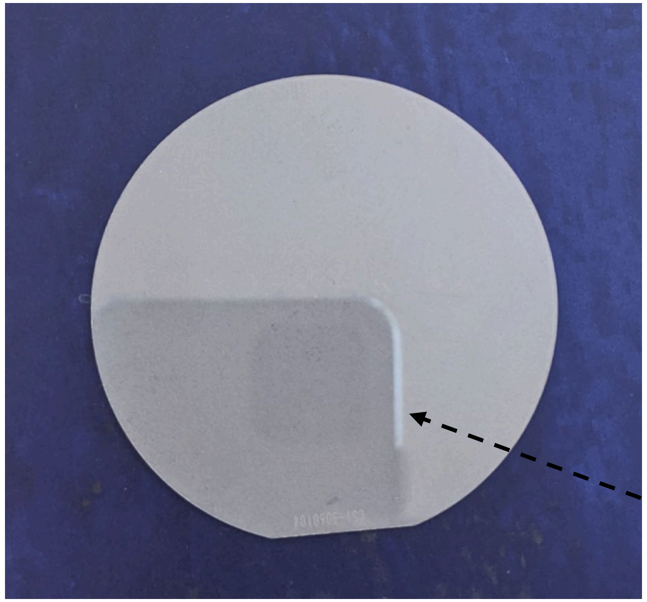


Image of phone camera

Very Unlikely — Very Likely

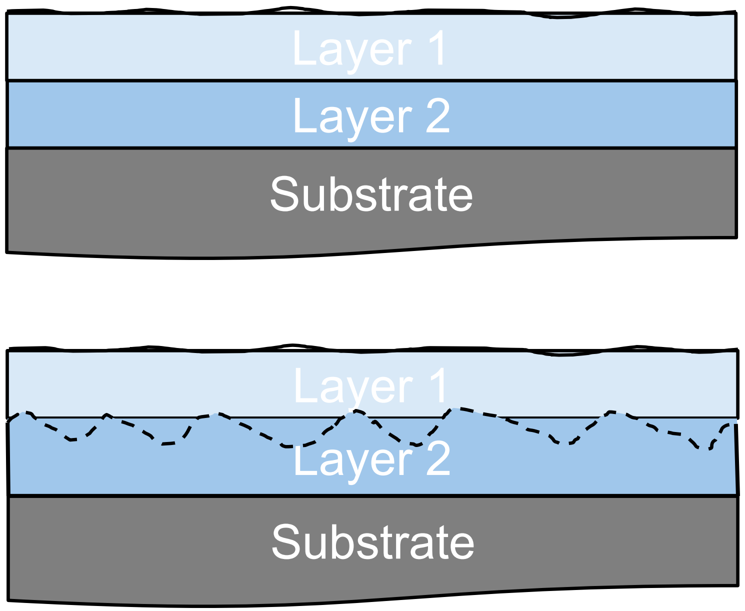
- Film thickness less than 1000nm
- Roughness less than 2nm
- Good lateral homogeneity

https://commons.wikimedia.org/w/index.php?curid=2971205
https://commons.wikimedia.org/w/index.php?curid=928106

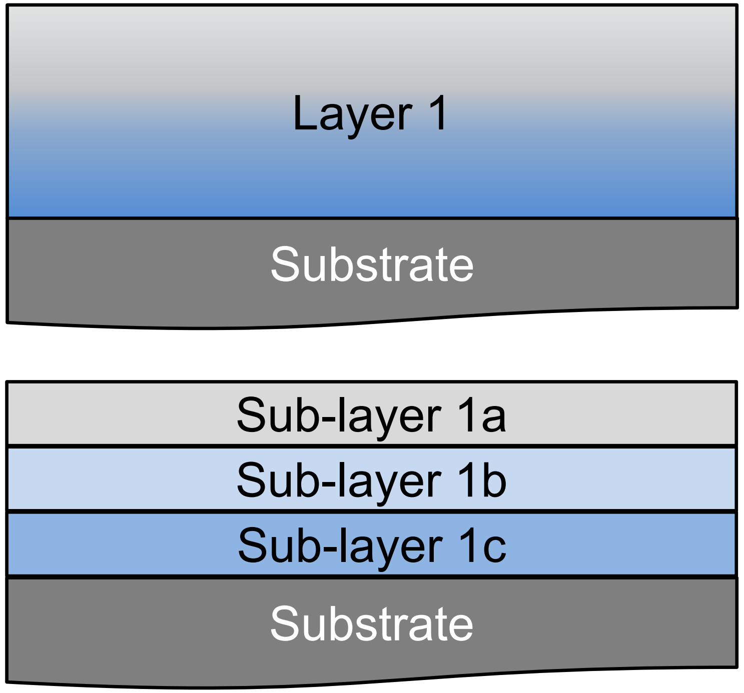
Density Gradients in XRR

Vertical gradients may exist in the layers and often can be successfully modeled in more than one way

(Electron) Density gradients
due to roughness

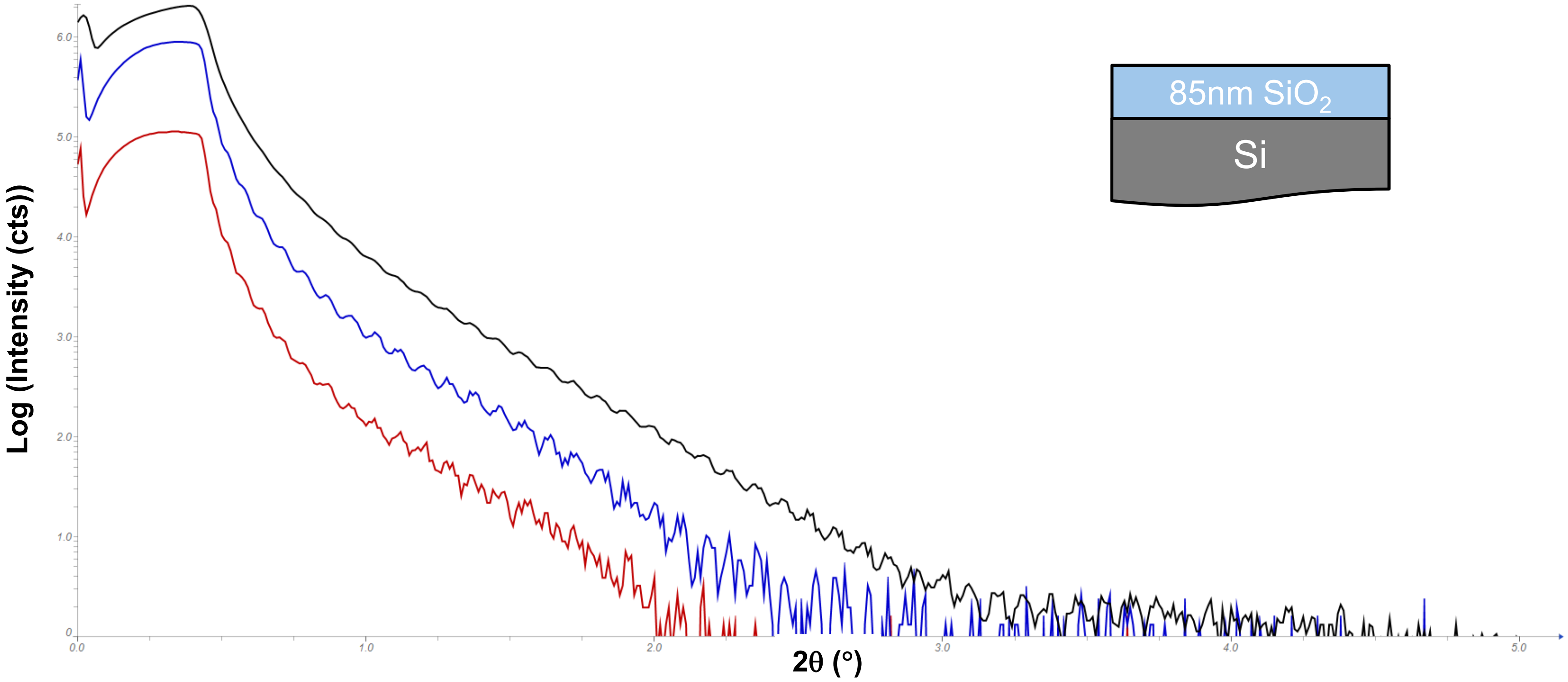


(Electron) Density gradients
modeled in two ways



Intensity Effects of Slits and Monochromator

The use of narrower slits or a monochromator to increase resolution causes significant overall intensity decreases

