COVALENT METROLOGY

Welcome

covalentmetrology.com

COVALENT METROLOGY



Covalent Metrology



- **Disruptive New Platform for Outsourced Measurement, Test & Characterization**
  - 32 team members (13 PhDs)
  - 9,500 ft^2 lab in Sunnyvale (and growing)
  - 35 in-house instruments (and growing); >$10M in instruments
  - 7 instrument partnerships
    (ThermoFisher, Rigaku, JA Woollam, Anton Paar, PVA TePla, Asylum Research / Oxford Instruments, Keyence)
  - 11 partner labs
- **>300 customers** (80% repeat)

Covalent Technical Groups and Organization

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

Speaker Introduction



Dr. Max Junda

- Senior Member of Technical Staff, Optical Characterization Techniques
  - Spectral Ellipsometry, Laser Confocal, Chromatic Confocal, White Light Interferometry, Patterned Light 3D Measurements
- Ellipsometry experience spanning 7 instrument types: THz to vacuum UV spectral ranges in both *in situ* and *ex situ* measurement modes
- Postdoctoral research used SE to explore vacuum deposition techniques for fabrication and optimization of thin-film photovoltaics
- Received PhD from the University of Toledo, and a BS from Middlebury College, both in Physics

Outline

COVALENT METROLOGY

1. Ellipsometry Review
2. Optical Modeling Toolkit
3. Application Examples

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1 Ellipsometry Review

2 Optical Modeling Toolkit

3 Application Examples

Instrument Partner: J. A. Woollam Company

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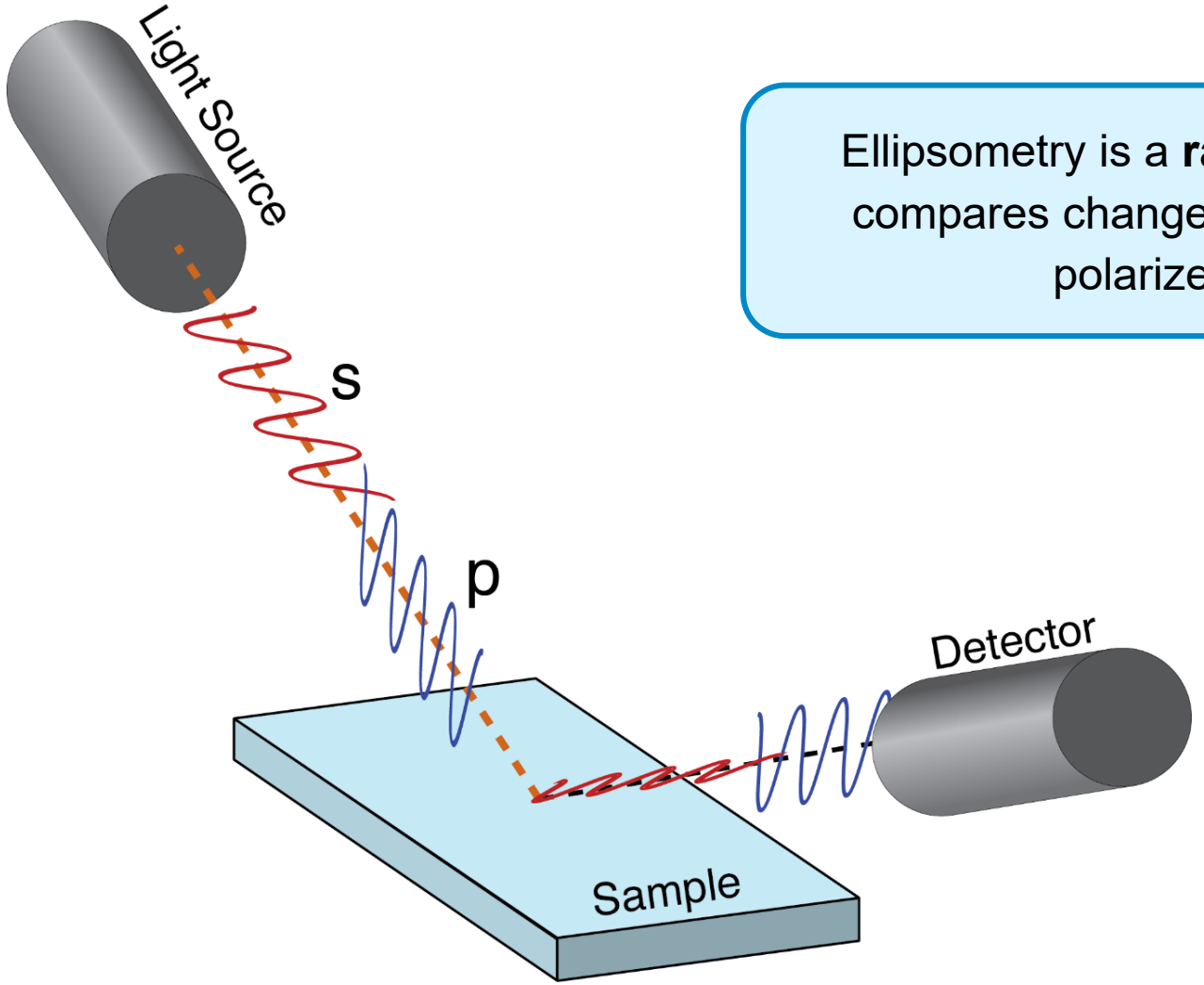
J.A. Woollam Co.
Ellipsometry Solutions

CompleteEASE 6

J.A. Woollam

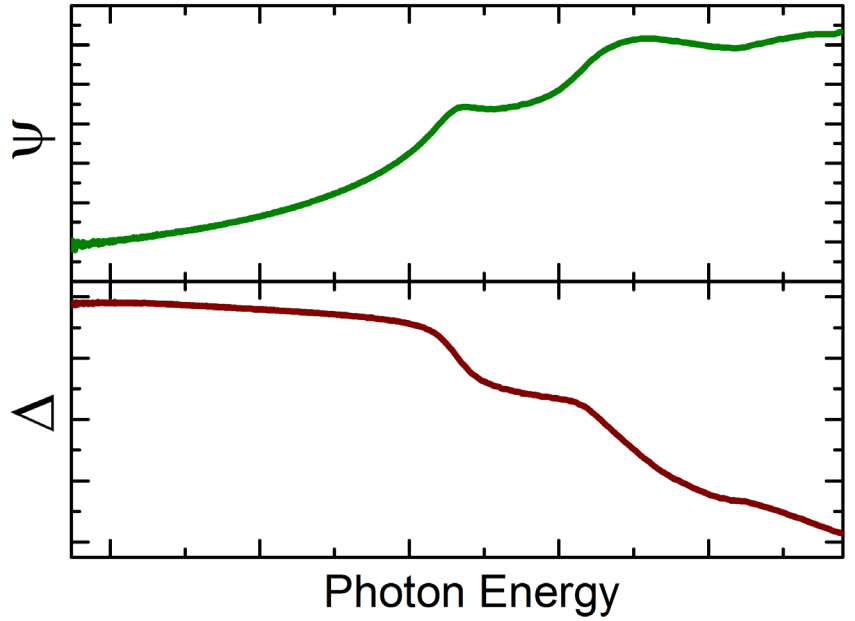
Measurement Schematic

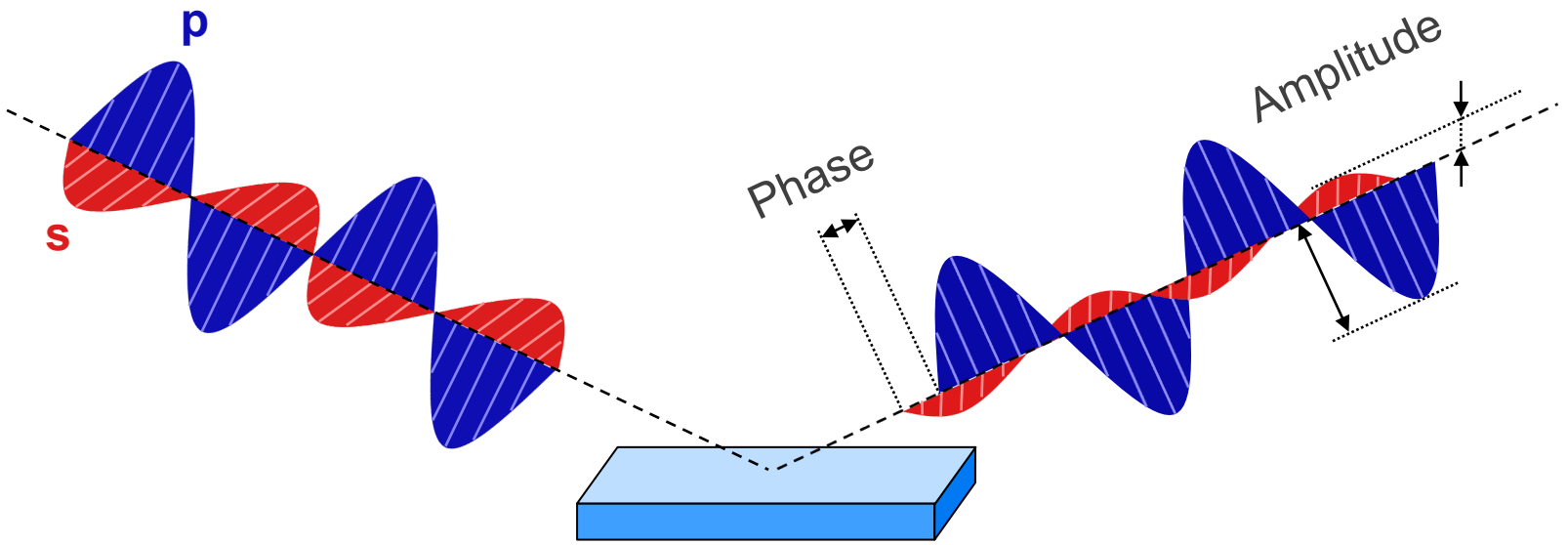


Ellipsometry is a **ratio-based** measurement that compares changes to **phase** and **amplitude** of polarized light components

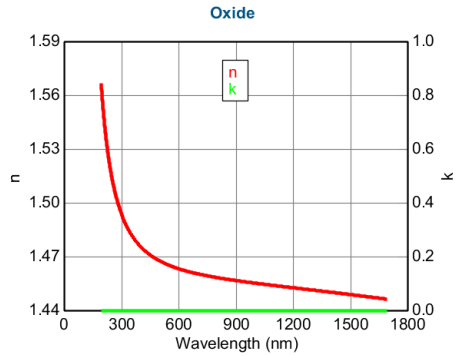
Psi and Delta

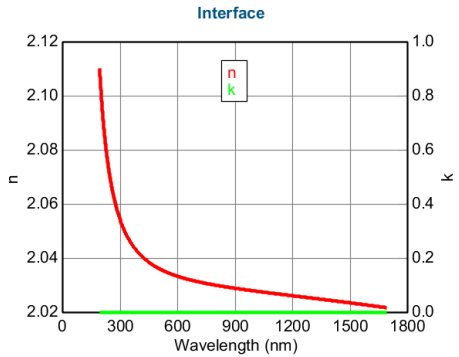
- Two values are measured at each wavelength:
  - ψ: amplitude ratio between **p** & **s** polarization
  - Δ: phase shift between **p** & **s** polarization

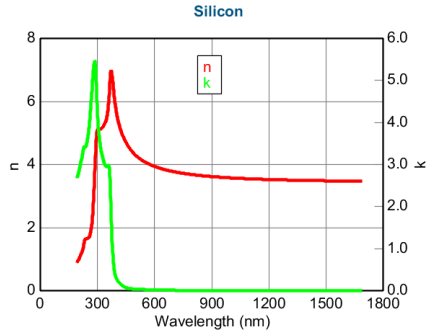




Model-Generated Data

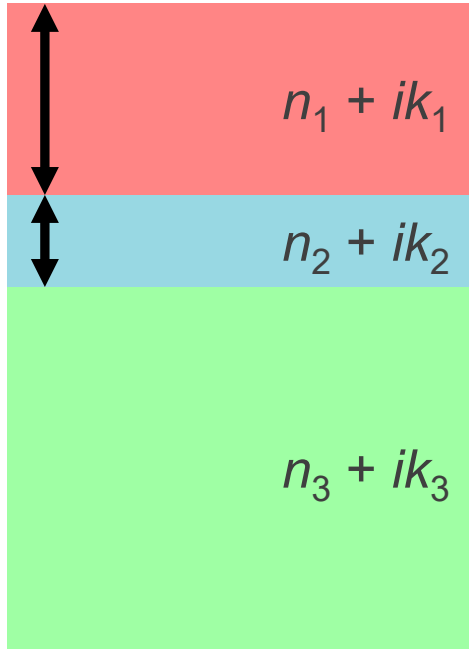


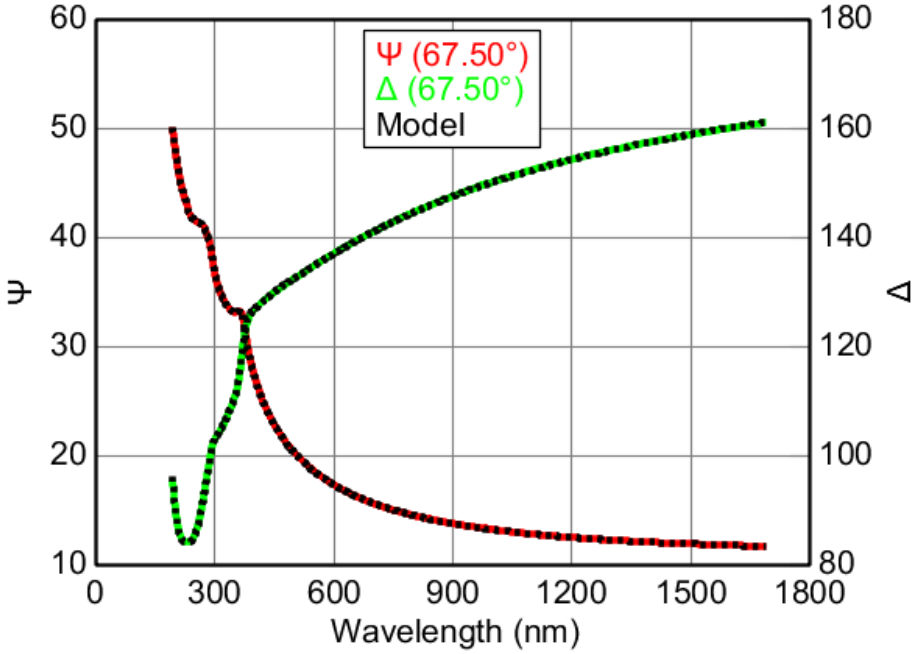




What is an optical model?

