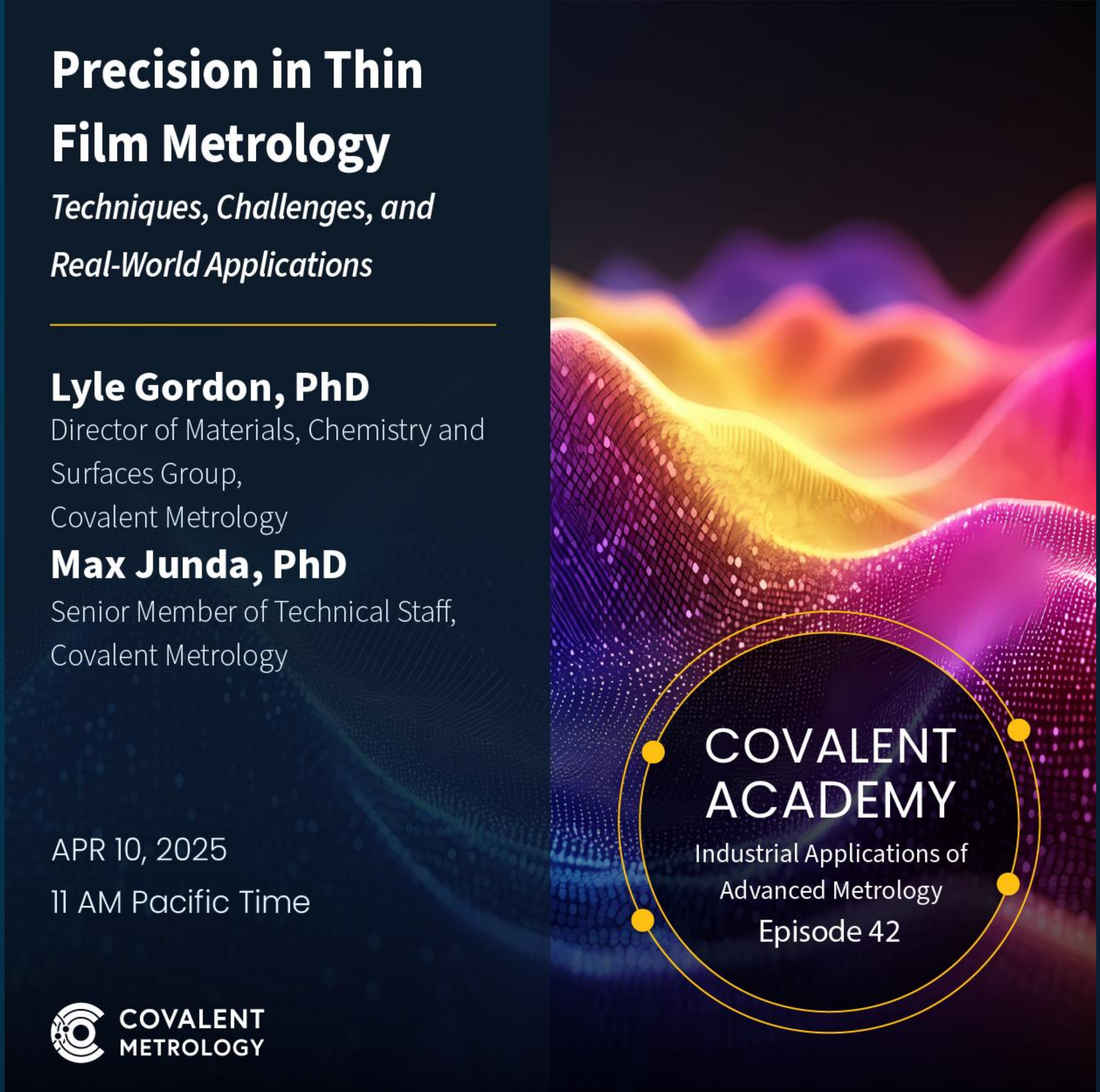
Welcome

to today's episode:



Precision in Thin Film Metrology

Techniques, Challenges, and Real-World Applications

Lyle Gordon, PhD
Director of Materials, Chemistry and Surfaces Group,
Covalent Metrology

Max Junda, PhD
Senior Member of Technical Staff,
Covalent Metrology

APR 10, 2025
11 AM Pacific Time

COVALENT METROLOGY

COVALENT ACADEMY
Industrial Applications of Advanced Metrology
Episode 42

Introducing today's speakers

Dr. Lyle Gordon

Director of Materials, Chemistry & Surfaces Group,
Covalent Metrology

Dr. Gordon joined Covalent in 2023 where he manages the Materials, Chemistry and Surfaces group, overseeing X-ray metrology, analytical chemistry, and surface science. He has extensive expertise in time-of-flight mass spectrometry, particularly related to his work on atom probe tomography. Dr. Gordon completed his Bachelor of Applied Science at the University of Toronto, his PhD in Materials Science at Northwestern University, and was a postdoctoral fellow for the Department of Energy at Pacific Northwest National Lab.

Dr. Max Junda

Senior Member of Technical Staff,
Covalent Metrology

Dr. Junda joined Covalent in 2019 to grow and develop the company's ellipsometry offerings. Since then, he has helped provide measurement, modeling, and consulting services to over 110 separate customers across many industries. Max's particular interest is in developing customized data collection and optical modeling methods that expand the areas where ellipsometry can be used beyond typical film thickness and optical property measurement. He enjoys working directly with customers to understand their specific problems and data needs so that the experiments can be tailored to each application.

COVALENT
METROLOGY

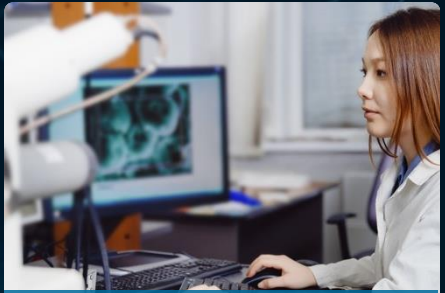
Modern, digitally-empowered analytical services platform delivering quality data and expert analysis to accelerate advanced materials and device innovation.

Comprehensive Solutions Stack

- *50+ Cutting-edge instruments in-house, 150+ Techniques*
- *Analytical Services*
- *Advanced Modeling*
- *Method Development*
- *Custom Consulting Solutions*

High-touch, High-Quality Services

- *Ionic Membership Program*
- *Enterprise Metrology Solutions*
- *Instant, Secure Access to Data and Reports*
- *Expanding Toolkit in Custom Digital Platform*



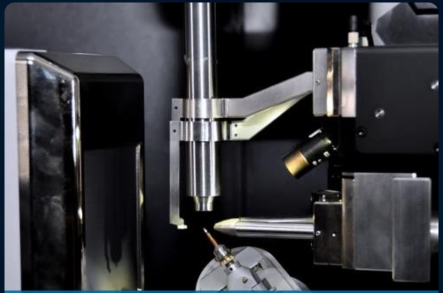
Flexible Business Models

- *LiveView™ (real-time collaboration)*
- *Co-op and Tool-share Opportunities*
- *Training and Certification on Instrumentation*
- *Laboratory Audits*

Rigaku
Anton Paar
ThermoFisher SCIENTIFIC
ASYLUM RESEARCH an Oxford Instruments company
KEYENCE
J.A. Woollam
TA Instruments
PVA TePla
TELEDYNE CETAC TECHNOLOGIES Everywhereyoulook™
HORIBA Scientific
Digital Surf

Rich Network of Partnerships

- *Partner to World's Leading Instrument Manufacturers and Labs*
- *Expanding access to Advanced Instruments and Analysis Tools*
- *Lab Connections and Applications Learning*



Who We Are, Who We Serve

- *50+ People, 14 PhDs*
- *Comprehensive, Modern Analytical Capabilities*
- *Headquarter Lab in Sunnyvale, CA*
- *600+ Clients, 15-30 new clients / week*

Covalent's Analytical Services & Technical Groups

Enterprise Metrology Solutions: Failure Analysis & EM

Electron Microscopy

- S/TEM
  with EDS; EELS; Electron Diffraction; SAED
- FIB-SEM & HR-SEM
  with EDS; EBSD; 3D Tomography
- Lamella Preparation
  incl. specialized lift-outs

Failure Analysis

- DPA / Mechanical X-section
- Dye & Pry Test
- EBIC / OBIC failure analysis
- Hot Spot Detection
- NIR / IR Imaging
- Emission Microscopy
- Root-Cause Failure Analysis

Materials, Chemicals, and Surfaces

Microscopy & Profilometry

- Chromatic Aberration
- Digital Optical Microscopy
- Laser Scanning Confocal Microscopy
- White Light Interferometry
- Scanning Acoustic Microscopy (SAM)

Analytical Chemistry

- Mass Spectroscopy:
  ICP-MS and LA-ICP-MS; GCMS
- ICP-OES / GDOES
- Raman
- NMR (solid / liquid + 1,2,3 nuclei)
- XPS, UPS, ISS
- SIMS, TOF-SIMS

X-ray Characterization

- X-Ray Diffraction (XRD)
- X-Ray Reflectometry (XRR)
- Micro-computed X-ray Tomography (Micro-CT)
- 2D / 2.5D / 3D X-ray Inspection & X-ray Radiography
- ED-XRF / WD-XRF

Mechanical Testing

- AFM & Advanced AFM Modes (EFM, KPFM, MFM, PFM, PiFM)
- Nano-indent / Nano-scratch
- Rheometry / Viscosity
- DMA / TMA (bend/stretch/compression)
- Tensile testing

Misc. Material Properties

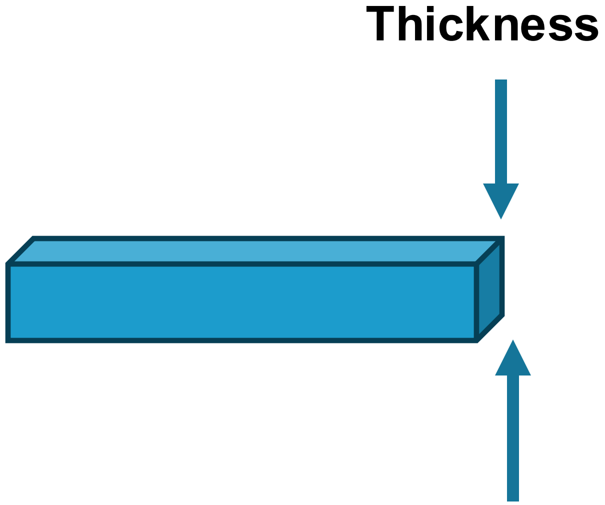
- Thermal Analysis: DSC, TGA
- Surface Zeta Potential
- Porometry / Pycnometry
- Gas Adsorption / Chemisorption
- Foam Density / Skeletal Density / Tap Density
- Particle Analysis: DLS / ELS / size distribution / zeta potential

Optical Characterization

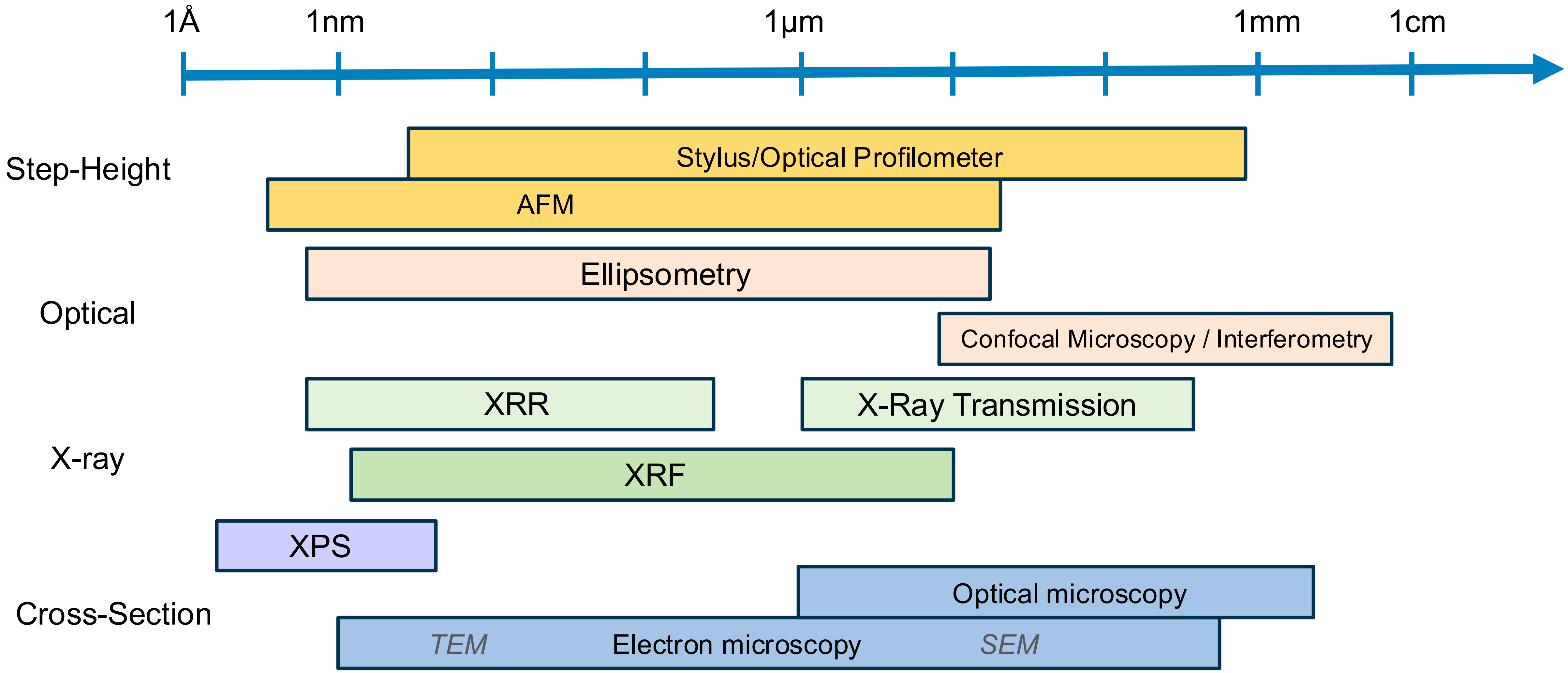
- Fourier Transformed Infrared Spectroscopy (FTIR and ATR-FTIR)
- **Spectral Ellipsometry & Advanced Optical Modeling**
- UV-Vis-NIR Spectroscopy