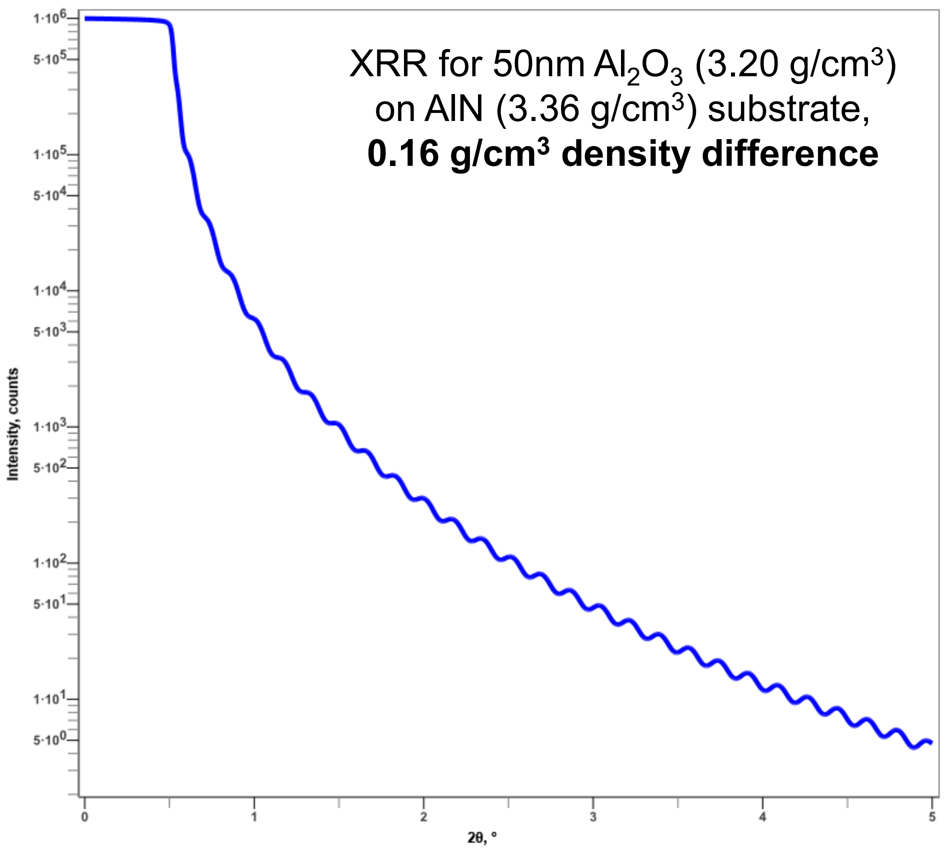


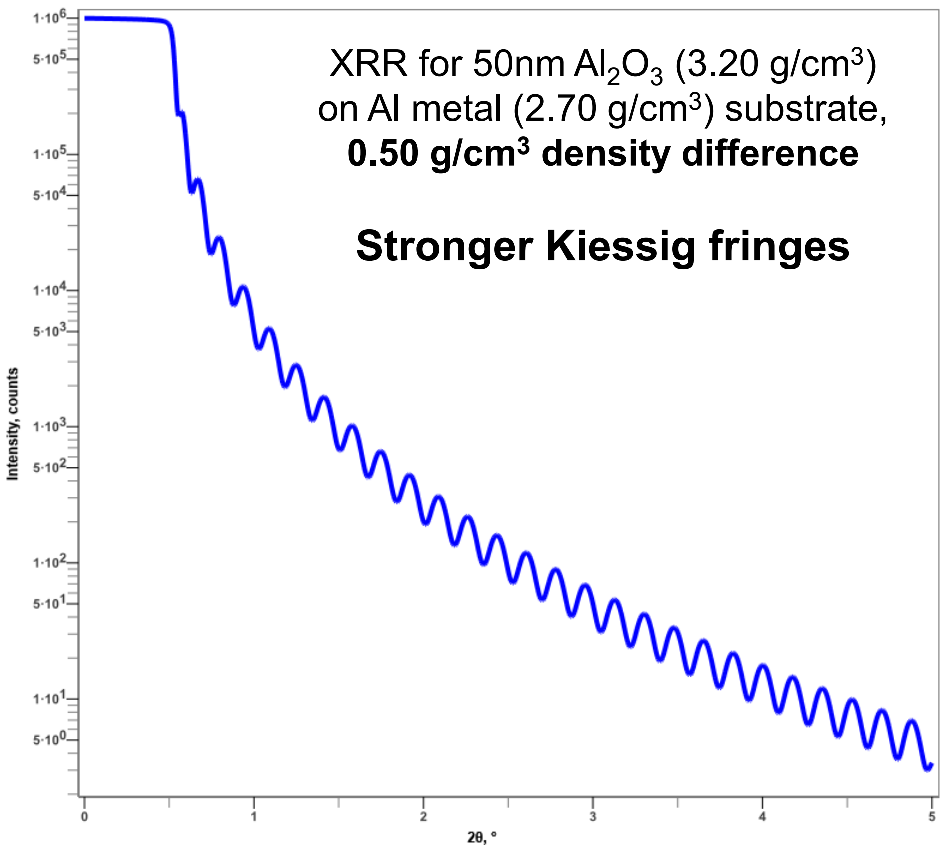
Scan Detail	Max Intensity (cps)	Relative Max Intensity (%)
Larger slits	5.40E+07	100%
Smaller slits	2.30E+07	43%
Ge(220)x2 Monochromator	2.90E+06	5%