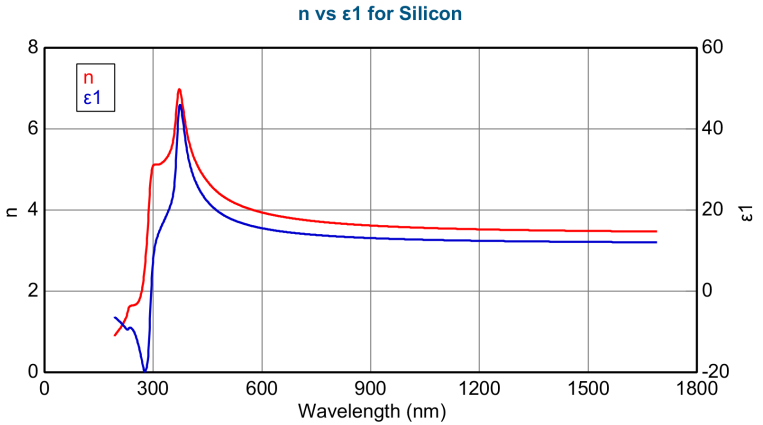
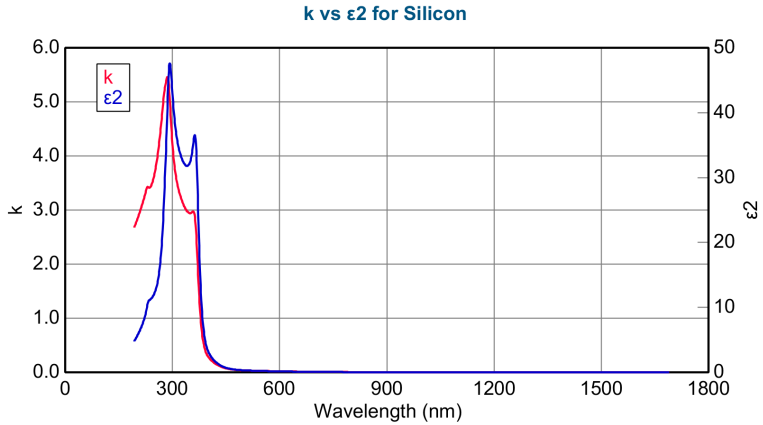
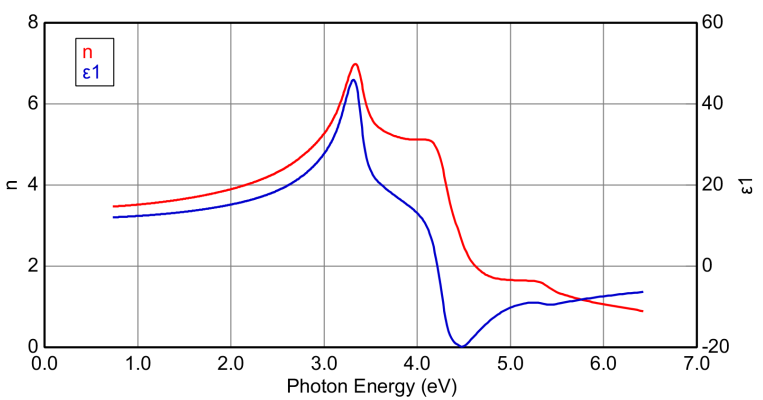
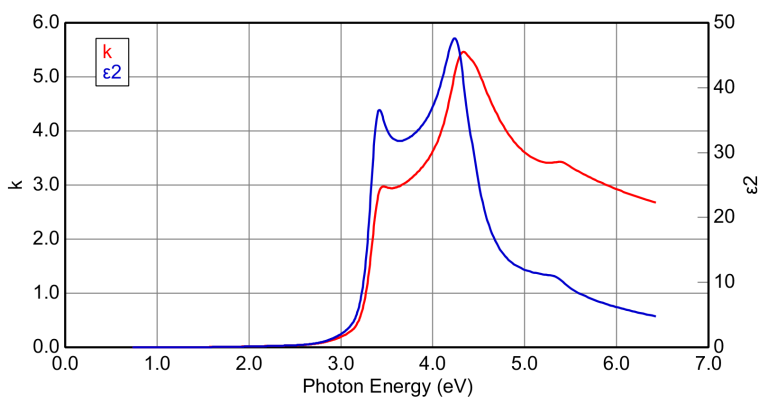
Spectra (n, k) (ε_1, ε_2) that describe layers and are used to mathematically calculate expected polarization effects (ψ, Δ)





Optical Properties

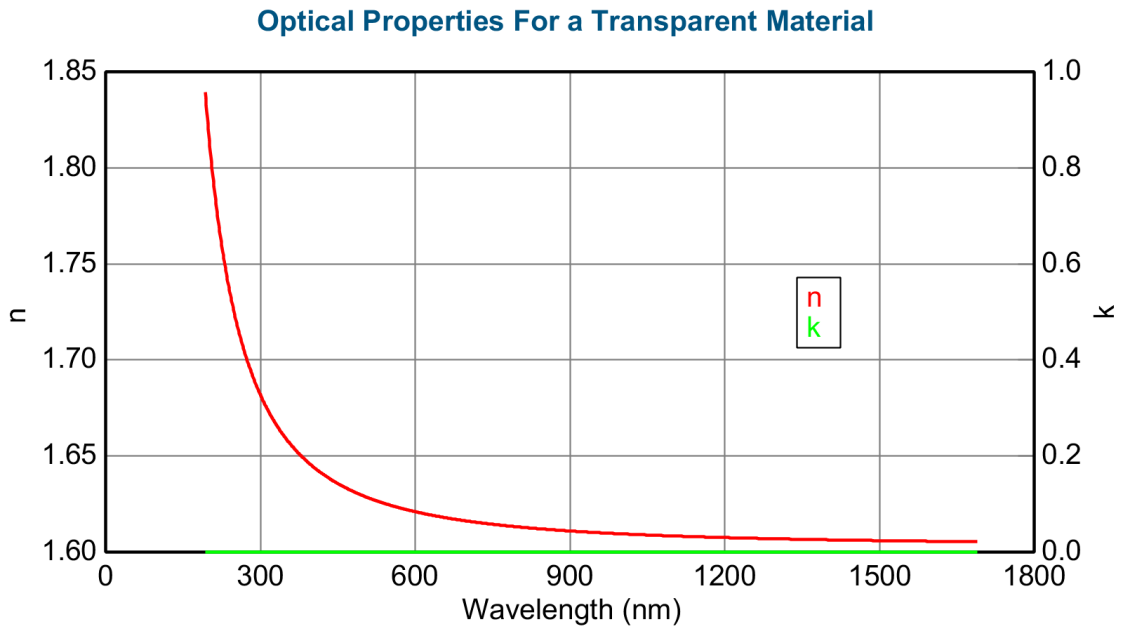
$$n + ik \quad \varepsilon_1 + i\varepsilon_2$$

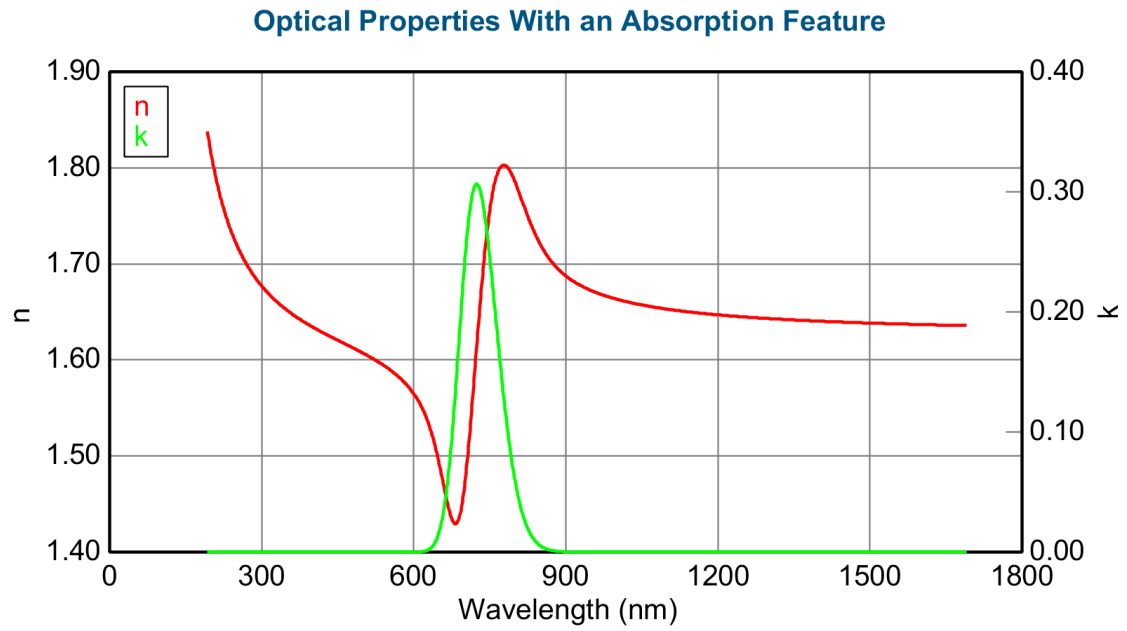
	Real Part	Imaginary Part
Vs. Wavelength		
Vs. Photon Energy		

Optical Properties

$$n + ik \quad \varepsilon_1 + i\varepsilon_2$$

- The imaginary part (k or ε_2) can never be negative
- There is a physical and mathematical relationship between (n & k) and (ε_1 & ε_2)
  - Kramer's Kronig relation

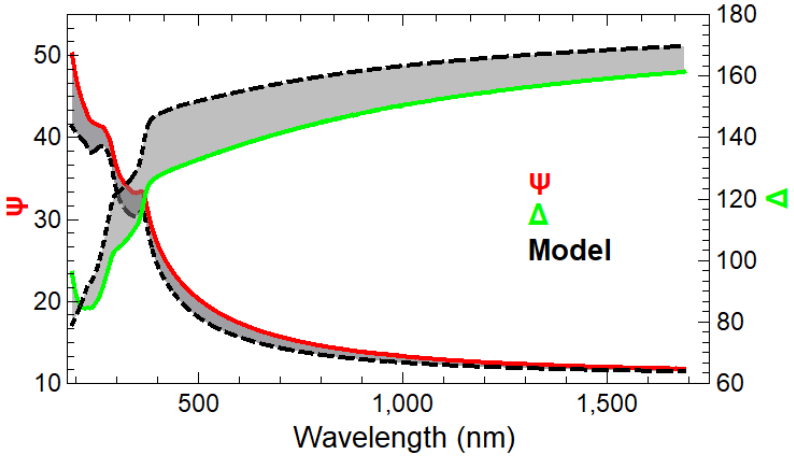


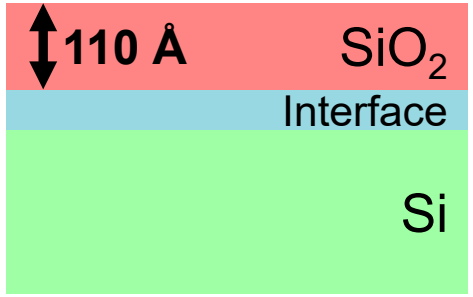


Mean Square Error (MSE) Function

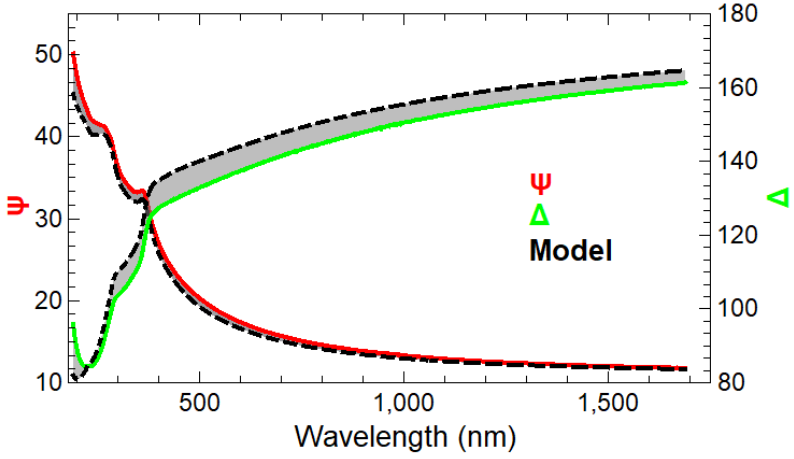
- The error function quantifies the mismatch between the model-generated and measured data
- Can be thought of as the area between the two curves
- Fit algorithms iteratively adjust model parameters to minimize MSE

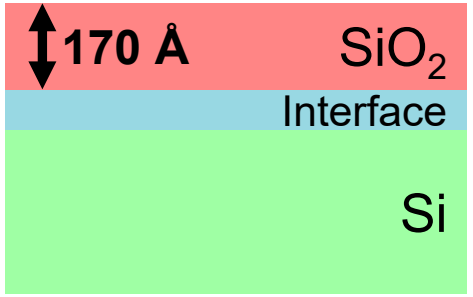
MSE = 103.274



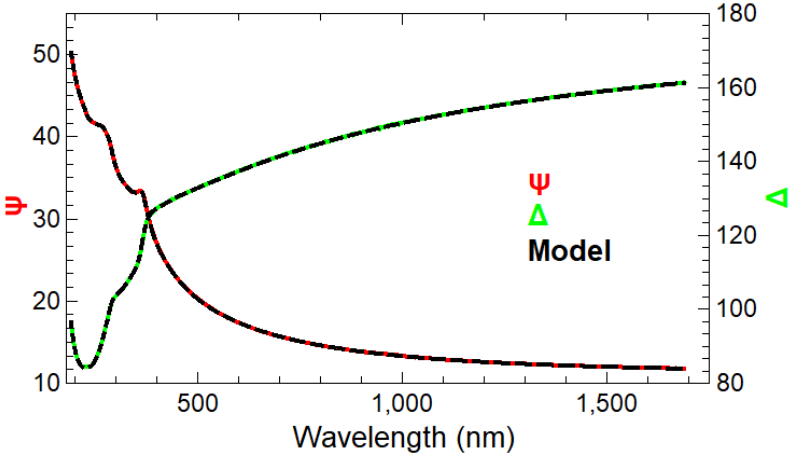


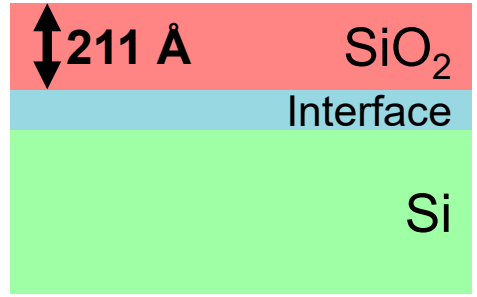
MSE = 42.354





MSE = 0.831





Mean Square Error (MSE) Continued

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$$MSE_{NCS} = \sqrt{\frac{1}{3n-m}\sum_{i=1}^{n}\left[\left(\frac{N_{E_i}-N_{G_i}}{.001}\right)^2+\left(\frac{C_{E_i}-C_{G_i}}{.001}\right)^2+\left(\frac{S_{E_i}-S_{G_i}}{.001}\right)^2\right]}$$

$$N = \cos(2\psi) \qquad C = \sin(2\psi)\cos(\Delta) \qquad S = \sin(2\psi)\sin(\Delta)$$

- Many different MSE functions can be used since, ultimately, these aren't specifically unique to ellipsometry and would be part of any fit algorithm
- More broadly, the fit algorithm also produces other measures of the "goodness" of a fit:
  - **Error bars** on individual fit parameters (from statistical 90% confidence limit)
  - Parameter **correlation coefficients**

Outline

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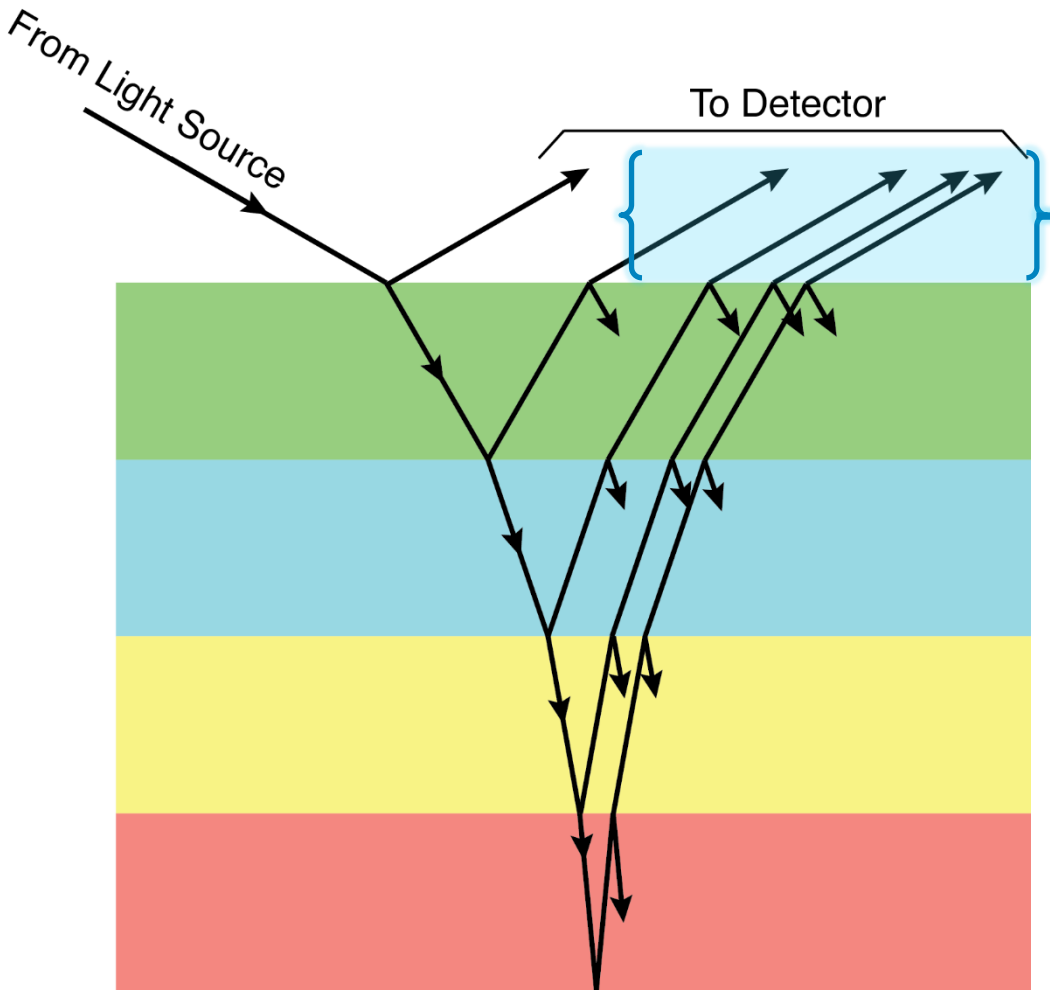
1 Ellipsometry Review