Thickness Measurements

- Film thickness plays a critical role in many applications
  - Corrosion resistance
  - Semiconductor devices
  - Optical and electrical properties
  - And many more…
- Thickness measurements are widely used
  - Optimize and control etching and growth processes
  - Understand functional materials
  - Reverse engineering
- Applicability and limitations of selected techniques
  - using a few **case studies** and **practical examples** from different industries
- Q&A with Covalent Experts



Thickness varies over many orders of magnitude



1Å
1nm
1µm
1mm
1cm

Step-Height
Stylus/Optical Profilometer
AFM

Optical
Ellipsometry
Confocal Microscopy / Interferometry

X-ray
XRR
X-Ray Transmission
XRF
XPS

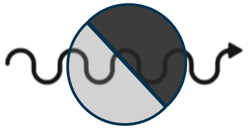
Cross-Section
Optical microscopy
TEM Electron microscopy *SEM*

Not exhaustive list

How to measure – Key deciding factors

1. Substrate exposed (or masked)

2. Optically transparent



3. Prior knowledge for modeling

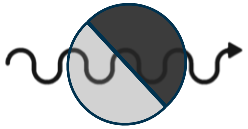
?

4. Everything else

How to measure – Key deciding factors

→ 1. Substrate exposed (or masked)

2. Optically transparent



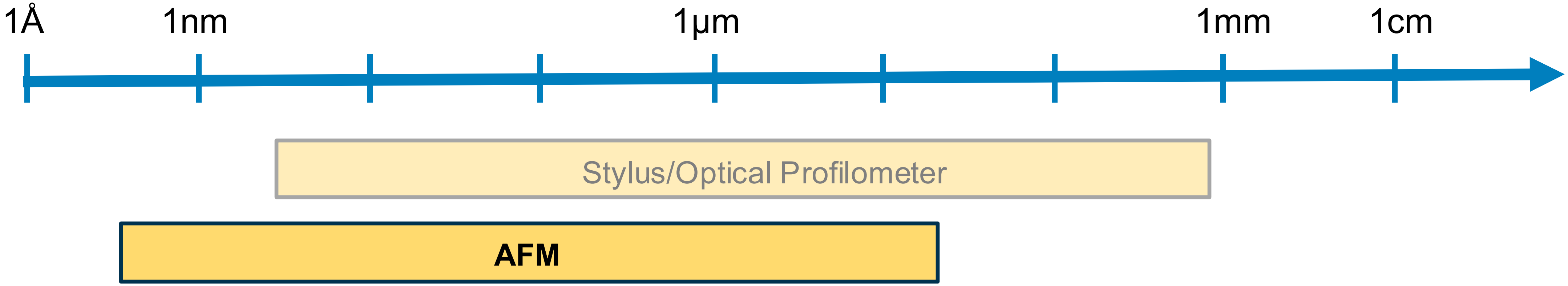
3. Prior knowledge for modeling

?

4. Everything else

Step Height

- What is the expected film thickness?



Step Height

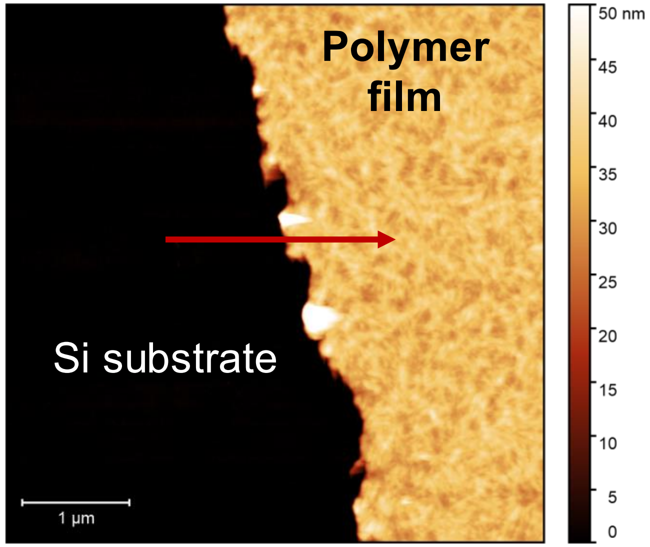
- Narrow applicability: requires exposed substrate
  - Simple and widely used
  - If you are growing the film, you may be able to mask an area of the substrate
- Measure the height difference from the top of the film to the substrate
  - Optical or stylus profilometry for thicker films, AFM for very thin

Additional Info
Surface roughness

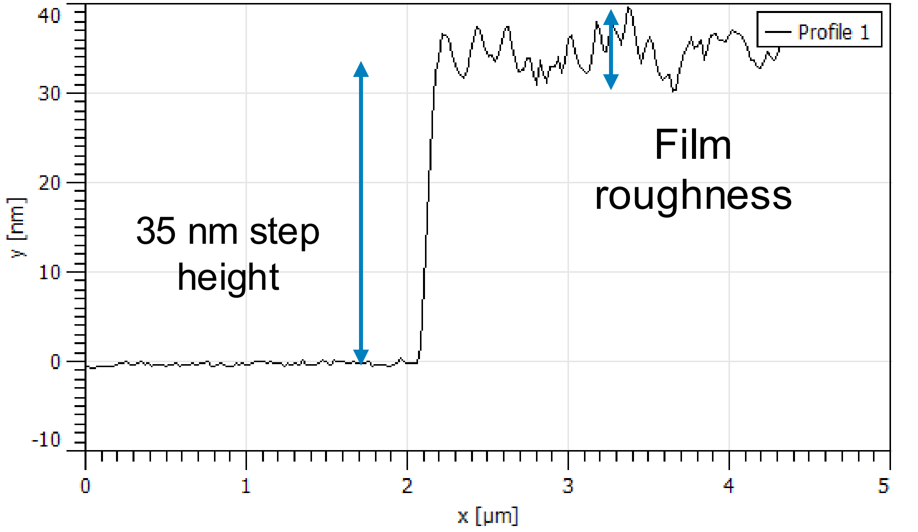
Example:
Soft polymer film was scratched away from a region of the substrate

Limitations:

- Requires exposed substrate



AFM results from Max Schrock

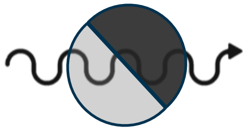


Method	Applicability	Benefits	Limitations
Step Height	Films with exposed substrates	Simple and direct measurement	Requires exposed substrate, stylus can scratch sample

How to measure – Key deciding factors

1. Substrate exposed (or masked)

→ 2. Optically transparent



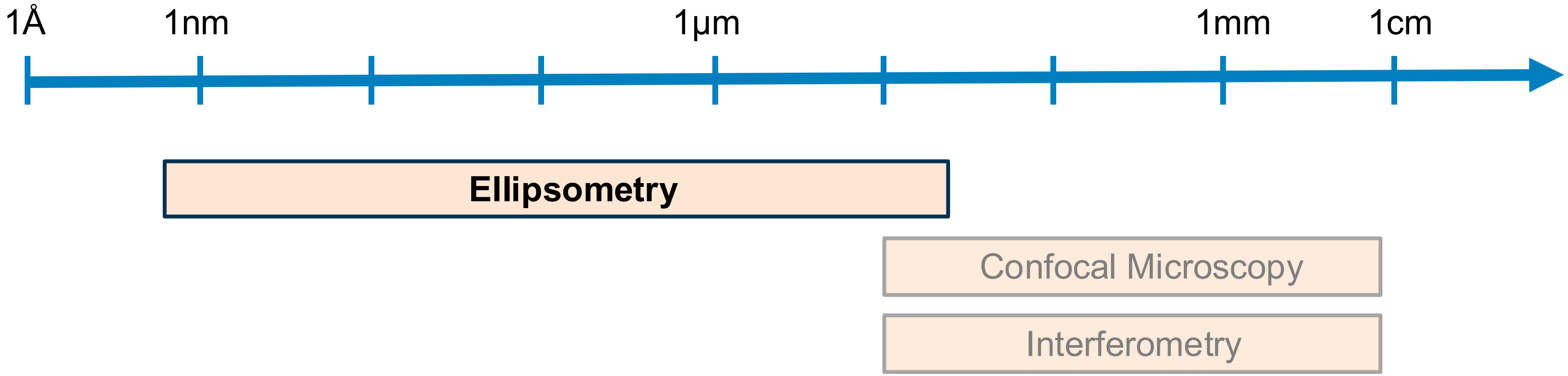
→ 3. Prior knowledge for modeling

?

4. Everything else

Optically transparent films

- What is the expected film thickness?



1Å

1nm

1µm

1mm

1cm

Ellipsometry

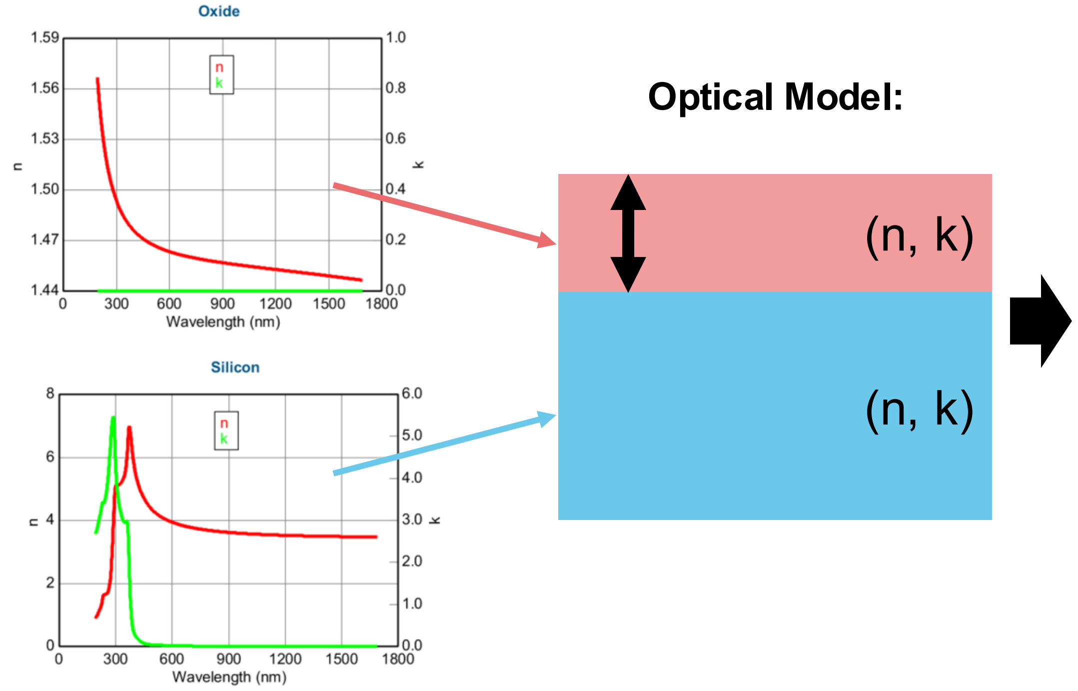
Confocal Microscopy

Interferometry

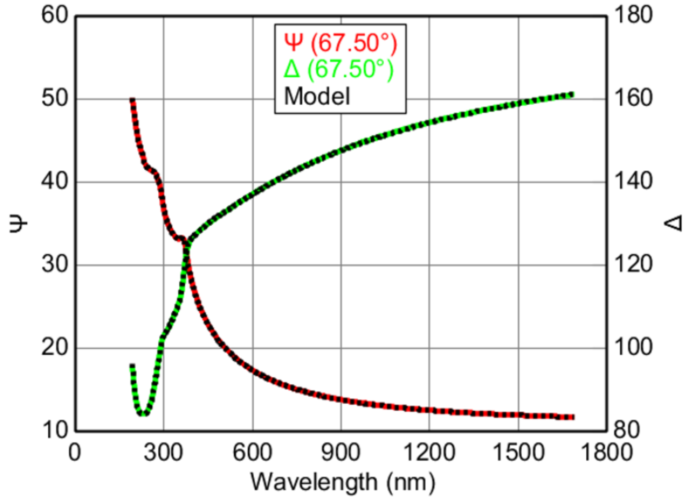
Ellipsometry Overview

Optical Property Spectra:

Optical Model:



Fit to Measured Raw Data:



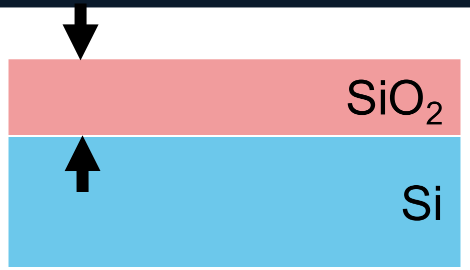
Limitations and Feasibility Vary For Different Cases:

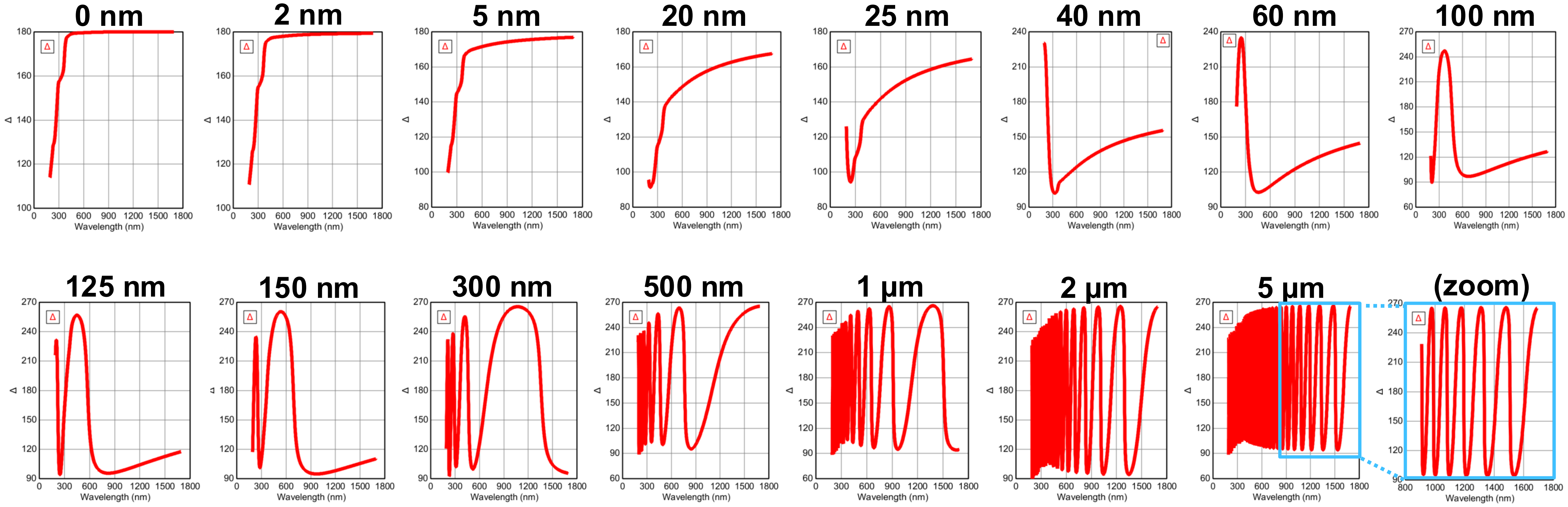
- Very thin films (<5 nm)
- Very thick films (>5μm)
- Absorbing films (e.g. metals)
- Multilayer Stacks

Method	Applicability	Benefits	Limitations
Ellipsometry	Somewhat optically transparent films	Quick, non-destructive, measure optical properties, mapping	Needs transparency and knowledge of the layers for **modeling**

Ellipsometry Thickness Sensitivity: Thin Film Interference

Example of raw ellipsometry data with varying film thickness




0 nm

2 nm

5 nm

20 nm

25 nm

40 nm

60 nm

100 nm

125 nm

150 nm

300 nm

500 nm

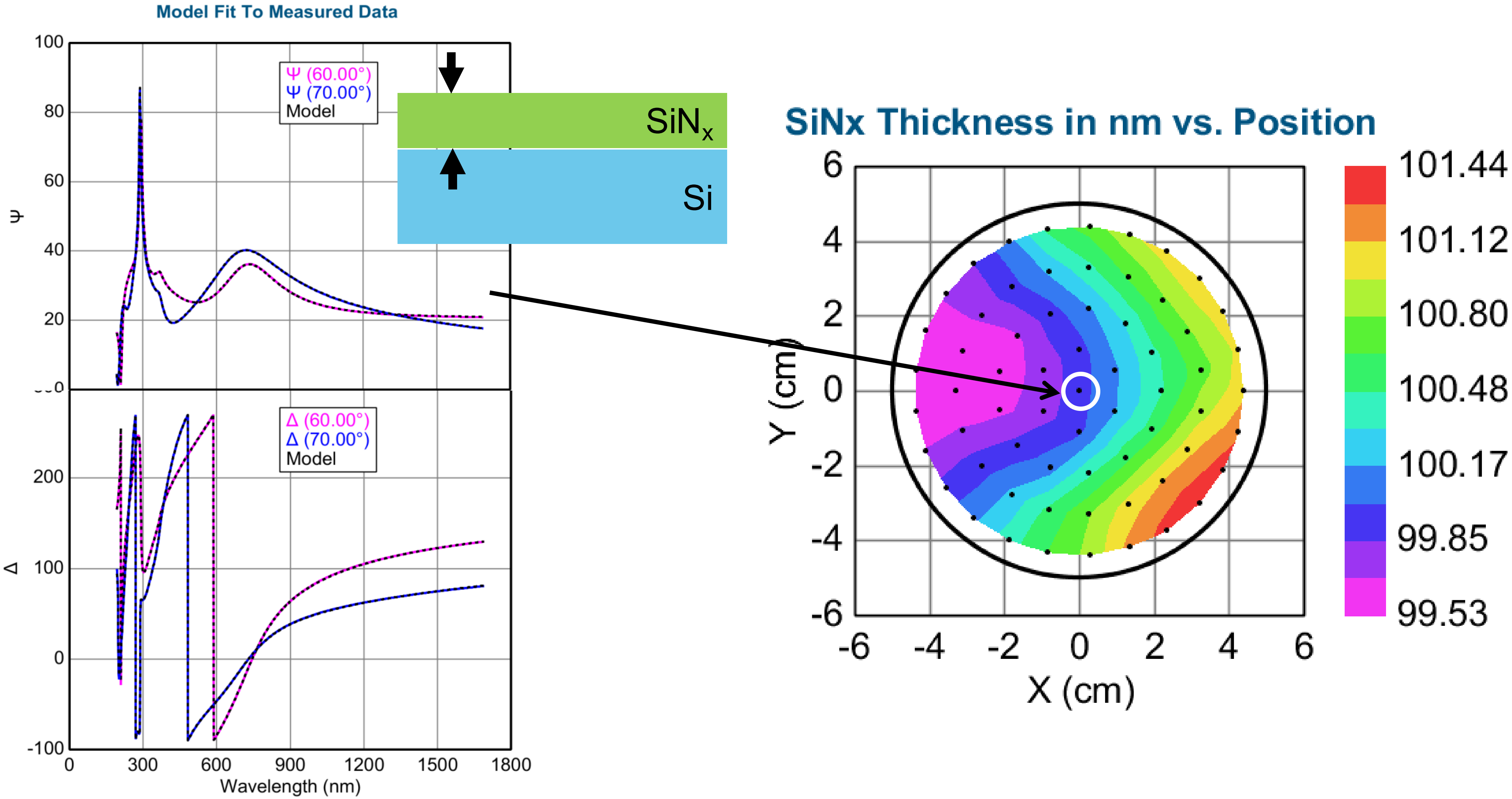
1 µm

2 µm

5 µm

(zoom)

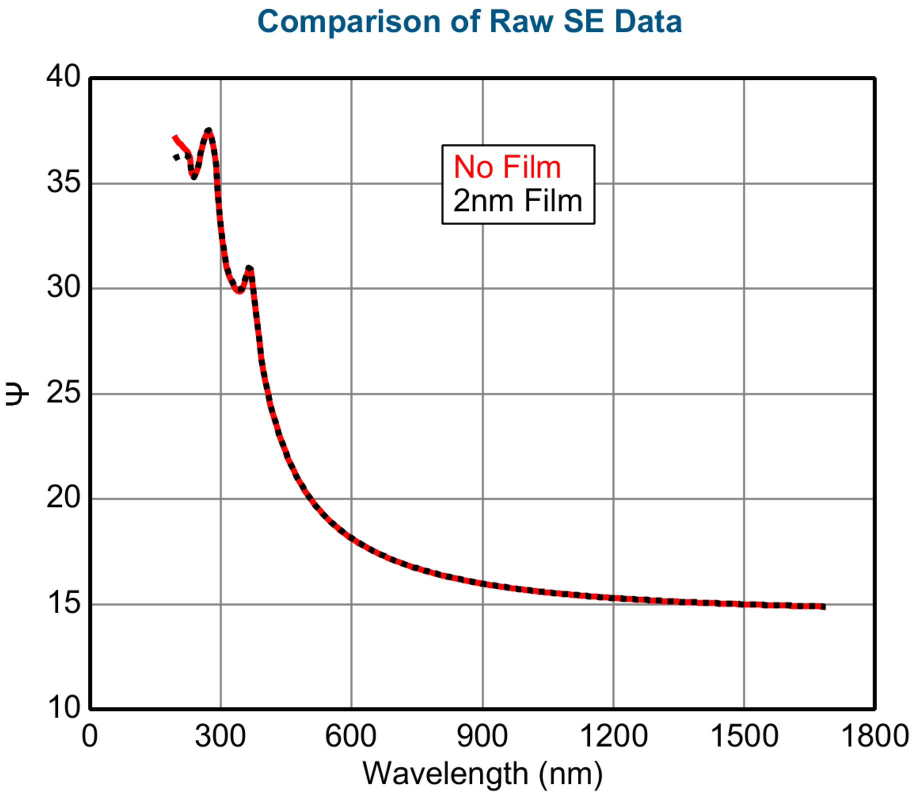
Basic Example: SiN on Si Wafer

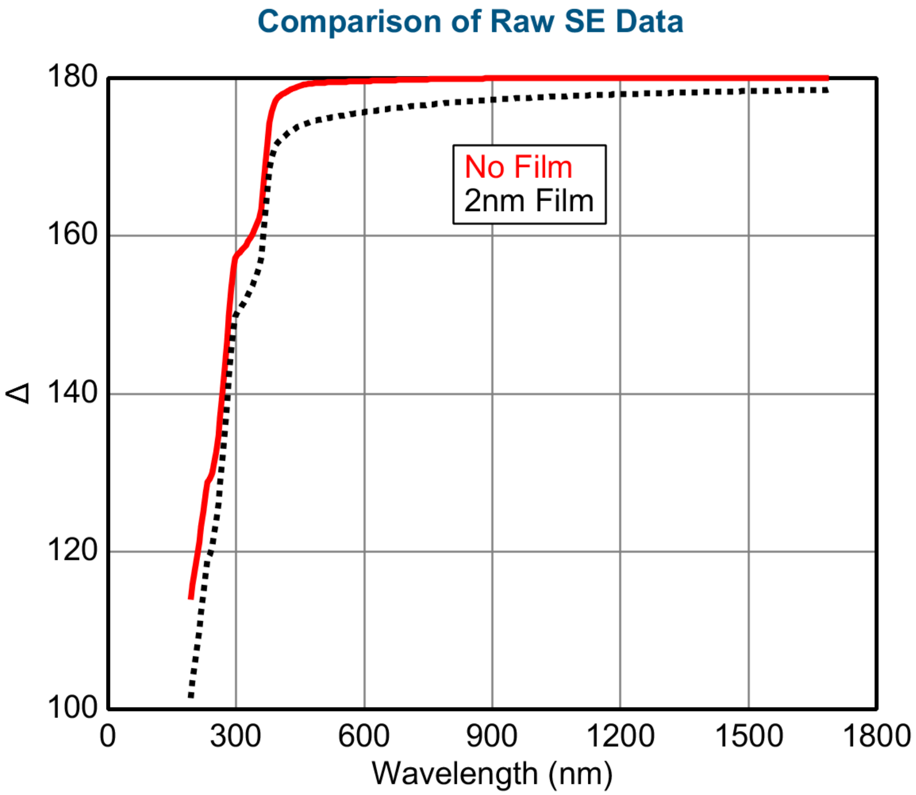


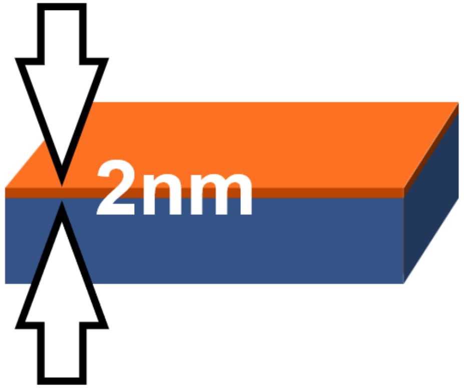
Model Fit To Measured Data

SiNx Thickness in nm vs. Position

Very Thin Film (<5 nm)