Density Contrast and Kiessig Fringes

COVALENT METROLOGY

Greater density contrast results in stronger Kiessig fringes

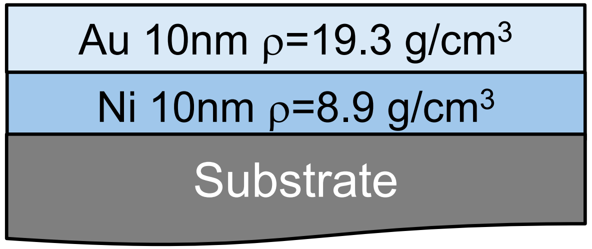




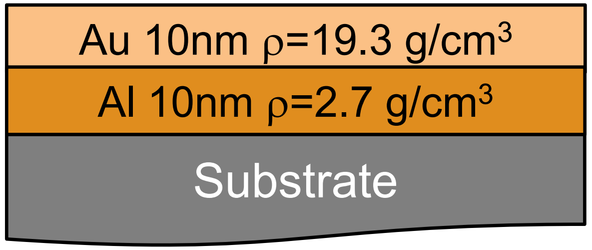
Depth of Information vs Critical Angle

COVALENT METROLOGY

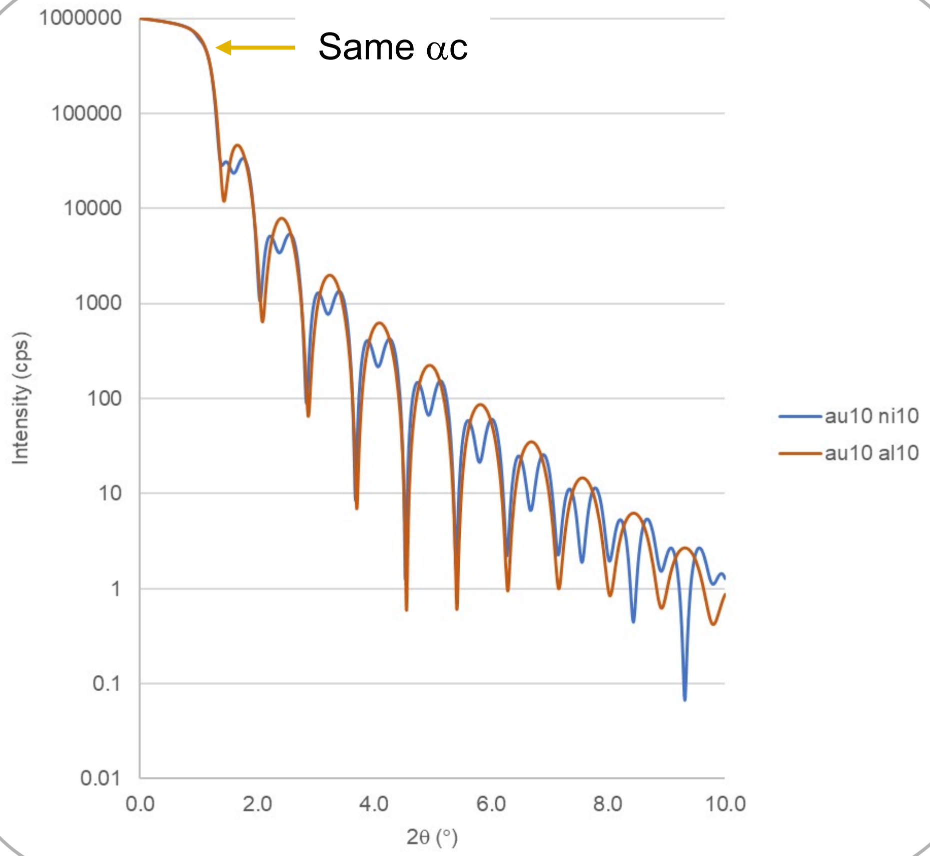
The critical angle is influenced more heavily by upper, dense layers than underlayers



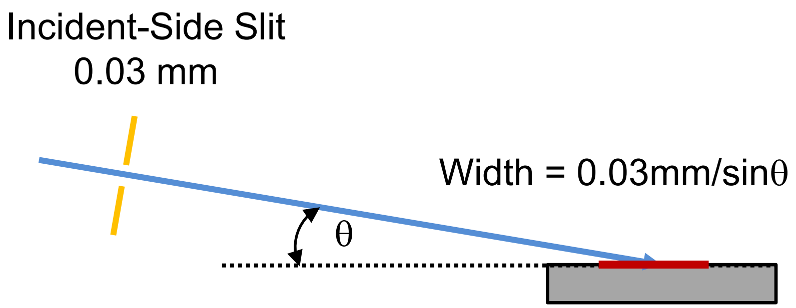
Δ Density = 10.4 g/cm³

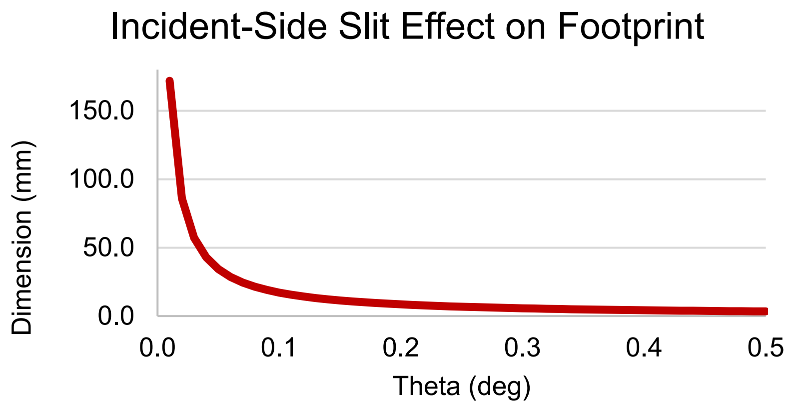


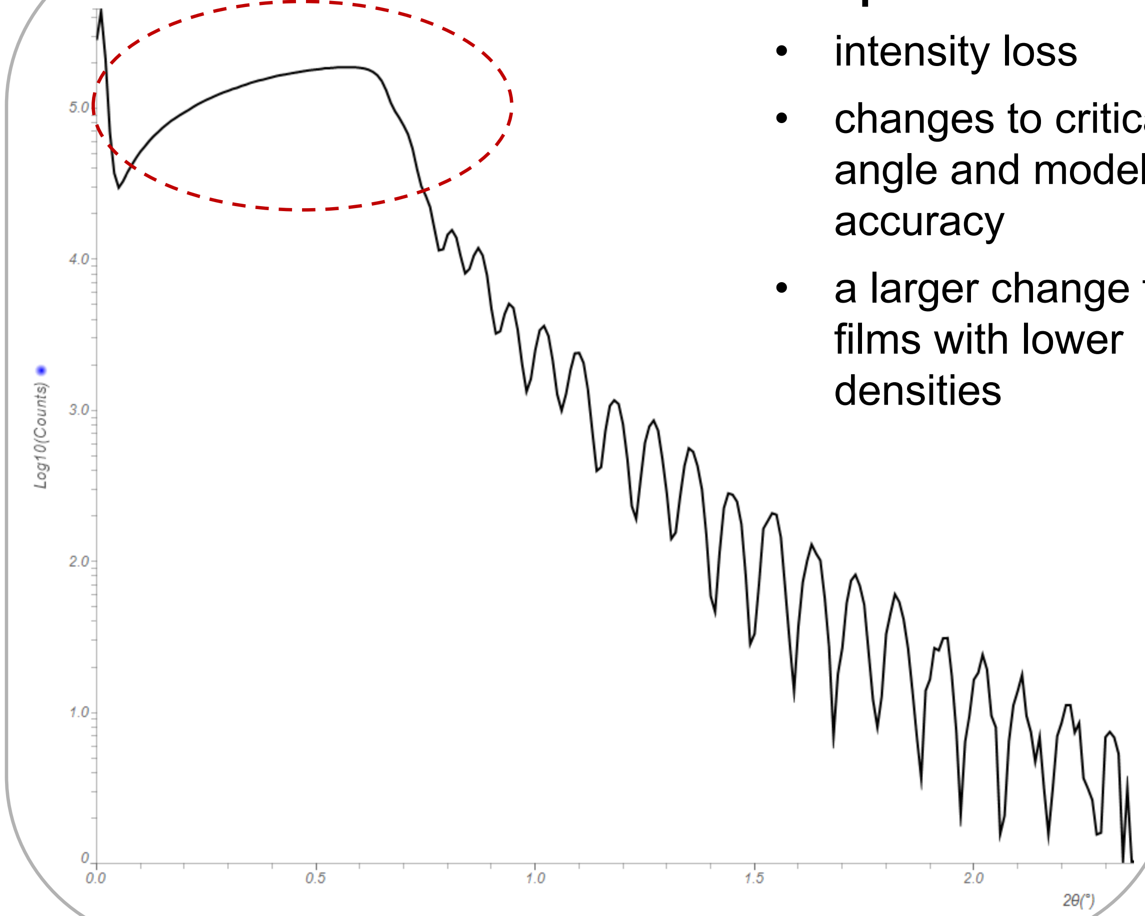
Δ Density = 16.6 g/cm³



X-Ray Beam Size on Sample





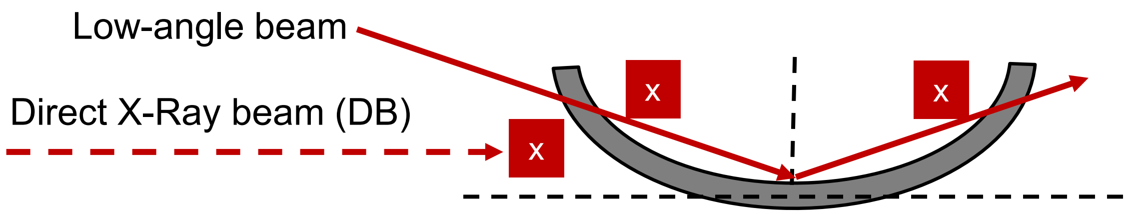


A beam larger than sample can cause:

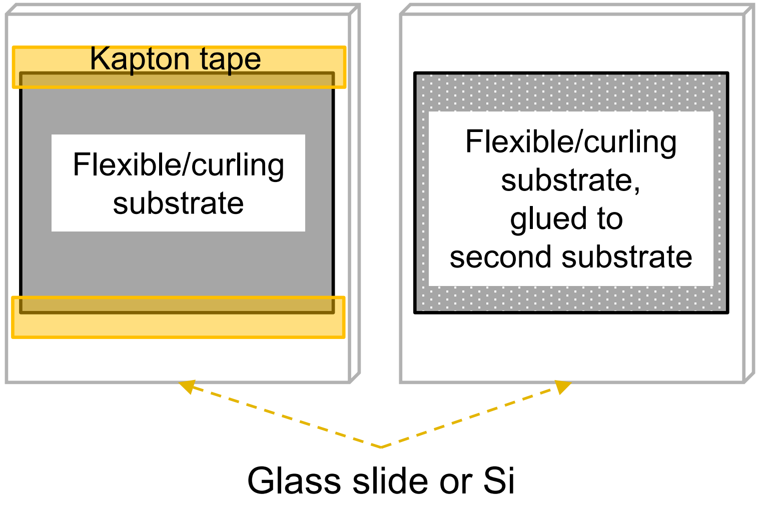
- intensity loss
- changes to critical angle and modeling accuracy
- a larger change for films with lower densities

Curling and Flexible Substrates

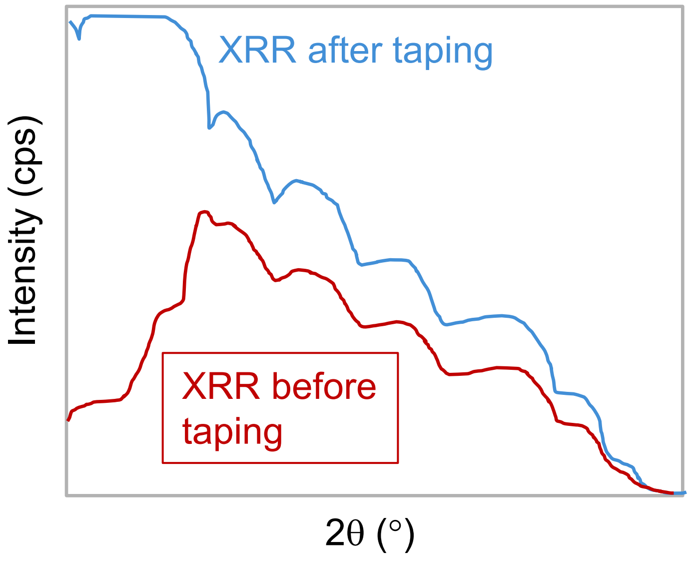
Flexible/curling substrate edges cause alignment error and can block low- angle measurements



Simple Mounting Approaches

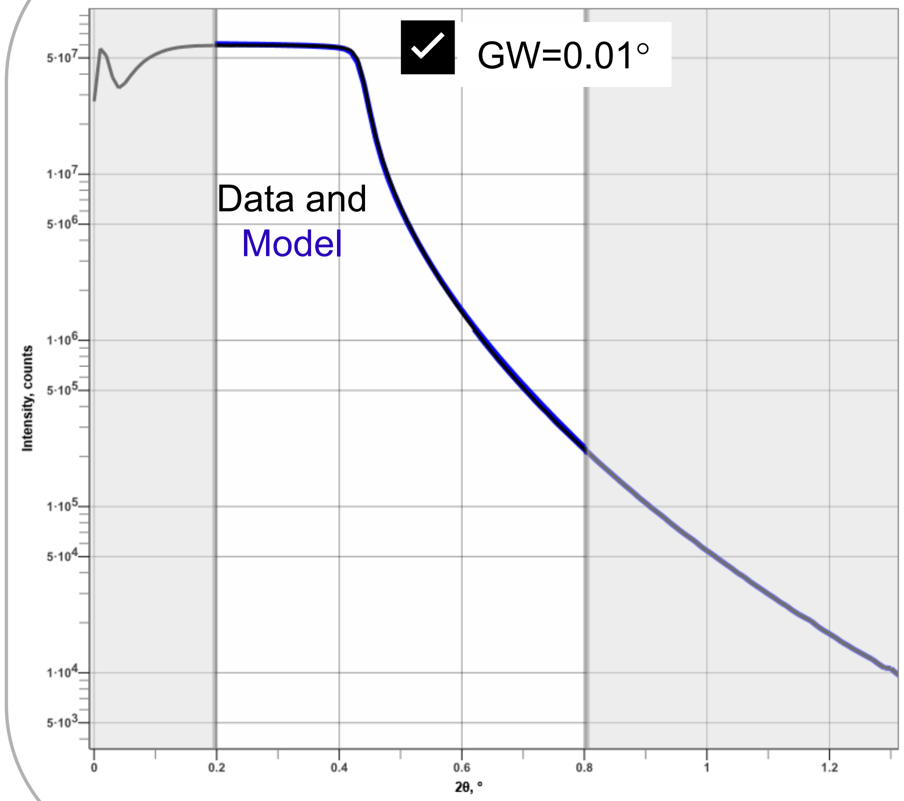


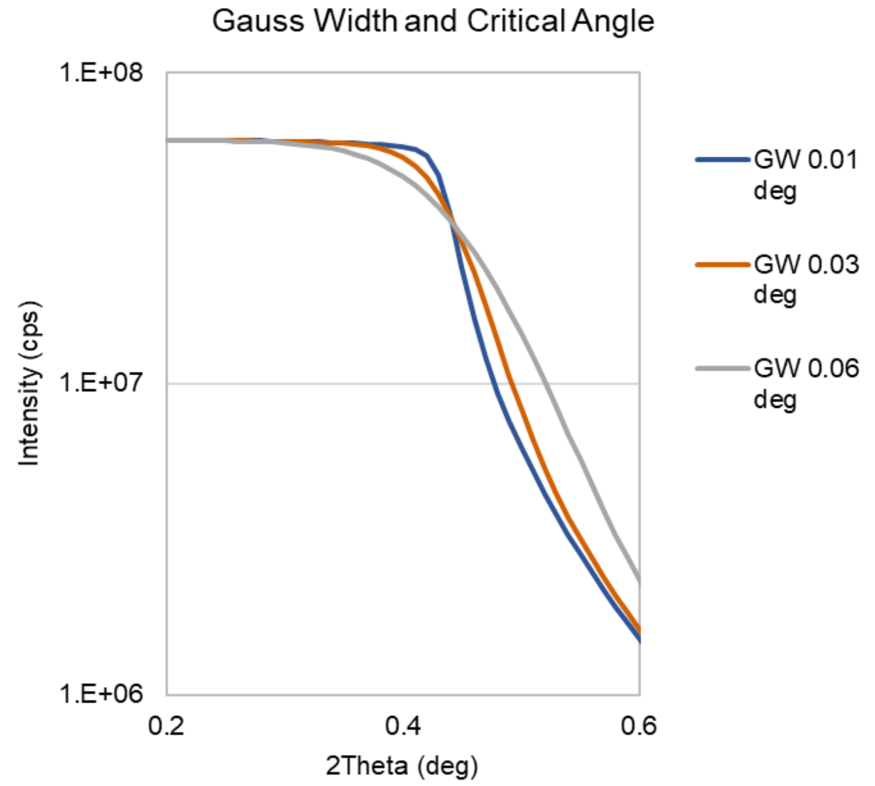
Sample with Curling Substrate



The Gaussian Width of your XRR system can be found from data from a clean, bare substrate