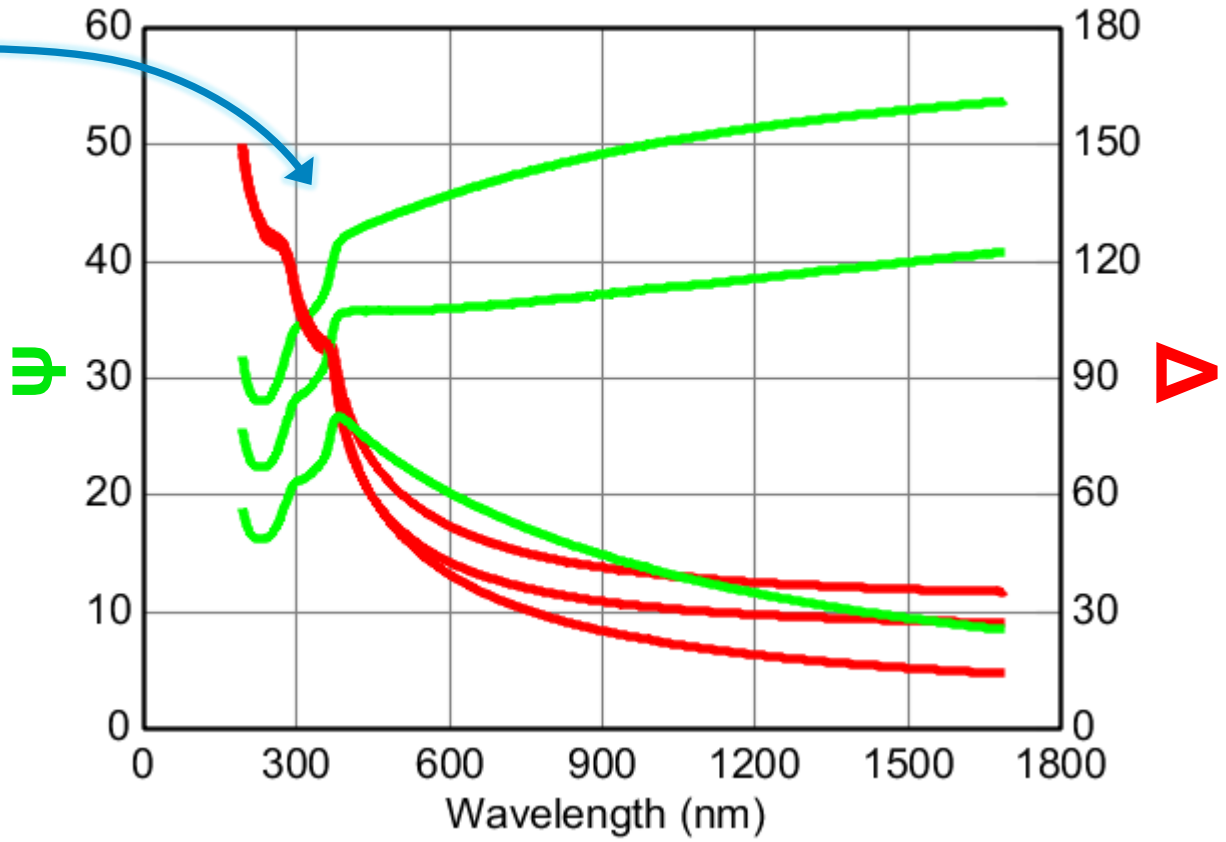
2 Optical Modeling Toolkit

3 Application Examples

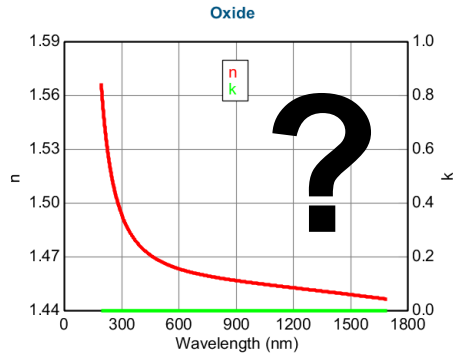
Information Content of a Measurement

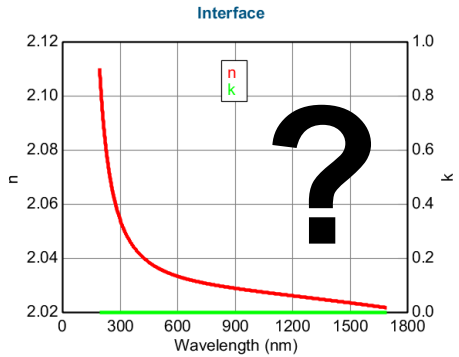
ψ and Δ contain a superposition of the entire interaction "history" of the light beam with the sample

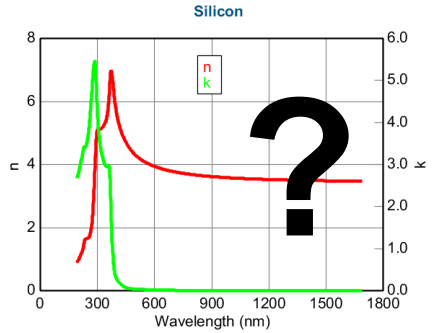


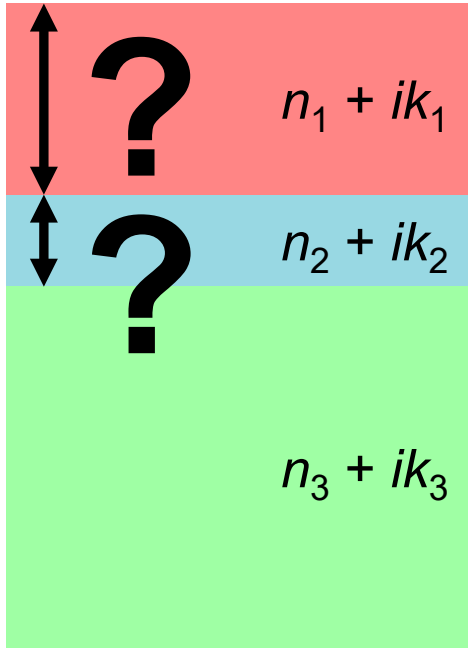


Problem: Too Many "Unknowns"

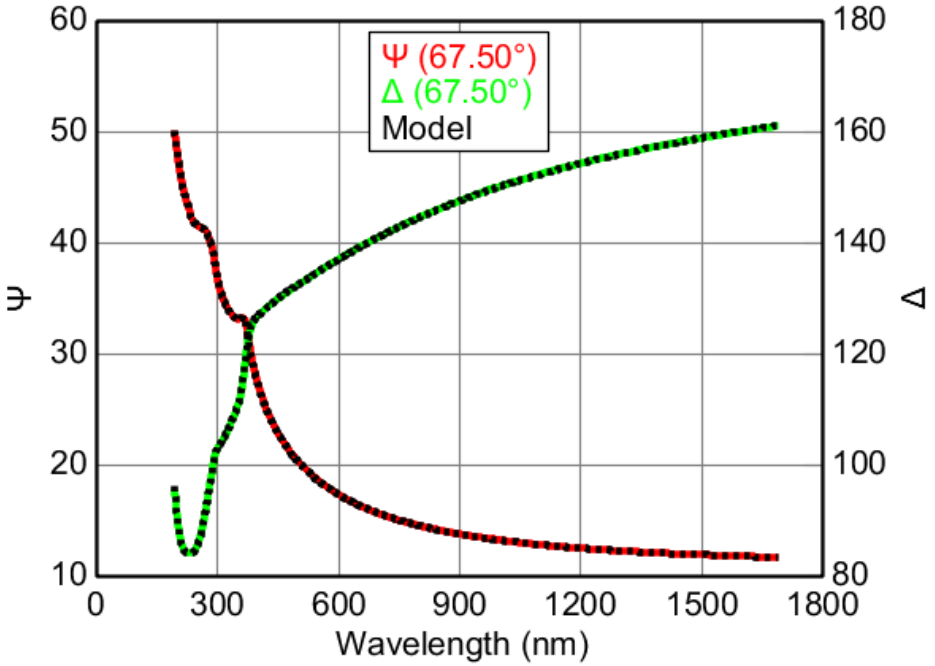








1083 Wavelengths Measured



Reducing “Unknowns”

Reducing the number of unknown parameters to obtain **unique (i.e. correct)** results is accomplished in several ways

- **For a single measurement:**
  - Use modeling techniques that constrain unknowns
  - Use previously published optical properties for a given material

- **With multiple measurements:**
  - Design strategic experiment involving multiple samples and SE measurements
  - Determine layer thickness using alternate technique (SEM/TEM cross section, step height profilometry, etc.)

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Modeling “Toolkit”

- **With multiple measurements:**
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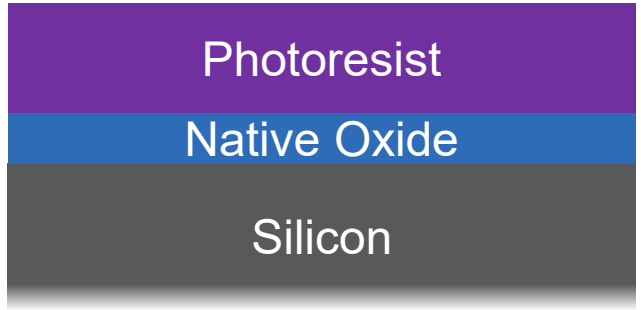
  - Determine layer thickness using alternate technique (SEM/TEM cross section, step height profilometry, etc.)

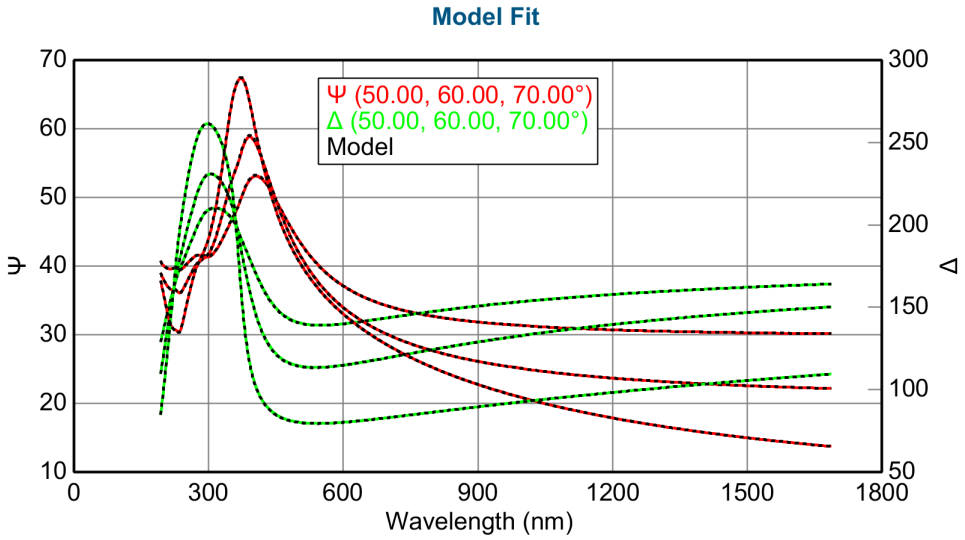
Direct Numerical Inversion

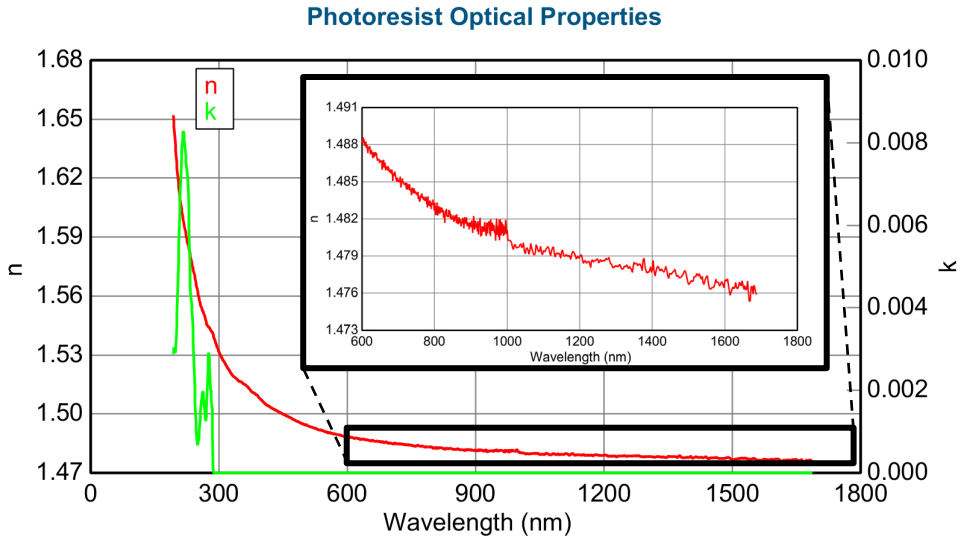
- Applicable when all but one set of optical properties are known
- Also called “wavelength-by-wavelength” fitting since it essentially converts the (ψ, Δ) spectrum into an (n, k) spectrum



Examples:

- Bulk material slab with “perfect” surface
- Single film of known thickness on known substrate

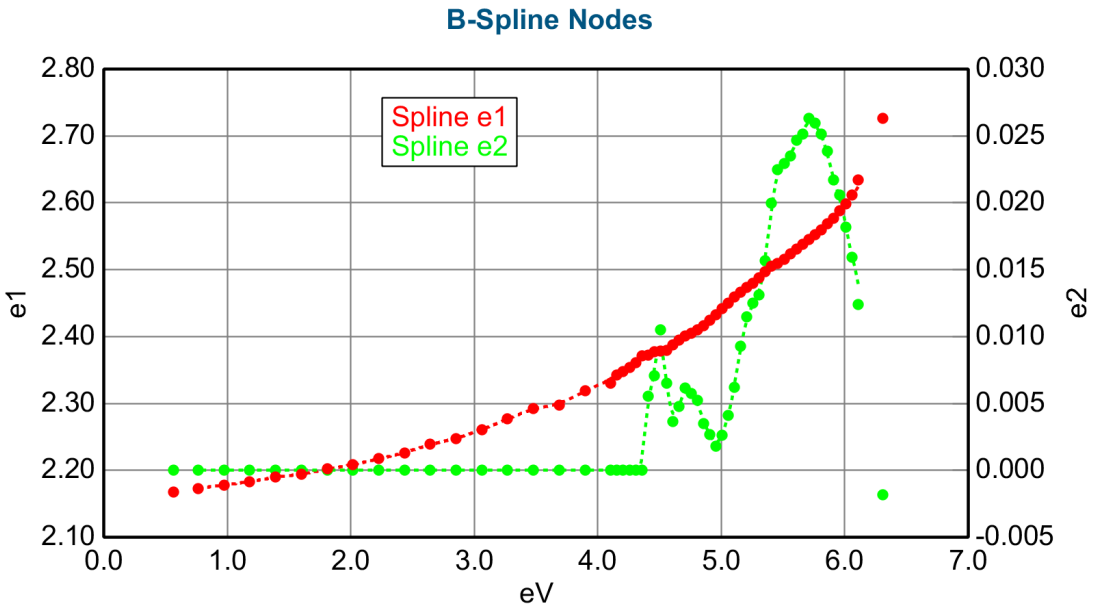


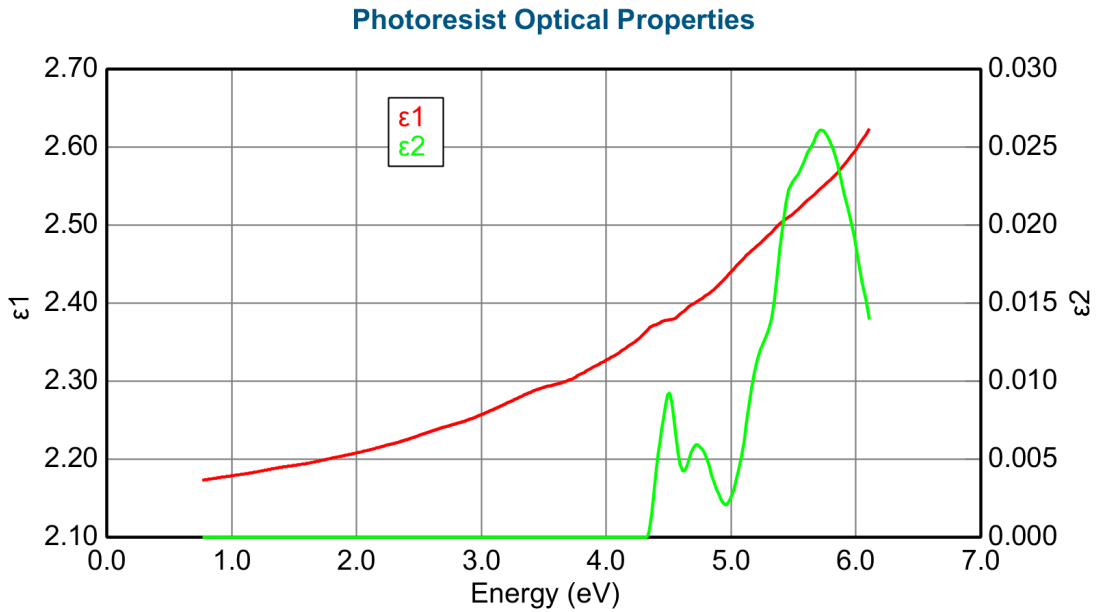


More Information: W. G. Oldham, *Surf. Sci.,* **16**, 97 (1969)