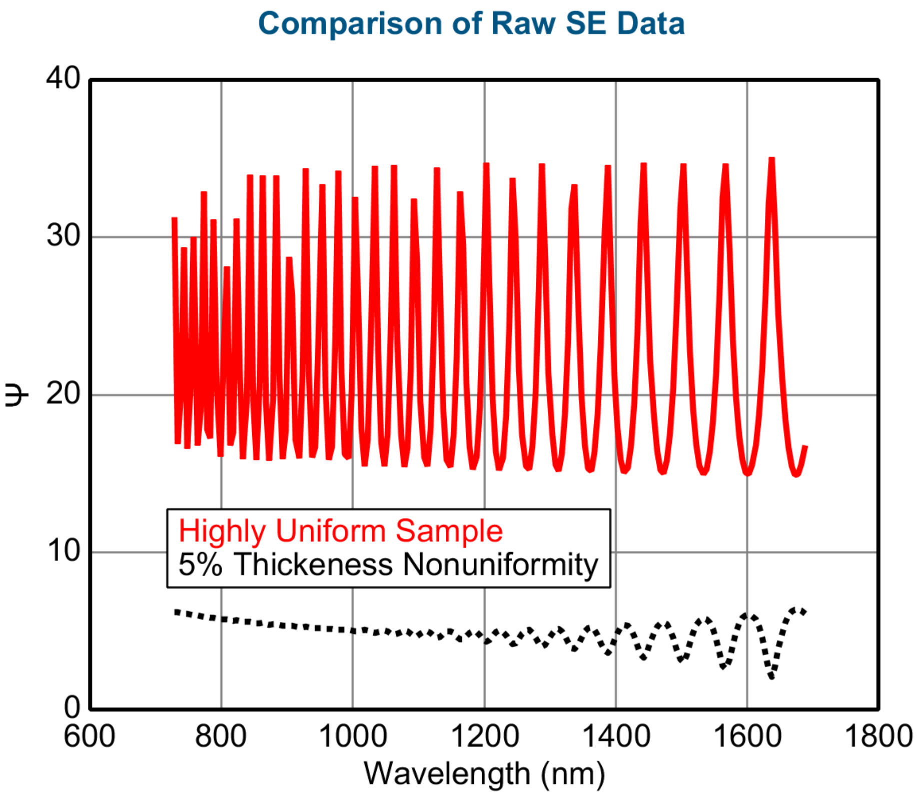


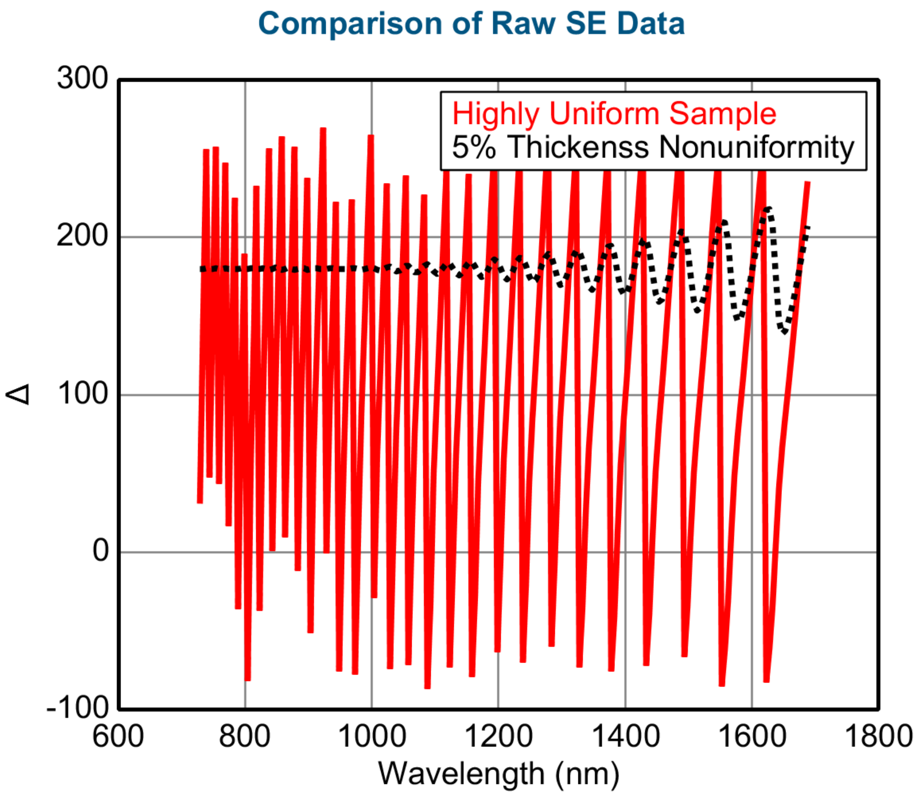


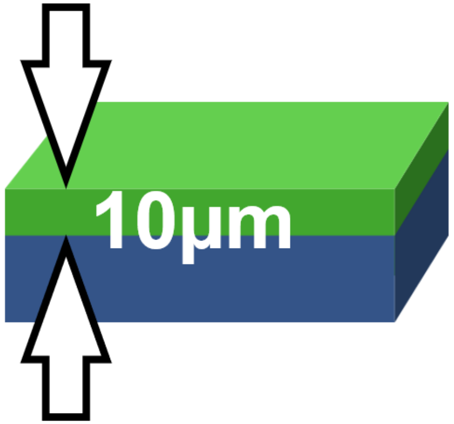
Limitations:

- Negligible sensitivity to film index of refraction.
- Film and substrate indices of refraction must be sufficiently different from each other.

Very Thick Film (>5 µm)



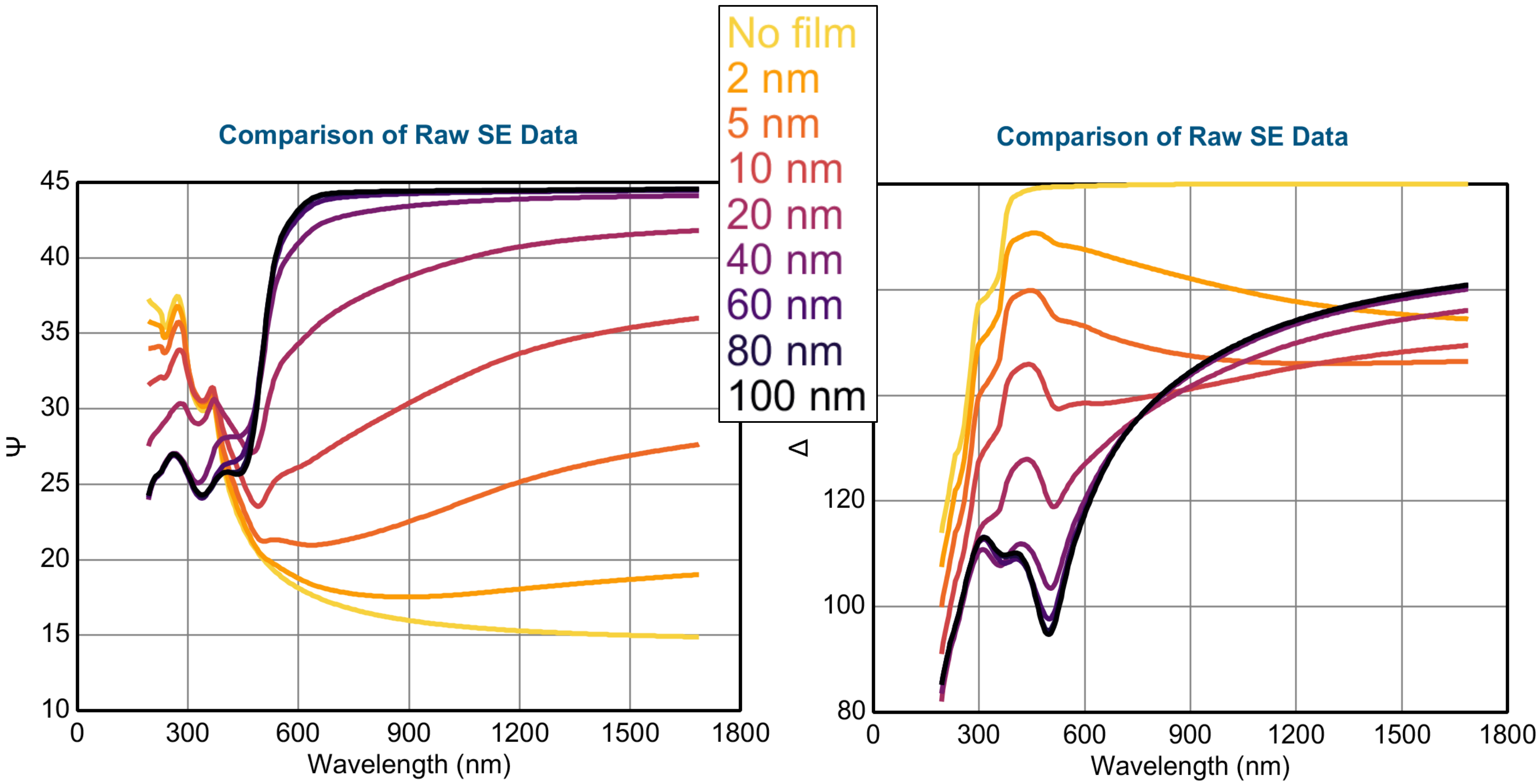




Limitations:

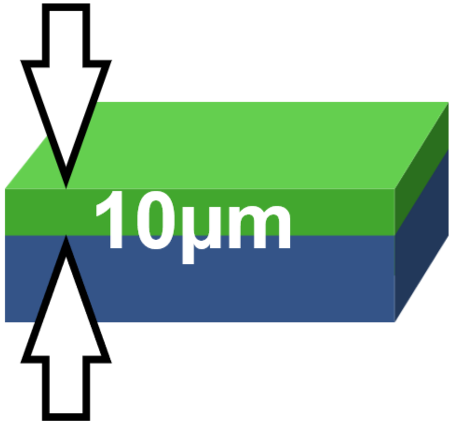
- Accurate optical modeling can be complicated.
- Highly sensitive to any nonuniformities or inhomogeneities.