Fused Quartz Plate



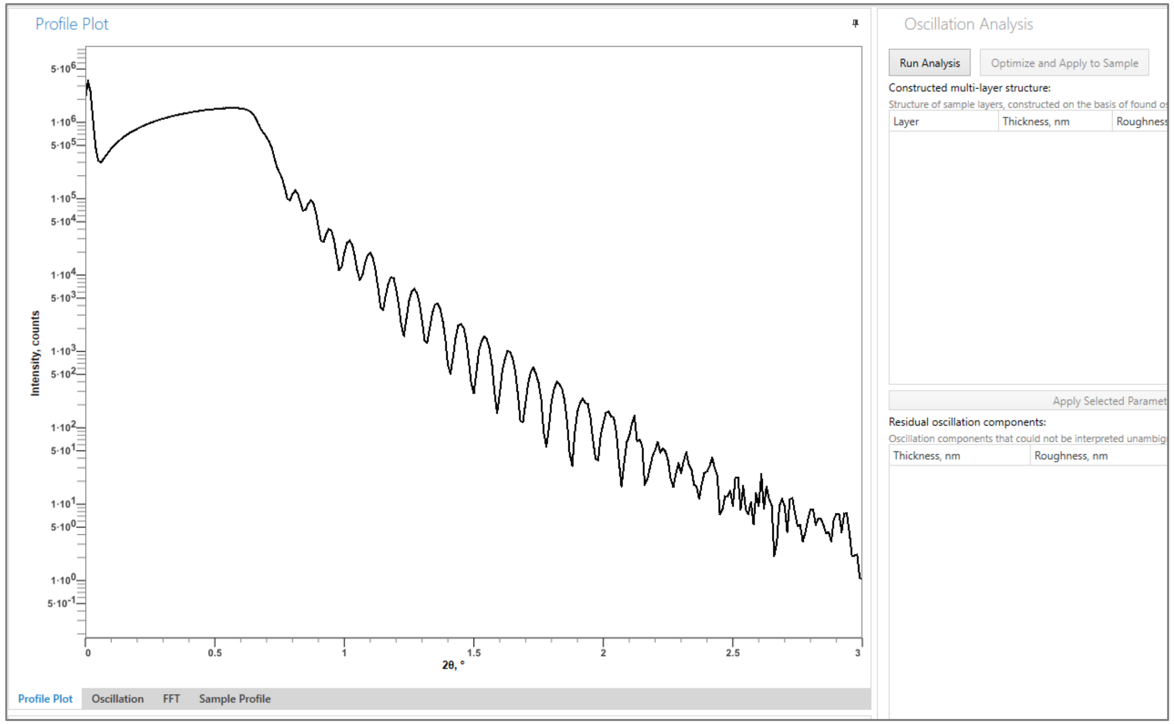


XRR Modeling Software and Inputs

COVALENT METROLOGY

Rigaku SmartLab Studio II XRR Modeling Software

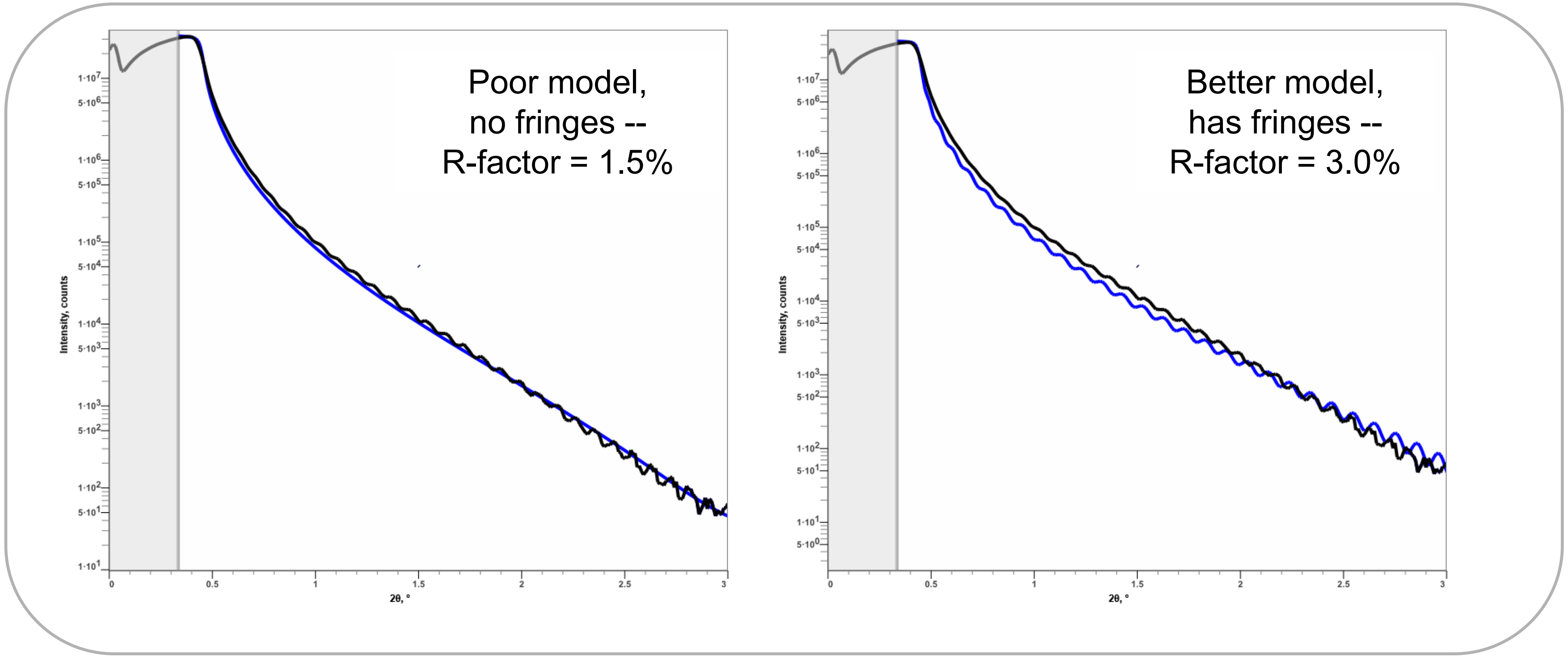
Robust program with oscillation analysis available



- **<u>Model Input</u>:**
  - Scan file
  - **Layer material (chemistry)**
  - Approximate layer thicknesses (nm)
  - Approximate layer roughnesses (nm)
  - Gaussian Width (deg)
- Fit algorithms to choose from:
  - **Genetic Algorithm**
  - Nelder-Mead
  - Quasi-Newton
- **<u>Model Output</u>:**
  - **Refined layer thicknesses (nm)**
  - **Refined layer densities (g/cm^3)**
  - **Refined layer roughnesses (nm)**
  - **Error values for layer parameters**
  - **Overlay plot of simulation and data**

Accuracy of Thicknesses, Densities, and Roughnesses

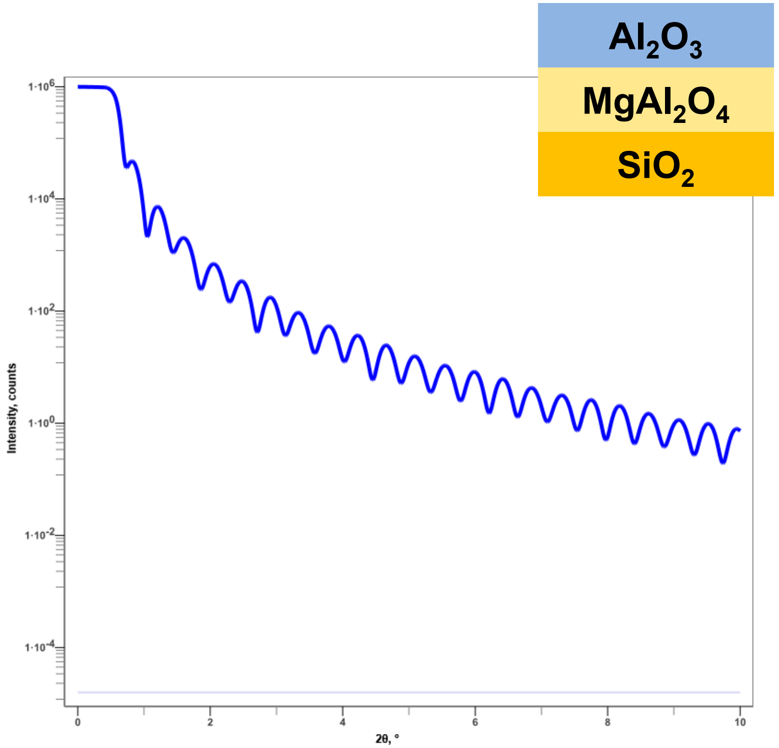
The quality of the model fit cannot be assessed purely mathematically