Basis Spline Function

- Uses a basis spline function to describe lineshape of optical properties
- Spline function is the sum of many localized polynomial segments
- **Key feature:** reduces unknowns by using “nodes” that control lineshape for surrounding region





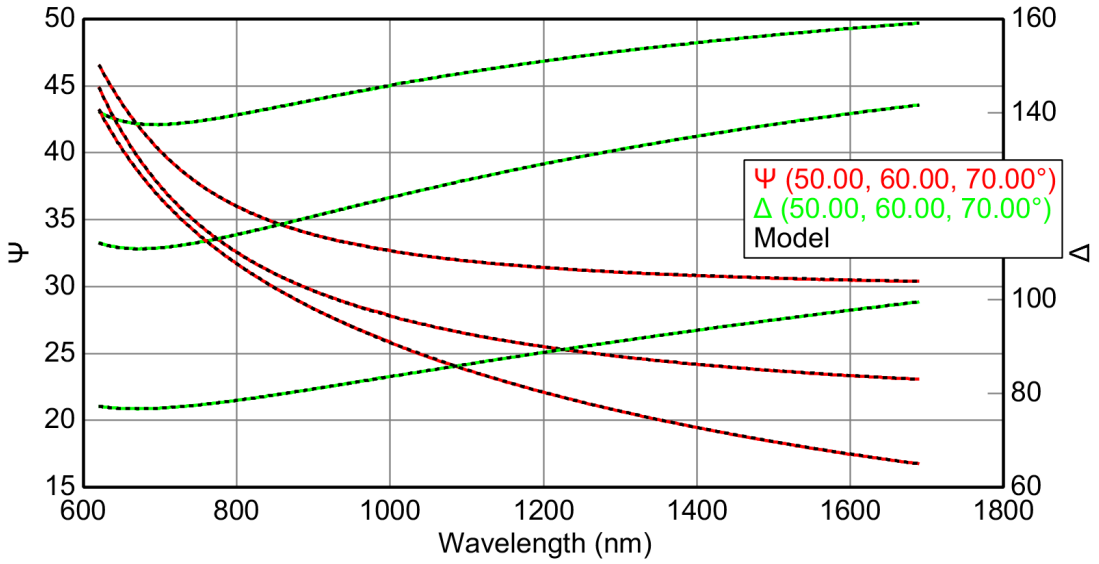
More Information: B. Johs and J. S. Hale, *Phys. Status Solidi A* **205**, 715 (2008)

Cauchy

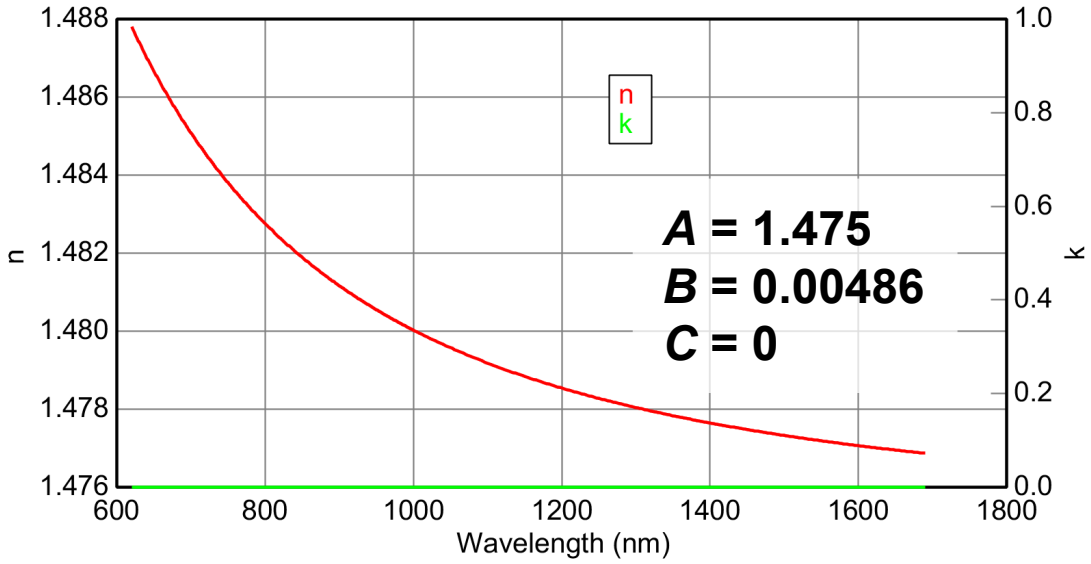
- Uses polynomial to describe dispersion for a nominally transparent material
- **Key feature:** reduces unknowns by using describing smooth dispersion curve with just a few parameters

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}$$

Model Fit

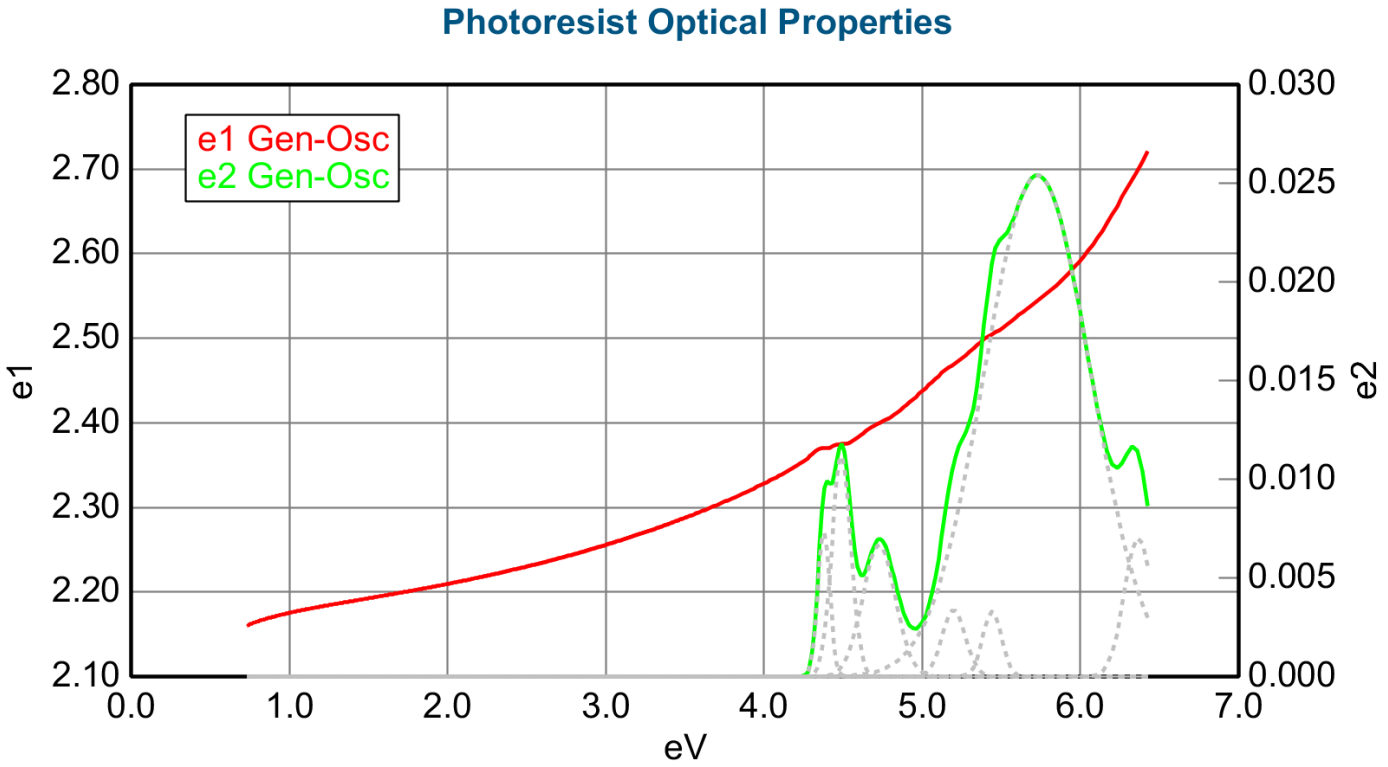


Photoresist Optical Properties (Where Transparent)



Oscillator-Based Parameterization

- Describes lineshape of optical properties as a sum of individual “oscillators”
- Each oscillator is described by an equation that is based upon the physics of the related phenomena (electrical conduction, vibration, electronic transition, etc.)
- **Key Feature:** reduces unknowns since lineshape is described entirely by a sum of oscillator equations



Effective Medium Approximations

Surface Roughness



Composite Materials



Interfaces

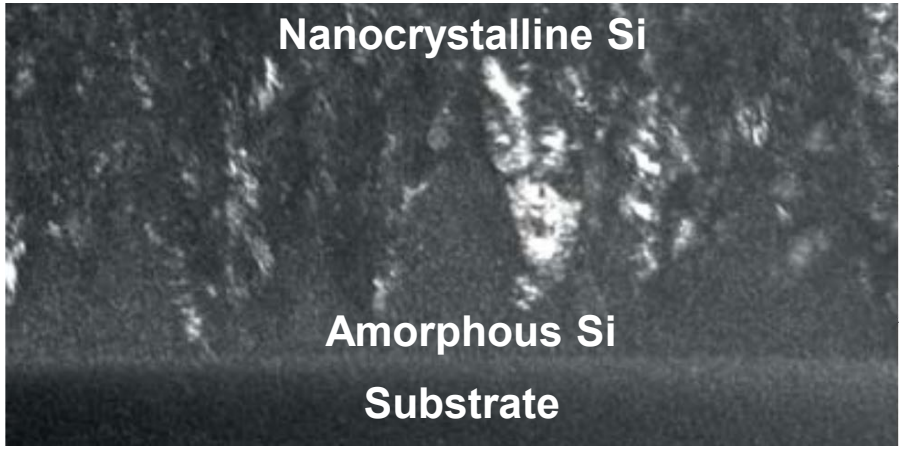


Image: Podraza, N. *et al.*, *Phys. Stat. Sol. (A)* **205**, 892 (2008).

Effective Medium Approximations

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Surface Roughness

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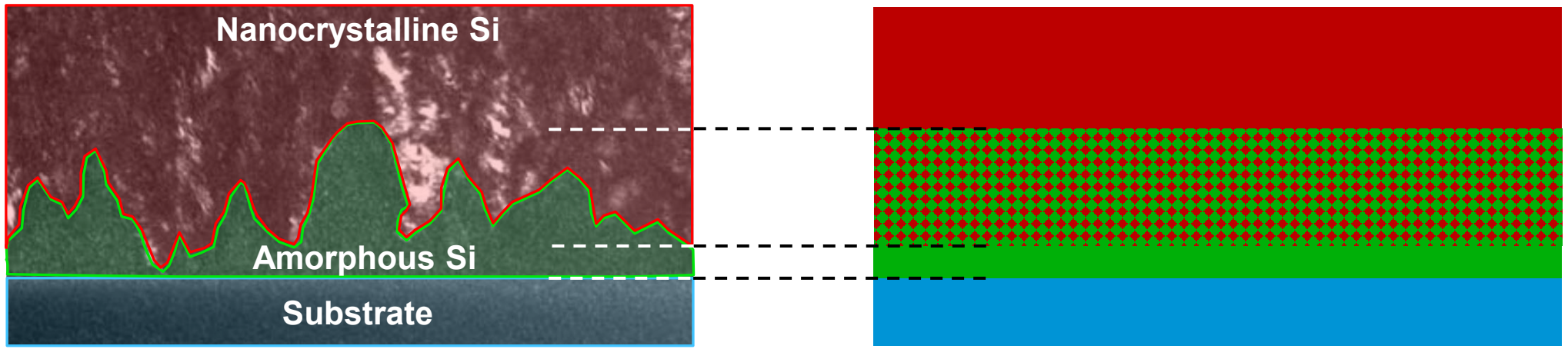
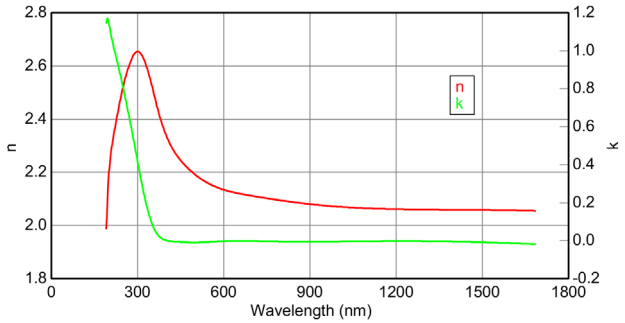


Image: Podraza, N. *et al.*, *Phys. Stat. Sol. (A)* **205**, 892 (2008).