Thin Metal Films



Comparison of Raw SE Data

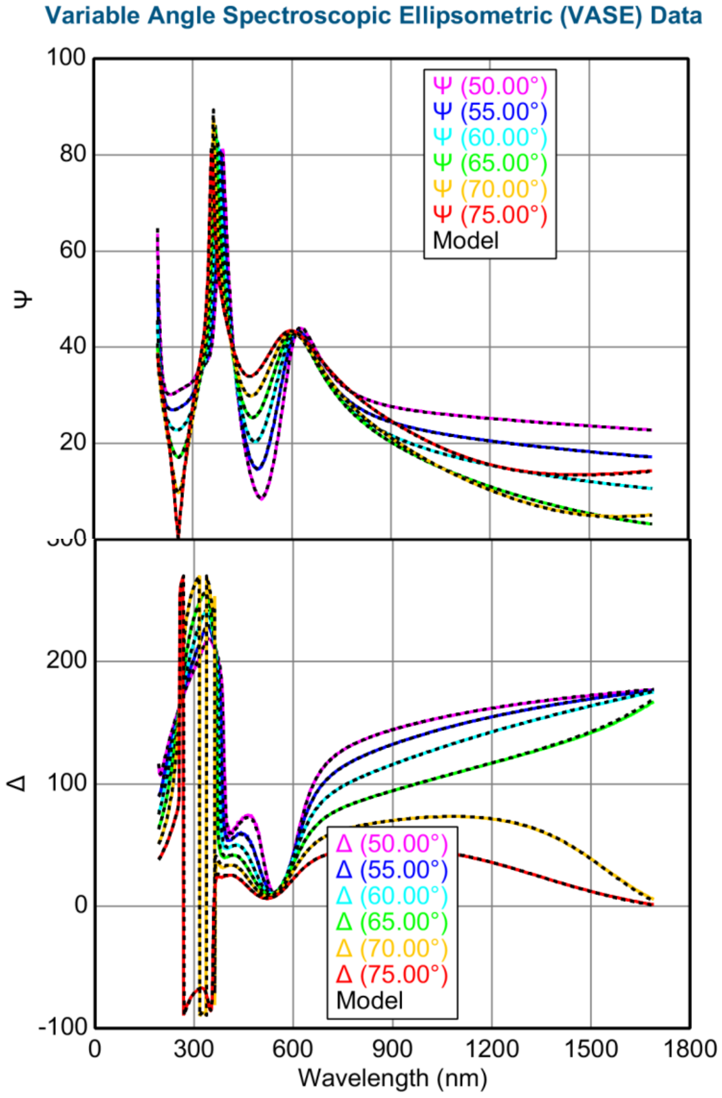
Comparison of Raw SE Data



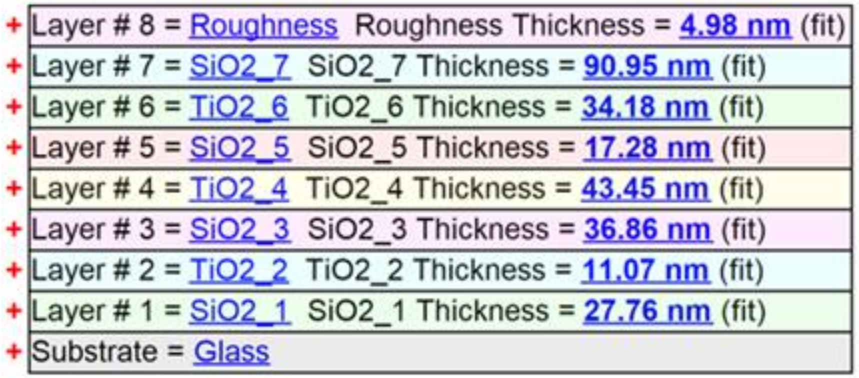
Limitations:

- Can only measure thin films that aren't fully opaque.
- Ambiguity between metal optical properties and thickness.

Multi-Layer Film Stacks

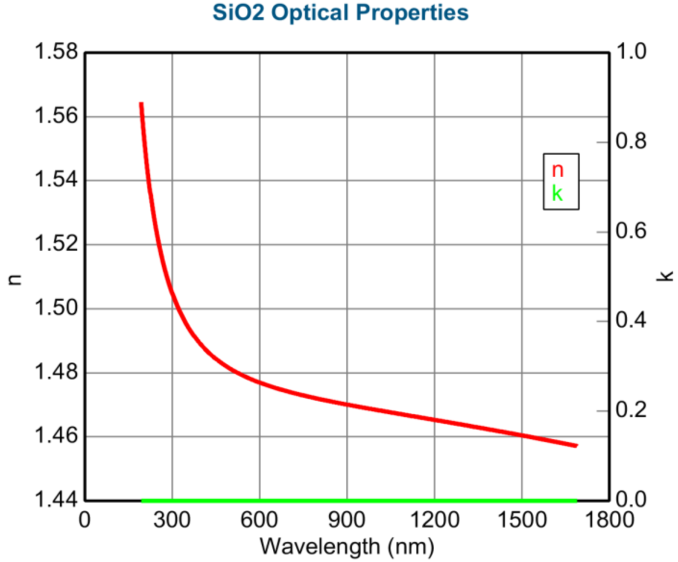

Variable Angle Spectroscopic Ellipsometric (VASE) Data

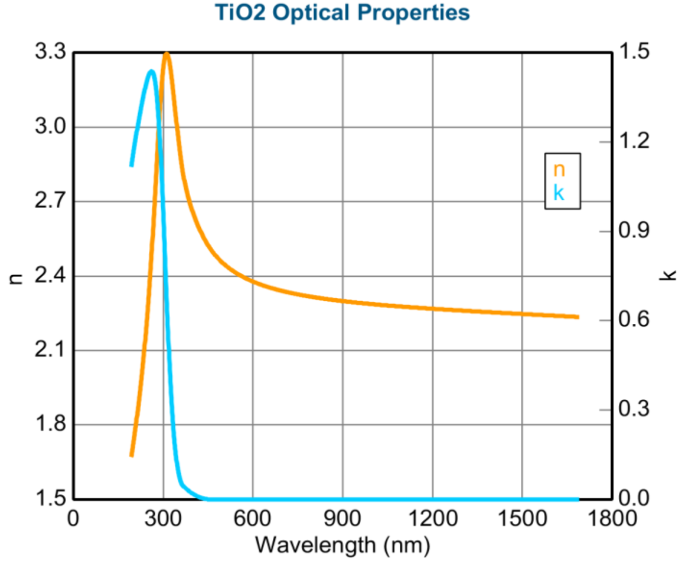
Anti-Reflection Coating:


\+ Layer # 8 = Roughness Roughness Thickness = 4.98 nm (fit)
\+ Layer # 7 = SiO2_7 SiO2_7 Thickness = 90.95 nm (fit)
\+ Layer # 6 = TiO2_6 TiO2_6 Thickness = 34.18 nm (fit)
\+ Layer # 5 = SiO2_5 SiO2_5 Thickness = 17.28 nm (fit)
\+ Layer # 4 = TiO2_4 TiO2_4 Thickness = 43.45 nm (fit)
\+ Layer # 3 = SiO2_3 SiO2_3 Thickness = 36.86 nm (fit)
\+ Layer # 2 = TiO2_2 TiO2_2 Thickness = 11.07 nm (fit)
\+ Layer # 1 = SiO2_1 SiO2_1 Thickness = 27.76 nm (fit)
\+ Substrate = Glass

Limitations:

- Need to know approximate stack structure to build representative optical model.
- Cannot have more than a few layers with unknown optical properties.

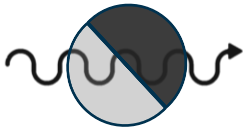

SiO2 Optical Properties


TiO2 Optical Properties

How to measure – Key deciding factors

1. Substrate exposed (or masked)

→ 2. **Not optically transparent**



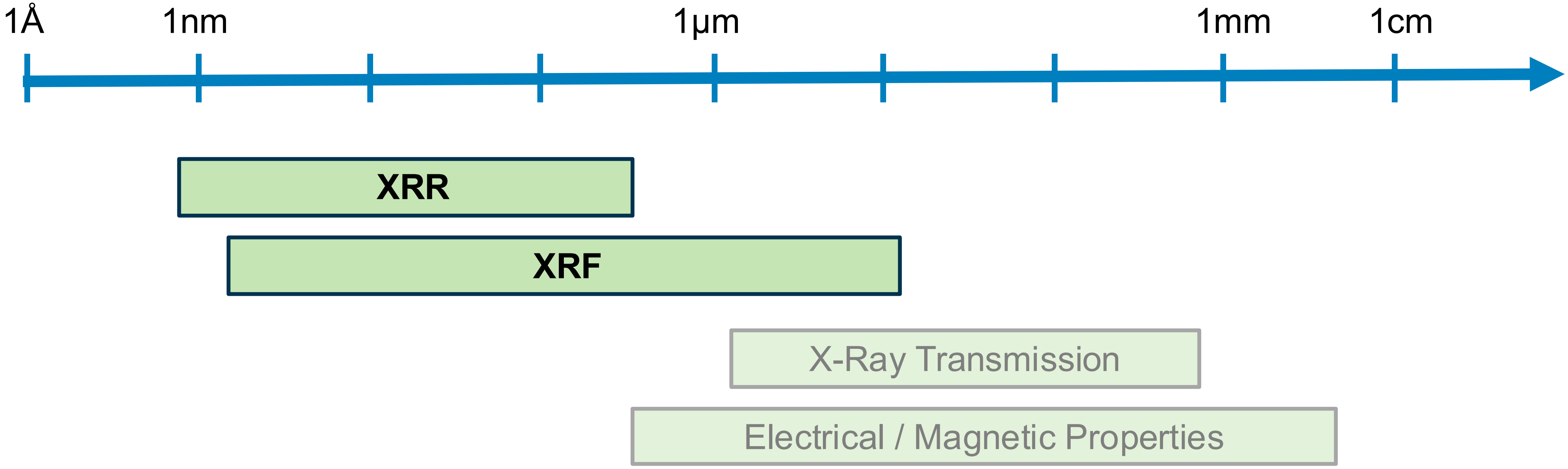
3. Prior knowledge for modeling

?

4. *Everything else*

Absorbing / Non-transparent films

- What is the expected film thickness?



Strongly absorbing/non-transparent films

- Many materials and coatings **strongly absorb UV/VIS/NIR** light; can limit optical methods
- However, these materials are **transparent to X-rays**
- Thickness can be measured by specular X-ray reflectivity (**XRR**) or by the intensity of characteristic X-ray fluorescence (**XRF**) from specific elements in the coating

X-ray Reflectivity (XRR)

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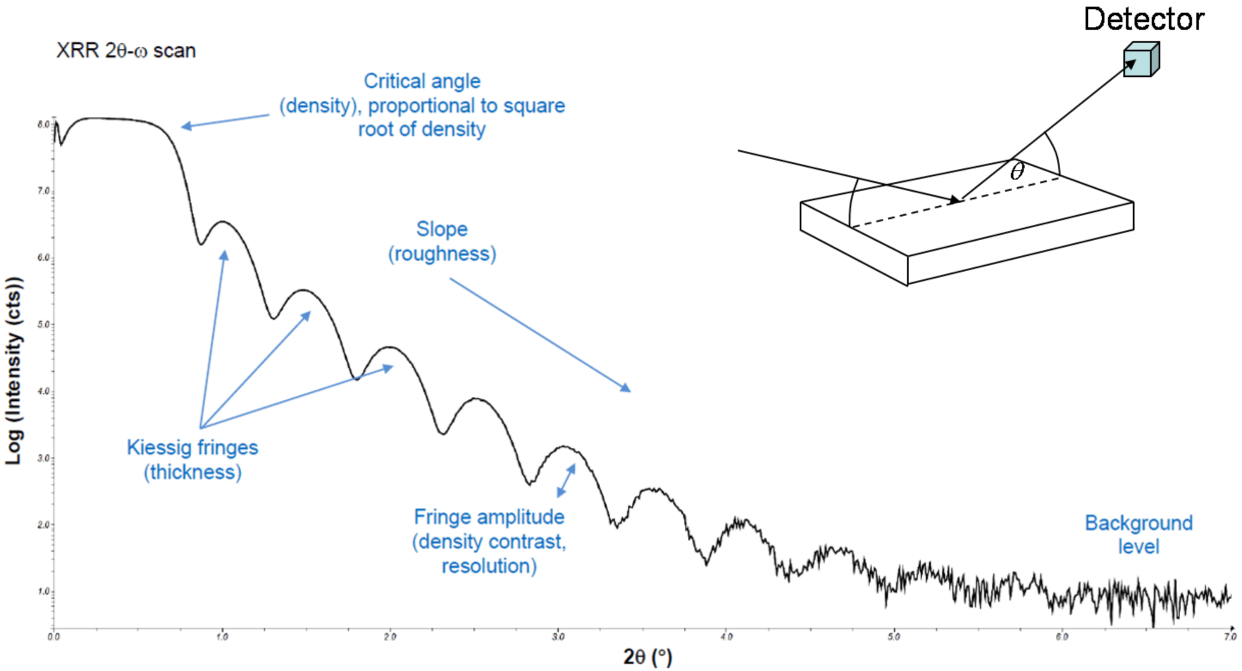
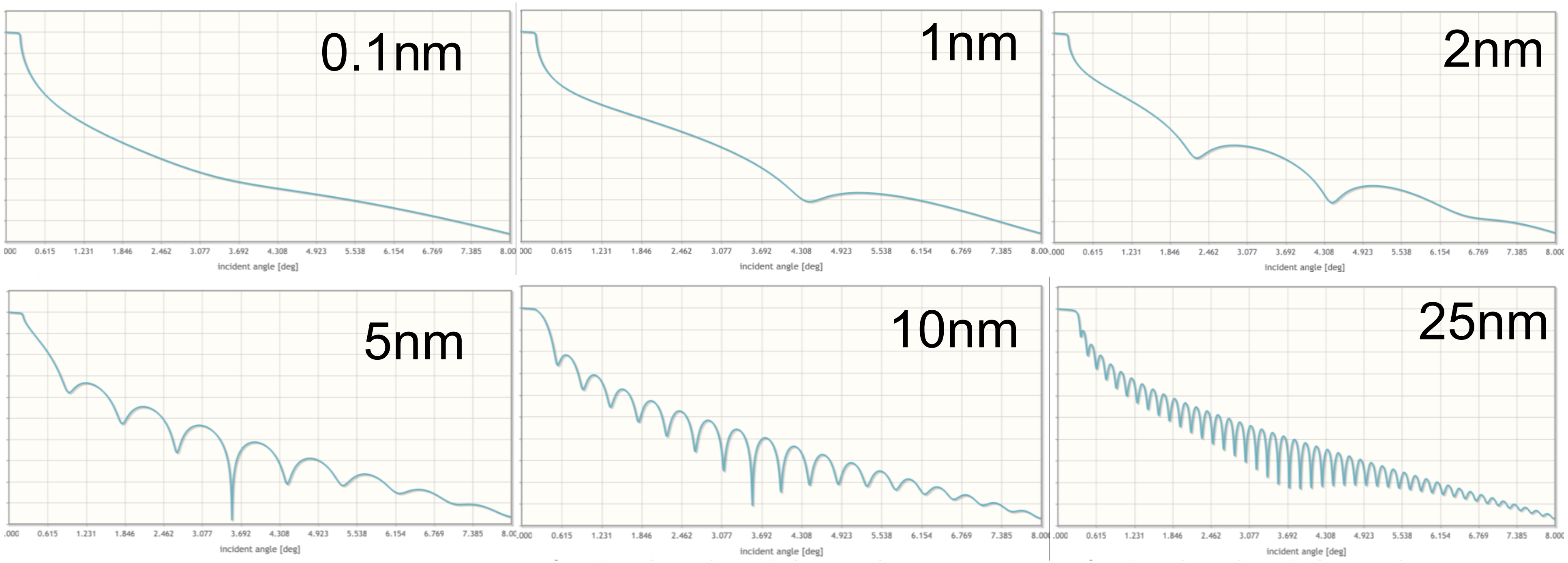
Additional Info
Surface roughness &
density of layers