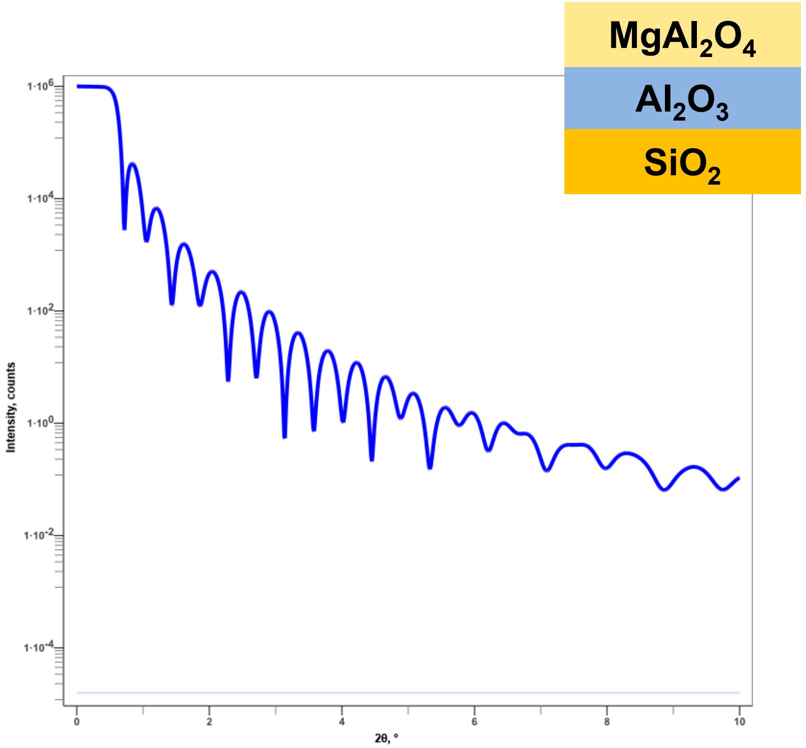


- Fitting error estimates for thicknesses, densities, and roughnesses are given by the Studio II software and are usually ± 0.01 or better
- NIST and NMIJ are working on a prototype certified XRR wafer for absolute error assessment as wafer stability and certification error ranges are issues to be solved

Effects of Layer Order

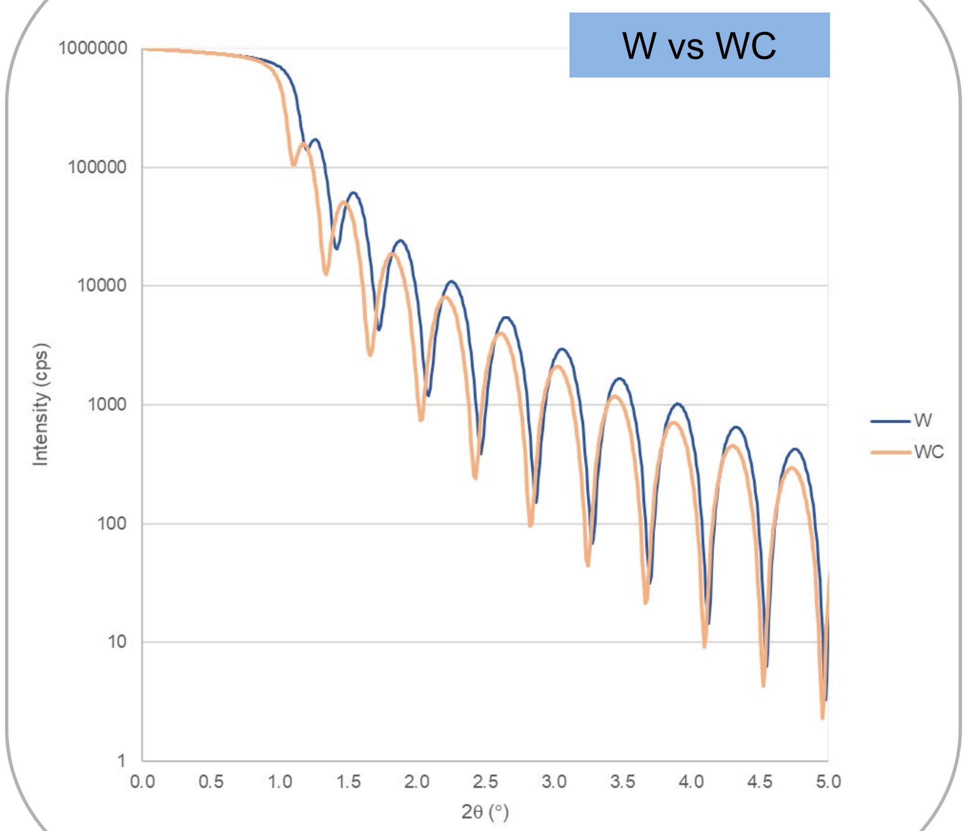
Density contrast and absorption effects change the XRR pattern when layer order is altered

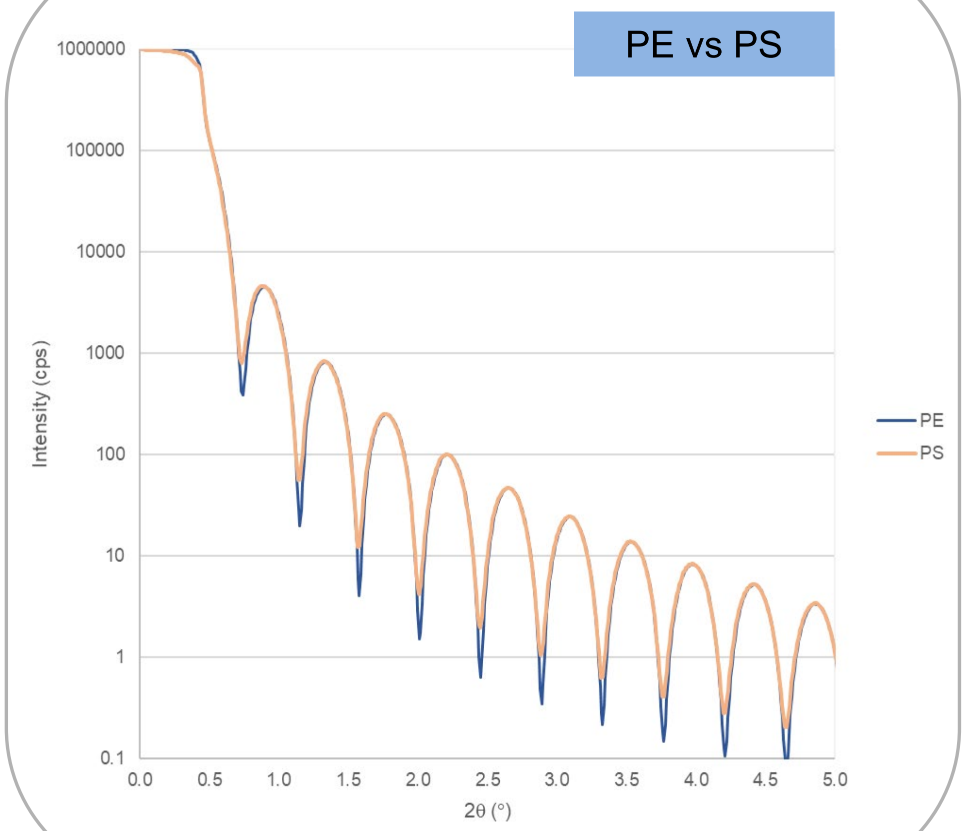




Impact of Inaccurate Layer Chemistry

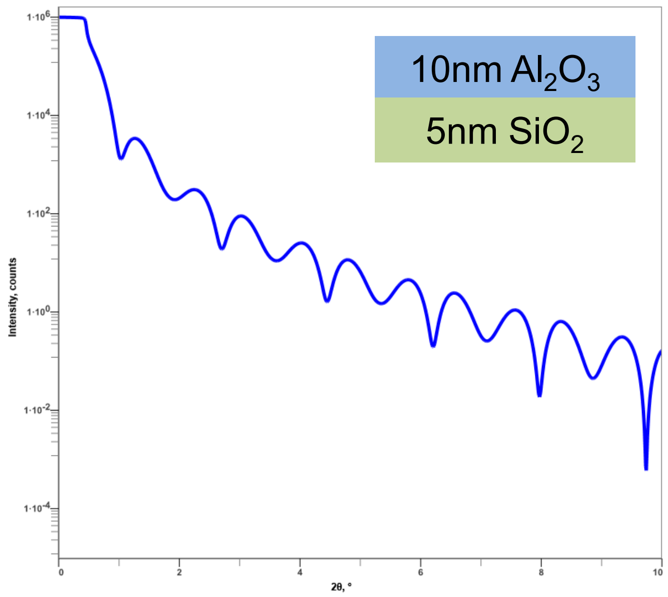
The impact of inaccurate layer chemistry depends on the materials involved