Effective Medium Approximations

- Effective medium approximations (EMAs) mathematically describe the effective composite optical properties of two distinct materials that are “mixed”
- Different types used for specific mixing situations (Bruggeman, Maxwell-Garnet, Linear, etc)

Film Optical Properties



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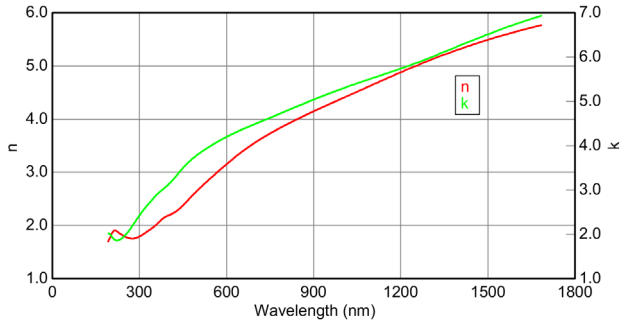
Substrate Optical Properties



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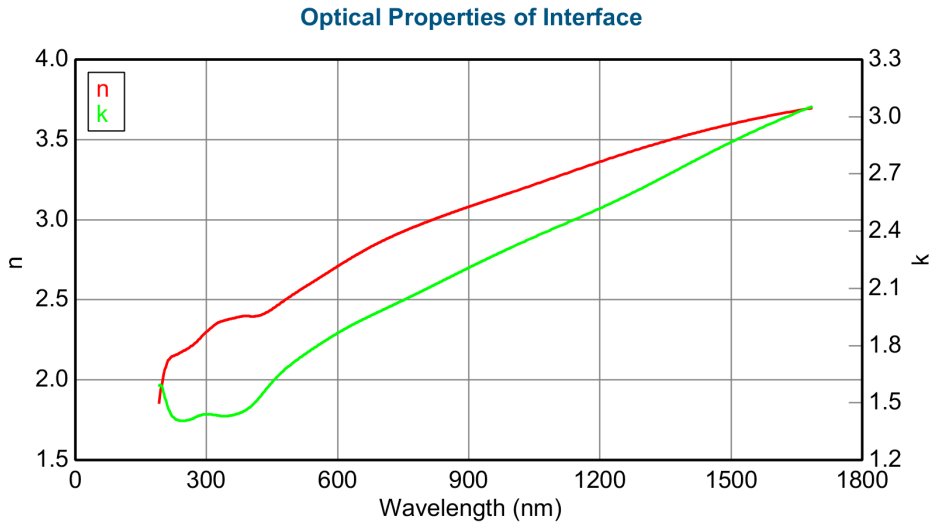

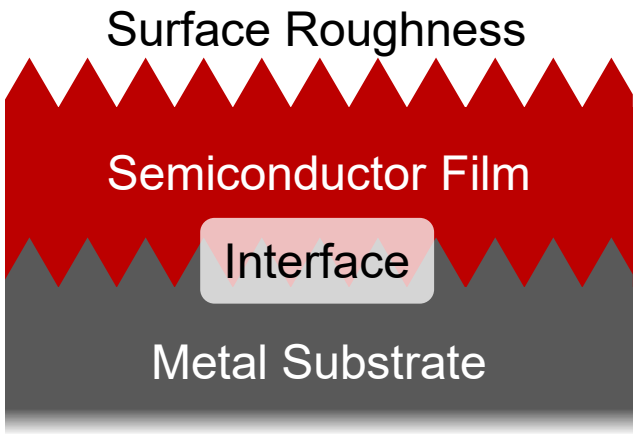

Bruggeman EMA: $$f_a \frac{\varepsilon_a - \varepsilon}{\varepsilon_a + 2\varepsilon} + (1 - f_a) \frac{\varepsilon_b - \varepsilon}{\varepsilon_b + 2\varepsilon} = 0$$

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COVALENT METROLOGY

1 Ellipsometry Review

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3 Application Examples

Example 1: “Well Behaved” Samples

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Samples:	Photoresist on Si wafers
Problem:	Determine photoresist optical properties and use to check for **(1) optical uniformity** and **(2) thickness uniformity**
Experiment Design:	• Measure bare Si wafer to characterize substrate • Fit optical properties of each film at center with multiple AOI • Use these optical properties to map film thickness uniformity

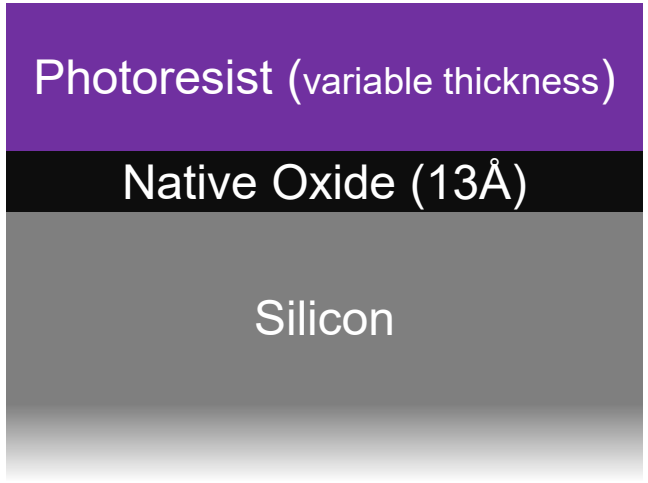
Modeling Strategy

1 – Model Bare Si Substrate:

- An uncoated substrate is measured to determine unique native oxide thickness for this batch of wafers
- “Known” (literature reference) optical properties used for Si and oxide

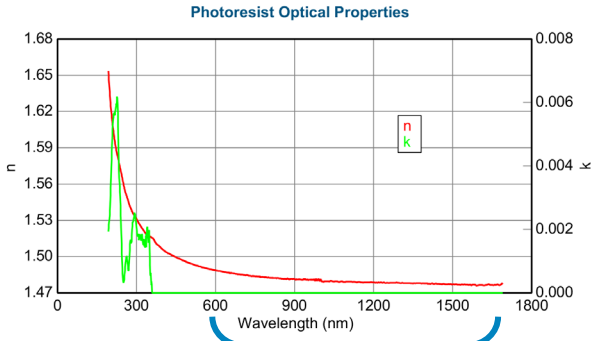
2 – Model Photoresist:

- Fit to center at multiple angles:
  - Start with transparent portion of spectrum **(Cauchy)**
  - Expand to absorbing region **(B-Spline)**
  - Fit “final” optical properties **(Wavelength-by-wavelength)**
- Use full range optical properties to fit full wafer map

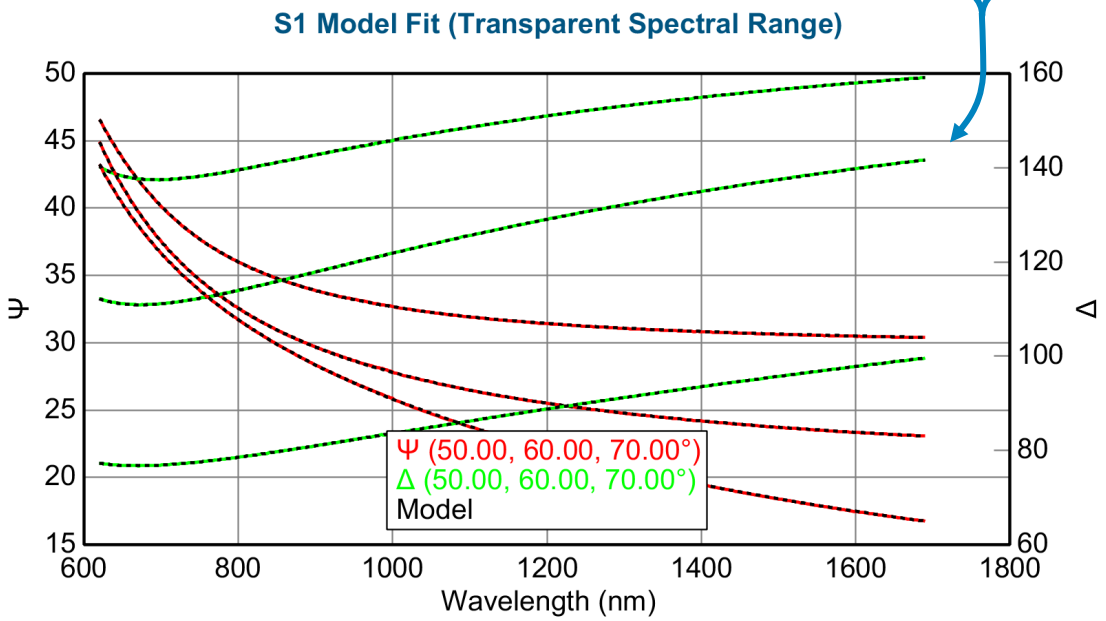


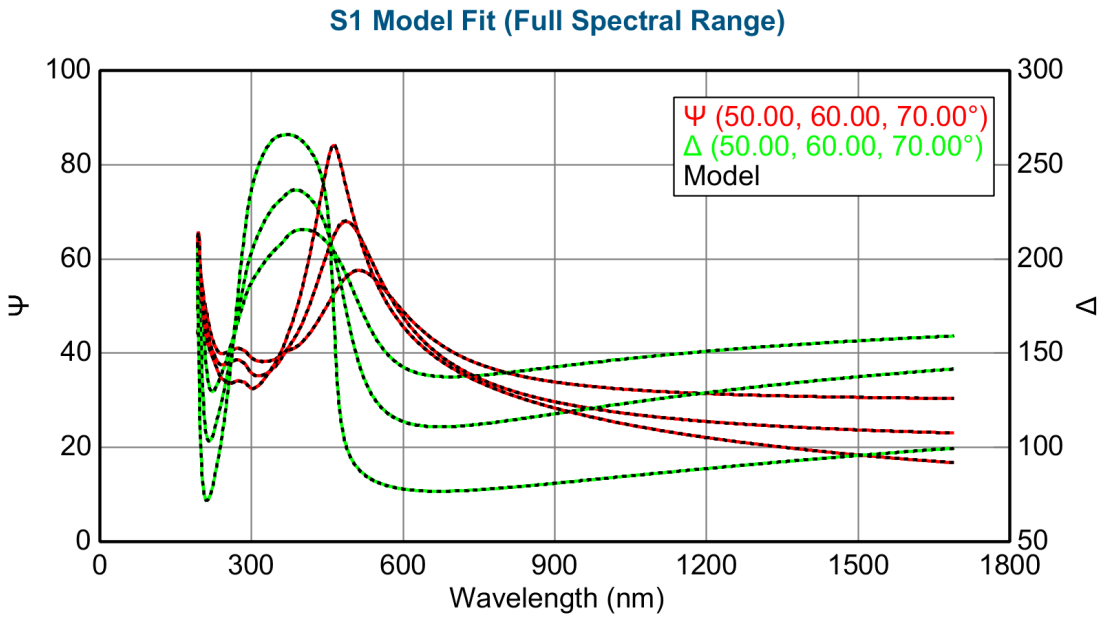
Modeling Strategy

First stage of modeling for determining film thickness at center:



Second stage of modeling for determining full spectral range optical properties:

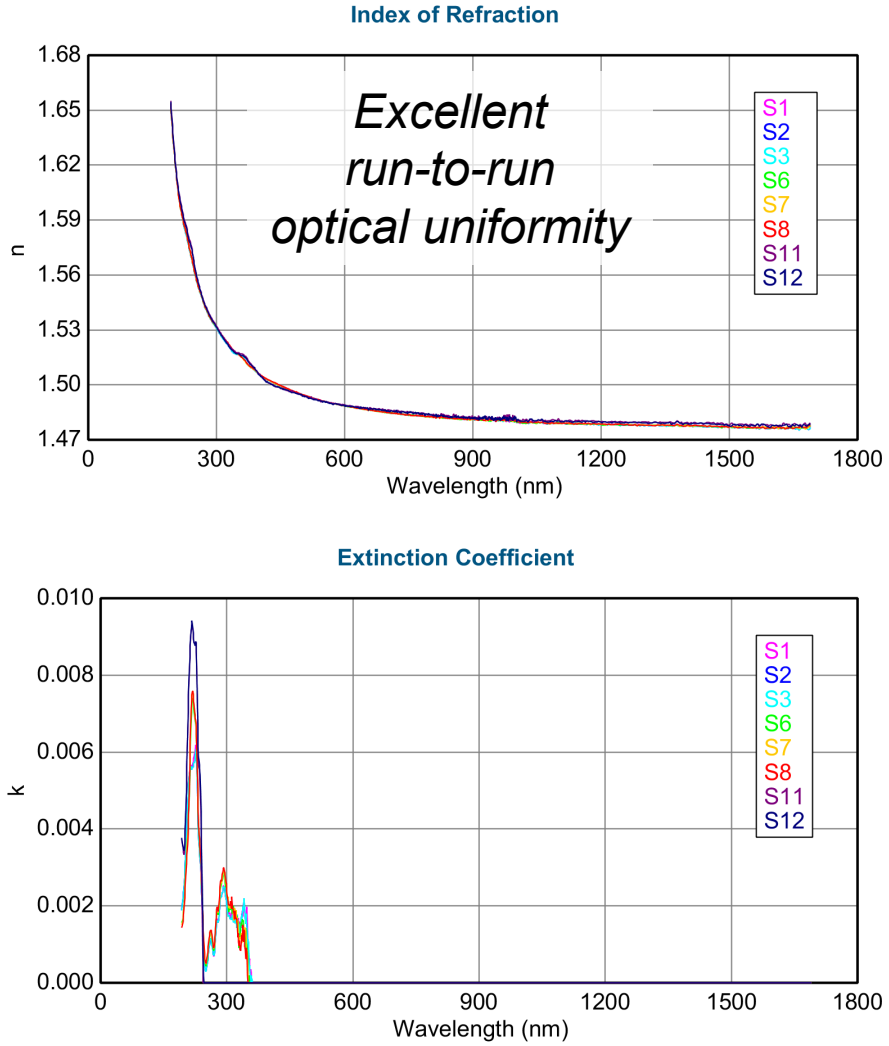




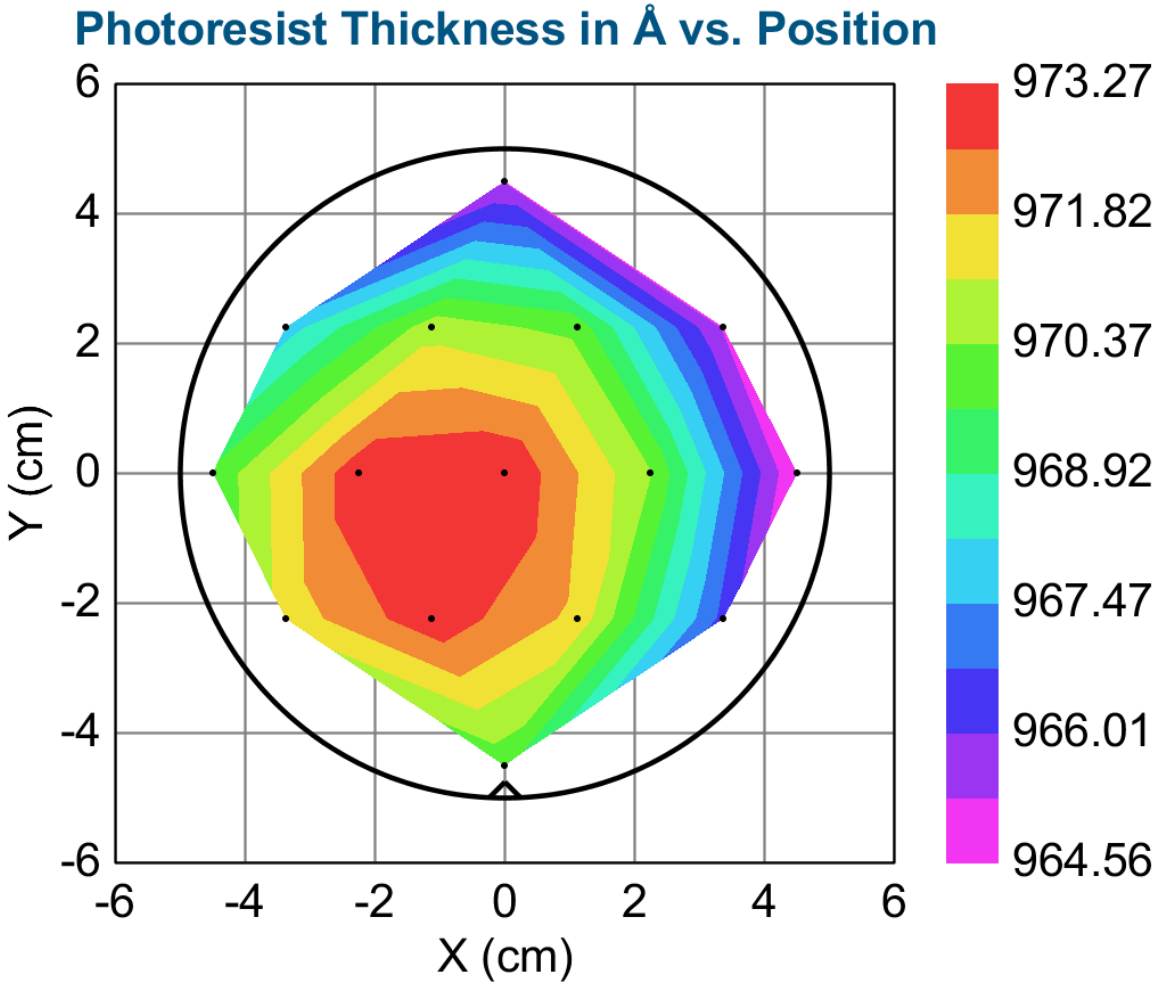
Cauchy

B-Spline

Final Optical Properties and Thickness Map



Wavelength-by-wavelength



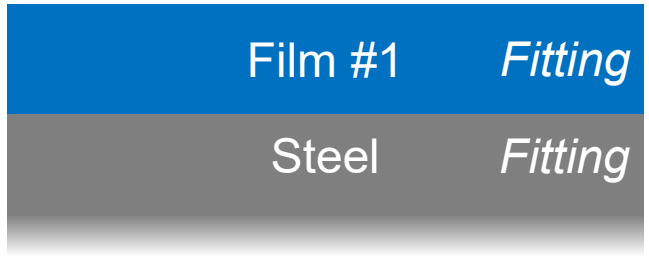
Example 2: Non-Uniform Samples

COVALENT METROLOGY

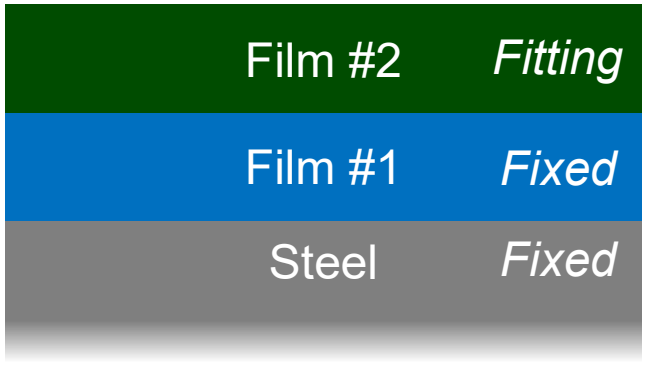
Samples:	Bi-layer Film Stack
Problem:	Early-stage process development where optical properties, uniformity, and film thickness provide process feedback
Experiment Design:	• Initially measure simpler sample: lower film on substrate • Use this as underlying model for complete bi-layer sample to model overlying film of primary interest