- Specular X-ray reflection from surfaces
- Interference induces oscillation (fringes) in the reflectivity signal
- Very high precision, applicable to optically opaque films

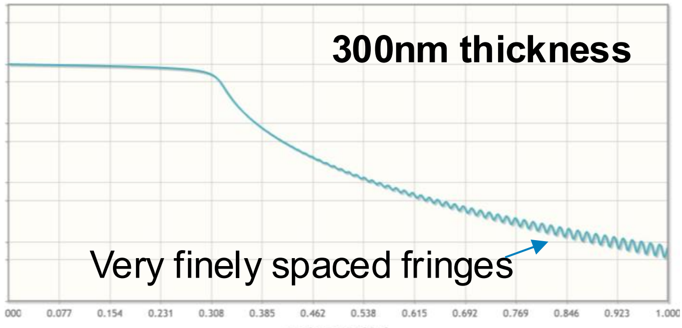


Method	Applicability	Benefits	Limitations
XRR	Thin smooth films	High precision, applicable to optically opaque films, models are typically simple, mapping	Needs very smooth films (<3nm) and thin (<300nm typically), modeling requires some knowledge of the sample

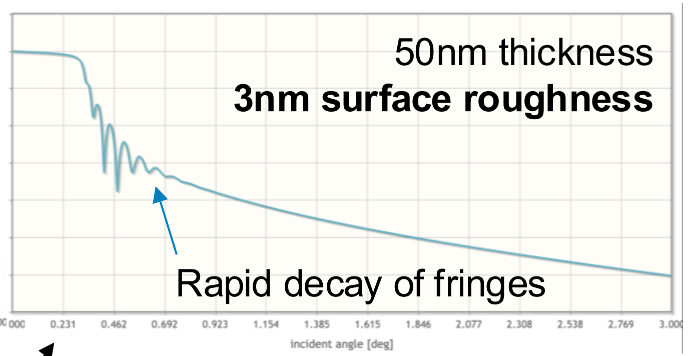
XRR film thickness sensitivity and limitations



- **Excellent sensitivity**
- Fringe frequency independent of optical properties or density


300nm thickness

Very finely spaced fringes


50nm thickness

3nm surface roughness

Rapid decay of fringes

zoomed in on lower angle region

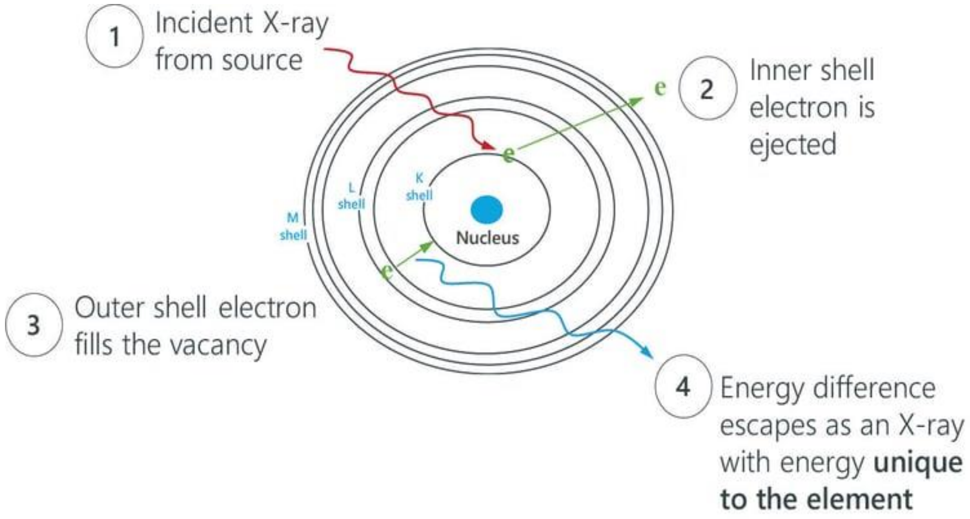
Limitations:

- Thicker films (>200nm) require higher resolution to resolve fringes
- Roughness rapidly reduces fringe intensity

X-ray fluorescence

Additional Info
Chemical composition

- Characteristic X-rays emitted during X-ray irradiation
- Emissions proportional to the amount of each element, typically correlated with thickness
- Widely applied to metal coatings
- Less strict roughness requirements than XRR
- Can measure multiple microns thickness
- Operates in serial wavelength dispersive or parallel energy dispersive mode



Limitations:

- Can require standards for quantification
- Energy dispersive detectors can suffer from overlapping peaks due to limited resolution

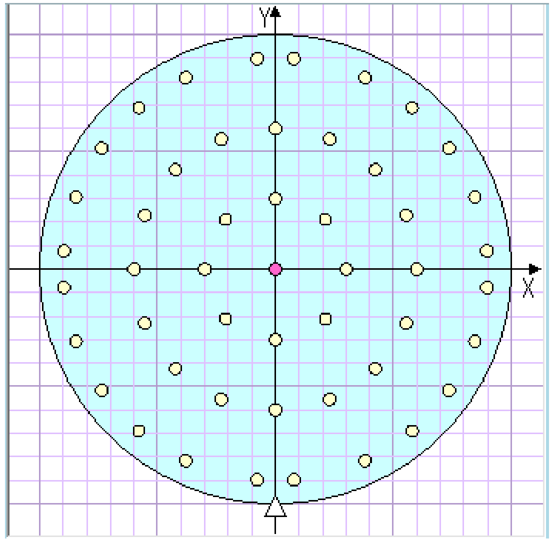
Method	Applicability	Benefits	Limitations
XRF	Typically, metal-containing coatings	Inexpensive, fast, mapping	Thickness measurement typically requires calibration standards

XRR & XRF

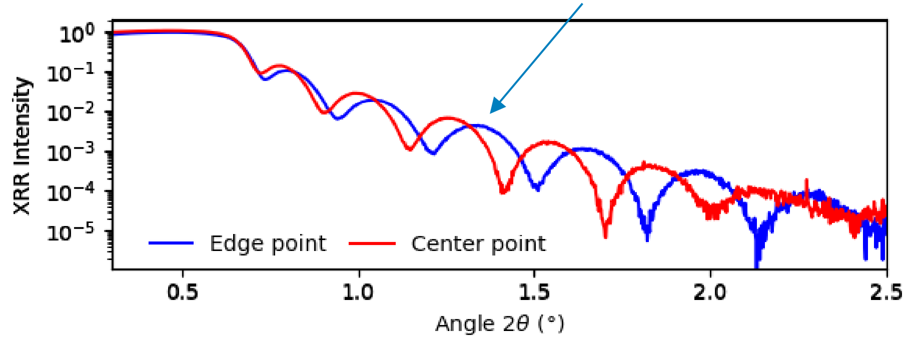
Rigaku XTRAIA MF-3000

XRR patterns and XRF signals were collected at 49 locations across a 300mm wafer featuring a 25nm TiN film.