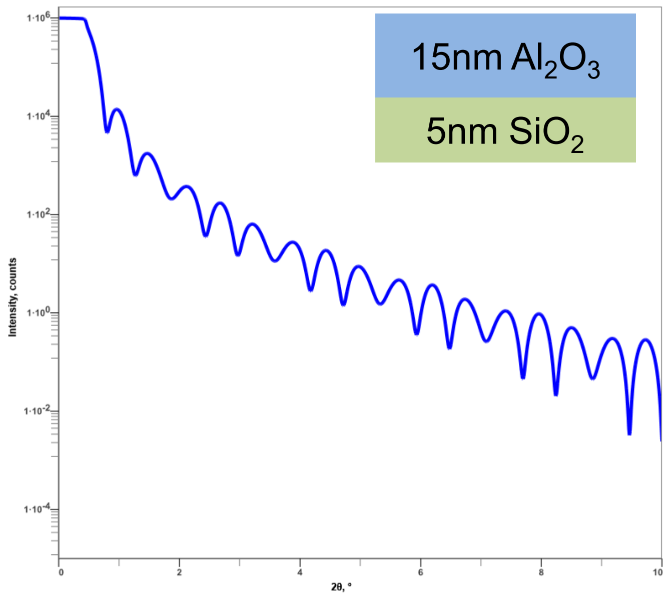


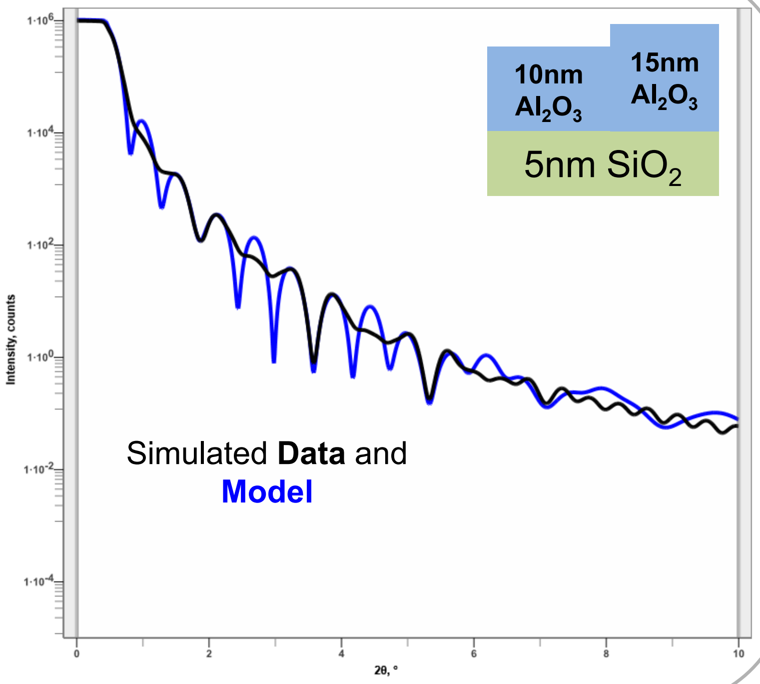


Lateral Inhomogeneity Can Be an Issue

Lateral inhomogeneity cannot be successfully incorporated into XRR models





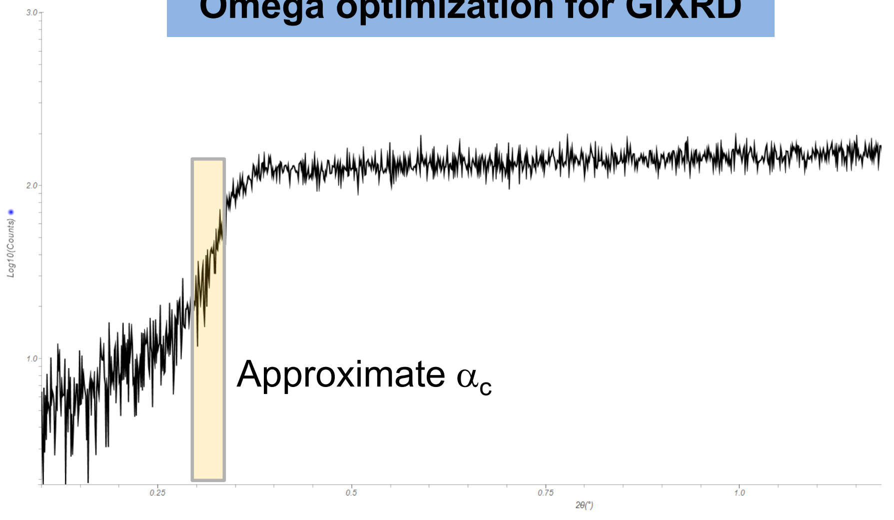


Complimentary Techniques of XRR and GIXRD

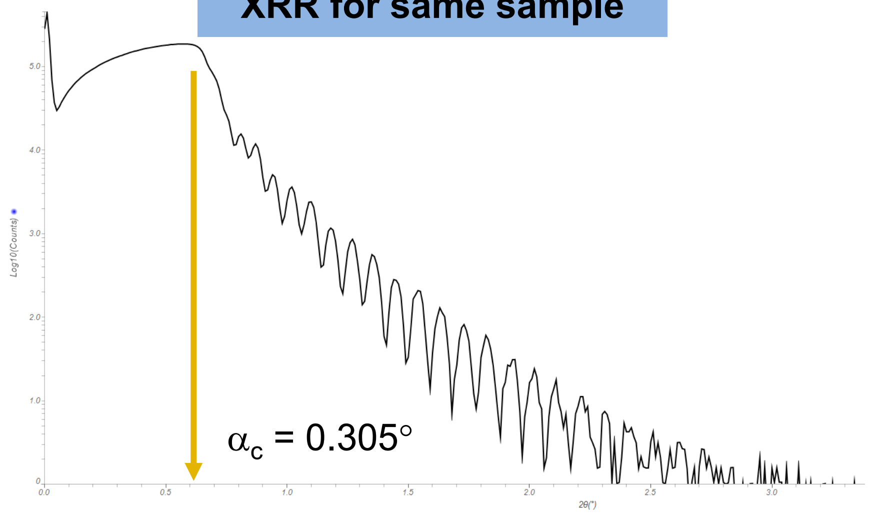
XRR can be used to:

- Determine the **critical angle (ω_c)** accurately
- Provide **evidence of film presence and thickness** regardless of crystallinity

Omega optimization for GIXRD



XRR for same sample



Troubleshooting

Identified Issue	Causes and Steps
Alignment Steps Fail	Reposition or remount sample Check Omega scan for curvature effects (may need to reduce sample size) Check roughness with visual test or profilometry (may be too rough)
XRR scan intensity too low under 0.4° 2Theta	If flexible substrate, remount (may need to glue entire sample back to substrate) If sample size is less than 20mm x 20mm, consider a larger sample Run alignment steps again Check Omega scan for curvature effects (may need to reduce sample size)
XRR scan has weak fringes	This is expected if density contrast is low Collect scan with narrower slits If film is thick (especially 400nm and greater), run using incident-side monochromator Lateral inhomogeneity may be the cause
XRR scan is without fringes	Collect scan with narrower slits If film is thick (especially 400nm and greater), run using incident-side monochromator Check that there is a film present Lateral inhomogeneity may be the cause