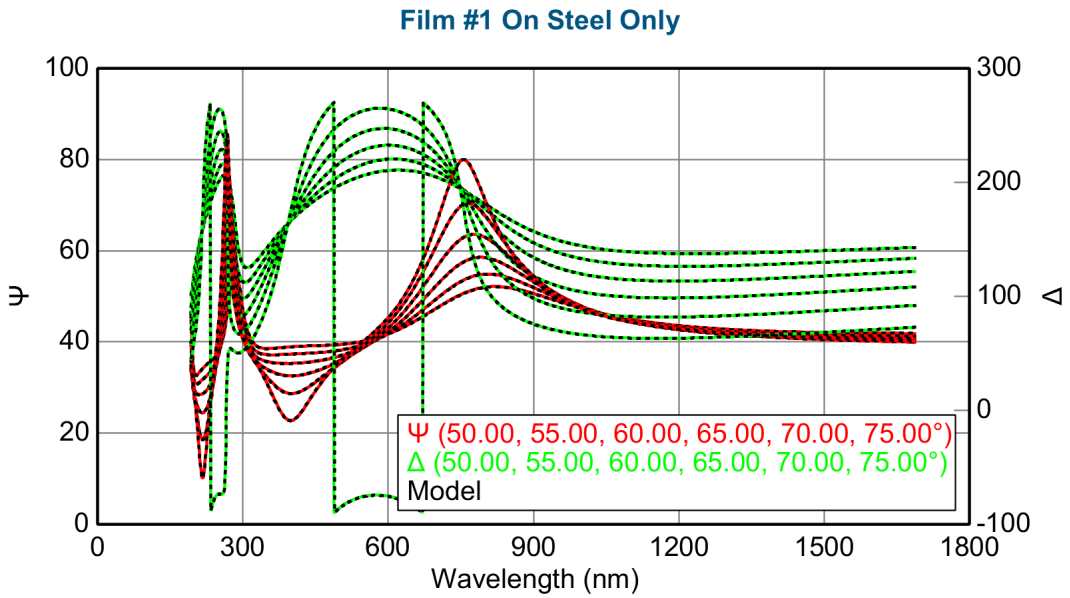
Modeling Strategy: Start With Simpler Sample

First, measure and model this:



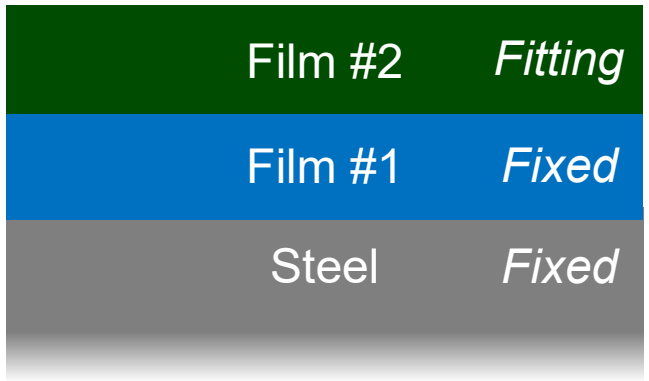
Then, use previous results to measure and model this:

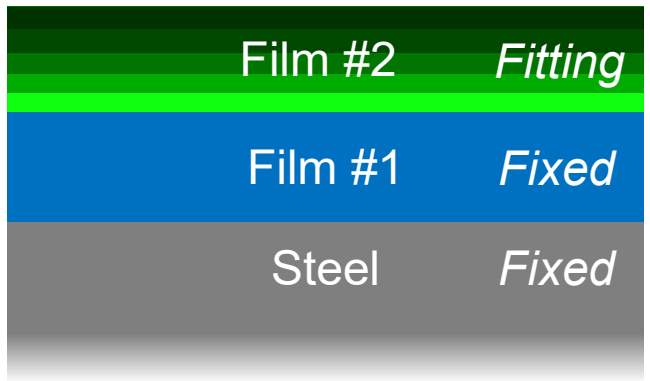




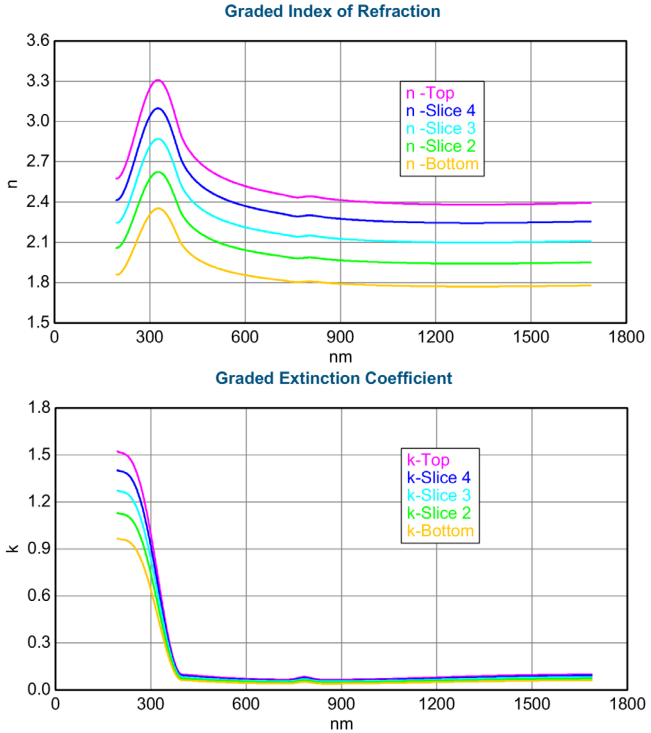
For complex, multi-layered samples with many unknowns, strategically designing a sequence of samples and measurements is key

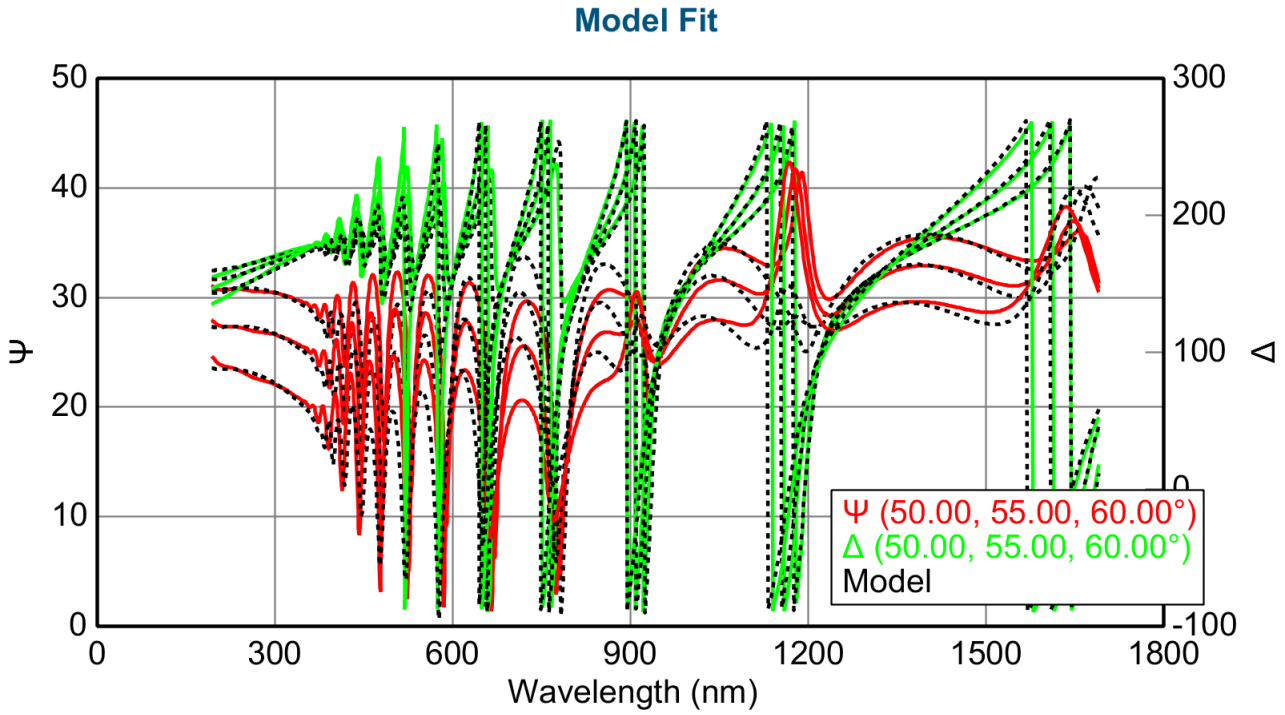
(Upper) Film #2 Inhomogeneity





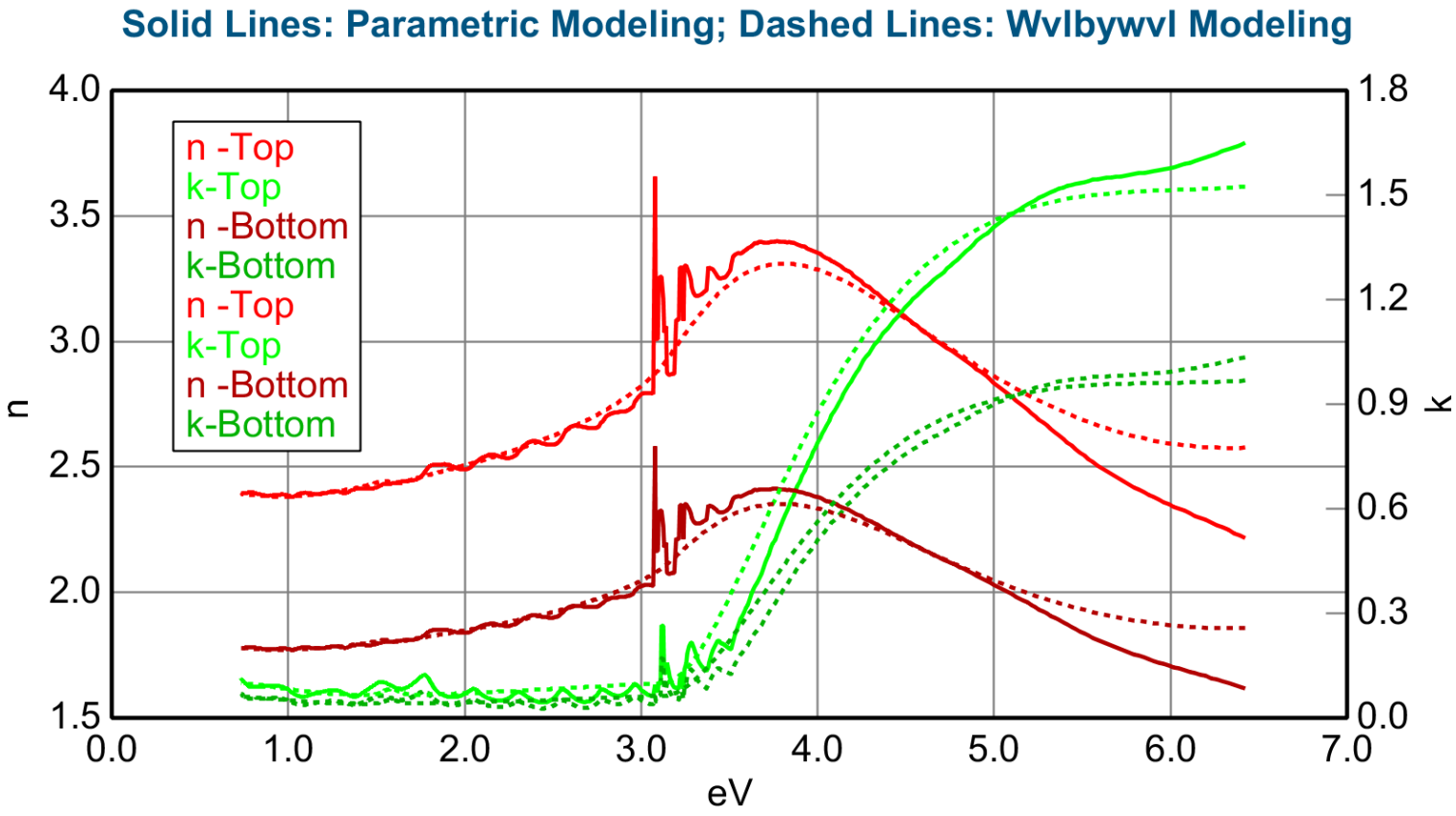
- Film #2 described as parametric sum of oscillators
- Divided into 5 sub-layers to model optical inhomogeneity with thickness





“Sanity Check”

- Initial modeling used parameterized, oscillator-based description of optical properties to reduce unknowns and degrees of freedom
- As a model check, a full wavelength-by-wavelength fitting can be executed as a check of model accuracy
- Convergence between the two optical modeling approaches is evidence of model accuracy



Example 3: Inaccurate Substrate Properties

COVALENT METROLOGY

Samples: Thin film on fused silica

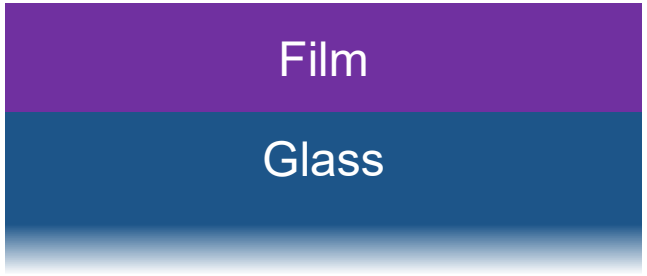
Problem: Need accurate film optical properties

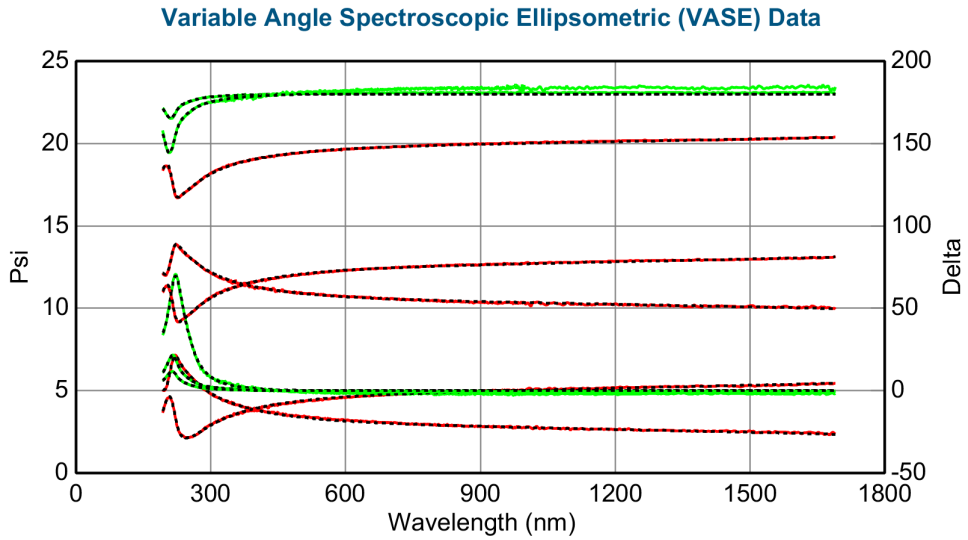
Experiment Design:

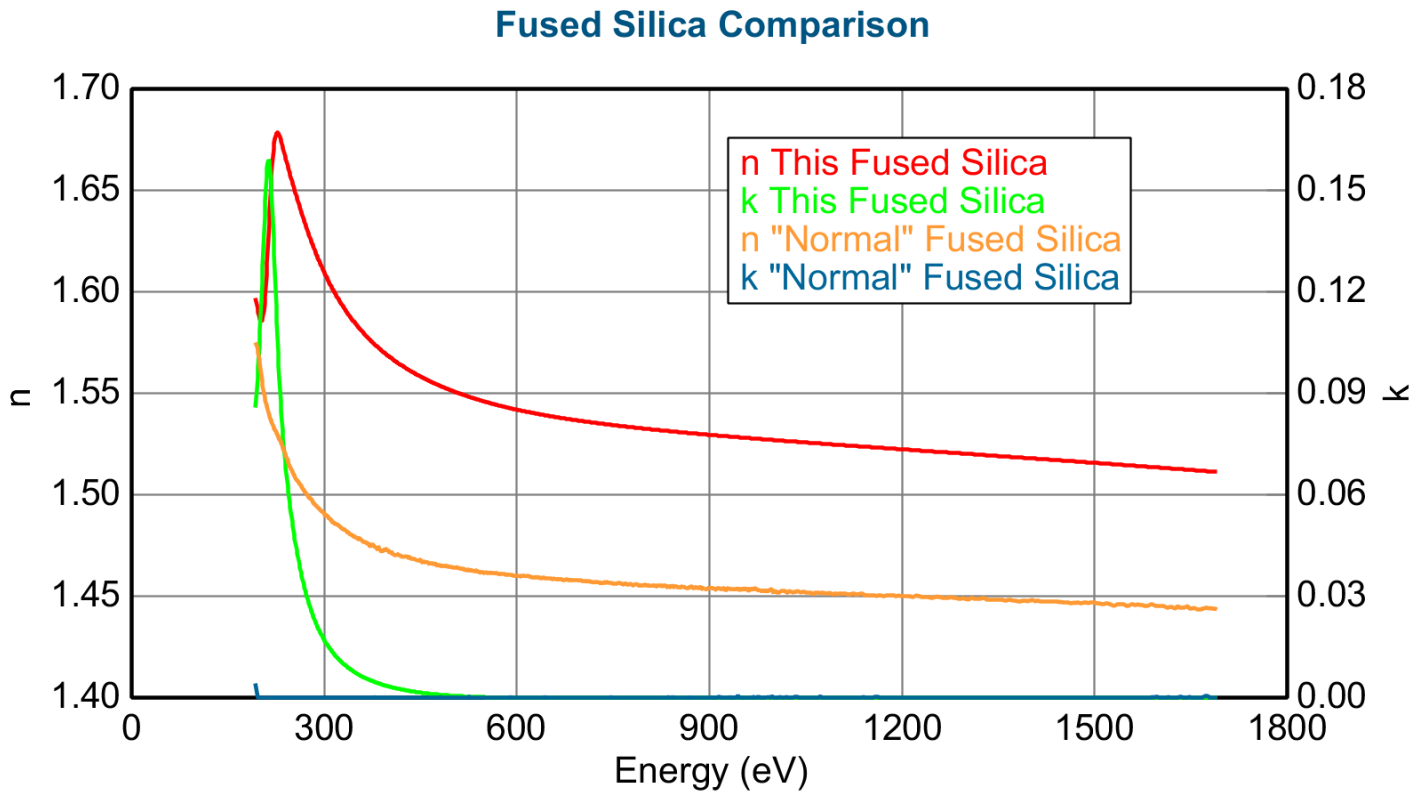
- Measure and model bare fused silica
- Use this to model measurements of film

Fused Silica Substrate

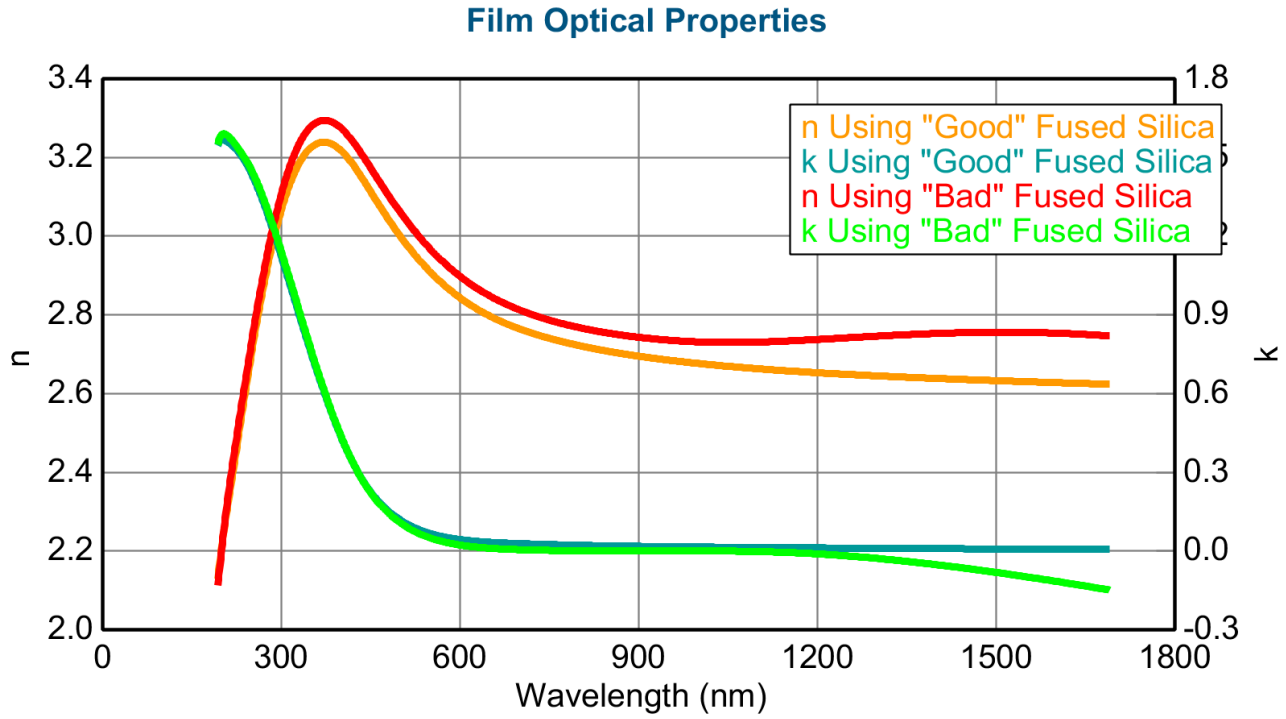
Model as a single bulk layer of material using a parametric sum of oscillators

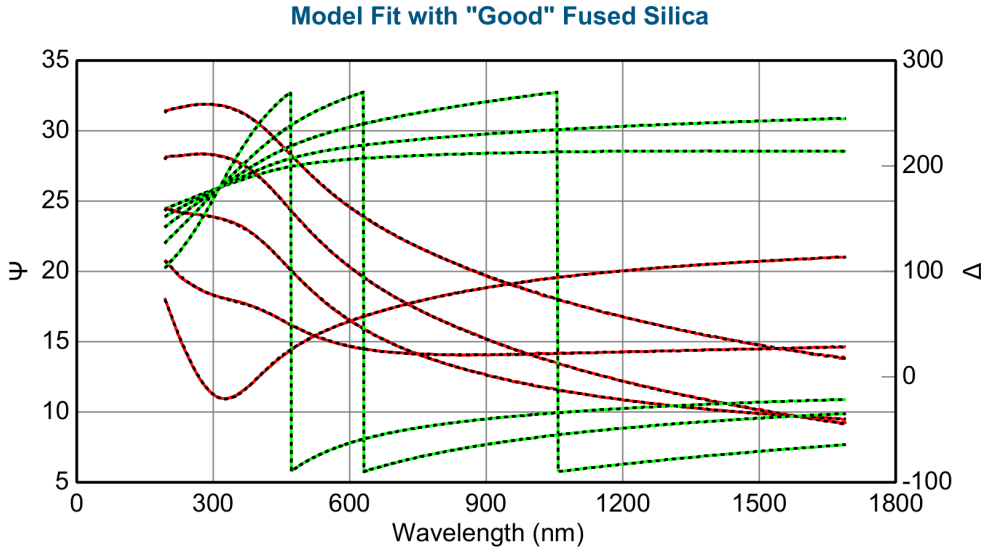


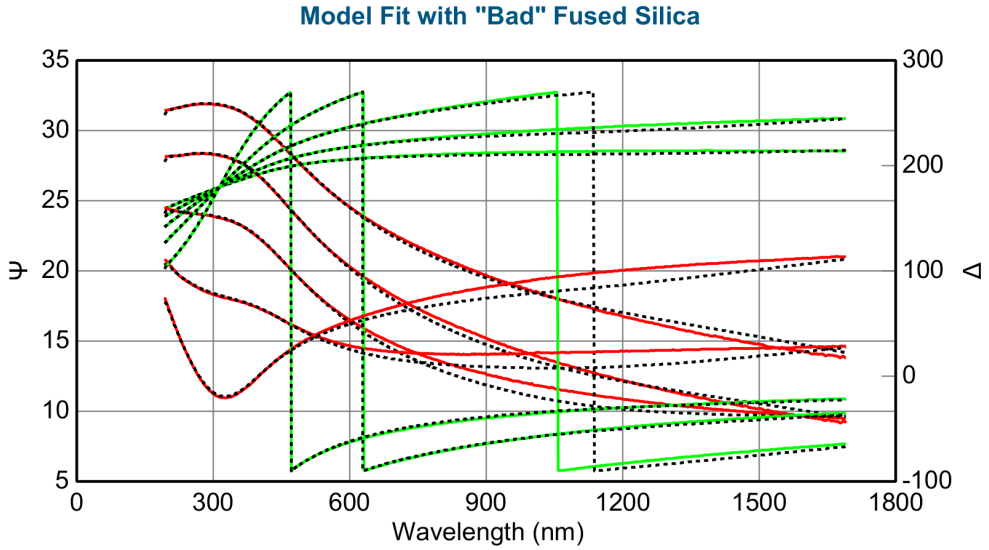




Thin Film Optical Properties







- Accuracy in optical properties of substrate affect accuracy of optical properties in overlying film
- Incorrect substrate optical properties results in unphysical behavior (i.e. k negative) in the film optical properties

Example 4: Unexpected Optical Absorption

COVALENT METROLOGY

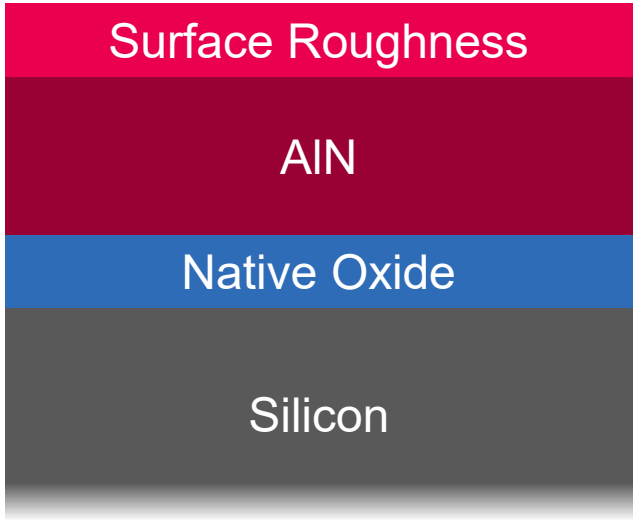
Samples: Thick (few μm) AlN film on Si wafer

Problem: Need to determine film thickness uniformity over wafer

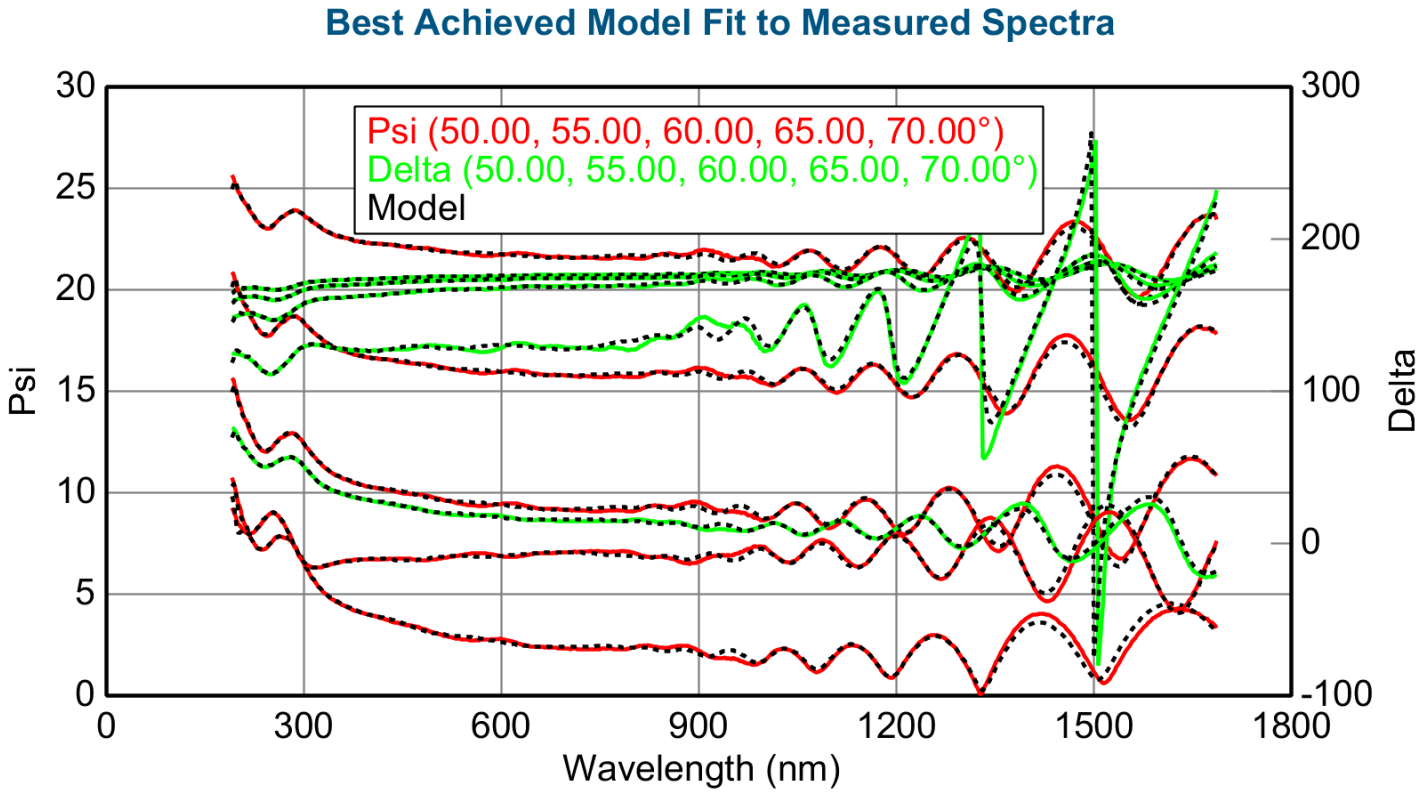
Experiment Design:

- Collect full sample mapping measurements
- Determine appropriate model and optical properties from center measurement location
- Use to fit full map for spatial thickness uniformity