WAFER MAP

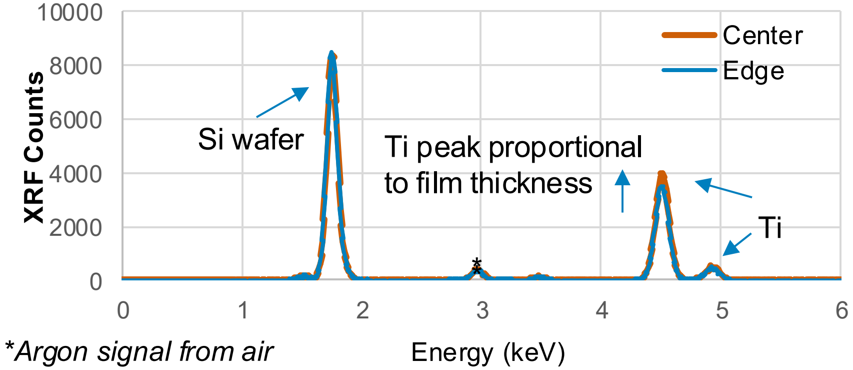


XRR



Fringe frequency difference indicates difference in film thickness

XRF



Ti peak proportional to film thickness

*Argon signal from air

From Data to Insight: Analyzing TiN Thickness Variation

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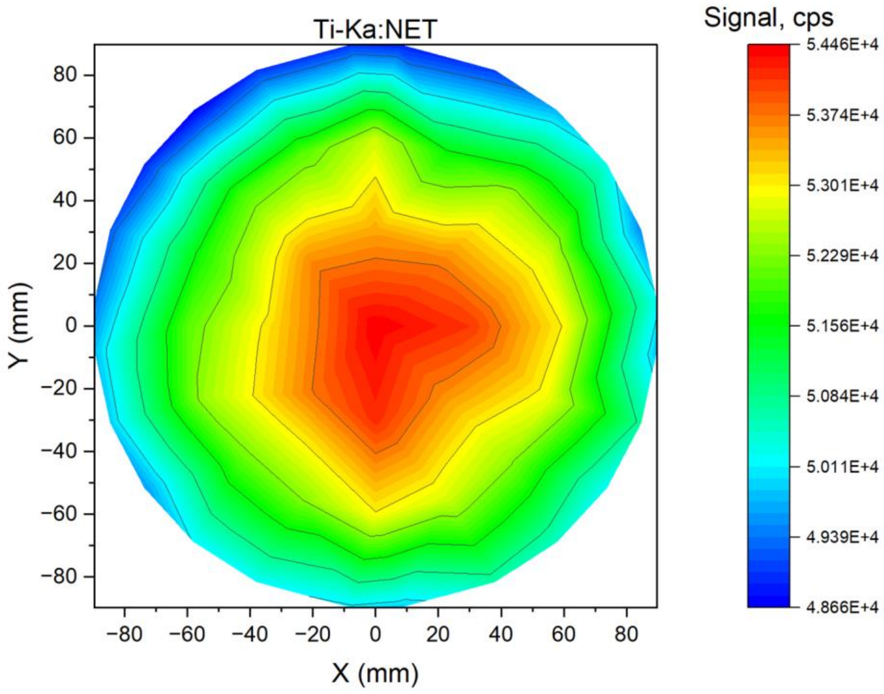
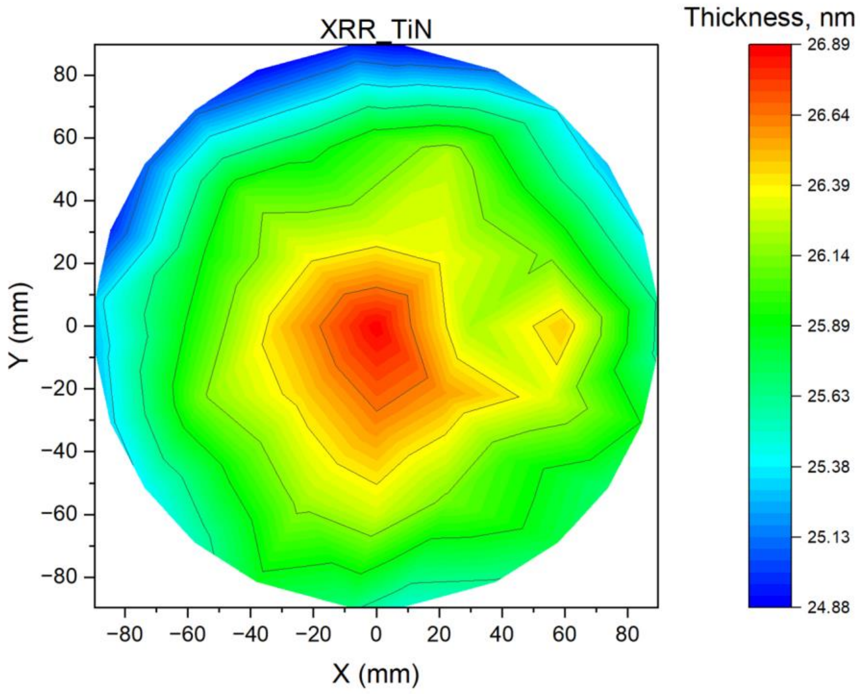
XRF

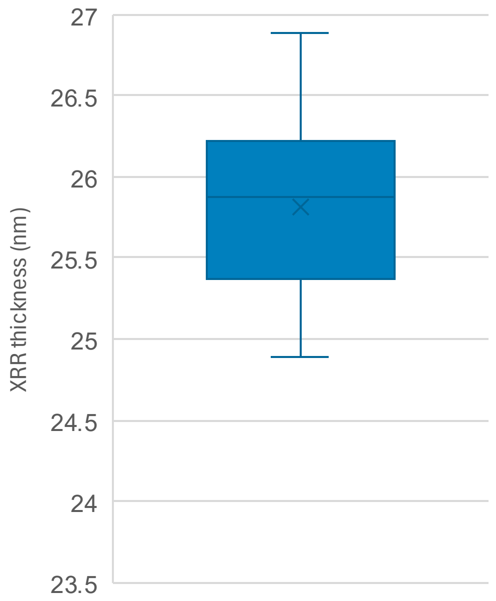


XRR



Film Thickness Variation:

Statistical Distribution Across 300mm Wafer



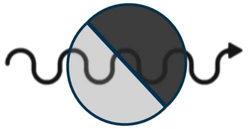
25.8 +/- 0.5nm (1σ)

The data reveals measurable variation in film thickness across the wafer, demonstrating the tool's capability for high-resolution, full-wafer uniformity analysis.

How to measure – Key deciding factors

1. Substrate exposed (or masked)

2. Optically transparent

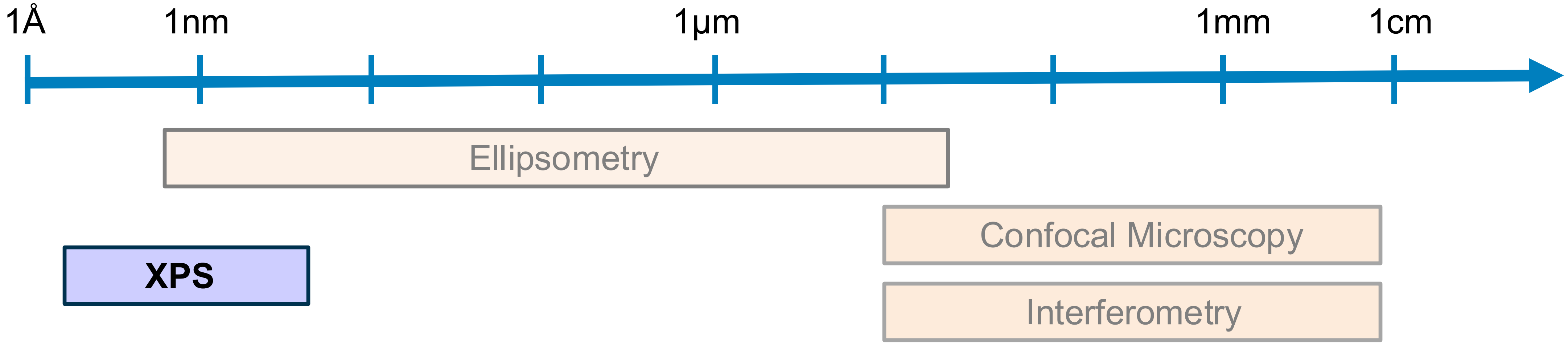


→ **3. Prior knowledge for modeling**

?

4. Everything else

Very thin transparent oxides, ex. native oxides

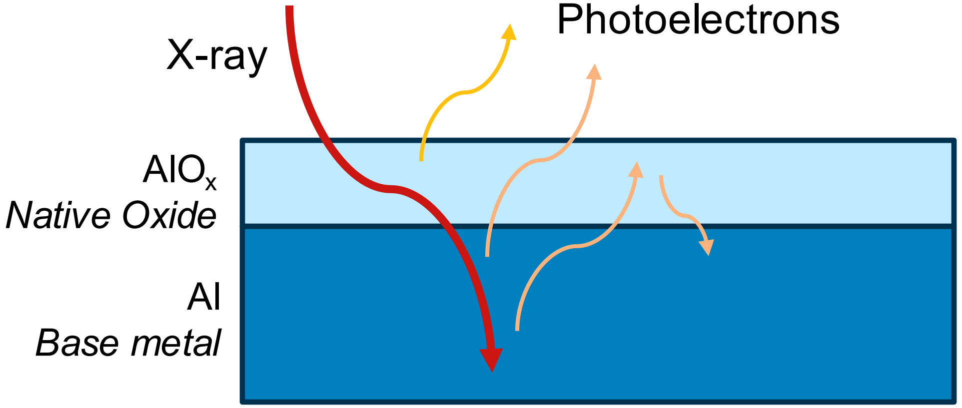


- Optically transparent – but often don't have good set of optical properties for modeling
- Very thin
- Form naturally on many materials (ex. Aluminum, Silicon) in air
    - if you try to mask off an area they will grow when the mask is removed
- They often form on rough metal surfaces unsuitable for XRR
- Contain light elements not suitable for XRF

X-ray photoelectron spectroscopy (XPS)

Additional Info
Surface Chemistry
Contamination

- XPS measures the binding energy (BE) of electrons in the sample
  - Sensitive to local coordination environment
  - Metal-Metal or Metal-Oxygen bonds have different BE
- The surface native oxide layer scatters the photoelectrons emitted from the substrate
  - Thickness calculated using inelastic mean free path (IMFP) of electrons in the oxide

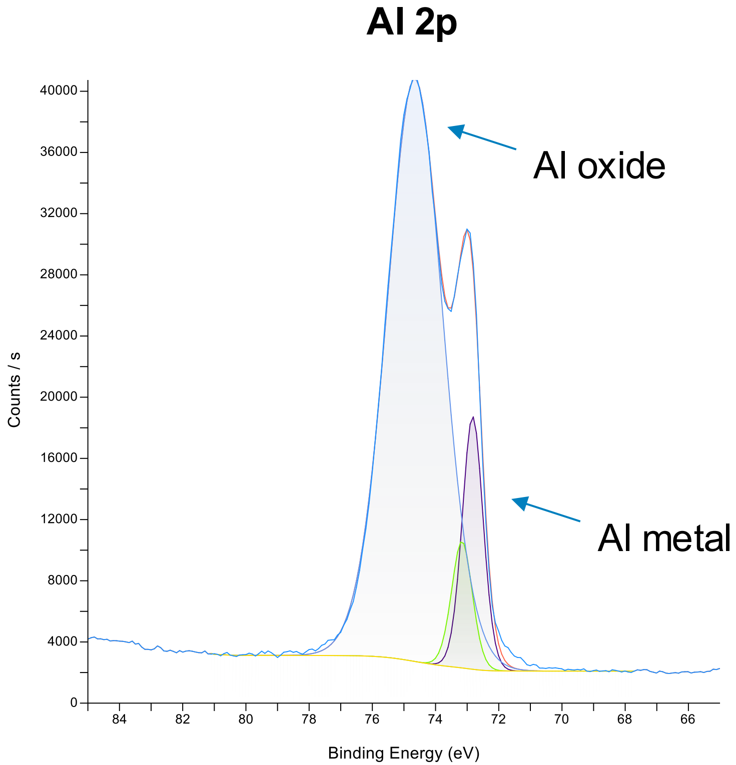


Limitations:

- Very thin films <10nm
- Requires known IMFP

Method	Applicability	Benefits	Limitations
XPS	Very thin layers where the coating and the substrate share an element, ex. native oxides	Can measure very thin native oxides	Requires binding energy difference and known inelastic mean free path, limited to very thin <~5-8nm films.

XPS



	Al	
	metal	oxide
Binding Energy (eV)	72.8	74.7
Area %	13.9	86.1

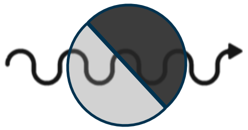
Thickness calculated to be 6.3nm

Method	Applicability	Benefits	Limitations
XPS	Very thin layers where the coating and the substrate share an element, ex. native oxides	Can measure very thin native oxides	Requires binding energy difference and known inelastic mean free path, limited to very thin <~5-8nm films.

How to measure – Key deciding factors

1. Substrate exposed (or masked)

2. Optically transparent



3. Prior knowledge for modeling

?

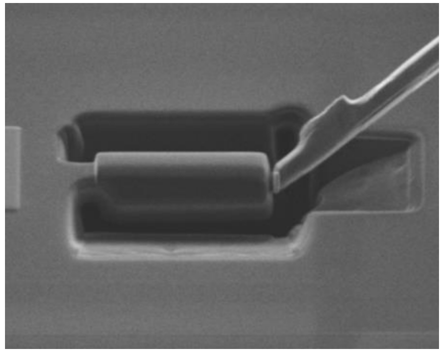
→ *4. Everything else*

Cross-section

- Gold standard in many application
  - directly visualize each the layer and measure thickness
  - Widely applicable
    - Requires minimal prior knowledge
    - complex unknown stacks possible
    - No limitations on optical transparency
- Many ways to prepare and image cross-sections
  - Embedding and polishing
  - Slicing with a blade or microtome
  - Focused ion beam
- Discuss two case studies from two industries