Acknowledgements

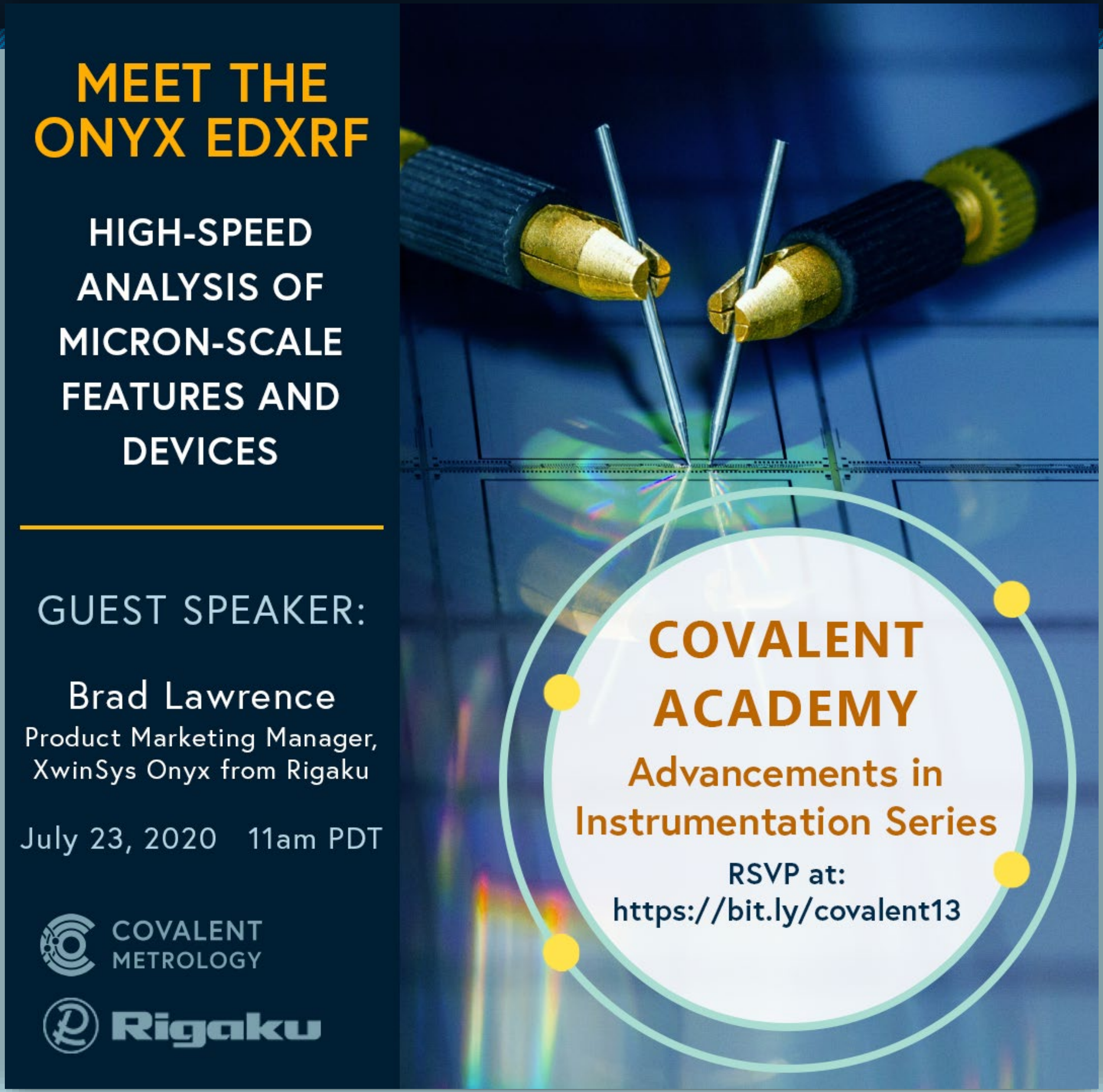
Appreciation and thanks to:

Rigaku

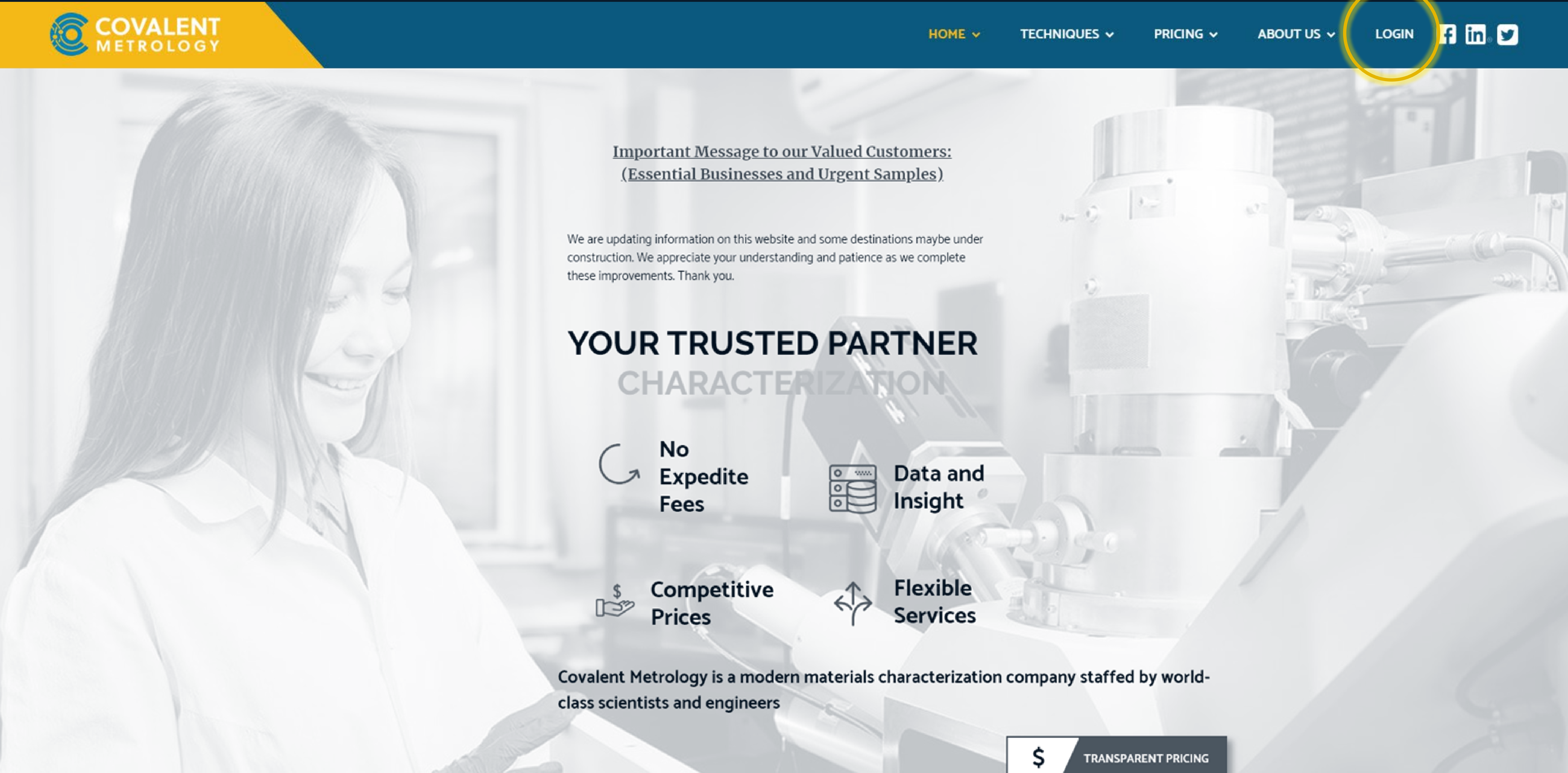
Alluxa

and my colleagues at COVALENT METROLOGY

Up next on Covalent Academy…



Covalent Community



Covalent Community

COVALENT METROLOGY

Data Portal

Customer Access to Data & Community Content

The DATA PORTAL is used by Customers and Lab Partners for uploading and downloading data. It requires two-factor authentication and advanced password protection. Data Portal users have complete access through their home page on the portal to all Community content, <u>and do not require a separate Community account</u>.

Covalent Community

All Other Users

The COVALENT COMMUNITY PORTAL requires password entry. It contains webinar and other metrology and characterization-related content that we believe would be useful and educational for the materials science innovation community. It does <u>not</u> provide access to any customer data and should only be used by individuals that are not Covalent customers or lab partners.

COVALENT METROLOGY

covalentmetrology.com