Modeling Center of Wafer

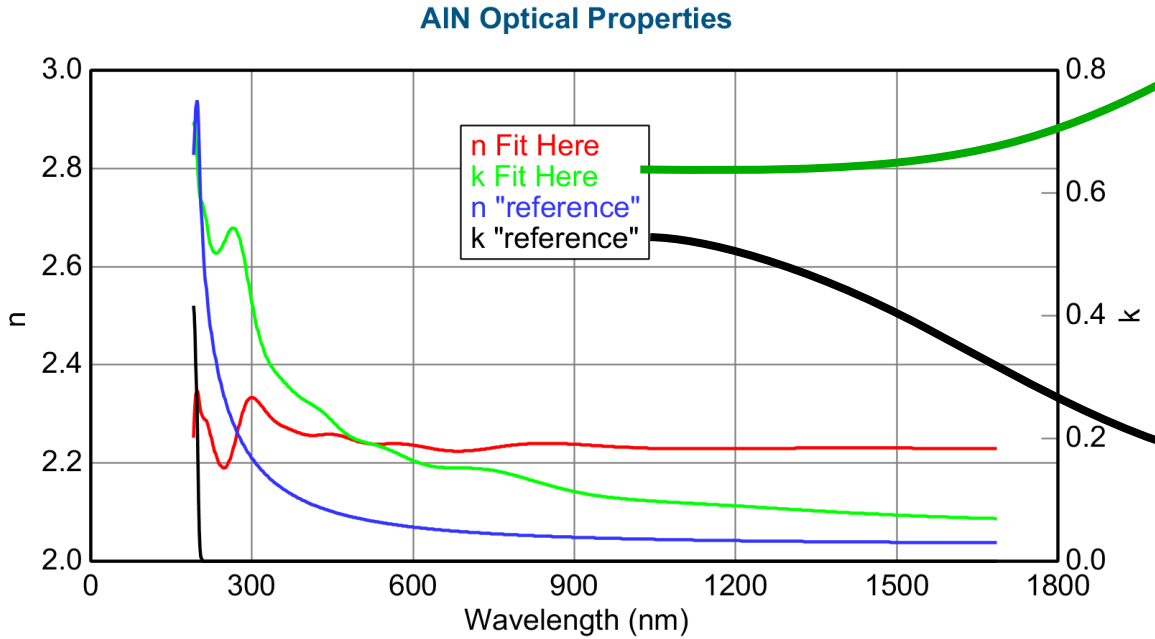


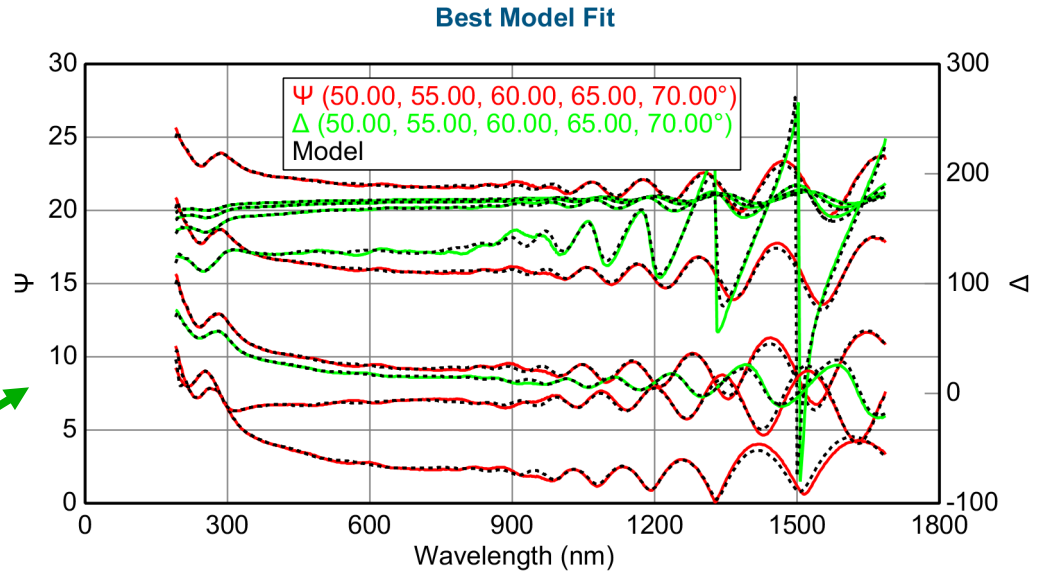
- AlN film was determined to be very thick (nearly 3 µm)
- AlN described by a B-Spline function
- Surface roughness described by Bruggeman EMA with 50% / 50% material ratio AlN and void

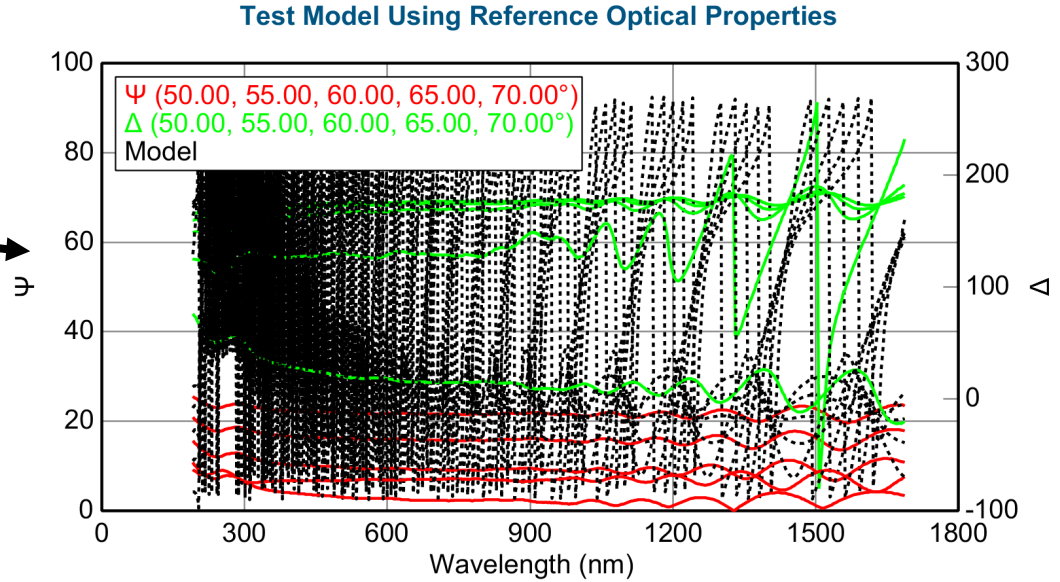


AlN Optical Properties

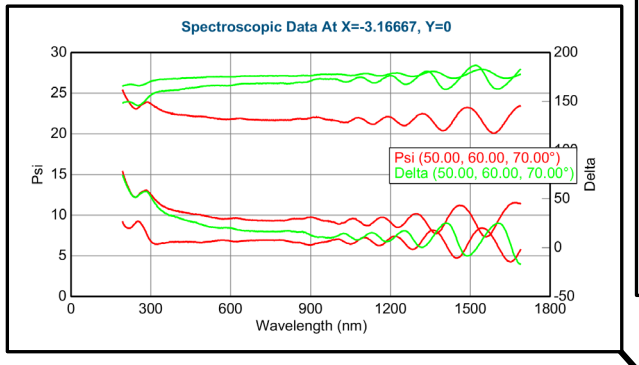
- Compared to “reference” AlN optical properties, this material has unexpected optical absorption across spectrum

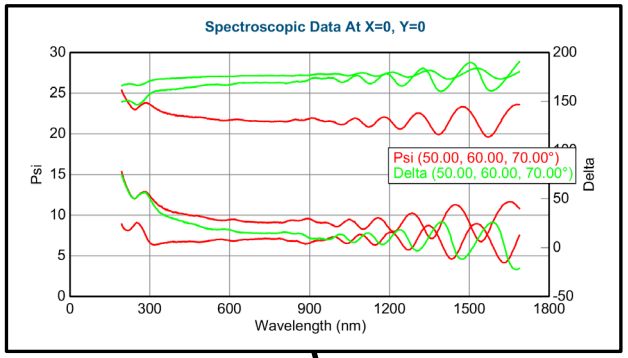


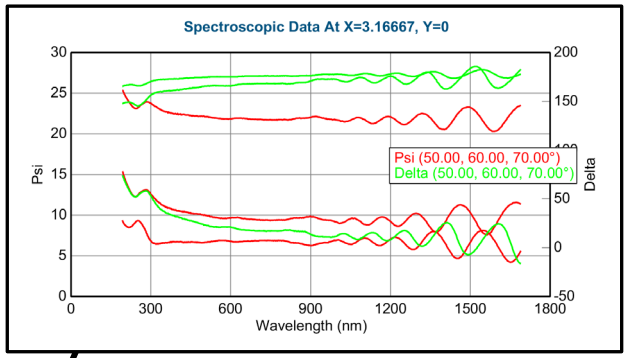


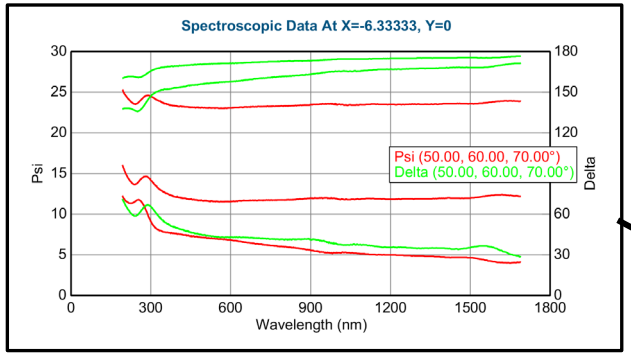


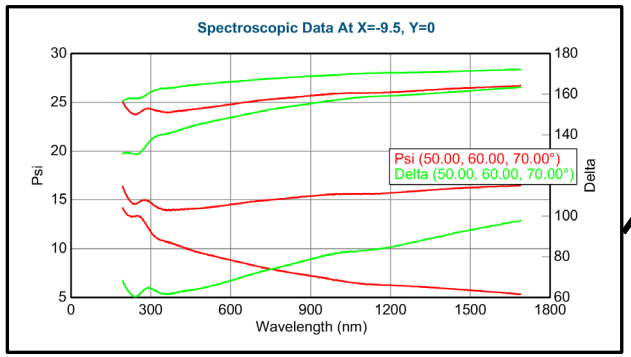
Loss of Thickness Sensitivity Due to Absorption

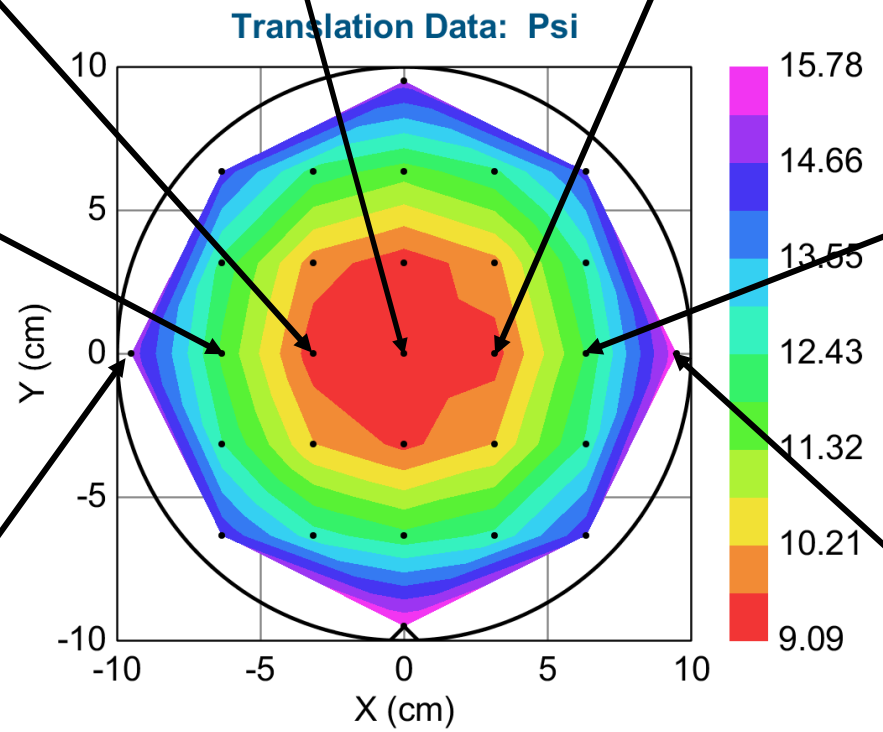


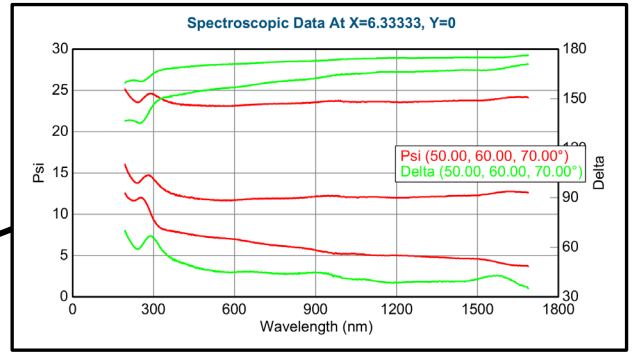


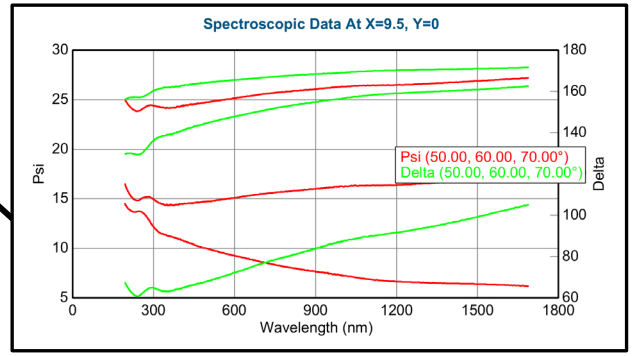












No thin film interference at measurements near edge of film suggests film becomes more absorbing away from the center

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COVALENT METROLOGY

SEEING IN SOUND

ULTRASONIC IMAGING WITH SCANNING ACOUSTIC MICROSCOPY

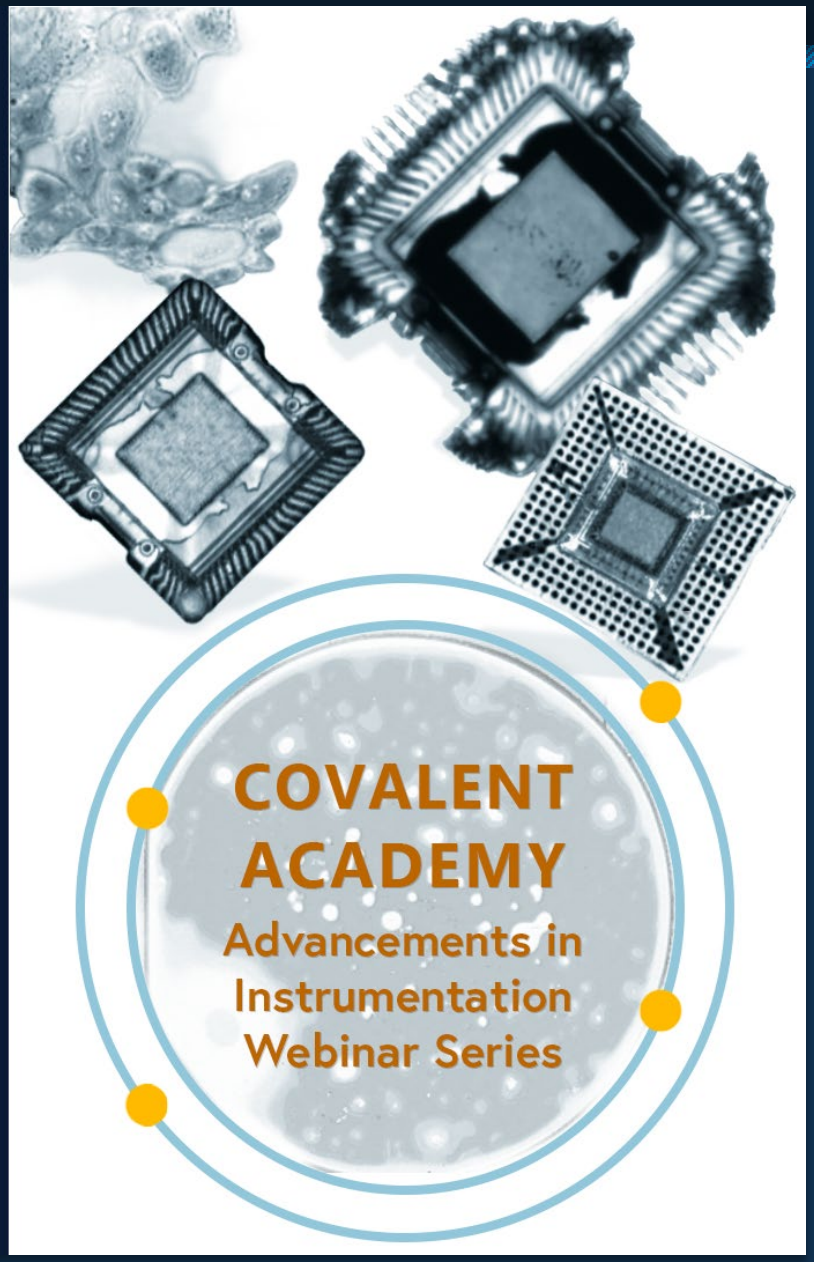
GUEST SPEAKER:

Lisa Logan
Applications Manager, SAM
from PVA TePla

Aug 20, 2020 11am PDT

COVALENT METROLOGY

PVA TePla



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Q & A Session

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