Additional Info
Sub-surface features/defects


Epoxy embedded and polished cross-section

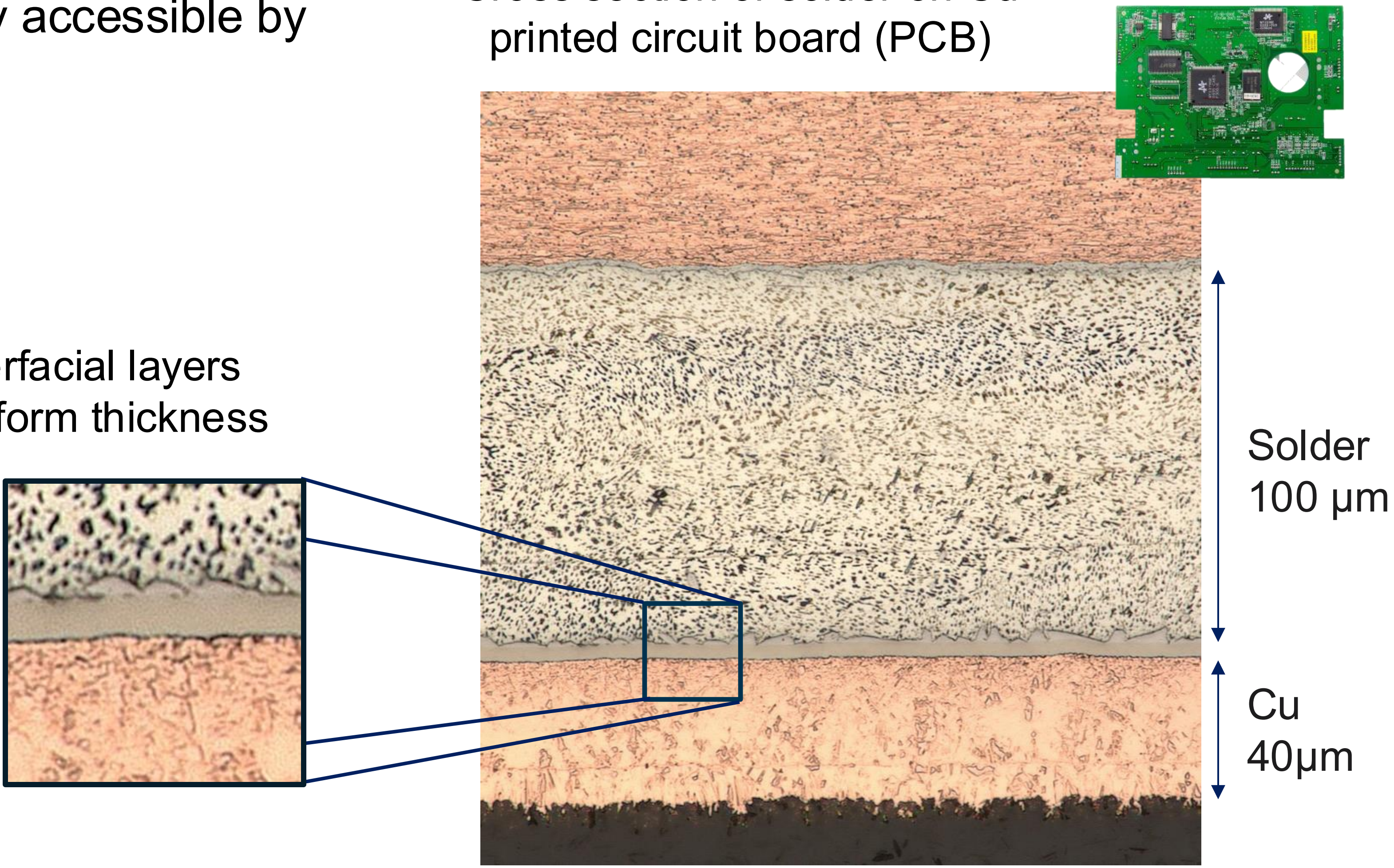

FIB preparation of TEM cross-section sample

Method	Applicability	Benefits	Limitations
Cross-Section	Almost anything	You can see all the layers	Destructive

Metal Films – nickel on a printed circuit board using optical microscopy

- Buried opaque layers, only accessible by cross-section

Cross section of solder on Cu printed circuit board (PCB)



Additional interfacial layers visible, non-uniform thickness

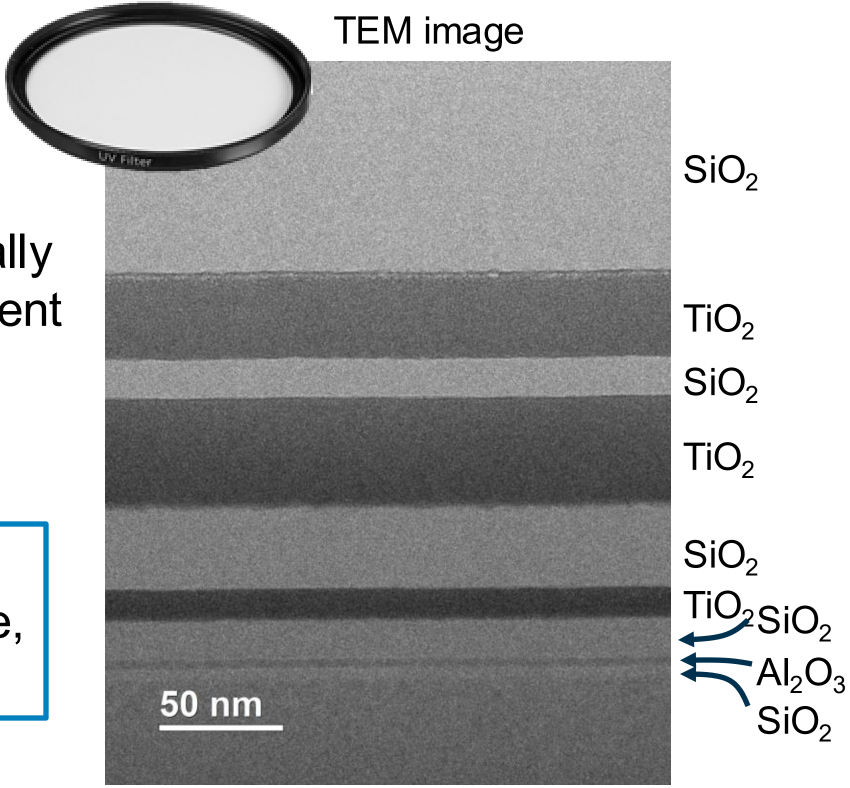
Additional Info
Defects, microstructure

Multilayer antireflection coating: Camera filter using TEM-EDS

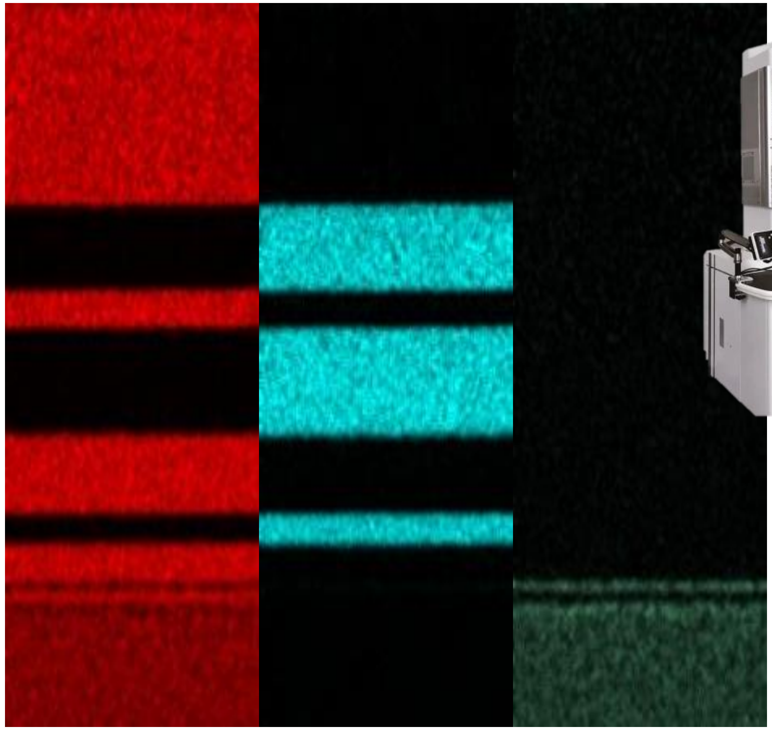
- Camera filter
- 9 alternating layers
  - 3-90nm thickness
  - Measured individually
- Inform model development for ellipsometry

Additional Info
Defects, nanostructure, interfacial roughness

TEM image



TEM-EDS
Elemental Maps



JEOL JEM-F200

	SiO_2	Al_2O_3	SiO_2	TiO_2	SiO_2	TiO_2	SiO_2	TiO_2	SiO_2
Thickness (nm)	5.0	3.2	15.4	12.3	32.6	43.2	15.9	34.7	85.2

substrate → *surface*


Thank
You
köszönöm
Suksama
Asante
Shukria
Vinaka
Maake
Dhanyavadagalu
Gratias Tibi
Terima Kasih
Dankscheen
спасибо
Kiitos
ارك شك
Manana
Dankon
Suksama
Diolch i Chi
Dank Je
Mauruuru
Biyan
Taiku
நன்றி
Dziekuje
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Arigato
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Gracias
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cảm ơn bạn
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Ua Tsaug Rau Koj
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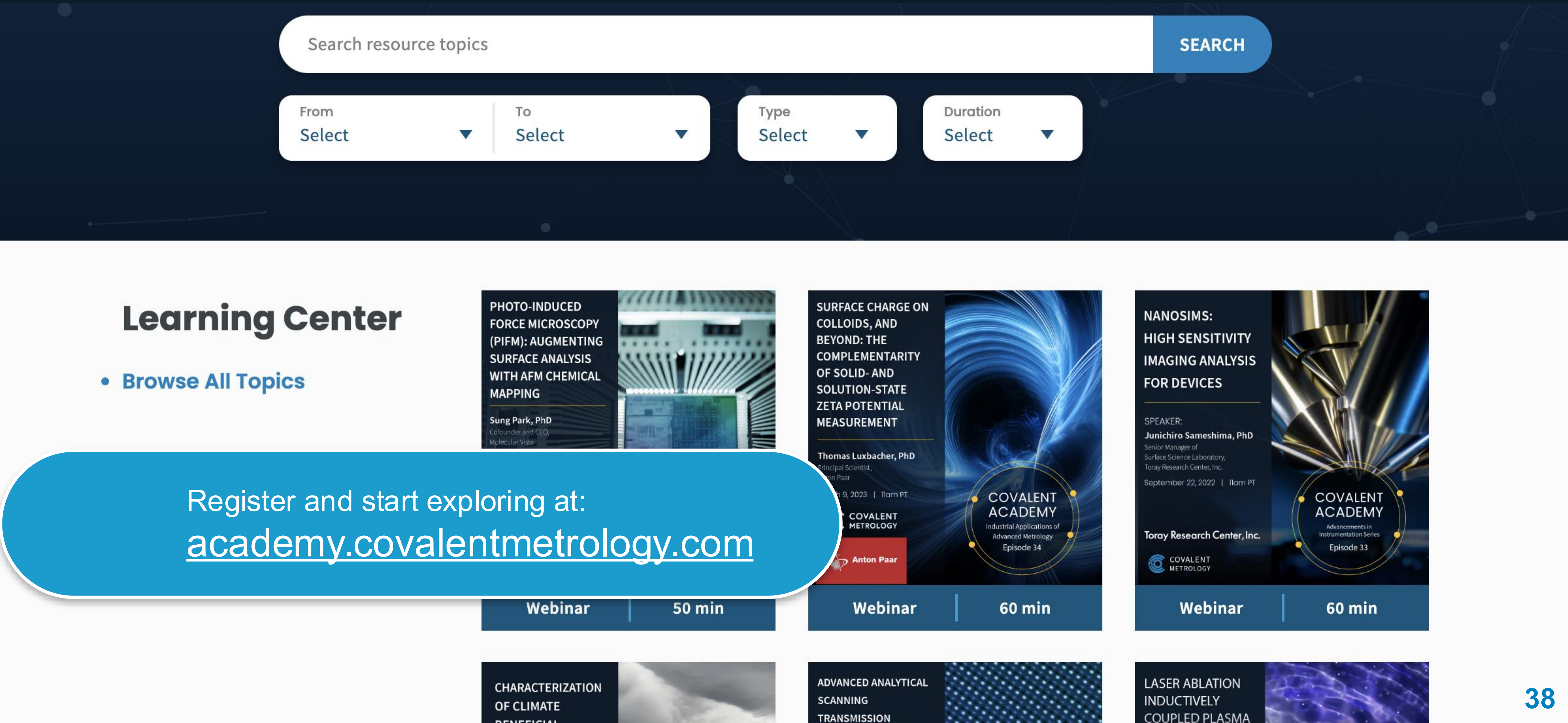

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Q & A
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Thank you.

Summary

Method	**Applicability**	**Benefits**	**Limitations**	***Additional Information***
Step Height	Films with exposed substrates	Simple and direct measurement	Requires exposed substrate, can scratch the sample	*Surface Roughness*
Cross-Section	Almost anything	You can see all the layers	Destructive	*Sub-surface features/defects*
Ellipsometry	Somewhat optically transparent films	Quick, non-destructive, measure optical properties, mapping	Needs transparency and knowledge of the layers to model	*Optical properties*
XRR	Thin smooth films	High precision, applicable to opaque metal layers, mapping	Limited thickness range ~1-300nm with low roughness (<5nm); modeling can require knowledge of the sample	*Surface roughness & density of layers*
XRF	Typically, metal-containing coatings	Inexpensive, fast, mapping	Thickness measurement typically requires calibration standards	*Chemical composition*
XPS	Very thin layers where the coating and the substrate share an element, ex. native oxides	Can measure very thin native oxides	Requires binding energy difference and known inelastic mean free path, limited to very thin <~5-8nm films	*Surface chemistry and contamination*
Many other techniques are available if your sample doesn't fit in these categories. Please reach out to discuss your application				